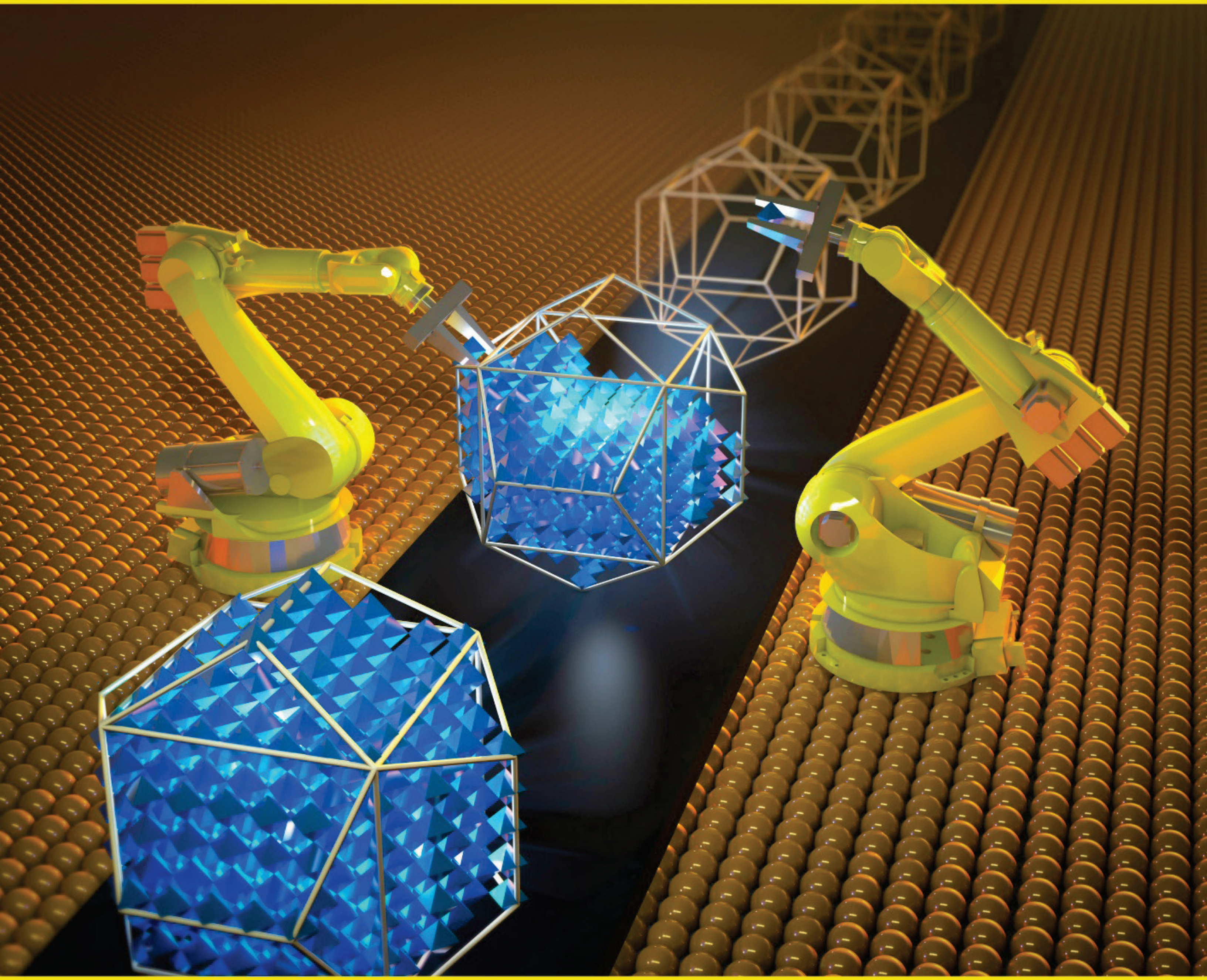
Basic Research Needs for

Transformative Manufacturing



Factual Document for the Basic Energy Sciences Workshop on Basic Research Needs for Transformative Manufacturing

March 9 – 11, 2020

Cover and back images are courtesy of Oak Ridge National Laboratory. Sidebar images are courtesy of Oak Ridge National Laboratory and Argonne National Laboratory and are reprinted with permission from the American Chemical Society and Springer/Nature.

Factual Document for the Basic Energy Sciences Basic Research Needs Workshop for Transformative Manufacturing

Chair – Cynthia Jenks, Argonne National Laboratory
Co-Chair – Ho Nyung Lee, Oak Ridge National Laboratory
Co-Chair – Jennifer Lewis, Harvard University

Panos Datskos, National Renewable Energy Laboratory; Joe Cresko, Department of Energy; William Morrow, Lawrence Berkeley National Laboratory; Greg Krumdick, Argonne National Laboratory

with contributions from

T. Kaarsberg (DOE)
M. Klembara (DOE)
K. R. Kort (DOE)
G. J. Leong (DOE)
T. Miller (DOE)
K. Peretti (DOE)
D. Berry (NREL)
A. Burrell (NREL)
A. Carpenter (NREL)
S. Hammond (NREL)
M. Ringer (NREL)
M. Ulsh (NREL)
S. Seetharaman (NREL/CSM)
S. Chaudhuri (Argonne)
J. Chen (Argonne)
S. Darling (Argonne)
M. U. Demirtas (Argonne)
J. W. Elam (Argonne)
D. Graziano (Argonne)
A. Harris (Argonne)
N. Iloeje (Argonne)
T. Krause (Argonne)
J.Kopasz (Argonne)
C. Negri (Argonne)
K. Pupek (Argonne)
J. Spangenberger (Argonne)
M. Stan (Argonne)
D. Singh (Argonne)
V. Srinivasan (Argonne)
M. Wang (Argonne)
M. Wu (Argonne)
Y. Zhang (Argonne)
A. Naskar (ORNL)
V. Kunc (ORNL)
T. Kurfess (ORNL)
N. Lavrik (ORNL)
L. Love (ORNL)
S. U. Nimbalkar (ORNL)
V. Paquit (ORNL)
M. Theodore (ORNL)
S. Smith (ORNL)
C. Tsouris (ORNL)
J. Turner (ORNL)
D. Wood (ORNL)
V. Battaglia (LBNL)
P. Rao (LBNL)
A. Weber (LBNL)
P. Randall Schunk (SNL)
J. Greenblatt (Emerging Future, LLC)

Abbreviations, Acronyms, and Initialisms

2D	two-dimensional
3D	three-dimensional
5G	fifth generation
ACSM	atomic or close-to-atomic scale manufacturing
ACGIH	American Conference of Governmental Industrial Hygienists
AEO	Annual Energy Outlook
AFM	atomic force microscopy
AHSS	advanced high-strength steel
AI/ML/DL	artificial intelligence/machine learning/deep learning
ALD	atomic layer deposition
ALE	atomic layer etching
AM	additive manufacturing
AMO	DOE Advanced Manufacturing Office
Argonne	Argonne National Laboratory
ANSI	American National Standards Institute
APS	Advanced Photon Source
BCE	before common era
Bgal/d	billion gallons per day
BPP	bipolar plate
BOF	basic oxygen furnace
Btu	British thermal unit
$CaCO_3$	limestone
CAD	computer-assisted design
CALPHAD	CALculation of PHAse Diagrams
CCS	carbon capture and storage
CF	carbon fiber
CFL	compact fluorescent lighting
CFRP	carbon fiber–reinforced polymer
CFTF	Carbon Fiber Technology Facility
CM	critical materials
CNC	computer numerical control
CO_2	carbon dioxide
CSE	coal sourced electricity
CSM	Colorado School of Mines

DL	deep learning
DOE	US Department of Energy
DPN	dip pen nanolithography
EBL	electron beam lithography
EES	electrochemical energy storage
EHS	Environment, Health and Safety
EIA	US Energy Information Administration
ELB	electron beam lithography
ESH	environmental, safety, and health
EU	European Union
EV	electric vehicle
FC	fuel cell
Fe	iron
FET	field effect transistors
FDP	fossil resource depletion potential
GDL	gas diffusion layer
GHG	greenhouse gas
Gt	gigatons
GWP	global warming potential
HDL	hydrogen depassivation lithography
HPC	high-performance computing
HTP	human toxicity potential
IACMI	Institute for Advanced Composites Manufacturing Innovation
IBC	international building code
ICEV	internal combustion engine vehicle
IR	infrared
IoT	internet of things
kHz	kilohertz
LBNL	Lawrence Berkeley National Laboratory
LCOE	levelized cost of electricity
LED	light-emitting diodes
Li	lithium
Li-ion	lithium-ion
LIB	lithium-ion battery
$LiFePO_4$	lithium iron phosphate
LiNCM	lithium nickel cobalt manganese

MCMC	Monte Carlo Markov Chain
MDF	Manufacturing Demonstration Facility
MDP	mineral resource depletion potential
MEA	membrane electrode assembly
ML	machine learning
MLD	molecular layer deposition
MT	metric ton
NDE	nondestructive evaluation
NdFeB	neodymium-iron-boron
NFPA	National Fire Protection Association
NG	natural gas sourced electricity
NIOSH	National Institute for Occupational Safety and Health
NREL	National Renewable Energy Laboratory
OLED	organic light-emitting diode
ORNL	Oak Ridge National Laboratory
OSHA	Occupational Safety and Health Administration
PAN	polyacrylonitrile
PCM	phase change material
PEMFC	polymer electrolyte membrane fuel cell
PFE	printed flexible electronics
PGM	platinum-group metal
PI	process intensification
PM	particulate matter
Pt	platinum
PV	photovoltaic
quad	quadrillion
R&D	research and development
R2R	roll-to-roll
Re-X	reduce, reuse, remanufacture, and recycle
REE	rare-earth elements
RFID	radio frequency identification antenna
SAXS	small-angle x-ray scattering
SB	scanning beam
SCM	supplementary cementitious material
SDS	safety data sheet
Si	silicon

SI	system integration
SNL	Sandia National Laboratories
SP	scanning probe
SSB	solid-state battery
STM	scanning tunneling microscopy
US	United States
WHP	waste heat-to-power
WHR	waste-heat recovery
X-T-P	composition, temperature, and pressure

Table of Contents

List of Figures

List of Tables

1 Manufacturing Background and Overview

1.1 Introduction

In the beginning of the twentieth century, post–industrial-revolution manufacturing was able to achieve mass production. Figure 1 shows manufacturing paradigms from Before the Common Era (BCE) to the present day (Chen et al. 2015). As manufacturing evolves, it takes different forms. Over time, advances in technological developments enable manufacturing systems to evolve and become more automated, computerized, and complex—gradually improving productivity and energy efficiency (Kusiak 2018).

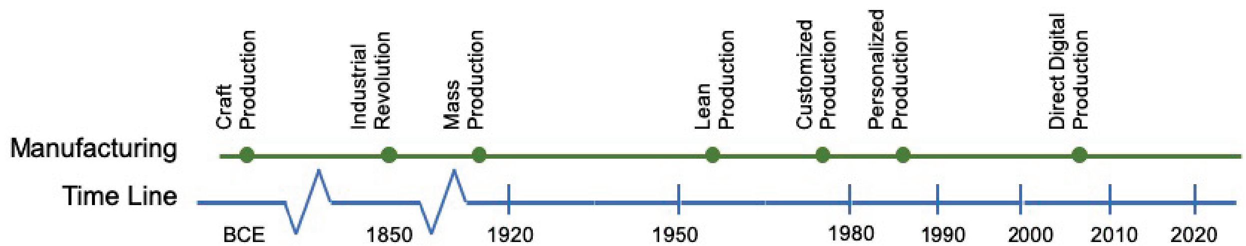


Figure 1. Selected view of the manufacturing timeline of different paradigms over the centuries. (*Source*: adapted from *J. Clean Prod.*, 107(615), D. Chen et al., Direct digital manufacturing: definition, evolution, and sustainability implications, 615, 2015, with permission from Elsevier)

The emergence of advanced manufacturing technologies and the desire for more customized products influence the scale and distribution of manufacturing. Manufacturing requires a level of understanding of the relationships between structure and function across many spatial scales (Figure 2). With many gaps in the scientific foundations for these complex processes, basic scientific research is necessary to fill these knowledge gaps and enable creation of manufacturing approaches with the specific characteristics required for next-generation energy technologies. Further, next-generation manufacturing should enable a low-carbon, high-efficiency, environmentally sustainable future, which requires a strong disciplinary base and sustained support for new scientific discoveries. To understand the current state of the art in manufacturing, the existing capabilities for synthesis, characterization, manufacturing scale-up, and performance validation of new processes is discussed. As factories adopt increasingly more data-driven systems and become fully networked, they will employ automation, sensing, and control systems to achieve higher productivity and better competitiveness.

The manufacturing process tends to be broad and includes product design (Figure 2). It uses models and physical systems to help characterize, quantify, and understand uncertainties at different length and time scales; improve energy efficiency; increase performance; and reduce cost. These models and physical systems must be verified and validated before the manufacturing of the product begins. Manufacturing supply chains and all phases of the product life cycle—including end-of-life considerations—are important in manufacturing.

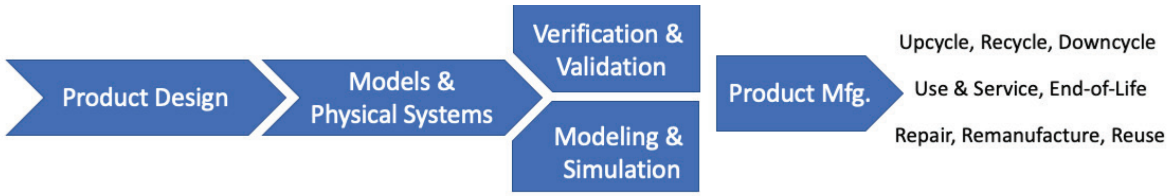


Figure 2. The various steps in the manufacturing process are linked, often are complex, and require an understanding of the relationships between structure and function, and across many spatial scales. (*Source*: Panos Dotskos, National Renewable Energy Laboratory)

A variety of manufacturing processes convert raw materials to finished products. These processes present opportunities to enhance throughput, reduce waste, and decrease wasted energy to achieve greater efficiencies. Production can be defined as the transformation of raw materials into products by a series of processes with energy applications, each of which effects well-defined changes in the physical or chemical characteristics of the raw materials (Dano 1966). Manufacturing systems depend on the availability of raw materials for the particular processes that create products (Figure 3) (Cresko 2019). Manufactured goods have significant energy impacts on the economy-wide system—including the transportation and building sectors as well as energy production and delivery. Within the industrial sector, *supply-chain* systems comprise the networks of facilities and operations associated with moving materials through a particular industry, from the extraction of raw materials to the production of finished goods in manufacturing facilities. *Production/facility systems* represent individual, goods-producing facilities such as a petroleum refinery or a vehicle-manufacturing plant. Production facility systems integrate manufacturing tools, on-site energy flows and generation, and energy-management strategies into a single workflow to manufacture finished goods. The narrowest systems group is *manufacturing systems*, such as curing tools and chemical distillers. Manufacturing systems include industrial equipment used for process and non-process unit operations.

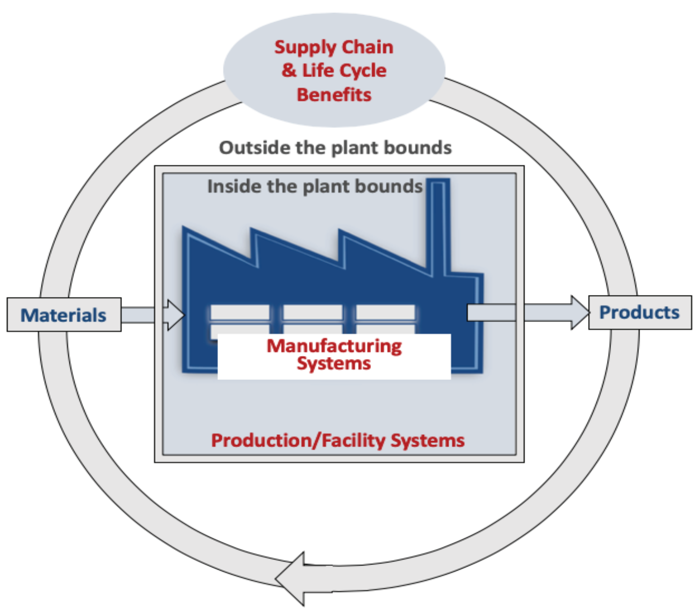


Figure 3. Manufacturing systems rely on the supply chain ensuring raw material availability to produce goods. In addition, considering the life cycle benefits tends to lead to a circular economy (*Source*: J. Cresko, DOE Advanced Manufacturing Office)

Basic research, technological innovations, and changes in manufacturing environments affect both short-term performance and long-term sustainability (Ghobakhloo 2018). When future directions and options in technology are unclear, appropriate strategies are needed to support planning for interacting with upcoming technological developments. Understanding the effect of catalyzing research, development, and adoption of energy-related advanced manufacturing technologies and practices is crucial because this work will be essential in driving US economic competitiveness and energy productivity.

Several national initiatives and programs are under way. Manufacturing, USA,[1] a public-private network of 16 manufacturing institutes, focuses on applied research and technology development projects to solve industry's toughest challenges and to train people in advanced manufacturing skills. More specifically, the US Department of Energy Advanced Manufacturing Office (DOE AMO) manages six of these institutes and is a leader in the support of applied research and development (R&D) for manufacturing. Currently, however, there is no parallel basic science strategy to underpin the efforts of these applied research activities. This presents a huge opportunity to undertake manufacturing and engineering sciences research that can inspire the next generation of manufacturing technologies, accelerate innovation, and transform manufacturing. Understanding the basic research needs, advancing scientific foundations, and developing innovative technologies and technical training are essential to move these advances from the laboratory to the factory floor.

1.2 Role of Manufacturing in the US Economy

Starting with basic economics, raising the standard of living requires an increase in wealth and purchasing power. Traditionally, countries have achieved this by creating new value and increasing productivity. Clearly, this is a difficult task for solely a service economy. Thus, manufacturing leads to more exports and jobs, and helps raise living standards more than any other sector does.[2] Productivity improvements and price moderation in manufacturing are key contributors in the improvement of living standards in the United States. Further, increased productivity helps keep wages and benefits in the manufacturing sector higher than the average.

Manufacturing is a fundamental part of the US economy, and it has been a key pathway to economic development. Manufacturing creates jobs and services that depend on manufactured goods. Additionally, global trade is based primarily on goods, not services. Manufacturing generates 11% of US gross domestic product (valued at over $2.3 trillion in 2018),[3] employs over 12 million Americans,[4] represents ~8.5% of the total workforce, and pays 12% more than other jobs pay.[5] US manufacturing uses approximately 25% of the energy consumed nationally.[6] According to the Bureau of Economic Analysis,[7] manufactured goods account for half of US exports. In fact, world trade in manufactured goods has more than doubled between 2000 and 2017—increasing from $4.8 trillion to $12.2 trillion. The US share of world trade in manufactured goods[8] has grown from 7.6% in 2002 to 8.7% in 2017.

---

[1] https://www.manufacturingusa.com.

[2] https://www.areadevelopment.com/advanced-manufacturing/q3-2016/importance-manufacturing-to-US-economy-909033.shtml.

[3] https://www.bea.gov/system/files/2019-10/gdpind219_2.pdf.

[4] https://www.bls.gov/iag/tgs/iag31-33.htm.

[5] https://www.thebalance.com/u-s-manufacturing-what-it-is-statistics-and-outlook-3305575.

[6] https://www.energy.gov/sites/prod/files/2019/07/f64/011-AMO%20Strategic%20Analysis%20-%202019%20Peer%20Review.pdf.

[7] https://www.thebalance.com/bureau-of-economic-analysis-3305976.

[8] https://www.wto.org/english/res_e/statis_e/wts2018_e/wts2018_e.pdf.

Compared with other industries, manufacturing has been efficient in delivering value-added goods. It takes about 5.8 full-time equivalent manufacturing jobs to achieve $1 million in the value-added arena, as compared with 7.7 for both transportation and services and 16.9 for retail trade.[9] Productivity improvements—including the increased use of computers, robotics, and other efficient processes—will require new jobs that use computer-related skills to manage the efficient automated processes. For example, although the chemical industry is directly responsible for creating more than 500,000 jobs, it is indirectly responsible for creating an additional 7 jobs in a different segment of the economy for every job created by the business of chemistry.[10]

Improving the productivity and energy efficiency of US manufacturing, along with reducing life cycle energy and resource impacts of manufactured goods, and strengthening and advancing the US manufacturing workforce, are crucial elements in ensuring the role of manufacturing in the economy and maintaining the traditional position of the United States: leadership and security.

1.3 Energy and Environmental Impact of Manufacturing

US manufacturing accounts for around one-quarter of primary energy use in the United States, and has an annual energy bill in the vicinity of about $150 billion.[11] Developing cost-effective technology that improves the productivity of US manufacturing and reduces the energy intensity of manufacturing processes is necessary for the US industrial sector to remain competitive.

It is important to frame the basic R&D challenges in manufacturing and identify the current trends in manufacturing technology R&D. The AMO within the Office of Energy Efficiency and Renewable Energy (EERE) focuses on two primary opportunity categories:

- Energy Efficiency (or Energy Intensity), and
- Life Cycle and Resource Impacts (or Use Intensity).

1.3.1 Intensity Metrics and Definitions

Intensity metrics are a useful way to characterize the resources required to produce a product. Developing and communicating such a metric has many benefits, including tracking the impact of a technology or process innovation, identifying areas where research is needed, allowing for benchmarking and comparisons, and providing a general understanding of the role of various resources in an operation.

"Energy intensity" refers to the amount of energy input required for a unit of production output—the ratio of energy inputs per unit of production output (e.g., Joules/kg). Because industry uses energy to manufacture products, improving manufacturing energy intensity is most often achieved through the development of new, more efficient processes; better integration of existing processes; or recovery of waste heat for use in a secondary process, which also reduces overall energy demand. Because energy is a major source of emissions in industry, improving manufacturing energy intensity also can reduce emissions. Although scientific breakthroughs can improve energy use up to thermodynamic limits,

[9] https://mapifoundation.org/manufacturing-facts/2016/9/13/how-important-is-us-manufacturing-today.
[10] https://www.americanchemistry.com/GBC2019.pdf.
[11] https://www.eia.gov/consumption/manufacturing/data/2014/pdf/table7_9.pdfhttp://www.eia.gov/consumption.

energy use is necessary for industry and manufacturing production, and energy intensity improvements alone will not eliminate emissions.

"Carbon intensity" refers to net changes in atmospheric carbon concentration that occur during the production of a unit of output—it is the ratio of net sum total of fuel use plus material transformations to carbon emissions per unit of production output (e.g., $MtCO_2eq/kg$). The carbon content of energy resources and raw material input is a source of carbon emissions in industry. Improving the carbon intensity of manufacturing through choice of fuel and raw material input can reduce emissions beyond the reductions enabled through energy-intensity improvements alone. Reducing the carbon intensity of manufacturing, however, often depends on scientific breakthroughs in materials and chemical sciences or processing technologies, or gaining a better understanding of complex interactions between materials and the Earth's biosphere (Smil 2003).

"Use intensity" refers to how a unit of production output is used—it can include a broad array of uses per unit of production output (e.g., end-of-life extension/kg; Δenergy/avoided; Δemissions/avoided). Often enabled by scientific breakthroughs in material properties, product substitutions, and creative innovations that eliminate inefficiencies and wastes, better use of production outputs can avoid inefficient energy consumption and associated emissions entirely. For example, scientific breakthroughs in material properties can improve the durability of a product and extend the usable life or optimize the strength of a material to be used for new applications and recovered from existing products. The circular economy envisions the capability to reduce, reuse, remanufacture, and recycle (Re-X) all materials, which would dramatically decrease the need for virgin materials. In some applications, materials are used in excess of what is needed given their properties. Many construction projects, for example, use significantly more cement and steel than is needed for building integrity.

Using only the material needed is referred to as "material efficiency," which also can be applied to the additive manufacturing (AM) process because of its use of only the material needed for the final product design. Enabling products should be designed for easy Re-X; this is an opportunity for science to drive the circular economy forward.

"Water intensity," as defined in this report, compares the characteristics of the water being used with the desired output of the process using the water (e.g., million gallons per ton of product, water temperature rise per degree of material cooling). In trying to quantify this metric, however, many challenges associated with characterizing manufacturing water use arise.

Although these intensity measures are in separate categories, there can be interconnections between the various measures. For example, a manufacturing process change could impact both energy intensity and water intensity. And if the process change included a switch in input energy type (e.g., from natural gas to electricity), this could impact carbon intensity.

1.3.2 Energy Use and Intensity

The manufacturing sector is of particular importance to DOE not only because of the considerable amount of energy currently consumed, but also because advanced manufacturing technologies have the potential to provide a competitive advantage over the practices that presently are widely in use, to reduce life cycle energy impacts of manufactured products.

The manufacturing sector draws on a diverse set of energy resources to serve the variety of end uses that transform raw materials into manufactured products. Steam and fuel energy are used during thermal processes such as melting, smelting, curing, and drying. Electricity is used to drive motors in pumps, fans, compressors, and materials-handling equipment. Manufacturing facilities also consume energy in non-process applications such as space heating and lighting. Figure 4 illustrates the distribution of on-site manufacturing energy use by energy and fuel type.

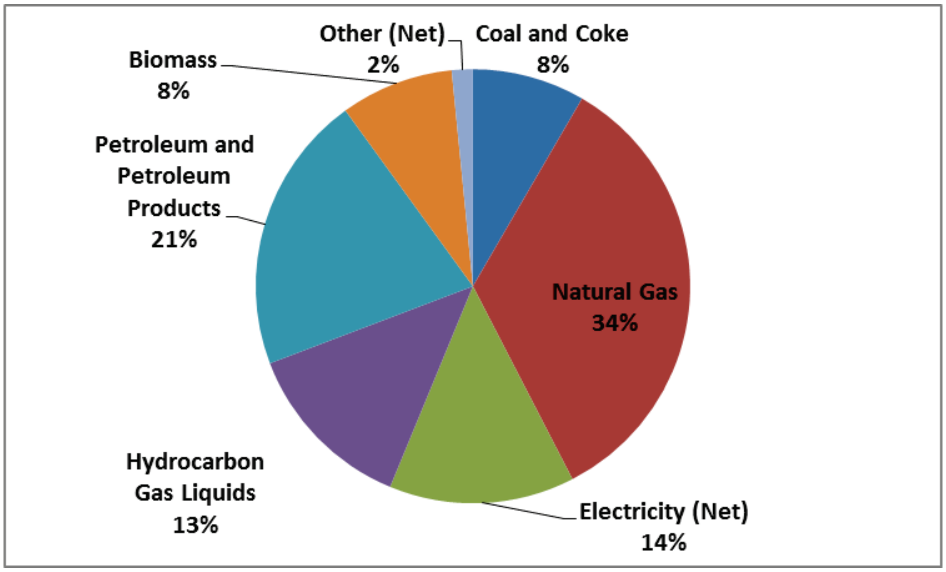


Figure 4. Energy consumption in the manufacturing sector by fuel type in 2014.[12] (*Source*: 2014 EIA Manufacturing Energy Consumption Survey, Table 1.5)

DOE AMO has mapped the flow of energy supply, demand, and losses, as well as GHG combustion emissions, from fuel use in 15 diverse US manufacturing industry-specific "Energy and Carbon Footprints"[13] using the latest US EIA Manufacturing Energy Consumption Survey[14] data for 2014.[15] As shown in Table 1, these 15 sectors constituted about 94% of primary energy and 95% of on-site energy.

Table 1. US Manufacturing Energy and Carbon Footprints in 2014

Manufacturing Industry	Primary Site Energy		On-Site	
	Energy (Tbtu)	CO_2 ($MMtCO_2e$)	Energy (Tbtu)	CO_2 ($MMtCO_2e$)
Alumina and Aluminum	469	25	242	7
Cement	371	28	298	22
Chemicals	4,542	260	3,527	163

[12] Derived from U.S. Energy Information Administration, "2014 Manufacturing Energy Consumption Survey," Table 1.5. https://www.eia.gov/consumption/manufacturing/data/2014/pdf/table1_5.pdf.
[13] https://www.energy.gov/eere/amo/manufacturing-energy-and-carbon-footprints-2014-mecs.
[14] https://www.eia.gov/consumption/manufacturing/.
[15] https://www.eia.gov/consumption/manufacturing/data/2014/.

Table 1. US Manufacturing Energy and Carbon Footprints in 2014 (continued)

Manufacturing Industry	Primary Site Energy		On-Site	
	Energy (Tbtu)	CO_2 ($MMtCO_2e$)	Energy (Tbtu)	CO_2 ($MMtCO_2e$)
Computers, Electronics, and Electrical Equipment	526	28	232	4
Fabricated Metals	620	33	344	11
Food and Beverage	1,762	96	1,209	49
Forest Products (Pulp and Paper)	2,995	92	2,473	47
Foundries	206	10	118	3
Glass	300	16	208	9
Iron and Steel	1,524	62	1,084	26
Machinery	319	17	164	5
Petroleum Refining	3,744	248	3,373	211
Plastics and Rubber Products	662	36	294	6
Textiles	245	14	132	4
Transportation Equipment	616	33	318	9
Subtotal	**18,901**	**998**	**14,016**	**576**
All Manufacturing	**20,008**	**1,064**	**14,759**	**609**
Subtotal % of All Manufacturing	*94%*	*94%*	*95%*	*95%*

The AMO has also undertaken a series of energy bandwidth studies for a number of energy-intensive manufacturing subsectors. Using energy intensity and annual production data, these studies assess the current typical energy use, the potential for improvement if state-of-the-art technologies were deployed, and the potential for future energy savings if next-generation technologies under development were realized. The differences between these ranges are termed "energy bandwidths." The results inform which manufacturing industries, processes, and subprocesses are the most energy intensive and which offer the greatest savings opportunities from technology advancements. Data also can feed into other analytical studies to explain the contribution of the manufacturing phase of the product to the net life cycle energy impacts of end-use products.

Bandwidth analyses have been completed for these subsectors:

- Chemicals;
- Petroleum refining;
- Pulp and paper;
- Iron and steel;
- Food and beverage;
- Cement;
- Glass;
- Plastics and rubber products; and
- Water desalination.

Bandwidth analysis represent the potential savings that could be attained through successful deployment of applied technologies that are under development worldwide.[16] In addition to these studies, bandwidth

[16] For a summary of manufacturing sector energy bandwidth reports, see Section 3.2.1, Manufacturing Energy Savings Potential, Table 6.

reports have been completed to analyze manufacturing energy use for a series of lightweight materials, including carbon fiber–reinforced polymer composites, glass fiber–reinforced polymer composites, aluminum, advanced high-strength steel, magnesium, and titanium.[17]

Analysis of the opportunities to improve energy intensity typically considers individual components, processes, or products (Granade et al. 2009). Efficiency, however, must be defined more broadly because manufacturing energy use is a function of the processes that transform material inputs into manufactured products and the volume of materials processed (Laitner et al. 2012). DOE has invested in the development of methodologies and tools[18] to evaluate the energy embodied in materials, allowing for a more expansive approach to traditional energy-efficiency analysis by considering energy through the full life cycle,[19] where the energy reduction potential is equivalent to the magnitude of traditionally defined energy efficiency (Cooper et al. 2017).

Looking forward, the US Energy Information Administration's (EIA) *Annual Energy Outlook* 2019 reference case forecasts that US manufacturing output will continue to grow over the next 10 years but will slightly lag behind overall growth in the US gross domestic product. Even though manufacturing energy intensity is expected to continue to decrease, energy consumption in manufacturing is expected to grow at a much greater rate than in the economy as a whole (EIA 2019).

1.3.2.1 Process Efficiency

A wide array of process technologies and manufacturing operations is used to convert raw materials to finished products—often through long sequences of intermediate product forms. In the United States, nearly 300,000 manufacturing establishments produce a broad range of products.[20] Opportunities for process-efficiency improvements exist throughout these supply chains—from fundamental commodities, such as metals and chemicals, to sophisticated final-use products such as automobiles and high-technology devices.

Efficiency gains can be attained by improving the technologies and energy-management strategies used at manufacturing plants, making these facilities more efficient. Energy use at industrial facilities can be grouped into five key clusters of equipment:

- **Process heating systems**, including furnaces, ovens, kilns, evaporators, and dryers;
- **Motor-driven systems,** including pumps, fans, compressors, and materials handling and processing equipment;
- **Other process systems,** including electrochemical systems and process cooling equipment;
- **Non-process systems,** including facility heating, ventilation, and air conditioning, facility lighting, and on-site transportation; and
- **Steam systems and other on-site generation**, including conventional boilers, cogeneration (combined heat and power) equipment, and other equipment for on-site electricity generation such as solar or geothermal energy.

[17] https://energy.gov/eere/amo/energy-analysis-data-and-reports.
[18] For example: https://greet.es.anl.gov/; https://www.nrel.gov/manufacturing/mfi-modeling-tool.htm.
[19] A product life cycle can be defined as the sequential stages that are required to extract, convert, and otherwise process natural resources into finished products that are used until they ultimately are retired at their end of life.
[20] https://www.census.gov/data/tables/2012/econ/census/manufacturing-reports.html.

1.3.2.2 Process Intensification

Process intensification (PI) uses new techniques and equipment that reduce the energy consumption, equipment size, production-to-capacity ratio, or waste production associated with a manufacturing or processing plant (Stankiewicz and Moulijn 2000). An example is the replacement of unit operations with compact, task-integrated devices that combine many operations into a single piece of equipment. Systems integration will ensure that the full benefits of process intensification are attained.

Although PI has its origins in chemicals processing, PI strategies are not limited to the chemicals industry. Similar process technologies are widely used in many of the energy-intensive industries—including metals refining and manufacturing, pulp and paper, and food processing—and PI and system integration approaches have broad applicability. Manufacturing processes for a range of applications benefit from fewer processing steps, low-energy processes and unit operations, hybridization, and more integrated equipment and systems designs. Benefits beyond energy efficiency include the ability to be more flexible with production output, leading to improved productivity and production resilience.

Modular equipment designs can improve energy efficiency, reduce capital costs, and simplify plant construction. PI techniques also can be used to take advantage of alternative forms and sources of energy that provide benefits in terms of energy efficiency, reduced environmental impact, and cost. For example, microwave heating could accelerate certain chemical processes by several orders of magnitude compared with conventional methods (Gedye, Smith, and Westaway 1988). Centrifugal field, ultrasound, and electric field energy could provide additional opportunities. Flexibility needed by new fuels and feedstocks could be accommodated by integrating energy-conversion technologies into process equipment. The use of natural gas in the industrial sector as a fuel and chemical feedstock is projected to grow substantially in the coming years, as natural gas has become a plentiful and low-cost resource in the United States within the past few decades (EIA 2019).

Materials transformations in thermally intensive systems (process heating) are candidates for lower–thermal-budget technologies that reduce the energy requirements of materials processing. Thermal PI approaches that improve the properties of manufactured products can have a significant effect on energy consumption, given that more than 7 quads of manufacturing energy use annually is attributable to processes involving heating (almost 70% of all process energy use), with approximately 34% of that energy lost as waste heat, accounting for more than 2,500 trillion Btu annually (DOE 2014a). Systems integration of energy-saving technologies—such as WHR and cogeneration of heat and power—could be applied in many industries and has the potential for broad impact. Improvements in industry-specific technologies offer additional benefits, especially in the energy-intensive manufacturing industries (i.e., food, paper, bulk chemicals, refining, glass, cement, iron, steel, aluminum) that collectively account for about two-thirds of all end-use industrial energy (EIA 2019).

1.3.2.3 Waste Heat Recovery

Industrial process heating is essential in the manufacture of most consumer and industrial products, including goods made from metal, plastic, rubber, concrete, glass, and ceramics. The manufacture of steel, for example, involves a combination of smelting, metal melting, and various heat-treatment steps. During polymer fabrication, fluid heating typically is used to distill a petroleum feedstock and to cure the final polymer product. Common to all process heating applications is the generation and transfer of heat. Industrial waste heat generally is found in four forms:

- Sensible heat of solids, liquids, and gases;
- Latent heat contained in water vapor or other type of vapors and gases;
- Radiation and convection from hot surfaces; and
- Direct contact conduction (in a few instances).

The largest and most visible sources of waste heat for most industries are exhaust/flue gases and heated air from heating systems. High-temperature exhaust gases from furnaces, boilers, heaters, and dryers, for example, can contain 20% to 60% of the total heat supplied to the system. Although waste heat in the form of exhaust gases is readily recognized, waste heat in industrial heating processes also can be found within heated water or liquids, hot products, and high-temperature surfaces.

Improvements in current waste heat recovery (WHR) technologies are needed before they will be widely accepted in industrial facilities. Industrial users demand equipment lifetimes of several years, low maintenance and cleaning requirements, and consistent and reliable performance over acceptable life. For low-temperature waste-heat streams (i.e., less than 600°F), low heat-transfer rates and large recovery equipment footprints are major barriers. For high-temperature waste-heat streams (i.e., more than 1,200°F), materials must withstand high-temperature gases that could be contaminated with particulate matter or corrosive chemicals. Key R&D topics include the following:

- Hot-gas cleaning technologies that can remove contaminants from waste-heat streams, allowing long-term operation of heat-recovery equipment and avoiding service interruptions for cleaning;
- Advanced materials that can withstand high-temperature waste-heat sources;
- Compact heat exchangers to reduce the size or footprint of the heat-recovery equipment;
- Secondary heat-recovery technologies to supplement and enhance the performance of primary WHR equipment;
- Integrated heat-recovery technologies that combine heating elements with heat-recovery equipment, eliminating the need for hot-air piping and external heat-recovery equipment;
- Innovative condensing heat exchangers for gases having high moisture levels and particulates, such as the waste streams discharged from paper- and food-production equipment;
- Liquid-to-liquid heat exchangers for heat recovery from wastewater containing contaminants; and
- Thermoelectric generators for electricity production from otherwise unusable waste-heat streams.

The recovery and reduction of waste heat generated in manufacturing systems offers one of the best methods for reducing energy intensity in manufacturing plants. Waste heat can be recycled either by redirecting the waste stream for use in other thermal processes (e.g., flue gases from a furnace could be used to preheat a lower-temperature drying oven) or by converting the waste heat to electricity. In some cases, the technologies and hardware needed to economically recover waste heat in the form of hot gas, liquid, or water already are available. Most industrial plants, however, do not take advantage of these utilities. According to EIA Manufacturing Energy Consumption Survey data, only approximately 6% of US manufacturing facilities were using any type of WHR as of 2010 (EIA 2013). Table 2 shows potential recoverable waste heat in five industries from various sources with harsh environments.

Table 2. Recoverable Waste Heat from Various Sources with Harsh Environments in Five Major Industries (*Source*: Vance et al. 2019)

Industry	Waste Heat Source	WHR Technology/ System Status	Production** (MM Tons/Year)	WHR Potential Terawatt Hours/Year*			Exhaust Gas Flow
				Sensible	Chemical	Total	
Steel	Blast furnace gases	Available and widely used—partial WHR	25.4	3.8	42.31	46.1	Constant
	Electric arc furnace exhaust gases	Available, not widely used—partial WHR	49.64	6.15	7.89	14.05	Varying
	Basic oxygen process	Available, not widely used—partial WHR	29.16	1.2	6.83	8.03	Varying
Glass	Flat glass	Available for air-fuel combustion only and widely used—partial WHR***	5.00	3.63	Negligible	3.63	Constant
	Container glass	Available for air-fuel combustion only and widely used—partial WHR***	10.00	5.7	Negligible	5.7	Constant
	Glass fiber (all types)	Available for air-fuel combustion only and partially used—partial WHR***	3.00	1.07	Negligible	1.07	Constant
	Specialty glass	Available for partial WHR but rarely used	2.00	2.23	Negligible	2.23	Constant
Aluminum	Aluminum melting furnaces (fuel fired)	Available, not widely used—partial WHR	10.00	4.7	Small—site specific	4.7	Constant
	Anode baking	Available but NOT demonstrated	2.22	0.55	Small—site specific	0.55	Constant
	Calcining	Available but NOT demonstrated	Data not currently available				

Table 2. Recoverable Waste Heat from Various Sources with Harsh Environments in Five Major Industries (*Source*: Vance et al. 2019) (continued)

Industry	Waste Heat Source	WHR Technology/ System Status	Production** (MM Tons/Year)	WHR Potential Terawatt Hours/Year*			Exhaust Gas Flow
				Sensible	Chemical	Total	
Cement (Clinker)	Cement kiln exhaust gases from modern clinker-making operation	Available, not widely used—partial WHR	76	17.15	Negligible	17.15	Constant
Lime	Lime kiln exhaust gases based on commonly used rotary kiln –type operation	Available, not widely used–partial WHR	18.3	10.45	Negligible	10.45	Constant
Total (terawatt-hours/year) = 113.6							

* For a few waste-heat sources (particularly in steel, aluminum, and glass industries), a small quantity of waste heat already is being recovered.
** Production data for steel industry are from 2016, glass industry from 2002, aluminum industry from 2012, and cement and lime industry from 2016.
*** WHR technologies currently not available/used for oxy-fuel fired systems

1.3.3 Material Use and Intensity

Earth has a limited amount of natural resources. Using raw materials in a way that maximizes their useful life and their value to society is critical. Minimizing the energy requirements to transform raw materials into usable products through manufacturing and good societal decisions is also important. Efficient material production and product manufacturing are key to making goods that will efficiently perform their tasks. In addition, there are opportunities to enable the recycling, remanufacturing and reuse of materials to reduce raw materials needs. The economic and energy intensity recycling, remanufacturing and reuse of materials must be considered. The following sections discuss topic areas related to use and material intensity.

1.3.3.1 Reduce, Reuse, Remanufacture, and Recycle

The life of a product follows typical stages from production to end of life. As shown in Figure 5, natural resources are extracted and refined, materials are processed, parts are manufactured, and products are assembled. The product then is used and eventually reaches the end of its useful life. Each stage of a product's life involves one or more of the following inputs: cost, energy, consumption of natural resources, creation of wastes, and time. The most benefit can be gained by reusing a product, because this requires the fewest quantities of these inputs. The next-best scenario is to remanufacture a product, which could require the machining or replacement of one or more of its parts. Recycling the materials in a product reduces the natural resource burden and potentially could reduce the cost.

The materials generated by recycling a product might not always be used to make the same product. In some instances, this results in downcycling, where the new product has a lesser quality, value, or function. Alternatively, recycled materials can be upcycled into products of higher quality, value, or function. The ability to upcycle a product is preferred, but many times it requires excessive inputs that outweigh the benefits of the upcycled product.

Additionally, reduced material content can provide cost and environmental impact reductions. Another method that sometimes falls into recycling is burning materials for energy recovery. Products can be designed to enable easier disassembly for remanufacture and recycling. New manufacturing technologies also can allow for the use of new, more recyclable materials in a product.

All of these principles—with the exception of reuse—rely on manufacturing concepts to enable their full benefit.

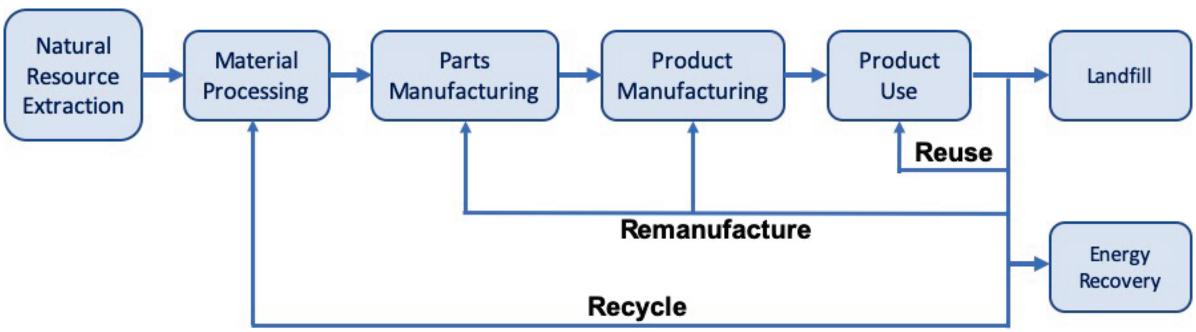


Figure 5. The life of a product follows a typical set of stages from production to end of life. (*Source*: Jeff Spangenberger, Argonne National Laboratory)

1.3.3.2 Circular Economy

In a circular economy manufactured products are designed and optimized for a cycle of disassembly, remanufacture, and reuse at the end of use resulting in a reduction in waste and potential for reduction in need for raw material inputs. The circular economy is a complex system problem and must be restorative or regenerative by design and, ideally, also reduce energy use in addition to raw material needs. Of course, designing products that are compatible with the circular economy can be challenging. The world economy is 8.6% circular[21] in that global recovery and reuse account for only 8.6% of the materials that are entering the economy. Opportunities exist to increase that percentage. Section 3.5 further discusses end of life issues and the circular economy.

[21] The Circularity Gap Report, 2019 (https://www.legacy.circularity-gap.world/2019).

1.3.3.3 Material Efficiency

Material efficiency is an essential concept to make manufacturing more cost effective and to minimize environmental impacts. Material efficiency is related to the concepts discussed in Section 1.3.3.2, as shown in Figure 6. Material efficiency starts in the design phase of a product's life. A product should be designed to meet its performance targets with the minimum material requirements. Designing a product for long life and recyclability also results in efficient material use. During manufacture, minimal material loss and waste should be targeted.

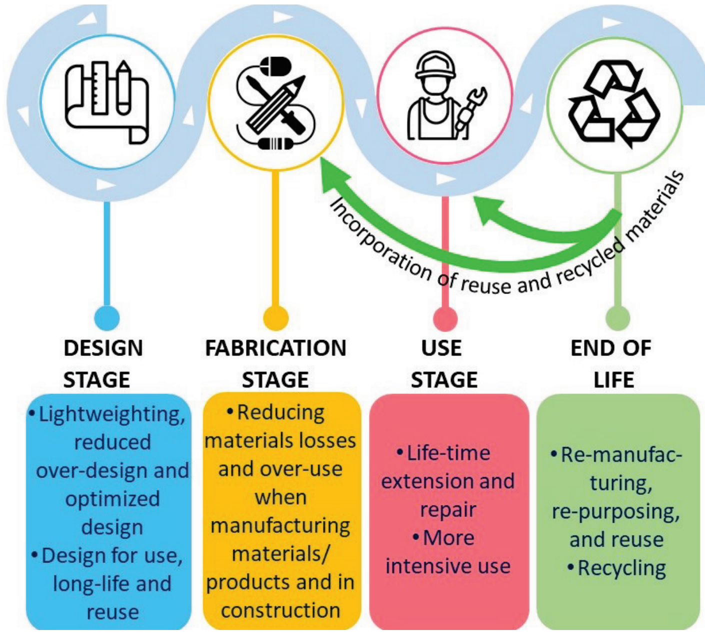


Figure 6. Material efficiency strategies across the value chain (International Energy Agency).

(*Source*: IEA 2019, *Material efficiency in clean energy transitions*, IEA, Paris), all rights reserved)

Efficiently using the materials in a product reduces the natural resources consumed while the product still performs the necessary tasks. In addition to simply using less material, material efficiency involves the sourcing of materials that are produced and manufactured more efficiently. The ability to manufacture with more material options for a product allows engineers to make the best choice. Design tools and advanced manufacturing technologies help to push the boundaries of this concept and, in many cases, enable a material to be used more efficiently in a product or make a material capable of being used that otherwise would not be used.

1.3.3.4 Material Substitution

Material substitution is the concept of replacing one material with another to obtain a benefit in cost, performance, or environmental impact. This can include a benefit gained during the manufacturing phase, the use phase, or the end of life of a product. The use of recycled content is a common example of material substitution. Content recycling is done primarily for reduced cost and environmental impacts. Another example is the lightweighting of vehicles. Using plastics in vehicles can reduce the overall weight of a vehicle, which improves fuel efficiency during the use phase. Careful consideration of substitutions must be made, however. For example, a material could be cheaper to use in a product, but the performance or environmental impacts might be compromised. Additionally, a material could be used that might not be recyclable or could prevent a product from being remanufactured. The design must consider all of these issues before a material is selected. Material substitution also can be used as a means of securing a steady supply chain.

1.3.4 Environmental Emissions and Intensity

As previously indicated in Table 1, manufacturing accounted for an estimated 1,064 $MMtCO_2e$ from its use of fuels in 2014. Process changes, through efficiency improvements or changes to fundamental unit operations, provide opportunities to reduce CO_2 emissions—both those inherent in the primary energy inputs and those released by material transformations. Additionally, feedstock substitution could reduce or eliminate emissions in a manufacturing process. For example, selecting a reduction process that replaces coke with hydrogen eliminates the CO_2 byproduct. Similarly, bio-derived feeds can have a lower emissions than similar chemicals derived from petroleum processing if the emissions from producing bio-derived feeds is lower. The use of on-site renewables, notably from solar sources, could directly or indirectly provide power for processes to replace purchased electricity or generate emission-free feedstock materials.

Advancements in manufactured products can also reduce energy use and associated emissions in their end uses. Reducing use intensity is related to the emissions and energy that are avoided by consuming less primary feed to generate materials, often enabled by the material properties. Material properties could include durability that extends the usable life of a product, or strength that allows a fraction of the material to be used for a given application. The circular economy envisions the reuse, repair, and recycling of all materials, which would dramatically decrease primary feedstock use.

1.3.5 Water Use and Intensity

The energy sector and the industrial sector both require substantial water input in wide-ranging water intensities to produce diverse products (Figure 7). According to the US Geological Survey (Dieter et al. 2018a; Kenny et al. 2017; Maupin et al. 2017), about 180 billion gallons per day (Bgal/d) of water was withdrawn for use in US agricultural and industrial manufacturing during 2005. This decreased to 165 Bgal/d in 2010 and 162 Bgal/d in 2015. The US manufacturing sector used 18.2 Bgal/d of water in production operations in 2005, as estimated by the US Census Bureau (Becker 2016), which accounts for ~4.4% of total water withdrawals in the United States. Separately, thermoelectric generation and industrial production were estimated to account for 195 Bgal/d and 25 Bgal/d of water use in 2014, respectively (DOE 2014b). Water resources vary regionally; in many cases, manufacturing is increasingly constrained by limited fit-for-purpose water availability, especially in water-stressed areas. Competing uses strain available water resources and raise the specter of resource depletion and environmental degradation.

Water is used throughout the manufacturing sector, often in ways that are critical to operations. Among other things, water is used for its superior and predictable thermal properties (steam generation, cooling), as a solvent (rinsing, cleaning), to transmit force (hydraulic applications), as part of a product being made, and for general domestic and landscaping purposes. For many operations, losing access to water means slowing or stopping production. Despite this situation, water conservation is in its infancy—particularly compared with energy conservation. Water conservation's relatively early state can be attributed to a myriad of economic, practical, and technological issues. Water generally is a very small percentage of production costs. Often, water-conservation projects do not meet return-on-investment requirements. Most manufacturing water used is self-supplied, which reduces incentives to conserve, and water efficiency also is not a widespread goal for technology developers. Several national assessments document changes of water intensity in US manufacturing over the past two decades, with a focus on energy (Dieter et al. 2018a; Wu, M. Mintz, Wang, and Arora 2009; Macknick, Newmark, Heath, and Hallett 2011; DOE 2014b).

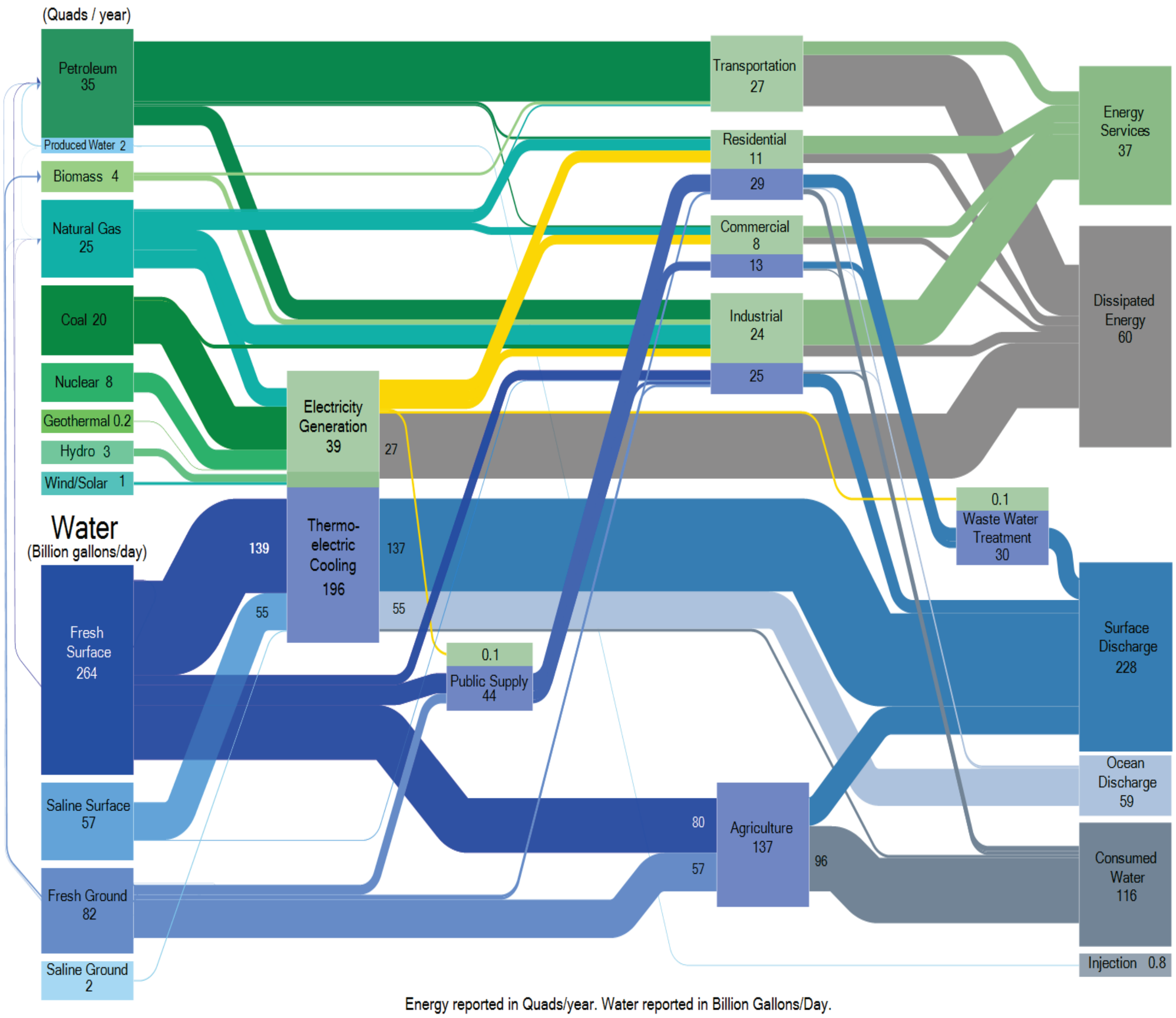
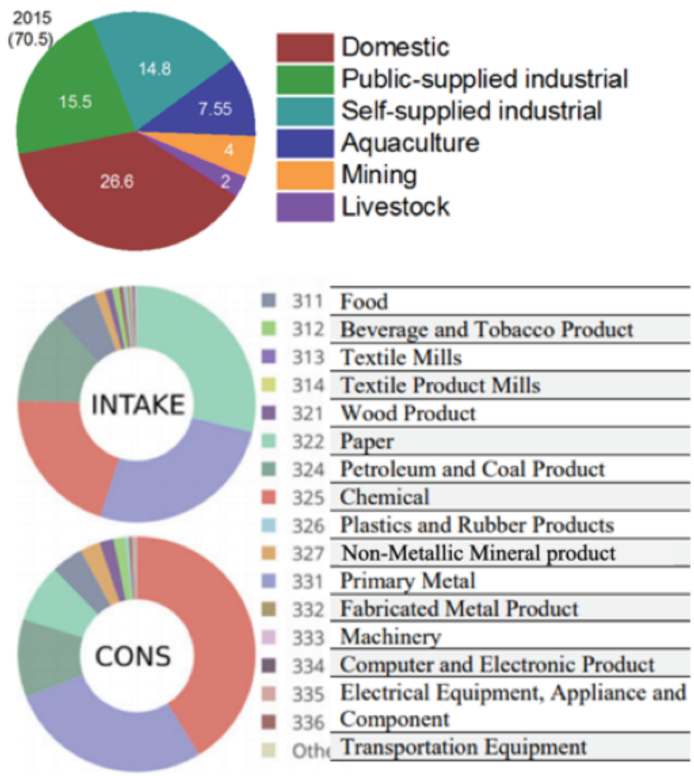


Figure 7. Energy-water interdependence in the United States. (*Source*: DOE, The Water-Energy Nexus: Challenges and Opportunities, 2014)

Challenges and opportunities in the water-energy nexus are analyzed based on dominant technologies in a DOE report (DOE 2014b). These studies evaluated the water intensity of major fuel pathways such as coal-to-electricity; gasoline from Canadian oil sands, Saudi Arabian crude, and US conventional crude; and agricultural feedstock to biofuels. Water is consumed through life cycle stages of production, including drilling, raw-material extraction, and feedstock processing and conversion. Globally, the concept of water footprint and virtual water has emerged as an analysis framework that characterizes water intensity by capturing water use (direct and embedded) throughout the entire supply chain. It is recognized by the Food and Agriculture Organization of the United Nations and industrial sectors, and has been used to develop industry applications of water-footprint assessment that span from manufacturing of food, fuel, and chemical products (Figure 8) (Dieter et al. 2018b; Mekonnen and Hoekstra 2010; Philippot et al. 2019; Rao, Sholes, Morrow, and Cresko 2017).



Item	Water Footprint (Gallons)
Steel (1 ton)	62,000
Plastic (1 ton)	44,000
Passenger car	39,090
Lithium ion battery pack in an electric vehicle	> 7,400
Paper (1 ton)	> 4,500
Cement (1 ton)	1,360
Leather shoes (1 pair)	3,626
Smart mobile phone	3,190
Cotton (1 lb)	1,320
Bovine meat (2 lb)	2,036
Butter (1 lb)	1,015
Pig meat (1 lb)	790
Chicken meat (1 lb)	571
Milk (1 lb)	135
Beer (1 gallon)	47
Petroleum refining (1 gallon)	2.5

Figure 8. Estimated use (in billion gallons per day) of virtual water in United States in 2015 with respect to the types of water users. (*Source*: upper-left panel: US Geological Survey; data extracted from Dieter et al. 2018b, USGS no. 1441; lower left panel: adapted from Rao, Sholes, Morrow, and Cresko 2017, DOE; right pane: data extracted from Mekonnen and Hoekstra 2010 and Philippot et al. 2019)

In the past decade, industrial water use decreased slightly from 34.5 Bgal/d in 2005 to 30.3 Bgal/d in 2015 because of increasingly efficient manufacturing practices (Dieter et al. 2018a; Kenny et al. 2017; Maupin et al. 2017). A breakdown of US manufacturing total water withdrawals and consumption by subsectors (Rao, Sholes, Morrow, and Cresko 2017) suggests that the following industrial subsectors have the greatest water withdrawals (corresponding North American Industry Classification System codes are given in parentheses): pulp and paper (322), primary metals (331), chemicals (325), petroleum refining (324), and food (311). These sectors also have the greatest water consumption, but in a different ranking of chemicals, primary metals, petroleum refining, pulp and paper, and food. Note that each industry has its own unique requirements for water quality, ranging from flexible water chemistry in boilers to extreme purification in semiconductor manufacturing.

Water consumption for feedstock and fuel production varies considerably by region, type of feedstock, soil and climatic condition, production technology, and extent of steam-recycling and water-management methods (DOE 2006; Wu, M. Mintz, Wang, and Arora 2009). Although thermoelectric power generation accounts for a majority of freshwater withdrawal, its energy requirements are relatively small. In contrast, the industrial sector demands a significant amount of both energy and water input (Figure 8). Therefore, reducing water intensity while maintaining or improving energy efficiency is critical for the industrial sector. A DOE-led Water-Energy Nexus assessment further identified opportunities to jointly reduce energy and water in manufacturing for specific industrial sectors based on watershed impact (Cresko 2017). Given the diversity of production processes and geospatial locations, a water footprint customized to a specific production process and region would provide geospatial-relevant environmental impact assessment. Growing concern about water resource stress motivates process design to incorporate freshwater resource availability, in addition to energy-efficiency process economics and infrastructure considerations, in technology R&D for manufacturing.

The water footprint of manufactured goods falls in a wide range from a few gallons to thousands of gallons.[22] For example, manufacturing an average passenger car can consume about 39,090 gallons of water, and 3,190 gallons are needed to make a smart phone (Mekonnen and Hoekstra 2010; Philippot et al. 2019). Regarding water sources in 2015, freshwater accounted for 87% of total withdrawals (both surface water and groundwater), and the remaining 13% was saline water. Of the total, 82% self-supplied industrial withdrawals came from surface water, 94% (6%) of which were freshwater (saline water) (Mekonnen and Hoekstra 2010; Philippot et al. 2019). Industrial water use is a major source of wastewater, as toxic pollutants are released to freshwater from fabricating, processing, and washing products (Mekonnen and Hoekstra 2010; Philippot et al. 2019).

American manufacturing competitiveness is challenged by water-intensive manufacturing industries, such as forest products, food and beverage, chemicals, and petroleum refining. These and other manufacturing industries have the potential to benefit from reduced cost and to improve energy efficiency with water-reuse and wastewater-treatment technologies, and water replacements. Water can be used for energy-production applications such as in thermoelectric-cooling and hydropower technologies, for example, as well as in energy-demand applications such as industrial process cooling and industrial WHR. Improving recyclability of water and increasing water reuse within the manufacturing sector could lead to significant savings in resource recovery. Likewise, reducing water demand will lead to considerable energy saving by reducing the amount of energy required for heating and pumping water.

A major challenge in increasing water-conservation efforts is the lack of information for characterizing water use. Unlike energy information—which is gathered by the US DOE EIA for several economic sectors—there is no centralized collection of water-use information in the United States. The nearest analog to the EIA's collection is the US Geological Survey's compilation of state-level water reporting. This is published every 5 years and does not break down manufacturing information by individual subsectors (e.g., primary metals, food, and transportation equipment). Given the diversity in how manufacturers use water, the data provide limited insights into manufacturing water-use characteristics. At the individual facility level, water use by process or end use is seldom tracked. In many cases, tracking of water across the entire facility is not comprehensive. For example, self-supplied water (water not obtained from a public supply)—estimated to be 75% of all manufacturing water use—might not be tracked because it is not purchased (Solley, Pierce, and Perlman 1998; Maupin et al. 2014). The lack of information hinders developing metrics to track impacts of technologies; understanding which sectors, processes, and equipment would benefit most from scientific and technological improvements; and generally guiding research efforts. In short, "what matters" when it comes to water use is difficult to determine, and what matters might not be uniformly true across all manufacturing subsectors. This is unlike energy, for which certain usage characteristics are universally important (e.g., waste, cost, consumption).

[22] https://www.watercalculator.org/footprint/the-hidden-water-in-everyday-products

2 Advanced Manufacturing State of the Art

Recent advances in digital manufacturing, computing, and sensors are leading to significant opportunities for manufacturers and impacting many manufacturing processes. For example, advances in edge computing are expected to impact a number of manufacturing processes. With such developments, however, come additional challenges for handling the large amounts of data produced, security of that data, and challenges to update the current manufacturing infrastructure. Digital manufacturing and associated opportunities and challenges are discussed in first section, followed by specific examples of the current state of the art of standard manufacturing processes.

2.1 Digital Manufacturing

In addition to advances in digital hardware (e.g., the industrial "Internet of Things") and software research, progress in the area of digital manufacturing depends on advances in the use of big data (e.g., machine learning, or ML) and optimization analytics (e.g., Bayesian optimization and Industry 4.0) and related contributions from scientific disciplines, including materials science, chemistry, and mathematics.

2.1.1 Cloud/Fog/Edge Capabilities and Utilization

This topic targets the use of cloud, fog (between edge and cloud), and edge storage and processing. Significant work has been done in this area in both cloud and edge processing. In particular, smart sensors are sensors that have integrated processing capabilities. In reality, these smart sensors primarily are low-cost processors augmented with sensors. In general, there are two types of processors that are used: real-time type controllers such as Arduino and the programmable real-time unit on the Beagle Bone Black, and more powerful microprocessor-based systems such as Raspberry Pi and X486 systems. Microprocessor systems typically run an operating system, with the most prevalent being Linux, Android, and Windows.

The biggest challenge in this area is determining where data processing and storage should happen. For example, accelerometers are widely used for rotating system diagnostics and prognostics (e.g., motors, bearings, turbines, pumps). For rolling element bearings on typical machine tools, sample rates on the order of tens of kilohertz (kHz) are required for both frequency and time-based diagnostics and prognostics. Clearly, within a short time, a single sensor can generate a significant amount of data (megabytes to gigabytes). A simple set of Fourier transforms can reduce these large data sets into several key frequency components that are critical for rolling element diagnostics and prognostics. Thus, several megabytes of raw data can be converted into tens of bytes of data representing these key frequencies. Note that such processing can be—and often is—considered a form of data compression. If processing is executed at the sensor, a larger amount of storage is needed at the sensor as well as a processor. These increased requirements result in greater power demands at the point of measurement. In general, if the sensor is powered via a wire, then the power requirements are not critical. If the sensor is battery powered, however, then power requirements must be carefully considered. Conversely, if raw data are sent to the cloud, then greater bandwidth data transmission is necessary. Increased bandwidth has increased power requirements as well as infrastructure costs. Also, if significant numbers of sensors are employed, then the bandwidth of the plant infrastructure (e.g., the WAN/Wi-Fi of a production facility) can easily be taxed and exceeded.

There are still gaps and technical challenges and the research activity has focused on understanding the best practices for designing such a sensor system (Chavarría-Barrientos, Batres, Wright, and Molina

2018; Guinard, Trifa, Mattern, and Wilde 2011). Although a single approach or answer does not exist, efforts have been focused on finding an optimal solution for a particular set of circumstances. Furthermore, understanding how to shift from one set of practices to another set is based on available technology or the particular need (Menon, Shah, and Coutroubis 2018; Singh and Willcox 2018). The answer relies on requirements-related issues such as power consumption, battery energy storage capacity, and the ability to provide power to the smart sensor (perhaps in a wireless mode). The impact of improved batteries, lower-power processors, and optimized power models for batteries as well as processor/sensor power consumption cannot be underestimated (Cherupalli et al. 2017; Liu, L. et al. 2017).

As the needs and capabilities change, the ability for the system to shift from edge operations to cloud operations will change, and an understanding of where the processing and data storage should take place and how it will affect digital manufacturing will be needed. It will require quantifying criterial and processing/storage capabilities that are necessary to shift processing from the edge to the cloud as more processing needs change (Raileanu, Borangiu, Morariu, and Iacob 2018; Borangiu et al. 2019). This might be due to new models or needs arising after initial deployment of smart sensor suites. This enables a system seamlessly to shift from edge to cloud and back when needed while the operation is executed in a secure fashion. Finally, privacy regulations are stricter in the European Union (EU) (Schoch 2016) than in the rest of the world. Therefore, meeting the privacy guidelines given the existing and future regulations is important in the future of digital manufacturing in utilizing current and future cloud, fog, and edge capabilities. Note that data generated from these sensors (in either processed or raw form) are a cornerstone to digital twin and artificial intelligence/machine learning/deep learning (AI/ML/DL) based models that are formulated from significant empirical data.

2.1.2 Data Structure and Architecture Needs

The data structure and architecture area covers the communication, processing, and storage required for digital manufacturing operations. As significantly more data are generated, the question arises as to how the data will be transmitted, stored, processed, used, and shared. Some overlap with cloud/fog/edge issues exists, but data structure and architecture requirements focus on operational requirements at different abstract levels such as sensor, machine, work cell, production line, plant, enterprise, and ecosystem. Some work has been done in this area, but there is not even a basic understanding of how much data will be generated and how much of that data will be stored. For example, just a fraction of sensor information available on a single computer numerical control (CNC) machine tool can easily generate megabytes of data per hour, and a moderate-sized plant can regularly generate terabytes of data on a weekly basis, if not daily.

The appropriate quantity and resolution of data to be stored for various processes also must be understood. Such a determination will be based on process characteristics such as time constants. For example, mechanical systems such as robots and machine tools have time constants on the order of milliseconds, requiring sample frequencies of tens of kilohertz, and petrochemical systems can have time constants on the order of minutes—requiring data acquisition rates on the order of seconds. The question arises, “Does one need to store every sensor signal for every product and every process?” The answer is not clear, as there are differences between a component for a low-cost power drill versus one for an artificial heart valve or an aircraft engine. The solution here will require significant uncertainty modeling, risk analysis, and process/product analytics. Of course, this type of statistical and probabilistic modeling lends itself well to large data sets and high-performance computer operations.

The architecture or structure of the stored data also will be critical, not only simply from a data-compression and resolution perspective, but also from the perspective of access. Data must be sorted and archived such that it can be efficiently, selectively, and accurately retrieved. These three points are critical. Efficient data retrieval relates to the ability to quickly access data. Selective data access relates to ensuring that the appropriate data are supplied to the appropriate entities. In other words, not everyone should have access to all data. How is this access controlled and the data appropriately protected? This question also is a significant aspect related to cybersecurity. Another critical issue is formatting the data so that it can accommodate high-performance computing and parallel operations. Finally, accurate data retrieval relates to providing the appropriate data to match the requestors' inquiry.

2.1.3 Infrastructure Requirements

As a follow-on to the data structure and architecture, supporting storage, processing, and communication (network) hardware for digital manufacturing operations must be better understood. Key topics include the sensor, machine, work cell, production line, plant, and enterprise. A question that must be answered is, how much data will be generated by a plant, and how quickly do those data need to be accessed by the entire enterprise or ecosystem? Initial data sets from hybrid (additive/subtractive) systems indicate that a single machine can easily generate megabytes of data per hour, if not more. For infrastructure, items to consider including the following.

- Processing large-scale data sets is critical to next-generation digital manufacturing. As with storage, it is anticipated that some combination of local and cloud-based capabilities will be employed. Distributed computing employing unused local computational resources in a plant to process the data is a major area of potential research. This concept is similar in nature to operations such as Search for Extraterrestrial Intelligence (SETI) that use free computing cycles of internet-connected computers for large-scale advanced analytics.
- The appropriate storage infrastructure and architecture for large quantities of manufacturing data are not clear. In general, it is anticipated that a combination of cloud and local storage will be used to address storage requirements. Issues related to cost, operations, flexibility, and security must be considered. Questions arise as to cost effectiveness, security, and accessibility of cloud operations versus local operations.
- Networking and data transmission are another critical research area. The key issue to be addressed for this topic is the secure and efficient transmission of significant amounts of data. Other topics in this area include networking and communication technology and its relationship to robust, resilient, secure, and error-free networking connectivity. Communication advances targeting areas such as low energy; low-/high-bandwidth operations; and secure, mesh, redundant, and resilient protocols are critical to the implementation of the digital manufacturing ecosystem. Standardization of secure sensor communication (e.g., MTConnect, Open Platform Communications Unified Architecture, Message Queuing Telemetry Transport, Representational State Transfer) also is critical to generating effective, affordable, and trusted communication paths among all levels of the manufacturing operation from sensors, to machines, to plant, and through the supply chain.
- Another significant area of research is to address the impact of the advent of fifth generation (5G) communication systems on the entire infrastructure and its specifications. If data transmission rates are increased by tenfold to hundredfold, how does this affect storage and processing, as well as overall architecture and protocol aspects of the digital manufacturing ecosystem?

- From an energy perspective, large server farms and computing systems consume significant amounts of energy. Thus, it is anticipated that results from this research area will have a direct impact on energy requirements for US manufacturing operations.

2.1.4 Cybersecurity

The cybersecurity topic area includes all security issues. There are two leading elements to cybersecurity in digital manufacturing that should be considered. The first is securing connected equipment, ensuring that manufacturing processes and information are tamperproof. This element of cybersecurity ensures that process and product information is secure, and that this information is not accessed inappropriately (i.e., stolen). Further, it prevents damage to manufacturing systems via sabotage and ensures that processes are not modified to produce inferior or defective components. Research in this area addresses appropriate protocols and practices necessary to ensure process, product, and information/data integrity. This area focuses on defending current operations and information.

The second area of research targets ensuring and validating process and product integrity. In this instance, a unique and verifiable digital passport that is part of or linked to a product's digital twin is generated. This provides products and processes with a digital signature ensuring that the part or process is tracked and appropriately documented. The documentation is linked to a product from its initial manufacture through its entire life cycle. It provides the digital signature or documentation that results in a part being born qualified and ultimately born certified. As an example, when a replacement turbine blade is ordered by an airline, the blade's unique digital passport ensures that the part is genuine (i.e., not a counterfeit part) and is manufactured and inspected to the appropriate specifications. Research in this area includes secure and verifiable data structures linked to both physical and cyber-physical characteristics of a product or process. For example, a 3D printed part might include a unique embedded quick-response code as an optical or other form of electromagnetic signature. Such a hidden quick-response code might be linked to a block-chain–type data stream that incorporates the entire manufacture and use history of the part.

2.1.5 Digital Twin/Data Analytics/Advanced Modeling

Digital twin/data analytics/advanced modeling addresses the use of data generated from the ubiquitous sensors available in manufacturing operations to develop and refine models in real-time or near–real-time fashions. This can be used for both validation and process modeling and optimization. Such models are supported by data gathered during the manufacturing process and can employ approaches from simple well-known physics-based mechanistic models to advanced AI/ML/DL approaches. A key area of research here is to reformulate many of the well-established manufacturing process models into parallelized formulations that are amenable to high-performance computing (HPC) operations. Of course, there are significant opportunities to model new manufacturing processes such as additive and hybrid (additive plus subtractive) manufacturing.

2.1.6 Low-Cost and Smart Sensor Development

Low-cost and smart sensor development and deployment is tightly linked to the cloud/fog/edge discussion. This includes multisensor fusion and processing, sensor array and matrix design, development, fabrication, and rapid integration into manufacturing operations. Other topics that fit into this area include sensor deployment, validation of sensors and fault tolerance, integration of sensors into products and processes, communication of sensors and sensor networks, distributed computing across smart sensors, and cybersecurity for smart sensors. This topic is strongly linked to digital twin/modeling, as the digital twin requires some form of empirical information to refine models whether analytical, numerical, or generated from AI/ML/DL approaches.

2.1.7 Industry 4.0

The term "Industry 4.0" was coined in the year 2011 by the German government as it formulated a strategy for developing high-tech industries (Mosconi 2015). The naming uses a software versioning style framing for an impending fourth industrial revolution and encompasses a wide-ranging collection of cyber-physical technologies (Bauernhansl 2016). In the past decade, the world has seen a steady growth in the use of industrial internet, interconnected sensors, actuators, autonomous systems, 3D printers, and the power of information and big data analytics in manufacturing. Similarly, this section on Industry 4.0 is used to capture the ongoing paradigm shift toward a digital interconnected industrial ecosystem and its potential impact on DOE's mission.

Industry 4.0 presents many important opportunities for transforming manufacturing for a sustainable and prosperous US economy. Although started in Germany, the concept was adopted by General Electric in North America by 2012. Many other countries have similar initiatives, including Japan, France, and, notably, China (called "Made in China 2025"). All industrial revolutions in the past have changed the nature of nations and transformed societies. The third industrial revolution was driven by a massive increase in demand as billions joined the middle class. It led to a global shift toward automation and programmable robots. Mass manufacturing of low-cost products in a few lead nations extended the supply chain to the furthest parts of the world. Greater efficiency and reduced cost in general did not lead to increased use of renewable sources of energy; the net emissions increased. The advent of machines and sensors connected to the internet or the industrial Internet of Things created a market for Industry 4.0. It has also gained momentum due to investment by major government and industrial players.

The drivers behind this transformation are well-known: increasing demand for agility in product development, digitization of business processes and production, shorter product life cycle, and smart machines to respond to this newly emerging paradigm of a "batch size of one." The growth in efficiency driven by the operationalization of big data and advanced analytics is estimated to reduce production cost, logistics cost, and quality-management costs. It is relatively certain that the job profiles at many workplaces will change. The slow rate of change in the US manufacturing sector is driven by certain skills-gaps preventing companies from adopting this model. This digital industrial revolution means decentralization of production and creation of a much more dynamic network of suppliers and manufacturers. This is an opportune juncture to drive research and innovation in the creation of this new industry for a resilient, technologically advanced, and sustainable future.

Additional focus on R&D will further improve the Industry 4.0 ecosystem. (Oztemel and Gursev 2020) Current trends include the following.

- Predictive science to optimize products and performance in combination with advanced materials, on-demand scale-up, and manufacturing process optimizations will be key to manufacturing dominance. In combination with rapid prototyping methods such as 3D printing, printed electronics, and fabrication capabilities, a direct digital thread can be established from design to production. The services for facilitating a shorter time from idea to finished product will be an important area of growth requiring the merging of applied science and engineering (Nikolic et al. 2017).

- Developments of algorithms for autonomous systems and human-machine interactions remain a nascent area of research. Industry 4.0 is significantly different from robotic arms and the automation of tasks. In a knowledge-based economy, human-machine collaboration can be key to improved efficiency and prosperity. Algorithms for human perception and virtualization of the work environment could build remote access into a collaborative work environment. Focus on safety and access to knowledge can increase productivity. Integration of cyber-physical systems and increasing on-demand and custom manufacturing can increase access to jobs (Ansari, Erol, and Sihn 2018).
- Smart machines require better AI and cyber infrastructure. More demands on the performance of cloud computing will be met partially by readily available 5G or WiFi-6. Discussions on 5G for science already have identified the need for better understanding of a fully connected work environment (Beekman 1998; Nardelli et al. 2019). Much of the testing on the use of wireless low-latency sensors on humans and the new generation of electronics will require major changes in printed flexible electronics, power sources, architecture for low-power devices, and new materials research for building a fully connected ecosystem of products in support of Industry 4.0 and related research domains (Matsuda, Fujimoto, Aoyama, and Mitsunaga 2019).
- Supply chain innovation for sustainable sourcing of raw materials and product recycling will become progressively more important. In a highly agile and custom manufacturing environment, much of the supply-chain management functions will be virtualized. This will bring new opportunities and challenges for current models of manufacturing. As manufacturing becomes more distributed, opportunities for local economies to participate will be important. This will require a complete rethinking of the current model for workforce training and entrepreneurship (Ivanov and Dolgui 2020; Tjahjono, Esplugues, Ares, and Pelaez 2017).
- Production of sensors and smart machines will be a growth area. This is an expanding area of opportunity for research to remain competitive and for gaining an understanding of the challenging performance demands for autonomy. Materials are becoming more complex. Often, control of micron and nanoscale domains produced in a fast-paced manufacturing environment is key to precision manufacturing. New imaging and metrology techniques should support integration of advanced materials and processes (Oztemel and Gursev 2020).
- Integration of renewable and smart grids will support a decentralized and on-demand economy. Product life cycle tracking is required for remanufacturing and creating technological building blocks for a circular economy (Ng and Ghobakhloo 2020; Stock and Seliger 2016).
- In an interconnected world of machines working in collaboration with humans, research on a new conceptual framework for cybersecurity is required. In a world where open-source data are rapidly increasing for making almost anything in a decentralized manufacturing ecosystem, protecting key design specifications, proprietary data, assets in the smart factories, and supply chain logistics for mission-critical products will continue to grow in importance (Cimini, Pirola, Pinto, and Cavalieri 2020; Jazdi 2014).

2.1.8 Relevance of Industry 4.0 to DOE's Mission

Manufacturing in an Industry 4.0 era is a strategic area where US leadership is closely linked to innovation in science and emerging technological areas. New modes of computing, robotics, sensors, applied mathematics, ML, and robust cyberinfrastructure are required for sustaining growth. Manufacturing of custom sensors and low-power edge computing devices is important for the future viability of many US corporations currently engaged in developing computer hardware and microprocessors for manufacturing industries. Manufacturing using big data and ML requires better hardware and computing response. Specialized hardware for improving response time for AI-enabled systems will mean less distance from computing capabilities and less dependence on centralized large supercomputers—except for large-scale simulations involving physics-based models. The ability of users to analyze and steer machines in an edge-to-exascale computing continuum will become a reality. Increasing demand for sustainable products will require renewed emphasis on improving circularity in using energy, water, raw materials, electronic waste, metals, plastics, and critical materials (CMs). Emphasis on creating a market through faster deployment of new science and engineering will help support a technologically superior workforce that is ready for maintaining US leadership in the era of Industry 4.0.

2.2 Example Manufacturing Processes and Use Cases

Many examples of manufacturing processes could be cited. Below are representative areas to aid in understanding manufacturing. in general. Examples chosen include additive manufacturing, chemical manufacturing, roll-to-roll manufacturing, composite materials manufacturing, energy storage production, metal processing, cement production, and atomically precise manufacturing processes.

2.2.1 Additive Manufacturing

The phrase "complexity is free" often is used to describe AM. In reality, all additive processes are very complex and lead to low reliability in terms of manufacturing. AM processes are based on incrementally manufacturing parts bit-by-bit, layer-by-layer. Conventional manufacturing processes typically control properties through one process (e.g., forging, casting) and geometry though a secondary process (e.g., turning, milling, stamping). AM attempts to do both at the same time. Most additive processes use some form of thermal energy to melt and fuse material. The incremental and time-varying phase change in material results in significant temperature gradients that manifest themselves in the form of residual stress leading to distortion. Further, the varying layer-manufacturing times (due to variations in geometry) result in large variations in cooling rates and material properties. There currently are seven AM technology categories (Thompson et al. 2016). Each of these processes has advantages and disadvantages.

- ***Vat photopolymerization*** uses ultraviolet light to site-specifically solidify the liquid, voxel-by-voxel, layer-by-layer, to create the final component.
- ***Powder bed fusion*** uses a focused energy source (laser or electron beam) to melt a metal powder in a bed of unfused material. The large thermal gradients (both within a single layer as well as layer-to-layer) manifest very complex internal stresses within the finished part.
- ***Binder jetting***, in a manner similar to powder bed fusion, manufactures a part layer-by-layer in a bed of powder. Rather than fusing the material with an energy source, however, a binder is deposited to chemically bond the particles. The part then is placed inside a furnace to sinter the particles and, in many cases, be back infiltrated with a secondary material. Although the residual stress is low during the manufacturing process, significant residual stresses can occur in the post-processing as a result of mechanical loads (unsupported cantilevers) as well as thermal loads (cooling of dissimilar materials in the back infiltrant).
- ***Material jetting*** is similar to binder jetting. In this process, the liquid material deposited is the working material rather than a binder holding a secondary material together. Ultraviolet light also is used.
- ***Sheet lamination*** refers to using ultrasonic energy to generate a solid-state bond between layers of thin metallic sheets. Unlike most additive processes, the sheet lamination process includes localized

in situ machining. There are two sources of manufacturing-based stresses: the ultrasonic welding of the metallic sheets and the in situ machining.

- ***Material extrusion*** is based on melting and depositing thermoplastic or thermoset materials. Thermal stresses are induced through the localized heating and cooling of the thermoplastic or the localized heating due to the exothermic curing of the resins.
- ***Direct energy deposition*** is similar to material extrusion. Rather than extruding a thermoset of thermoplastic, however, liquid metal is fused through localized welding.

Each process, however, experiences some form of incremental material fusion that results in residual stress. The two primary sources of residual stress in AM are chemically induced stress and thermally induced stress. The sources and influence of residual stress and varying microstructure in AM have been studied extensively; three effects that influence variations in microstructure and stress states were found.

The first effect is the design of the part. For each process, today's thermal models are too complex and fail to incorporate a path to identify geometric shapes that can lead to distortion as a function of the material and process. Design optimization tools—as a function of the manufacturing process—lack the underpinning understanding of the influence the process has on properties. A second effect is the process itself, specifically the tool path used to generate the path. Conventional slicing software is based on geometry. A graph theory approach to tool path generation enables integration of mechanical and thermal models in the slicing software to help identify tool paths to help control stress. Finally, slight modifications to the materials (both metals and composites) can influence the ability of the process to successfully and reliably manufacture parts.

Another aspect of AM is the potential for low-volume manufacturing. This result is a significant challenge: how to certify and qualify a part quantity of one. Conventional manufacturing certification and qualification is based on controlling processing parameters to ensure the same quality of material for every manufactured part. These design allowables are defined by extensive testing. As described previously, additive processes control geometry and properties at the same time. These properties can vary from part to part, and even can vary within a single part. New methods would enable certification and qualification based on prior knowledge of parts.

Finally, AM enables a very lean production cycle where products can change rapidly (because of the removal of long lead times associated with tooling). Microfactories can be established rapidly, inexpensively, and remotely. Products can be manufactured and delivered locally, thus reducing transportation requirements. One final challenge is the delivery of the feedstock (base material). An emerging trend is the exploration of "ore to more"—locally sourced resources (biomaterials, mining materials) and products. This trend has the potential to transform infrastructure, enabling all towns to have manufacturing and production capabilities. These factors lead to the democratization of production.

2.2.2 Chemical Manufacturing/PI/Decentralization

The major scientific research areas important to chemical manufacturing, chemical PI, and decentralized chemical processes include chemical engineering, chemistry (specifically catalysis and separation sciences), materials science, and condensed matter physics.

2.2.2.1 Chemical Manufacturing

More than 95% of all manufactured products rely on some form of industrial chemical process.[23] Chemical manufacturing and associated innovations require large amounts of energy and, for some energy-intensive chemical products, can account for up to 85% of the total production costs (EIA 2019). In 2018, the industrial sector was the second-largest end-use sector in energy consumption behind the transportation sector, with the chemical industry being the largest user of energy in the manufacturing sector.[24] The bulk chemicals industry consumed more than 7 quadrillion Btu in 2018 (both fuel and non-fuel feedstock[25]) and is projected to grow and consume more than 30% more energy in 2030, representing almost 10% of all US energy consumption.[26] Further, the sector-wide potential energy savings opportunities for energy consumed as fuel, as cited in the energy bandwidth studies for chemicals, are significant based on current typical practice versus both practical minimums and implementation of state-of-the-art technologies.[27]

From a broad perspective, approaches to chemical production can be broken down into two major classes: (1) synthesizing chemicals (e.g., hydrocarbons and alcohols by Fischer-Tropsch, ammonia by Haber-Bosch, ethylene by oxidative coupling of methane, plastics by polymerization); and (2) reforming chemicals (e.g., alkane dehydrogenation to olefins or dehydrocyclization to aromatics, petroleum cracking to fuels and chemicals, lignocellulosic biomass to biofuels). Because 80% to 90% of all chemical manufacturing relies on catalysts, any improvements to catalyst selectivity and reaction conversion potentially could have great impacts on energy use. Improved catalyst performance requires interdisciplinary approaches for catalyst design through computation and high-throughput technologies, enabling accelerated advancements through directed experimentation and validation.

Innovative technologies through reduced energy budgets (lower temperature and pressure processes), hybrid approaches, and PI can also reduce energy use in chemical manufacturing. Although large bulk-chemical processes are not new and will continue near-term due to existing capital investments, opportunities exist in the R&D space for innovative approaches that not only address the impact of renewables on the grid but also are flexible enough for dynamic operating inputs. Note that the increased amounts of domestic shale gas are making possible additional technological advancements—for example, converting methane to higher-value chemicals at the wellhead through modular chemical approaches that require new catalysts and reactor designs.

Moreover, because chemicals are a part of the supply chain for virtually any technology, improvements in this critical building block propagate across numerous other industries. It would be beneficial if this industry were involved in any recycle-by-design initiative. Likewise, given the current trend in high temperatures, high pressures, and corrosive environments associated with the chemical industry, there

[23] https://www.icca-chem.org/sustainable-development/; https://www.icca-chem.org/wp-content/uploads/2019/03/ICCA_EconomicAnalysis_Report_030819.pdf.
[24] https://www.eia.gov/totalenergy/data/monthly/pdf/flow/css_2018_energy.pdf.
[25] Non-fuel feedstock is the combustible energy converted to chemical products instead of being used as fuel; for example, the ethane in natural gas used to make ethylene in ethane crackers. Non-fuel feedstock can account for 50% or more of the fuel used in US chemical manufacturing. Even when the non-fuel feedstock is accounted for, however, the chemical sector is still the largest user of energy for fuel in the United States.
[26] Tables 1 and 28, 2019 Annual Energy Outlook. https://www.eia.gov/outlooks/archive/aeo19/tables_ref.php.
[27] https://www.energy.gov/sites/prod/files/2015/08/f26/chemical_bandwidth_report.pdf.

has been a focus on materials (McMillan 2003) that can better withstand these conditions that enable increased energy efficiency on a life cycle basis.

Finally, scaling up new chemical manufacturing technologies from bench-scale to lab-scale to pilot-scale with a successful demonstration of the technology is a challenge that requires deep understanding of scale-up science and reactor engineering based on specific chemical reactions. The extent to which a new technology has reduced the risk "enough" for industry to invest in further development is probably subjective, even with a successful demonstration at pilot-scale. Nevertheless, bridging the gaps between fundamental and applied technologies is vital to ultimate adoption.

2.2.2.2 Process Intensification

Deployment of PI technologies is challenged by scale-up issues, as well as by health, safety, and environmental impacts. Optimizing process performance requires consideration of conversion efficiency and selectivity, energy requirements, cost, and environmental impacts by considering molecular-level kinetics, thermodynamics, and transport phenomena (such as momentum, heat, and mass transfer). Van Gerven and Stankiewicz describe the fundamentals of PI with four guiding principles (Van Gerven and Stankiewicz 2009):

1. Maximize effectiveness of intramolecular and intermolecular events (e.g., dynamically changing conditions to attain kinetic regimes with higher conversion and selectivity);
2. Provide all molecules with the same process experience (e.g., plug flow reactor with uniform, no-gradient heating);
3. Optimize driving forces at all scales and maximize specific surface areas to which they apply (e.g., increasing heat transfer surface area through microchannel designs); and
4. Maximize synergistic effects from partial processes.

Overcoming challenges by considering some or all of these guiding principles at the molecular level likely will result in transformative solutions for new PI strategies (Tian, Demirel, Hasan, and Pistikopoulos 2016).

Development of new PI methods could integrate multiple processing steps and alternative energy sources. These developments will enable researchers to optimize mass, heat, and momentum transfer within a given process or unit, and thereby overcome challenges in the manufacture of products that otherwise could not be safely or successfully made. These advances also will make possible the handling of highly variable raw materials, as well as "fit for purpose" separations.

Applications of PI technologies should be evaluated in a decision matrix that includes their energy-saving potential, cost improvement and competitiveness, technology readiness, waste reduction (e.g., CO_2 emissions), and probability of overcoming existing barriers (e.g., process downtime, loss of productivity over time). A taxonomy of PI technologies as formulated by this decision matrix would enable researchers to determine the applicability of a specific PI technology across various fields, such as manufacturing, energy production, and water treatment. Assessment of a technology's readiness is a crucial part of overcoming challenges in scale-up that include design, fabrication, and integration of PI equipment and devices into the plant. This assessment can involve multiscale modeling and is required for the development of transfer functions from an initial molecular-level assessment up to pilot-scale testing in the context both of the PI equipment or method and the yields, conversions, and selectivity of specific chemistry and processes.

Development and scale-up of PI technologies that integrate separations with chemical conversions are important. The separations process is a highly cross-functional area in which PI technologies could provide effective solutions. Specifically, successfully applied PI technologies have the potential to decrease production costs, increase productivity, and reduce waste generation. Materials and process development strategies for reducing the cost of separations include, but are not limited to, replacing energy-intensive processes with low-energy, high-efficiency processes, and integrating process-intensification strategies with alternative energy sources.

In summary, the key focal areas in PI R&D are process integration and design, multiscale modeling and simulation, process development, and scale-up of PI equipment and methods. These efforts should be supported by systems integration and operability, as well as by modular design concepts.

2.2.2.3 Process Intensification in Decentralized Chemical Processes

Large-scale manufacturing processes might not be economical for new products with a short lifespan, and for volatile markets and distributed raw materials. In some cases, on-site storage, long-distance transportation of hazardous materials, and the requirements of meeting stringent regulations can challenge the operations at centralized plants.

New metrics would benefit evaluation of the sustainability and cost of production at production scale and making the decision with appropriate indicators to comprehend the selection of decentralized processes over large-scale manufacturing. Several drivers for this decision should be considered, including feedstock availability; logistics; market; health, safety, and environmental regulations; trade-offs for the benefits; and cost of production.

Scalable modular plants are possible because different technologies—such as electrochemical-based technology—can be integrated. These resources scale differently from traditional large-scale manufacturing plants; however, colocation with existing resources and needs is possible.

An important aspect is the use of renewable resources (decentralized production of chemicals from biomass or waste streams) and integration with renewable energy to use cost-effective solar-based power during certain times of the day. New modeling approaches to design systems that operate efficiently under different operating conditions would advance processing. Both a fundamental research component (e.g., new separation technologies/materials) and a modeling/analysis component (e.g., optimization models for supply-chain design) are necessary. The models, methods, and analyses that are necessary to design efficient systems all should be considered. Key questions are how low production costs can be achieved at small scale, and how multiple sites are integrated. Therefore, these questions take into account small-scale manufacturing and manufacturing supply chain.

2.2.3 Roll-to-Roll Manufacturing

Roll-to-roll (R2R) manufacturing is used to produce a wide range of products for various applications that span many industrial business sectors. It allows for significant cost savings, increased throughput, and high-speed manufacturing compared with batch processing or other stepwise processing routes. Several key technological hurdles must be overcome to further the understanding of R2R opportunities and enable science-driven advancement in this field, which will allow for increased penetration and significant advancement in domestic manufacturing competitiveness.

DOE has had a coordinated approach and multiyear investment in a multilaboratory collaboration that has advanced the field in some limited but key areas. As laid out in several publicly available reports, the overall R2R methodology has been in use for decades (Daniel et al. 2018; Daniel et al. 2019; Daniel et al. 2016). This continuous technique traditionally involves deposition of material(s) onto moving webs, carriers, or other continuous belt-fed or conveyor-based processes that enable successive processes to build a final version serving to support the deposited materials. Established methods that typify R2R include tape casting, silk-screen printing, reel-to-reel vacuum deposition/coating, and R2R lithography. Products supported by R2R manufacturing include microelectronics, electrochromic window films, PV films, fuel cells (FCs) for energy conversion, battery electrodes for energy storage, and barrier and membrane materials. Due to innovation in materials and process equipment, high-quality yet very low-cost multilayer technologies have the potential to be manufactured on a cost-competitive basis. To move energy-related products from high-cost niche applications to the commercial sector, a means must be available to enable manufacturing of these products in an affordable, cost-competitive manner. Fortunately, products such as FCs, thin- and medium-film PVs, batteries, electrochromic and piezoelectric films, water-separation membranes, and other energy-saving technologies readily lend themselves to manufacture using R2R approaches. A remaining challenge, however, is linking the materials (particles, polymers, solvents, additives) used in ink and slurry formulations, through the ink processing, coating, and drying processes, to the ultimate performance of the final R2R product—especially for a process that uses multilayer deposition to achieve the end product.

A typical R2R process has three steps:

- Mixing particles and various constituents in a slurry;
- Coating the ink/slurry mixture on a substrate; and
- Drying/curing and processing the coating.

Final performance of devices made via R2R processes is dependent on the active materials (e.g., electrochemical particles in battery; FC electrodes) and the device structure that stems from the governing component interactions within the various steps. A fundamental understanding of the underlying mechanisms and phenomena still is lacking, however, which is why industrial-scale R2R process development and manufacturing still largely is empirical in nature.

This section discusses R2R science and technology in the following five key areas:

1. Thin film and coating manufacturing
2. Deposition and patterning technologies
3. Precursors and inks
4. Multilayer processing
5. Metrology for inspection and control

2.2.3.1 Thin Film and Coating Manufacturing

As scientists continue to discover new and interesting properties of nanoparticles, interest in thin films and coatings grows nearly as fast as the number of potential applications. The materials can be exploited by coating on a web or amplified by coating different particles as layers, one on top of the other. The performance of the films can depend heavily on the physical morphology and stability of the films, which is somewhat dictated by the method of deposition, the chemical components, and the environments in which they operate. The properties of the particles are dictated by their crystallinity, or lack thereof—be it amorphous or polycrystalline—and the films are either deposited onto a carrying web or in layers where multiple functions complement each other.

Some of the techniques gaining in popularity are listed in Table 3, which is reproduced directly from the literature (Jilani, Abdel-Wahab, and Hammad 2017). It provides more in-depth descriptions of some selected techniques. This section provides a general summary of issues that cut across several techniques. The techniques can be split between physical and chemical deposition.

Regarding physical deposition, scientists rely on sublimation and resolidification of the material of interest onto the substrate of choice. These processes generally are performed in a vacuum and usually are split into two categories, evaporation and sputtering. Examples of evaporation techniques include vacuum-thermal evaporation, electron beam evaporation, laser beam evaporation, arc evaporation, molecular beam epitaxy, and ion plating evaporation. These sublimation modes depend upon a target of the given chemistry that is heated and then further energized by different means that lead to a gaseous phase. The atoms or molecules reconvene on a target held at a temperature where they deposit back to their solid form. Sputtering refers to striking a target with energetic molecules from a plasma or gas. The thickness of the films is dictated by the length of time the system is running. Running these systems in an R2R process could create complications, mainly because of the difficulties in maintaining a vacuum over a movable web and limiting the deposition to the web itself.

Table 3. Physical and Chemical Deposition Techniques (*Source*: Jilani, Abdel-Wahab, and Hammad 2017)

Physical Deposition	**Chemical Deposition**
Evaporation techniques	Sol-gel technique
• Vacuum thermal evaporation	Chemical bath deposition
• Electron beam evaporation	Spray pyrolysis technique
• Laser beam evaporation	Plating
• Arc evaporation	• Electroplating technique
• Molecular beam epitaxy	• Electroless deposition
• Ion plating evaporation	Chemical vapor deposition
Sputtering techniques	• Low pressure
• Direct current sputtering (DC sputtering)	• Plasma enhanced
• Radio frequency sputtering (RF sputtering)	• Atomic layer deposition

Chemical deposition processes rely on chemical reactions on surfaces to generate thin films. Reactive thin films are first created directly through a deposition process of a liquid precursor or, if more easily performed, are formed directly into substrates. Those films are then subjected to chemical environments that lead to a change in the surface chemistry, which leads to stable, solid, thin-film structures. Techniques include sol-gel, chemical bath, spray pyrolysis, plating, and chemical vapor deposition. These techniques can be somewhat less expensive than those employed by the physical-deposition techniques, which require focused, high-energy methods of energizing elements off a surface and directing them to another surface. Many of the chemical-deposition techniques, like the physical-deposition techniques, depend on low-pressure atmospheres and the ability to uniformly distribute and react the vapor phase with the substrate phase. All rely on engineering designs that lead to uniform molecular transport to the surface and uniform surface reactions.

2.2.3.2 Deposition and Patterning Technologies

Solution-deposition using R2R platforms is a practical approach to addressing the process and configuration complexity in terms of integrating physical and chemical vapor deposition with R2R processes. R2R solution coating and printing enables remarkable throughput (speed and area) that has been applied to manufacture high-quality films, patterned surfaces, and even multilayered devices and structures. The R2R platforms enable remarkable throughput (speed and area) of coating and printing technologies to manufacture high-quality films, patterned surfaces, and even multilayered devices and structures. Essentially, the combination of web-conveyance and deposition processes is a way to scale AM to meet the volume needs of transportation, grid, water, and other energy applications. Additive deposition includes solution-deposition processes such as continuous liquid film coating, ink jet printing, gravure and flexographic printing, electroless plating, and various forms of vapor-phase/particulate processing, such as chemical and physical vapor deposition, solid- or liquid-state spray coating, and xerography. Post-deposition, films and structures typically are processed thermally, optically, mechanically, or chemically to achieve the solid-state material microstructure intended to maximize performance. The full gamut of materials process science underpinning ceramics, polymer, or metals is critical to the R2R process design. Poor scientific understanding of the competition of physical rate processes at work during the deposition and solidification stages—all while undergoing rapid throughput from the unwind roll to the winding roll—usually thwarts new materials from being realized for energy applications.

The most common deposition technologies deployed on R2R platforms are by far liquid film coating with continuous, pre-metered extrusion of liquid inks and printing with soft lithography, flexographic, and gravure processes that transfer a pattern to a substrate from an "inked" template. For nearly a century, these technologies have been deployed in manufacturing of products ranging from tapes/adhesives to decorative and functional paints on flexible sheet metal for automobiles and appliances. In the past 10 years, these same technologies have been advanced for more precision applications that require small-feature printing of less than a micron, ultra-thin coatings of less than 100 nm, and controlled microstructure allowing them to serve as electrolyzers, membranes, or other functions. The deposition process is a complex interplay of fluid mechanics, capillarity, and structural mechanics with rheologically complex fluids. The effect of functional materials on the fluid, thermal, and interfacial properties complicates the connection of process-composition/structure-property understanding and severely complicates process stability. Suitable process stability and full control rest on data and models for a process that is extremely nonlinear and bounded by hydrodynamic instabilities, colloidal aggregation, de-wetting, and other forms of defects. To ease the translation of bench-top, lab-scale coating and soft-lithography printing materials and processes to a full-scale R2R conveyance system, understanding the process science underlying the plethora of physical rate processes involved is critical.

R2R processing for nanomanufacturing, electronics and packaging, energy storage, and water separation/purification technologies is topical and driven by many economic and societal factors that have been acknowledged by the National Science Foundation, NextFlex (for advancing flexible hybrid electronics), and DOE AMO. Underpinning all of these applications is the materials science of reliability (adhesion, residual stress, and degradation under environmental stressors), the materials and engineering challenges of precision through nanomaterials and registration of multilayers, and the materials science underpinning performance (dense metallization, porosity and microstructure control of membranes).

2.2.3.3 Precursors and Inks

Liquid film coating and printing requires adequate control of ink materials, rheological, wetting, and adhesive properties, as they directly influence the microstructural and physiochemical properties of the coating films and final devices. When performed under R2R configurations, ink rheological properties need to be further optimized to accommodate high-speed material transfer from the ink sources to the moving substrates.

The first step is to optimize ink material components and composition. Inks are usually made of functional ingredients, binders, additives, and solvents. Functional ingredients are micro- or nanometer-scale structures that provide the major serviceable attributes of the inks. Functional ingredients can be synthesized either through a top-down or a bottom-up approach. With a top-down method, the micro/nanostructures can be obtained by mechanical reduction (breaking down) of larger-size materials, for example, through ball milling, sonicating, and exfoliation. In bottom-up approaches, the small structures often are formed from precursors through wet chemical (precipitation or decomposition) or electrochemical reactions (electrospinning, electroplating). Additives are another group of functional materials that add serviceable characteristics to the inks, such as electrical conductivity and adhesion. Binders are primarily used as carrier materials that make the inks workable and add mechanical strength to the coating and film products. Binders influence electrostatic and steric interactions within the inks, which in turn influence ink stability and shelf life. Solvents are used to dissolve binders and disperse functional gradients. Binders and solvents must be compatible because poor solvent-polymer interaction can lead to the collapse of polymer segments that are not absorbed on to the surface of the functional ingredients (Sundararajan, Tyrer, and Bluhm 1982).

Ink material and composition designs influence applications such as lithium (Li) batteries, FCs, and printed flexible electronics (PFE). Liquid ink coating is used in other application domains, including low-temperature water electrolysis, CO_2 separation/reduction membranes, water-filtration membranes, and PV films.

2.2.3.4 Printed Flexible Electronics

PFEs are a new technology that fabricates electronic components and devices using direct-write methods such as ink jet printing, aerosol jet printing, gravure, flexographic, and screen printing. Plastic substrates such as polyimide and Kapton often are used to make the devices flexible and stretchable. This technology can fabricate electronic devices for a wide range of applications, including organic light-emitting diode (LED) displays, radio frequency identification antenna tags, solar cells, transparent electrodes, antennas, various sensors, and transistors. Because of the many potential applications, the ink materials for PFE are rich and diverse. A general formulation of PFE inks also contains four components: (1) inorganic nanomaterials, (2) polymer carriers, (3) additives, and (4) solvents. The inorganic nanomaterials provide the main functionalities of the prints, such as electrical conductivity, capacitance, bio-functionality, and optoelectronic properties. Further adjustment of ink properties (e.g., conductivity, adhesiveness, rheological properties) relies on additives and solvents. Table 4 lists some common nanomaterials (Wu, W. 2017). In most inks, the functional nanomaterials are in the format of micro- or nanoparticles. It has also been reported that nanowire conductive inks show higher electrical conductivity than nanoparticle inks, due to more continuous electron transport path makeup—a characteristic that stems from enhanced fiber overlapping (Hu et al. 2010). Recently, two-dimensional

(2D) nanosheets were developed to obtain beneficial semiconductor properties such as direct bandgaps (Lim et al. 2016).

Table 4. Nanomaterials Used in Printing Inks and Their Functions Toward Potential Applications (*Source:* Wu, W. 2017)

Nanomaterials	Function
Metallic nanostructures: Metallic nanostructures: Au, Ag, Pd, Cu, Ni	Electron conductors used for electrical interconnects
Transparent conducting oxide: Indium-tin oxide, zinc oxide–doped aluminum oxide, gallium oxide, indium oxide; tin oxide–doped fluorine, CdO	Transparent electrodes used for electronic displays and heat-reflecting coating for windows
Carbon nanostructures: graphite, carbon nanotube, graphene	High electron mobility electrodes used for thin-film transistors, and chemical and bio sensors
Semiconductor: ZnO, TiO_2, MoS_2, perovskite	Solar cells, field-effect transistors

After ink materials are optimized, the fluidic dynamic properties of the ink must be characterized and adjusted to enable a stable coating/printing window and a precise control of the coated film quality, such as surface conditions and microstructures. Ink viscosity and rheological properties often correspond to the workability and stability of the coating/printing processes. Poorly prepared inks can lead to non-uniform dispensing of inks and subsequent defects in coatings. Common defects include ribbing, neck-in, edge defects, streaks, bubbles, pinholes, and particle clusters. Ink fluidic dynamic properties depend on particle size, shape, loading ratio, and distribution in solutions, as well as the hydrodynamic interactions between particles.

Modeling and experimental testing of the ink rheological properties and the correlation between ink properties and coating parameters usually is conducted to help establish good process control. In terms of R2R coating/printing, the influence of a moving web on the coating behavior also is considered and studied. Effort also has been devoted to coating tools and process improvements to accommodate a broader range of inks and substrates, as well as multilayer coatings that handle the deposition of wet inks on the existing wet or dry coating layers. In situ real-time monitoring of the ink deposition on moving webs helps accelerate this improvement (discussed in the next two subsections).

2.2.3.5 Multilayer Processing

The majority of coatings for industrial and energy applications involve deposition of more than one layer, and these multiple layers often must be deposited simultaneously. These multilayer coatings can have specific structure, physical properties, and functionality to maximize product performance and minimize device cost. Processing of multilayer coatings must also meet operating cost targets based on energy efficiency and production line speed, as well as keep capital equipment costs at a reasonable level and still achieve the structure-function-performance goals. R2R processing is an excellent means to meet all these criteria, and this methodology is highly compatible with simultaneous multilayer deposition. Examples of highly successful approaches include slot-die, gravure, slide-die, curtain, knife, and flexographic coating. The existing major research gap, however, is that these methods have not been fully implemented in many advanced energy storage and conversion and electronics fields. Additionally, many of these methods have not been developed for multilayer coatings (i.e., three or more layers deposited simultaneously) with specific functionalities for the energy devices that will be needed during the remainder of the twenty-first century. The overarching goal for R2R multilayer processing should include a three-fold correlation of (1) effect of processing parameters on multilayer architectures, (2) effect of inherent multilayer physical properties on device and coating performance, and (3) materials and formulation chemistry implications on choice of coating deposition method.

Established R2R multilayer creation is a three-part process. The first part involves complex colloidal and formulation chemistry where liquid solvents (processing aids) are added to one or more solids (e.g., ceramics, polymers, metals) that can have secondary particle sizes ranging from 50 nm to 50 µm that are subsequently mixed together using sophisticated blending protocols. The second part consists of the deposition of the resulting system of colloids, suspensions, or dispersions onto a substrate carrier (e.g., metal foil, polymer film) using one of the coating methods mentioned above. The third step, which can involve the most physics and chemistry, is the consolidation of the layers and solvent removal through drying. To understand the linkage among these three phases of multilayer processing, mathematical models can be used. But many programs still must be modified for the novel material sets for advanced applications, such as FCs, intercalation batteries, flexible PV films, and PFEs. True understanding of the processing-structure-performance relationship of multilayer coating deposition cannot occur without development of these models, correlating them to experiments, and extending them to predictive capability. Therefore, the major scientific disciplines of interest for multilayer processing are chemical engineering, chemistry, mathematics, materials science, physics, and thermodynamics.

The future of R2R multilayer processing likely lies in eliminating the liquid solvents or replacing all organic solvents with pure water because of the increasing emphasis on green chemistry and enhanced international environmental standards (Yang, S. et al. 2019; Schmatz, Lang, and Reynolds 2019). In the former case, technologies such as ultraviolet and electron-beam curing of multilayer systems are being developed in which there is no solvent present at all; but the processing physics and chemistry understanding gap is even greater for these newer methods. Combination of the layer functionality of a set of layers into fewer layers is also a major future research direction, where the material types and architecture complexity will increase dramatically; the advantage will be reduced operating costs through fewer processing steps.

2.2.3.6 Metrology for Inspection and Control

R2R manufacturing platforms have been ubiquitously used for high-volume manufacturing of 2D material structures, composed generically of polymers, metals, carbons, and ceramics, in a vast range of applications. Adhesive coating; printing of text, patterns, and designs; paper and textile processing; photographic and magnetic films; and packaging materials are all applications that have heavily relied on and driven the advancement of R2R manufacturing equipment and methods. More recently, R2R platforms have been utilized for prototyping and manufacturing energy materials. Further understanding of how processing of raw materials, synthesized materials, and inks and slurries—via a vast array of R2R-enabled deposition, drying and curing processes—impacts the final layer morphology (in some cases multilayer morphology) and ultimate device performance are crucial needs.

Integral to the concept of high-volume manufacturing with R2R processes is the necessity for real-time in-line inspection. Clearly, from an ultimate product-quality perspective, these methods are important. At high throughputs, materials are consumed extremely rapidly, and detection of improper processing at the end of the line can result in low yields—especially for newly developed materials and products—and thus very high costs. Manufacturers of these materials need to understand critical-to-quality requirements for these materials. In some cases, these can be discrete or continuous variations in the

R2R processed materials, for example, voids, bubbles, or foreign particles. In some cases, these can be specific properties of the processed materials, such as, porosity, thickness, or active material loading. In many cases there are a combination of critical-to-quality requirements. Methods to measure these properties or detect these variations are typically known for small coupons of material sampled at the end of the line. The same methods, however, rarely are valid for in-line measurement while the "web" (continuous roll form of the material) is moving, under tension, and being conveyed on the roller system. Further—and critically—quality is not the only aspect impacting cost in these processes. In cases where expensive raw materials are used, in-line inspection is relied on to make sure that *too much* of the material is not being deposited, thus leading to a higher than necessary cost per unit of area. In cases such as coated electrode layers using platinum (Pt) group metal (PGM) catalysts, this control is critical. Beyond these aspects, in-line measurement techniques can be used as feedback loops for process controls. An example is the measurement of coat weight leading to direct feedback control of raw material feed rates in an R2R extrusion system.

The methods applicable to in-line inspection vary greatly depending on the material(s) and property to be measured. Most include an incident beam of some wavelength of electromagnetic radiation that is observed by a detector and processed into the target metric. Methods exist for a range of materials; however, they are in almost all cases single-point measurements. For many new energy materials, it is understood that discrete defects can be catastrophic to device performance, and thus most existing methods are not sufficient. Generally, needs related to improving in-line metrology for inspection and control include further understanding and development of methodologies for

- Imaging-based techniques (rather than single-point);
- Measurement of properties within multilayer structures;
- Measurement of a broader range of constituents (i.e., that are relevant to new energy materials);
- Ensuring precise alignment and registration of multiple layers or successive patterned depositions;
- Increased resolution and/or measurement of smaller structures;
- Increased data acquisition and processing rates; and
- ML, especially related to process feedback and control.

Addressing these challenges requires understanding the interplay between electromagnetic physics and material structures at high rates, under material stress (tension and movement on the R2R line) and, in most cases, at atmospheric conditions. It also requires developing fast, areal detector technologies; developing algorithms and hardware with increased computational speed; and incorporating advanced analysis tools and methods into manufacturing processes and data streams.

2.2.4 Composite Materials Manufacturing

Advances in chemical and process engineering and thermodynamics as well as in optimization (through both AI/ML and analytic approaches) will enable development of the next generation of composite materials technologies.

2.2.4.1 Polymer Composites

Polymers are lightweight soft materials and need stiffness and strength augmentation for their effective use in replacing metals. Polymer matrix composites have been used for many decades in various forms, with primary applications focused on high-performance applications and with limited consideration of cost.

Although currently carbon- and glass-fiber reinforced composites are clearly the volume leaders, using nanoscale reinforcing materials has the potential to play an important role. Polymer composite systems containing multiple phases, including inorganic or organic additives, can exhibit outstanding properties even at very low loading of additives. Often, these composites are composed of polymer matrices loaded with high-surface-area, nanometer-scale reinforcing particulates that enhance the properties of a polymer. Examples include a polymer matrix reinforced with nanoparticles such as exfoliated graphene sheets, carbon, and other inorganic nanotubes, nanorods, or platelets. These nanoparticles have unique structures and properties. For example, one-dimensional nanotubes made either of carbon (Endo et al. 1993) or inorganic materials (Tenne, Margulis, Genut, and Hodes 1992) have extraordinary mechanical properties. Carbon nanotubes (10–25 nm diameters and several micrometers long) have superior tensile modulus compared with inorganic nanotubes (Kis et al. 2003), although the latter have outstanding compressive modulus (Zhu et al. 2003). It has been shown that nanopillars made of polymers, depending on their diameter and surface energy, form unique self-assembled structures (Kang, Pokroy, Mahadevan, and Aizenberg 2010). Commercialization of polymer matrix nanocomposite materials has occurred over the past couple of decades for specific low-volume usages (Kumar, Benicewicz, Vaia, and Winey 2017). Mass production at lower cost is needed for its use in commodity products (Naskar, Keum, and Boe 2016).

Large structures that are cost sensitive are typically manufactured using glass or other lower-cost fiber reinforcement. Examples include marine or windmill blade production, where glass-fiber preforms typically are infused on single-sided molds to form a structure (Govignon, Kazmi, Hickey, and Bickerton 2011). Epoxy and vinyl-ester matrix materials are prevalent (Nhan et al. 2019). Vacuum-infusion processes require trade-offs with respect to lower fiber-volume fraction, as well as potentially higher void content; however, the process produces large structures at low cost and with minimal capital investment.

Smaller and geometrically complex structures can be produced rapidly using processes such as injection molding or compression molding (Yilmaz, Ellingham, and Turng 2019). Rapid processing typically requires the use of discontinuous fiber architectures and acceptance of flow form microstructure containing defects. Thermoplastic polymers with low-cost fiber reinforcements typically are used in injection molding, and low-cost thermosetting sheet molding compounds or bulk molding compounds are prevalent in automotive and consumer product manufacturing. Although these processes are rapid and result in low-cost products when used at high volume, the initial capital investment for molds and dies typically is substantial and frequently is prohibitive for small- and medium-size enterprises.

2.2.4.2 Nanocomposites

Apart from the issues with the manufacturing throughput, polymer-matrix nanocomposites impose another practical difficulty—dispersing reinforcing particles in a matrix via formation of an isotropic network of unorganized structures (e.g., randomly oriented exfoliated nanoparticles in a polymer matrix). If the particle-matrix adhesion is poor, then the particles tend to agglomerate and form a defect center. If the adhesion energy is high, the high surface-area-to-volume ratio of nanoparticles would allow a high-volume fraction of immobilized matrix to adhere to the particles. To tailor the adhesion energy between filler and matrix, either the particles or the matrices often are functionalized (Vaia and Giannelis 2001). Organic functionalization of nanoparticles enhances interfacial adhesion and allows formation of partially oriented nanocomposite morphology (Vickery, Patil, and Mann 2009). However, retaining the organic functionalities and the preferred orientation of incorporated, dispersed particles during thermal processing or fabrication of nanocomposite materials remains a challenge. Additionally, the mechanical shear in processing nanocomposites causes strain-induced desorption of matrix molecules from the nanoparticle surface, which allows formation of filler agglomerate (Schaefer and Justice 2007).

Building controlled anisotropy in polymer composites has been attempted in the past. For those studies, nanotubes or layered reinforcing units were preferred. Spherical particles such as carbon nano-onions or fullerene derivatives have the least surface contact with other particles and are expected to be easy to disperse. Spherical nanoparticles, however, cannot exhibit anisotropy; therefore, those have not been extensively used for nanocomposite manufacturing. In a specific study, carbon-nanotube-reinforced polycaprolactone formed a unique morphology where the nanotube acted as a "shish" (the skewer) and the polycaprolactone crystalline lamellae grown on the tubes acted as "kebab" (the food) The nanocomposite material exhibited an eight-fold improvement in tensile modulus at 0.2 wt % loading of nanotubes (Chatterjee, Mitchell, Hadjiev, and Krishnamoorti 2007). Formation of controlled hierarchical order in polymer nanocomposites remains difficult to achieve (Vaia and Maguire 2007).

Although hierarchically ordered architecture in polymer nanocomposites has yet to be obtained, tailoring the combination of matrix and particles has given a few successful functional nanocomposites. Kim and colleagues (2011), for example, prepared an outstanding material that showed greater hardness than pure calcite, using a synthetic biomineralization process involving formation of self-assembled calcite-copolymer micelle adducts (Kim et al. 2011). Further, Yoonessi and colleagues prepared networked nanocomposites with shape-memory properties (Yoonessi et al. 2012). Choice of matrix and particles and the morphology of the hybrid interface, whether having hierarchical architecture or not, are clearly very important for desired functionality. The major challenge in manufacturing polymer nanocomposite is achieving directed assembly of nanoparticles via controlling the processing conditions and their flow-induced viscoelasticity (Wan and Chen 2012). Larger aggregates of particles impose a key barrier to creating nanocomposites with better performance.

2.2.4.3 Advanced Thermoplastic Resin Systems for Composite Manufacturing

Thermoplastic resins have the potential to be lower in cost than epoxy and require less process heat to cure, thus reducing embodied energy. Depending on the polymer system, cure kinetics are so rapid that managing the exotherm is one of the predominant technical hurdles to commercialization. In some cases, thermoplastics with short fiber reinforcements that are simply injection molded are used. Polymerization cure in thermoplastic composite systems is much less prevalent than thermosets where a cure is required. Reducing cycle time is understood as the single most important economic driving force for commercial adoption.

2.2.4.4 Thermal Welding of Composite Structures

Although all megawatt-scale wind turbine blades currently are manufactured using thermoset resin systems, studies are under way in national laboratories, academia, and throughout the wind industry to incorporate thermoplastic resin systems into the production of wind blades. Although the initial goal was to evaluate using thermoplastics in wind turbine blade production for the potential to increase recyclability of blades at the end of their service life, the potential of a thermoplastic resin system's ability to enable thermal welding during the wind blade production stage is garnering increasing attention.

For wind turbine blades, the process of thermal welding could revolutionize the method in which blade components are joined in the factory—and potentially in the field as well. The current method of joining is to bond the various blade components together with an adhesive. This involves several process steps that, if not performed correctly, can lead to manufacturing defects in the blade structure that often result in field blade failures. In fact, the adhesive joints in wind turbine blades account for a significant portion of the overall field blade failures.

Currently, composites used for wind blades use thermoset matrices that cannot be thermally welded. Specific thermoplastic resin systems can overcome this limitation; thermoplastics can be thermally welded. The industry is targeting the use of current welding technologies such as resistance welding and induction welding. Limitations for welding composites are unknown at this point.

2.2.4.5 Fiber-Reinforced Composites

Attempts to reinforce polymer matrices by incorporating nanometer-scale reinforcing particles have shown sporadic success when an optimal dispersion of nanoparticles with preferred network and orientation is achieved. Commonly, such morphologies offer improved stiffness in the material but not necessarily an improvement in their failure strength. When used as reinforcing agents, large-diameter (micrometer scale) fibers (failure strength 3–7 GPa) provide significant enhancement in composite stiffness. Additionally, preferentially oriented fiber-reinforced composites offer significantly higher tensile strength along the fiber direction but have lower failure properties in the transverse direction due to the anisotropy caused by oriented fiber morphology. Various forming methods (Boisse, Hamila, and Madeo 2017) are followed to assemble the fibers (either in continuous or discontinuous form) before impregnating the preformed fiber structure with polymer resin and subsequent molding. Another approach with these fiber-reinforced polymer composites is free-form fabrication of products via 3D printing (Compton and Lewis 2014; Nguyen et al. 2018; Raney et al. 2018). Although structural composite materials exhibit potential impact to the automotive industry, the manufacturing protocol has not yet been widely adopted. Their manufacturing rate is simply not robust enough to compete with metal stamping (NRC 2012).

The market for carbon fiber (CF) and CF–reinforced plastic (CFRP) is strong and rapidly growing. The future demand growth rates vary from study to study; however, they all show that the demand for CF will more than double in the near future (Das, Warren, Wes, and Schexnayder 2016). Carbon fiber demand (Figure 9) is projected to increase with compound annual growth rates ranging from 10.8% to 15.7%; CFRP growth rates follow a similar pattern as CF. Regardless of what study is evaluated, growth-trend projections inarguably showed that aerospace, automotive, and wind energy are within the top five major sectors for CF and CFRP utilization, as shown in Figure 10.

Other major sectors are pressure vessels and civil engineering. These applications are the largest market for CFRP because of mass volume consumption and mainly because of the high-performance properties of CF and CFRP. Carbon fibers provide highly directional properties superior to metals and provide the maximum “lightweighting” potential and energy reduction in the aforementioned applications. Unfortunately, the market restraints and challenges are the cost of CF and its composites, insufficient production capacity to produce low-cost CF composites for large-volume mass market application, and limited research and technical advancements.

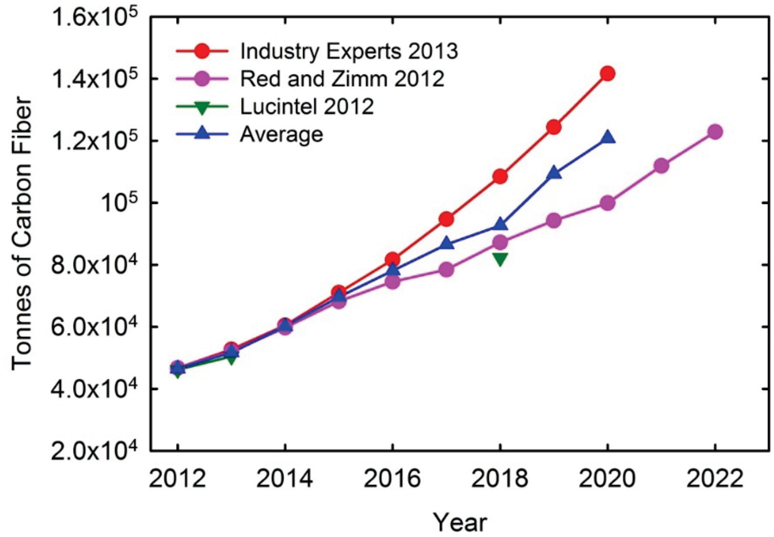


Figure 9. Annual forecasted[28] carbon fiber demand. (Source: S. Das et al., [Global Carbon Fiber Composites Supply Chain Competitiveness Analysis](), ORNL/SR-2016/100 | NREL/TP-6A50-66071)

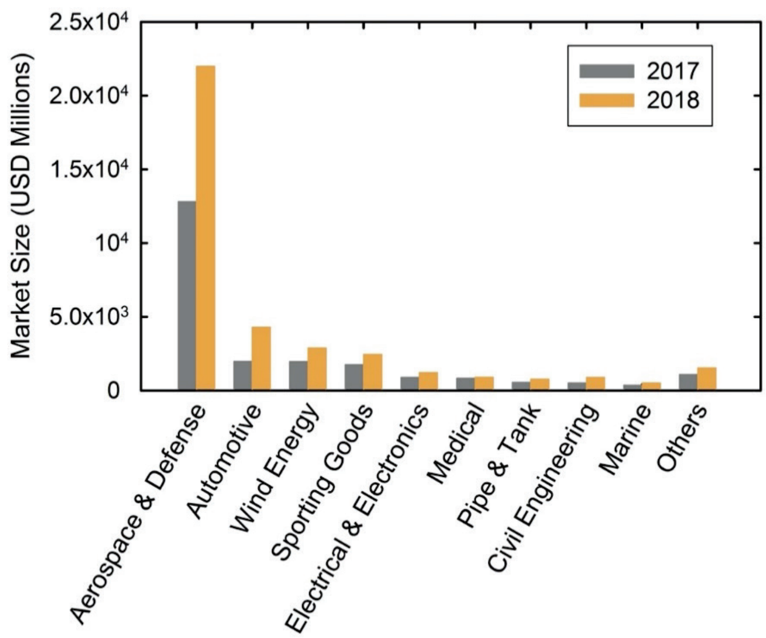


Figure 10. Aerospace and Defense to Dominate CFRP Market During the Forecast Period.[29]
Note: industries included under "Others" are oil and gas, 3D printing, moldings and compound, and other consumer goods. (*Source*: CF & CFRP Market . . . Global Forecast to 2020, MarketsandMarkets)

[28] Red, C., and P. Zimm (2012). "Global Market for Carbon Fiber Composites: Maintaining Competitiveness in the Evolving Materials Market." Composites World Carbon Fiber 2012 Conference, La Jolla, CA, December 4–6; Lucintel (2012), November. "*Growth Opportunities in the Global Carbon Fiber Market: 2013- 2018*" (Irving, TX: Industry Experts 2013). *Carbon Fibers and Carbon Fiber Reinforced Plastics (CFRP): A Global Market Overview*. Hyderabad: Industry Experts.
[29] https://www.marketsandmarkets.com/Market-Reports/carbon-fiber-composites-market-416.html.

Carbon fibers and CFRP currently are being implemented in mass volume applications; however, there are challenges from regulation to basic science associated with CF and CFRP in high-volume applications. The major drawbacks for CF and CFRP use in high-volume applications are

- High CF manufacturing costs and price;
- Availability, quality, and consistency of raw materials;
- CF and CFRP manufacturing processes are slow and energy intensive;
- Lack of understanding of process-parameter-structure-property-relationships;
- Health, safety, and environmental concerns when producing and handling composite materials; and
- Scaling of materials and/or processes.

The United States has a competitive composites industry with composite initiatives supported across national laboratories, academia, and consortia. For example, the Institute for Advanced Composites Manufacturing Innovation (IACMI)[30] is a partnership of industry, academic institutions, as well as federal, state, and local governments, working together to validate manufacturing technologies that respond to private industry's need for faster and more cost-effective, material, and energy-efficient composite manufacturing, including recycling at the end of product life. IACMI's R&D programs are driven by major industry participation with a focus on reducing technical risk and developing a robust supply chain to support a growing advanced composites industry.

As another example, Washington State University established a Composite Materials and Engineering Center[31] that is equipped to conduct research in composite materials development and manufacturing, as well as structural and durability testing. Michigan State University Composites materials and structure center,[32] led by Dr. Lawrence Drzal, is fully equipped with eight laboratories housing the latest instrumentation for the study of composite manufacturing, performance, and durability. Georgia Institute of Technology's Carbon and Multi-Functional Fiber Center[33] is focused on developing the next generation of CFs, and the Composites Manufacturing and Research Laboratory is used for a wide variety of research programs in composites manufacturing and testing. The University of Maine's Advanced Structures and Composites Center[34] is a world-leading, interdisciplinary center for research, education, and economic development encompassing material sciences, manufacturing, and the engineering of composites and structures. Argonne National Laboratory (Argonne) provides industry with the tools and opportunities to optimize AM polymer and composite materials and processes to fit specific applications.

Among various reinforcing fibers (metallic, ceramic, and organic), CFs exhibit an outstanding balance of strength, stiffness, and density. Therefore, CF is a preferred material for composite manufacturing. Unfortunately, CF manufacturing methods are cost intensive. Additionally, current technologies are capable of producing continuous carbon filaments with a micrometer diameter and with less than 10% of the tensile properties of theoretical predictions (Peng et al. 2008) These same materials, under different processing conditions, yield fibers with 90% of the tensile modulus of single-crystal graphite; but they have very low strength and break easily. Thus, attempts to increase the rigidity of carbon materials typically come at the cost of reduced breaking strength. To develop advanced carbon materials with both

[30] www.iacmi.org
[31] https://cmec.wsu.edu/who-we-are/.
[32] https://msu.edu/.
[33] http://www.acfrc.gatech.edu/brochure.html.
[34] https://composites.umaine.edu.

very high strength and modulus or other functionality demands a thorough understanding of structural evolution at each processing stage. Such an understanding will enable controlled long-range order in carbon and extraordinary performance. To that end, predictive design of advanced carbon precursor materials and their new methods will enable conversion to carbonized filaments.

Polymer composites used in aerospace and defense are primarily continuous CF–reinforced structures manufactured using demanding processes, resulting in high-performance materials with minimal defects. The manufacturing process starts with creating a pre-impregnated intermediate form with a well-defined fiber-matrix ratio and processing characteristics in a subsequent autoclave process. This pre-impregnated material is then cut and stacked into appropriate laminate sequence to form a laminate. Full cure and consolidation are achieved in an autoclave process, where elevated pressure and vacuum are used to generate essentially defect-free material. Capital investment and material tracking of time-sensitive feedstock for autoclave processing is significant.

In an attempt to reduce the manufacturing cost of CFs, Oak Ridge National Laboratory (ORNL) recently developed a high-throughput method (Jackson and Naskar 2019) for converting industrial-grade polymeric textile fibers to CFs. These manufactured CFs are not the best in terms of performance, but they are acceptable for semi-structural applications. Effective use of these CFs in the form of lightweight structures of composite materials with both acceptable failure strength and toughness can improve vehicle fuel efficiency (NRC 2011) and reduce GHG emissions. However, very high-melt viscosities of fiber-reinforced polymer matrix and breakage of reinforcing fibers during processing hinder rapid manufacturing of composite products. Therefore, to properly design and process fiber-reinforced polymer matrix composites, it is imperative to understand how multiscale interfacial structures affect the rheological properties. Only when there is a deeper understanding of the structure-processing-properties relationships of polymer matrix composites will development of novel structural soft-matter materials with enhanced mechanical properties for high-volume industrial applications be possible. For successful manufacturing of polymer matrix composites, the chemistry of structural fiber formation must be integrated with (1) technology of fiber manufacturing, reinforced polymer-AM, multifunctional and multimaterial composites; (2) modeling of composite failure and crashworthiness, and (3) interfacial science and engineering.

Market forecasts for composites continue to predict significant potential for carbon material as leading CF manufacturers are finding different ways to develop their businesses and expand the role of CF composites in an increasing number of applications. In 2019, global demand for CF increased to a healthy level of ~100,000 MT (Mazumdar 2020), consistent with the growth rate over the previous years. Surprisingly, three non-traditional new growing CF market segments—wind energy, automotive, and pressure vessels—combined contributed to 40% to 45% of the total 2019 market, significantly more than the traditional premium aerospace market share of 20%. Cost as well as embodied energy competitiveness of CF composites manufacturing (i.e., in terms of $/kg and MJ/kg) will be critical detriments to these non-traditional potentially big CF markets whose future growth rates are projected to more than double.

To further lead a drive to develop the potential of composites into new sectors, establishing strategic road-mapping actions is important, including the development of business and cost models, supply chains implementation, and development suitability for high-volume markets and addressing technology management (Koumoulos et al. 2019). The technology gaps must be identified and strategically designed to overcome these challenges. Technology gaps identified are

- Development, and scalability of low-cost, high-yield precursors;
- Energy-efficient precursor spinning processes;
- Development of environmentally friendly, highly reactive resin systems;
- Development of energy- and cost-efficient conversion technologies and processes;
- Development, and scalability of cost-effective high-rate, high-volume composite processes;
- Transfer packages from R&D to commercialization;
- CF and CFRP cost models for the various application areas;
- Development of new processes for handling and packaging low-cost CF;
- Basic research to develop structure-property relationship;
- Automation of manufacturing processes by advanced software tools; and
- Development of codes and standards for composites material.

There is an increasing urgency for R&D efforts to address the technological gaps in the CF and CFRP technology area. Currently, CF technology relies primarily on the production of high-performance CF from polyacrylonitrile. The key contributors to the high cost of CF manufacturing are the precursors and equipment required for conversion of precursors into CF. Half of the cost of CF manufacturing is the precursor; therefore, low-cost alternative and renewable precursors are an increasing necessity. New, innovative manufacturing processes for low-cost precursor development and conversion technologies hold the key to reducing CF cost for energy applications. Recent research is well on the way to adopting low-cost alternative precursors using lignin (Paulauskas et al. 2009), pitch[35] (Maeda, Zeng, Tokumitsu, and Mochida 1993), polyolefins (Maeda, Zeng, Tokumitsu, and Mochida 1993), precursor blends (Jiang et al. 2018), biomass[36] (Jiang et al. 2018; Milbrandt and Booth 2016), and textile precursors (Paulauskas et al. 2009). The Carbon Fiber Technology Facility (CFTF)[37] at ORNL, established in 2013, serves as a national testbed for government and commercial partners to scale up emerging CF technology. CFTF is DOE's only designated user facility for CF innovation. The CFTF facility provides a platform for identifying and scaling high-potential, low-cost raw materials, including textile, lignin, polymer, and hydrocarbon-based precursor for CF manufacturing. The CFTF supports the technology development and commercial deployment of CF in the United States for use in clean-energy applications. Additionally, research focuses on further understanding the kinetics of CF manufacturing, energy consumption, and environmental impact.

Additionally, other studies also are focused on the development of advanced and non-conventional plasma-based, e-beam based, microwave-based conversion technologies and have shown that it is possible to reduce energy costs by up to 90% in some cases (Koumoulos et al. 2019). Unfortunately, limited evidence exists for scaling such technologies for commercialization. The combination of precursor and conversion technologies R&D efforts can significantly reduce the energy and cost of CF and CFRP manufacturing. Thus, CF material costs make up a large portion of the cost associated with composite materials. Other factors to consider are the resin system, labor and machining, consumable and waste, production, and indirect costs.

The Manufacturing Demonstration Facility (MDF)[38] at ORNL, established in 2012, is DOE's only designated user facility focused on performing early-stage R&D to improve the energy and material

[35] https://www.compositesworld.com/news/coming-to-carbon-fiber-low-cost-mesophase-pitch-precursor.
[36] https://www.energy.gov/sites/prod/files/2017/05/f34/Biomass%20Conversion%20to%20Acrylonitrile%20Monomer-Precursor%20for%20the%20Production%20of%20Carbon%20Fibers.pdf.
[37] https://www.ornl.gov/facility/cftf.
[38] https://www.ornl.gov/facility/mdf/research-areas/carbon-fiber-composites.

efficiency, productivity, and competitiveness of American manufacturers. The MDF research portfolio focuses on manufacturing analysis and simulation, composites and polymer systems, metal powder systems, metrology and characterization, machine tooling, large-scale metal systems, and robotics and automation. The MDF has 110,000 sq. ft. of floor space and the facility houses integrated capabilities that drive the development of new materials, software, and systems for advanced manufacturing with the capacity to produce full-scale demonstration components.

To overcome the aforementioned challenges, development of cost- and energy-effective materials and technologies across the CF supply chain must be implemented to foresee mass production of composites. The technological impact will offer the breakthrough of CF and CFRP in long-awaited, cost-sensitive sectors.

Potential lightweighting opportunities in major growing nontraditional markets, that is, wind energy, automotive, and pressure vessels, provide overall system/life cycle cost and energy benefits as the use phase energy contributes more than 80% of the total life cycle energy of a lightweight vehicle. Embodied CF manufacturing energy is estimated to be ~1,200 MJ/kg, of which the precursor contributes ~50%.

For manufacturing CF energy competitiveness, ongoing R&D initiatives have been focused on alternative low-cost precursors, for example, lignin, low-textile acrylic fibers, and coal tar pitch, besides fiber conversion technologies. Additionally, CF composites recycling (both during part manufacturing and at end of life) is needed as the large nontraditional markets grow, besides the benefits of CF composites manufacturing energy competitiveness.

2.2.4.6 Manufacturing Process Optimization for Wind Turbine Blade Composites

The ongoing demand to reduce the levelized cost of electricity (LCOE) drives the wind industry to explore new technologies that will advance the state of the art for composite wind blade manufacturing. These new technologies span the range from new resins and fibers, to improved blade designs, to innovative manufacturing techniques. Since the introduction and widespread adoption of vacuum-assisted resin infusion techniques for blade making, however, there has been no significant change in the basic labor-intensive manufacturing process for wind blade production.

Regardless of the differing wind-blade designs and intricacies of blade manufacture, each blade requires on the order of 1,000 man-hours of labor input and exhibits cycle times that do not conform to an even multiple of typical eight-hour shifts. The latter requires the manufacturing floor to always be staffed for the worst-case scenario steps in the manufacturing process. To advance the state of the art of composite wind blade manufacturing, high-fidelity techno-economic and manufacturing floor simulation models of the overall blade-making process would assist in identifying opportunities to optimize and thereby reduce the cost of wind blades. The labor, overhead, and production cycle represent approximately 50% of the blade cost. Thus, these areas are prime targets for finding opportunities. The proposed models must consider the very rapid product refresh cycle (and concurrent consumption of capital), the desire for ever-longer blades, and the potential future composite technologies, such as CF and thermoplastics, that could affect the blade design and resulting manufacturing processes. The modeling also must consider the cash flow over a multiyear period so that the true value of improvements can be identified and used to justify capital investment in automation and other process changes.

The market in the United States for wind turbines is—and is expected to remain—strong with the focus that all original equipment manufacturers have on reducing the LCOE of wind turbine electricity and the continued demand for sustainable energy sources. Research can play an important role in providing guidance to blade manufacturers on opportunities for improvement, and potentially provide justification for significant investments in automation. Production of wind blades, now growing well beyond 60 m in length, has to a good degree shifted back to North America; however, jobs made possible by this market may be jeopardized if ongoing efforts are not made to improve productivity.

2.2.4.7 Automation of Composite Manufacturing

Wind turbine blade production has historically been a very labor-intensive operation. Starting with the manufacturing of kilowatt-size blades in the late 1970s and early 1980s and progressing through the current production of multimegawatt-size blades, both the molding and finishing operations of wind turbine blades have been performed primarily by specialized teams of personnel in composite manufacturing environments. Notable efforts over the course of the past few decades have been made to automate certain aspects of wind turbine blade production. These attempts at automation have had varying but usually very limited degrees of success. The efforts that have worked well focused on targeted automation for very specific blade manufacturing steps, such as root drilling.

2.2.4.7.1 Blade Molding Versus Blade Finishing

Wind turbine blade manufacturing generally is divided into two major phases—molding and finishing—usually taking place in separate areas of a blade-production facility. The molding operation, which typically centers around a large tooling set of wind blade skins (the high-pressure and low-pressure skins) as well as ancillary blade tooling (for shear webs, spar caps, root inserts, and trailing edge preforms), is the phase in which the composite blade structure is produced using constituent materials such as fiberglass, CF, balsa wood or foam core, thermoset or thermoplastic resin systems, and other materials. The labor steps involved in blade molding—such as tooling preparation, fabric laydown, core placement, infusion material layout, vacuum bagging, infusion, bonding, curing, and demolding—historically have been completed by specialized teams of laborers with time-efficient methods and with reasonable reliability.

The finishing operations of wind turbine blade production take place after the blade is demolded from the blade skin tooling and transferred to a different section of the factory. The finishing process steps for blade production—including trimming, overlay, oven post-curing, root cutting, root facing, root drilling, surface preparation, painting, and other operations—traditionally have been done at many separate finishing stations with specialized teams of finishing laborers. The blades in the finishing process are moved from one finishing station to another, often on blade carts and on tracks.

As mentioned, although there have been a few notable efforts to automate certain aspects of blade molding and blade finishing operations over the past two decades, these attempts too often have been met with very limited or no success. The broad feedback from many wind industry partners that have been involved in automation research and deployment in the past is that the efforts failed due to four main drawbacks: the high cost of automation equipment, the relative slow speed of automation processing, the low utilization factor of automation equipment, and the lack of automation technology to scale as blades grew in size and length over time. The first three of these drawbacks applied more to molding operations than to finishing operations. The molding operations were more complicated and thus required more complex and costly automation equipment; the speed to lay down fabric and adhesive was slow compared with teams of laborers; and the automation systems sat idle while the blade was being infused, cured, and demolded. By contrast, the targeted effort of automated blade finishing involved relatively less costly equipment, was more in line with the speed of finishing operations compared with laborers and operated for a high percentage of time as blades were cycled through the finishing stations in the factory. The fourth drawback mentioned—the difficulty of adapting and scaling automation solutions as blade sizes being produced grew—applied to both molding and finishing automation efforts. Because of these experiences, as well as promising emerging innovation in technology related to blade finishing and growing constraints in production factory finishing operations, the consensus among the vast majority of wind industry partners (both wind turbine original equipment manufacturers and blade manufacturers) is that the promise of transformational wind blade production automation lies in the area of blade finishing rather than blade molding.

2.2.4.7.2 Drawbacks of Traditional Blade Finishing

The finishing operations for wind turbine blades, mentioned briefly in the section above, have not changed much over the past three decades of blade production—as wind turbine blades have grown from about 9 m to more than 100 m in length. Although some continuous improvements have been realized in blade finishing as the industry has grown, significant drawbacks have remained, including the following:

- The finishing operations for blade manufacturing typically include up to 50% of the overall labor of producing a wind turbine blade. Furthermore, the labor for finishing operations as blades become longer is growing nonlinearly.
- The environment of blade finishing operations, especially compared with blade molding, has always presented environment, health, and safety challenges. Many of the finishing steps involve processes that produce fiberglass and other composite dust, incorporate traditional hand lay-up of composite laminates, and use materials—such as surface primers and paints—that can be hazardous to humans.
- Although different types of nondestructive evaluation (NDE) have been developed to improve the quality of wind turbine blades being produced at a factory, the current manual approach to NDE has limited the speed, efficiency, and efficacy of these processes in the finishing and inspection steps of the blade-manufacturing process.
- The human labor approach to many of the aspects of blade finishing often leads to variable results in the finished quality of wind turbine blades being shipped from the factory—which, in turn, sometimes results in reliability issues in the field for the blades. Although much of this has been controlled by better blade designs, improved manufacturing quality systems, and the development of global wind industry standards, an inherent variability still exists across wind turbine blade finishing operations due to the prevalence of human labor.
- Although it is relatively easy to move 20-, 30-, and 40-m blades from station to station in the traditional finishing process, the logistics of moving very large blades—now growing to more than 100 m long—around the factory is becoming increasingly challenging. Even more importantly, the floor space required to incorporate separate locations for individual blade finishing operations for such massive blades is driving an exponential increase in the required factory size and cost for wind turbine blade production.

2.2.4.7.3 Emerging Challenges in Manufacturing Automation and Blade Finishing

In addition to addressing some of the challenges associated with traditional methods of wind turbine blade finishing operations, the timing of research in the area of blade finishing automation could benefit from the emergence of advanced technology. For example, although previous attempts at automation in both the molding and finishing operations of wind blades traditionally have incorporated gantry-based systems, recent advances in robotic-arm solutions could transform the way that automation is used in blade finishing. Additionally, the recent innovation with respect to robotic arm end effectors, or end of arm tooling, especially in the areas of locating nonlinear and nonplanar surfaces, as well as regulating applied forces between robots and their environment, could greatly enhance the speed and resulting quality of the automated systems. Finally, the emergence of the potential use of autonomous mobile robots, a form of automatic guided vehicles, could vastly increase the flexibility and adaptability of automated wind turbine blade manufacturing systems as blades continue to grow in length and size.

2.2.4.7.4 Potential Benefits of Blade Finishing Automation Research

Research in this area should design and prepare for the prototyping of an automation system for wind turbine blades in the finishing operations of the blade production steps. This will potentially include such steps as trimming, leading edge and trailing edge shaping, tip shaping, post curing, root cutting, root facing, root drilling and composite surface finishing. Design of a "cell-based" approach to complete all these automated finishing steps in one location could reduce the need to move a very large wind turbine blade around the factory floor. The design of an automated system should build on recent advances in robotic technology (as described in the section above) deployed on either high-precision rail systems or autonomous mobile delivery systems.

The potential benefits that could result from the prototyping and eventual commercialization of an advanced cell-based automated wind turbine blade finishing production system include the following:

- The reduction in labor hours and labor costs for wind blade finishing;
- The removal of human labor from the challenging EHS conditions of the wind blade finishing production environment;
- The reduction of individual finishing step cycle times—leading to the reduction in overall blade finishing cycle time;
- The increase in the consistency and quality of the wind turbine blades produced in the factory—leading to increased reliability, better performance, and increased annual energy production in the field;
- The significant decrease in automated system costs from previous commercialization attempts;
- The incorporation of automated NDE into the finishing operation of wind turbine blades leading to increased quality of blades being deployed in the field;
- Enabling vastly superior flexibility of blade finishing automation to allow for the required adaptability of the systems to efficiently grow with quickly changing blade geometries;
- Transforming the current traditional blade finishing production to an automated, cell-based approach that will reduce factory floor space requirements and manufacturing factory costs; and
- Reducing the costs of wind turbine blades and ultimately the LCOE for wind power.

2.2.4.8 Low-Cost Carbon Fiber for Large Composite Structures

The recent transformative growth in the size of wind turbine rotors and blades provides unique challenges for the use of CFRP composites, and solutions are being actively pursued by the leading manufacturers. Several documented applications to date leverage CF to increase blade length without additional weight gain, thus effectively increasing the swept area and associated gains with greater energy output. Using large-diameter rotors for E-glass-based composite blades can also offer incentives to be innovative with the use of CF in the blade design. The approach of using CF in wind turbine blades can be in the form of selective or complete replacement of load-bearing fiber glass or, more likely, in the future, using a completely new blade design that optimizes the use of CF.

The types of external loading experienced by wind turbine blades include flapwise and edgewise bending, gravity and inertial forces, pitch acceleration-related loads, and torsional loading. The flapwise and edgewise bending loads cause most of the damage in wind blades and necessitate high longitudinal tensile and compressive strength and modulus, as well as high fatigue strength, to minimize progressive damage. Because of their high specific modulus and specific strength, CFs have an enormous technical advantage over the use of glass fiber for wind turbine blade applications. For example, an intermediate-modulus CF has a tensile modulus nearly four times that of glass fiber. Glass fiber at a single filament level is more prone to defects than CF, and it has much higher density. The fatigue life of aligned CF composites is also a big advantage. Despite these technical advantages, the wind energy sector has not realized the broad use of CF compared with E-glass fiber, largely because of perceived economics and potential difficulties with reliably securing large volumes of CF for non-aerospace applications.

Fiber-reinforced composites made of glass or carbon experience large reductions in compressive strength due to fiber misalignment and waviness. Current blade manufacturing processes incorporate a very heterogeneous microstructure and include using woven or stitched fabrics, balsa or foam core materials for reducing weight, and thick spar caps. Most blade manufacturers use vacuum-assisted resin transfer molding, which can also introduce process-induced waviness along in-plane and through-thickness directions. This potential misalignment reduces the baseline compressive composite properties dramatically because it predisposes fibers to buckling. The geometric changes along the thickness from ply drops, or use of joints and inclusions, further exacerbate these factors.

2.2.4.9 Polymer Composite Research Challenges

Research in this area should demonstrate the potential to significantly reduce the cost of turbine blades with CFRP structure. Applicability of textile CFs should be evaluated for use in pultruded spar cap elements as a path to cost reduction for utility-scale wind turbine blades. The materials of interest for pultruded spar cap elements are thermoset resins reinforced by CFs. Thermoset resins include epoxy, polyurethane, and vinyl ester.

A significant barrier for composite materials is the ability to recycle and reuse materials. This is particularly true for thermosetting materials containing lower-cost fibers that are not cost-effective to extract. The world is facing an increasing waste stream from end-of-life wind blades over the next several decades. Higher-end aerospace products are also being recycled; however, the industry and value streams are still not well established.

Research in polymer matrix composite manufacturing should help address the entire life cycle of composite structure, including handling, manufacturing, use, and recycling. In addition, the issue of scaling that is predominant in composite manufacturing requires further research. For example, experiments with micrograms of thermosetting polymer will have limited relevance to practical use because of the volume-dependent exotherms of most thermosetting materials. Experiments at scale, or mimicking manufacturing at scale, are crucial for wider adoption of composite materials.

2.2.5 Energy Storage Manufacturing

Energy storage can involve the conversion of one form of energy to another. For example, batteries convert chemical energy to electrical energy and vice versa; phase change materials maintain the thermal energy but convert heat from sensible to latent forms and vice versa; and electric capacitors convert electric to electric and are sometimes called “bidirectional energy storage.” The efficiency of the reversibility of the energy storage process is paramount for these materials along with cost effectiveness and durability. A full treatment of the state of the art of all forms of energy-storage technologies (including chemical, such as hydrogen, synfuels; mechanical, such as flywheels, compressed air, and pumped hydro; and magnetic, such as high-temperature superconducting materials for superconducting magnetic storage) is beyond the scope of this document.[39]

Advances in materials (including materials discovery, design, and synthesis; physical and mechanical behavior of materials; and materials science), processing science (including major scientific disciplines such as chemistry, mathematics, and physics), engineering (including chemical and computer engineering—for example, using AI and ML for the design of materials manufacturing processes), and thermodynamics are required to accelerate energy storage manufacturing R&D. New models of materials and processes based on experimental and computational data could predict material compositions of optimal functionality correlated to manufacturing-process conditions. One promising set of approaches that crosscut manufacturing research are high-throughput experimental methods—combined materials synthesis and characterization/measurement tools that enable rapid collection of data on the materials manufacturing process, including material composition, structure, and functional properties under process-relevant conditions.

2.2.5.1 Manufacturing of Electrochemical Energy Storage (EES) and Conversion Systems

Electrochemical systems long have been viewed as crucial to enabling the future of transportation and ensuring a resilient grid. From electrification of transport (on the roads and in the sky) to enabling devices that can store electricity, batteries and FCs are enabling technologies for the future. Electrochemical systems also share a common set of material and component challenges from a manufacturing perspective. The materials that serve as electrodes must be designed with exquisite morphological and structural precision to ensure performance, and the methods for manufacturing must ensure that the device maintains a low cost. These materials must be made into composite porous electrodes with different materials performing different functions (e.g., electron conductors, ion conductors, reaction sites).

These electrodes must be thin, be uniform, and have the right distribution of the various phases to ensure superior performance. Both devices require a separator—either polymeric or a hard solid—that ensures that the electrodes do not electronically short but allow high ion transport. Current collectors must be thin and low in cost, and also must be corrosion resistant and highly electronically conductive. Some batteries, such as flow batteries, also share the common need to bring in reactants from external tanks and require a means to remove the spent product, thus imposing the need for flow fields.

In other words, manufacturing challenges in these systems range from materials all the way to the device. The next section discusses the specific state of manufacturing for energy storage and FC systems.

2.2.5.2 Lithium Battery Manufacturing

Lithium batteries have been used widely in consumer electronics and electric vehicles. For Li battery manufacturing, the wet-coating process often is applied to produce battery cathodes. The cathode films

[39] https://www.eesi.org/papers/view/energy-storage-2019

are coated onto a current collector foil (e.g., copper foil, aluminum foil), followed by solvent evaporation in a drying zone. Various coating technologies can be used to deposit cathode coatings, including tape casting, slot die coating, and screen printing. Of note here is that slot die coating is attractive and widely used because of its production efficiency.

The functional ingredients of cathode inks are Li-ion conductors such as Ni-rich $LiNi_xMn_yCo_{1-x-y}O_2$ and Li Fe phosphate ($LiFePO_4$), and electron conductors such as carbon black. Polyvinylidene fluoride is a common binder material. Other binders, such as polyvinyl butyral, also are used because they offer better ink stability (Shen et al. 2019). Depending on binders, common solvents are toluene, tetrahydrofuran, and n-propanol. The future trend is to make water-based environmentally friendly solvents.

Lithium batteries are mainly divided into two categories: (1) Li-ion batteries (LIBs) and (2) solid-state lithium batteries (SSBs). Most traditional LIBs use liquid electrolytes, and SSBs have solid electrolytes that can introduce improved battery compactness, enhanced volumetric energy density, and increased safety and lifetime. With the development of all SSBs, the wet-coating process also is presented for solid electrolyte manufacturing because of its minimal restructuring requirements of existing production lines.

Other production methods, however, also are proposed, such as aerosol deposition (Hanft et al. 2018; Schnell et al. 2019) and film deposition, such as sputtering (Lobe et al. 2016). In terms of wet-coating methods, the inks are a mixture of solid inorganic Li+ conductors, such as aluminum-doped $Li_7La_3Zr_2O_{12}$; $Li_{0.05-3x}La_{0.5+x}TiO_3$; and $Li_{1+x}Al_xTi_{2-x}(PO_4)_3$; and a polymer electrolyte, for example, poly(ethylene oxide) and polystyrene-block-polyethylene oxide. Conducting Li salts—such as Li bis(trifluoromethanesulfonyl)imide—also can be introduced to further improve the ionic conductivity. In addition to functional ingredient materials and compositions, the microstructure and electrolyte-electrode interface also influence the ionic conductivity of the solid electrolyte. These microstructural features include particle size, interstices, particle aggregation, and percolation. Argonne and ORNL have developed nanoparticle and nanofiber electrolytes to improve the contact of electrolytes and microstructures by using flame spray pyrolysis and electrospinning methods.

2.2.5.2.1 Manufacturing Challenges in Electrochemical Energy Storage

Electrochemical energy storage (EES) technologies will enable increased variable renewable adoption (Kroposki et al. 2017) and significant reductions in CO_2 emissions from transportation (Needell, McNerney, Chang, and Trancik 2016). At its core, EES must efficiently time-shift electrical energy in a cost-effective fashion. The key factors that dominate the cost of EES systems are the choices of materials and the manufacturing methods of the storage system.

In all electrochemical systems, there are common components whose performance significantly impacts cost. Each cell of a battery has three components that receive the most attention, the anode, the cathode and the electrolyte. There are other components, however, such as the case, separator, current collectors, tabs, and seals that all impact performance and cost. Moreover, the assembly of these components into a functional device is a major manufacturing challenge that will continue to require innovation to improve performance and reduce costs.

Typically, EES devices are enclosed to ensure integrity. In lead acid batteries, a polymer enclosure contains the electrolyte. In Li-ion cells, this enclosure can be a ridged container (e.g., stainless steel) or a flexible polymer. In flow batteries, the enclosure is the tanks, and piping can range from polymers to high-quality stainless steel. Obviously, the nature of the casing directly effects the cost and energy density of the system. Manufacturing opportunities in the development of stronger, lighter materials that are compatible with the electrolytes—for example chemical compatibility and corrosion resistance using cheaper alternatives—are key targets.

Current collectors enable electrons to flow to an external circuit with a minimum of resistance. Manufacturing approaches to high-conductivity, light, thin, corrosion-resistant current collectors that are compatible with the EES chemistries will lead to improved performance and reduced cost. As an example, current anodes in LIBs use copper foil that ranges from 6 μm to 10 μm and aluminum in the 3 μm to 10 μm range. Thinner foils would reduce both volume and weight in cells, but the tensile strength of the foil is a limitation in the current R2R manufacturing (Yang, X. et al. 2019). Approaches to improve the strength of the thinner foil or new manufacturing processes that reduce tears and folds are desirable. Alternatively, it might be possible to develop new manufacturing approaches to materials that can replace the extant current collectors with advanced composites to reduce costs and improve performance.

Graphite is the current anode material in Li-ion systems. Manufacturing processes that improve the purification of natural graphite with less waste and less environmental impact would significantly mitigate cost issues and broaden the supply chain. Alternatively, manufacturing improvements in the production of synthetic graphite would significantly reduce costs. Silicon is a higher-capacity alternative to graphite in Li-ion cells. Although challenges remain in the development of effective Si anodes, low-cost routes to Si production, at scale, will be needed. The ultimate negative electrode in the Li-ion systems is Li metal itself (Cheng, Zhang, R., Zhao, and Zhang Q. 2017). Although Li metal foil is readily available, it is too thick for practical applications. Manufacturing approaches to thin-film Li anodes are needed to enable beyond–Li-ion chemistries.

Cathode materials are the most expensive component of an LIB. The most common manufacturing method is co-precipitation that leads to phase-pure products having a uniform particle size (Zheng, Wu, and Yang 2011). Improving energy density, reducing cost, and extending lifetimes of cathodes is a major research undertaking. As new approaches to balancing all of these variables develop—such as coatings (Shobana 2019) and new particle design (Harlow et al. 2019)—improved manufacturing techniques will be critical to keep costs down while improving performance.

Electrolytes provide the essential ion-conducting role in EES systems. The electrolyte must be compatible with both the low-potential negative electrode and the oxidizing positive cathode (Ue and Uosaki 2019). The development of new electrolyte compositions designed to increase energy density, prolong life, and improve safety continue to advance. New electrolytes that encompass liquids, solids (Zhao, Q., Stalin, Zhao, C.-Z., and Archer 2020), and composites (Li, S. et al. 2020) all are under development. In each case, the new approaches require new manufacturing routes and techniques to allow large-scale full cell production at a low cost. This is especially the case for the development of solid ceramic electrolytes, for which there is currently no scalable solution to manufacturability.

Keeping the positive and negative electrode physically separated is essential in a functional battery (Zhang, S. 2007). The separator can simply act as physical barrier, as in the porous polyethylene common in LIBs, or it can function as the ion conductor in flow batteries. As energy densities advance, the properties for the separator must improve. Advanced materials to resist high voltage, improve self-short resistance, and ensure compatibility with beyond–Li-ion systems require manufacturing methods for both the separator and the cells themselves. In the development of advanced ion conductors to improve flow battery performance and energy density, advanced polymers and composites are being explored (Tan et al. 2020). As with all materials development, efficient manufacturing will be the key to large-scale deployment of these new membranes.

Electrodes are the composite structures formed from the current collector and the anode or cathode material. Their manufacture requires control of materials compatibility, rheology, and electrode assembly (Wood III, Li, and Daniel 2015; Hawley and Li 2019). The state-of-the art technology in the formation of electrodes for LIBs is the slot-die coater in an R2R process (Schmitt et al. 2013). Future manufacturing challenges will include the production of electrodes that increase energy density, using new chemistries, or larger amounts of active materials. For example, thick electrodes provide improved energy density but result in limitations in ion diffusion through the electrode structure. Manufacturing routes that enable improved ion diffusion through ordered 3D electrodes might lead to functional thicker electrodes. Additionally, other coating approaches—for example AM, spray, electrophoretic, and solvent-free deposition techniques—could lead to lower-cost high-performing batteries.

2.2.5.2.2 Manufacturing Challenges for the Next Generation of Electrochemical Energy Storage

Beyond–Li-ion systems and advanced energy-storage approaches that currently are available only within research labs—such as SSBs, molten metal batteries, Li-sulfur batteries, Li-air batteries, multivalent batteries, and a myriad of other options—will not be compatible with today's manufacturing landscape. New challenges in materials compatibility necessitate innovative manufacturing advances. Completely new cell designs—such as totally solid-state systems—will require a complete redesign of the manufacturing process. If these low–technology-readiness-level chemistries are to compete with market leaders such as lead acid, Li-ion, and flow batteries, they must be produced at scale and at costs that give them the opportunity to break into the market. New materials development must be coupled with advances in manufacturing science that will enable the batteries of the future to quickly move from the lab to the market.

2.2.5.3 Fuel Cells

Fuel cells are a renewable energy generation technology applicable to a wide range of portable, stationary, and automotive power deliveries. Many FC technologies have been developed, including alkaline FCs, phosphoric acid FCs, solid oxide FCs, and polymer electrolyte membrane FCs (PEMFCs). Among different types of FCs, the PEMFC is the most extensively studied because of its low operating temperature, reduced catalyst loading, efficient proton transport, and ease of refueling.

The core component of PEMFCs is a membrane electrode assembly (MEA) that generates and conducts protons. The MEA consists of catalyst-coated anode and cathode layers that are separated by a proton exchange Nafion membrane. The catalyst coatings often are made of catalyst inks. The catalyst layer provides reaction sites for the oxidation of hydrogen on the anode as well as the reduction of oxygen on the cathode. Thus, catalytic efficacy at the interface between the catalyst layer and electrodes is critical and often is related to the loading and spatial distribution of the catalysts. Typical catalyst inks are a mixture of carbon-supported PGM catalysts, ionomer solutions in H+ form such as Nafion, and solvents (e.g., water, n-propanol). The microstructure of the catalyst inks is governed by interactions among Pt, carbon, and ionomer that can be evaluated by rheological and ultrasmall-angle x-ray scattering studies (Khandavalli et al. 2019). Due to the usage of expensive PGM catalysts, the MEA is the costliest component of an FC. Developing low-PGM catalyst layers—such as Pt-transition metal alloys, core-shell nanostructured catalysts, non–PGM-containing catalysts (Co_3O_4), and nanostructured ultrathin catalyst layers—without sacrificing FC performance has been widely investigated in recent years (Zhang, C., Shen, and Peng 2017; Stacy, Regmi, Leonard, and Fan 2017; Antolini 2018; Deng, R. et al. 2020).

2.2.5.3.1 Fuel Cell Manufacturing

Fuel cells efficiently convert fuels directly into electricity without combustion and are key power-production technology for building a competitive, secure, and sustainable clean-energy economy.[41] They offer a range of benefits, including greater energy efficiency and reduced air pollution, criteria pollutants, and less water use compared with combustion technologies. Fuel cells have been deployed in all energy sectors: commercial, residential, industrial, and transportation. Although FCs are competitive in some applications—such as for forklifts—broader commercialization would be greatly accelerated if the cost were reduced. Lack of large-volume manufacturing is one of the factors leading to their high cost. Increasing manufacturing volumes and scale is expected to lead to significant cost reductions to achieve cost competitiveness in certain markets, such as transportation.

An FC consists of an anode and a cathode separated by a liquid or solid electrolyte that transports charged particles between them. The fuel is introduced on the anode and the oxidant is introduced on the cathode. The amount of power produced by an FC depends upon several factors, such as FC type, cell size, the temperature at which it operates, and the pressure of the gases supplied to the cell. A single FC produces less than 1 V of electricity, which is generally insufficient for most applications. To meet the power requirements, individual cells are "stacked" together in a series to form an FC stack, as shown in Figure 11.

Six different types of FCs, differing in the type of electrolyte and operating temperature, have been commercialized: PEMFC, direct methanol, alkaline, phosphoric acid, molten carbonate, and solid oxide FCs. PEMFCs dominate in terms of both number of units shipped, accounting for more than 60% of the total numbers, and total number of megawatts shipped, accounting for more than 80% of the megawatts delivered in 2019.[42]

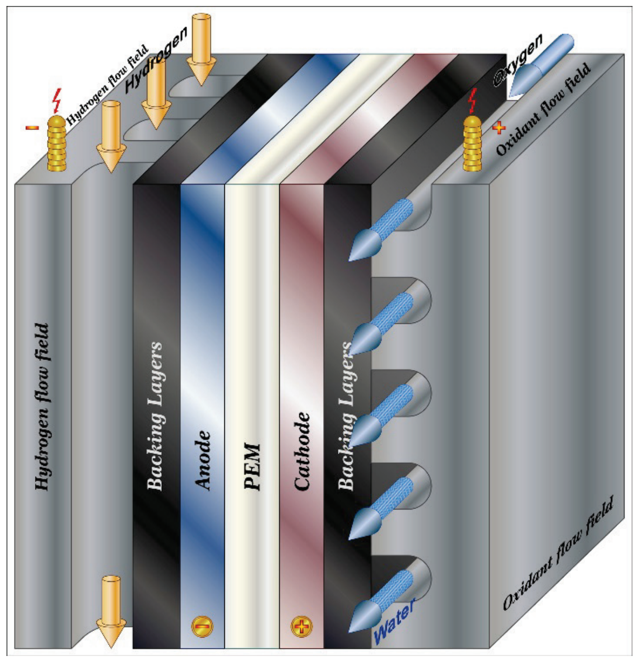


Figure 11. PEMFC stack showing individual cells "stacked" together.

(*Source*: F. A. Saad, Shutterstock)[40]

Light-duty vehicles are an emerging market for PEMFCs, with more than 8,000 FC vehicles sold or leased in California as of February 2020.[43] One of the challenges FC vehicles face is the high cost of the FC stack. Although R&D conducted by the DOE's Fuel Cell Technologies Office has reduced the

[40] https://www.plugpower.com.
[41] https://www.energy.gov/sites/prod/files/2017/05/f34/fcto_myrdd_fuel_cells.pdf.
[42] http://www.fuelcellindustryreview.com.
[43] https://cafcp.org/by_the_numbers.

projected cost of the automotive FC stack by 67% since 2006, further reduction is needed to reach the long-range cost target of $30/kW (Papageorgopoulos 2019). Analyses funded by the Fuel Cell Technologies Office estimate that the current cost of an FC stack of $210/kW at a production scale of 1,000 units per year would decrease to $50/kW and $40/kW at annual production of 100,000 and 500,000 units per year, respectively. Automotive FC stacks can contain upward of 400 cells to deliver roughly 100 kW of power.[44, 45] Achieving annual production volumes of 100,000 to 500,000 stacks would require the manufacturing of 40 million to 200 million individual cells. This is a substantial scale-up from today's volumes and presents numerous manufacturing challenges and opportunities.

At the heart of a PEMFC is the MEA that consists of the membrane, the anode and cathode catalyst layers, and the gas diffusion layers (GDLs) (Pollet et al. 2016). The membrane facilitates the transfer of protons from the anode to cathode while impeding the flow of electrons. The anode catalyst catalyzes the oxidation of hydrogen, and the cathode layer catalyzes the reduction of oxygen. The GDLs facilitate transport of reactants into the catalyst layer, as well as removal of product water.

Both the anode and cathode catalyst layers consist of Pt-based nanoparticles, either pure Pt or a Pt alloy, supported on a high–surface-area carbon substrate and mixed with a proton-conducting ionomer. The catalyst loading on the cathode side is significantly greater than the loading on the anode side because the oxygen-reduction reaction is considerably slower than the hydrogen-oxidation reaction on the anode. The catalyst is applied using an inking process to prevent the formation of catalyst aggregates and to optimize the dispersion. The resulting structure of the catalyst layer must provide a three-phase interface allowing connection of the gas phase, electronically conducting phase (the carbon), and ionic conducting phase at the Pt-based catalyst particle. To obtain the proper dispersion, the catalyst ink consists of a mixture of catalyst, ionomer, an organic solvent, and water. The catalyst ink can be applied directly or via a decal transfer to the membrane, forming a catalyst-coated membrane. For low-volume production, the catalyst ink can be applied using several techniques, including hand painting, air brushing, ultrasonic spray coating, or Mayer rod coating.

At higher production volumes, R2R processes such as single- or dual-sided simultaneous slot die coating processes are used. The catalyst-coated membranes and the GDL are then bonded together using an R2R hot press process. Alternatively, the catalyst can be applied directly to the GDL to form a gas diffusion electrode. The GDL is a sheet of carbon paper that is partially coated with polytetrafluoroethylene to prevent excessive water buildup and to facilitate gas diffusion. The gas diffusion electrode then is hot pressed to the membrane to form the MEA. Once formed, the MEA is then fed through cutters and slicers that trim it to the desired dimensions for insertion into the stack.

The MEAs are combined with bipolar plates to form a stack. The bipolar plates connect each of the individual cells electrically, provide flow-field channels for gaseous fuel and air to flow to the MEA and for removal of reaction products (water) from the cell, and keep the cells cool. Bipolar plates are formed in two halves and then are welded or joined together to provide internal coolant channels and external flow fields for the gases. Current estimates of bipolar plate costs indicate manufacturing costs account for more than 45% of the cost of the bipolar plate (Park et al. 2014).

[44] https://www.toyota.com/mirai/assets/core/Docs/Mirai%20Specs.pdf.
[45] https://www.hyundaiusa.com/nexo/specifications.aspx.

The design of the gas flow-field channels is complex, as shown in Figure 12, and significantly affects the performance of a stack. The two most common types of bipolar plates are carbon-based (graphite or a carbon composite) or metal (typically stainless steel or titanium). Carbon-based bipolar plates (BPP) are typically made by compression molding. Metal BPPs are made using sheet metal stamping techniques, typically progressive die stamping, given the complex design of the plates. Carbon-based BPPs have the advantage of higher formability, low contact resistance, and high chemical stability. However, processing times are longer than for metallic plates, and carbon plates are not as strong; so they tend to be thicker, thus leading to lower stack power densities. Metallic BPPs exhibit high mechanical strength and good electrical and thermal conductivity; however, corrosion is a problem because of the highly acidic conditions and thus requires the application of protective coating layers onto the metal plate. Additionally, stretch-formed flow fields formed by stamping of metallic steel BPPs cannot achieve the target geometries desirable for water management (narrow channels with tight curvature at the corners of narrow upstanding landings). This leads to flow field designs with increased pressures and pressure drops.

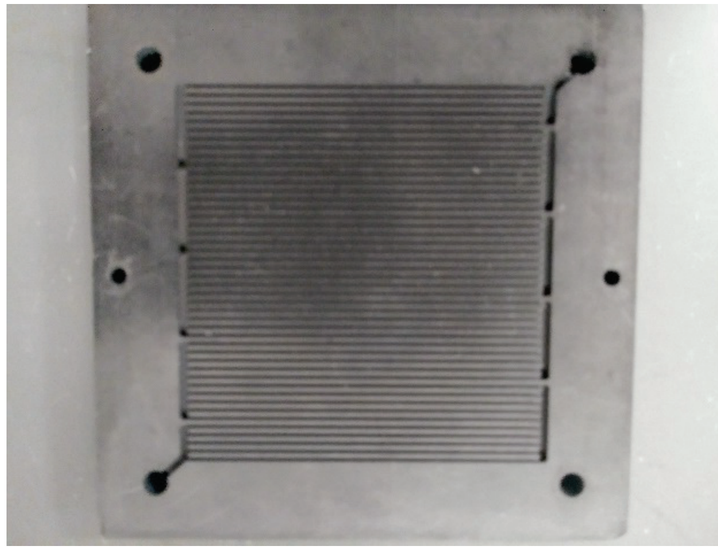


Figure 12. Bipolar plate showing complex gas-flow channels.

(*Source*: Fuel Cell Store, used by permission)

A high-throughput joining method is needed to join the two halves of the BPP, sealing the plates and forming the cooling channels. For metal plates, laser welding has been the baseline method of joining. Current welding speed was estimated at 0.2 to 5 min per BPP assembly because of the long weld lengths required. It was estimated that with multiple lasers per station and multiple stations, welding times of ~2 seconds per BPP assembly are achievable, although such short times have not yet been demonstrated (Kopasz, Benjamin, and Schenck 2017). An alternative approach is to use adhesive bonding of the plates. The adhesive bonding process can be done with faster projected processing speeds than welding. The downside is that seals may fail more frequently than is experienced for welding. Gaskets then are applied to the plate to seal the MEA to the BPPs, preventing leakage of gases (Napporn, Karpenko-Jereb, Pichler, and Hacker 2018). Using a traditional approach of sealing the MEA with an injection-molded gasket, Dana[46] estimated a throughput of 1 plate every 45 sec. At that rate, 21 injection-molding cells are required for a production rate of 30,000 vehicles per year (~10,000,000 MEAs) (Kopasz, Benjamin, and Schenck 2017). Finally, the end plates cap off and protect the stack by evenly distributing the compressive loads across the stack. The end plates interface with the current collector while electrically insulating the ends of the stack. Higher throughput is needed.

[46] https://www.dana.com/company/innovation-technology/dana-energizes-fuel-cell-components-with-more-than-20-years-of-research/.

2.2.5.3.2 Fuel Cell Manufacturing R&D Challenges

The FC performance is limited by the transport of oxygen to the Pt catalyst particles and transport of water out of the MEAs. Processes that can better control the distribution of Pt on the carbon supports and distribution of carbon particles and ionomer in the cathode catalyst layer offer the potential for higher performance by improving transport. A better understanding of the colloidal chemistry of the ink suspensions and how it relates to the deposited and final catalyst layer structures and charge and mass transport processes would be beneficial. Advanced manufacturing methods such as 3D printing also offer the potential to create unique catalyst layer structures that could improve transport and MEA performance; however, the high processing speeds required for applications like automotive vehicles should be kept in mind.

For BPPs, the main issues identified as limiting their use were forming limitations, cost, and corrosion concerns (Kopasz, Benjamin, and Schenck 2017). Formability of the steels used in current plates requires compromises in flow channel design that lead to suboptimal performance and multistep high-pressure stamping. Additionally, coating steps are needed for corrosion protection. The more corrosion-resistant materials are more expensive and, in the case of titanium, less formable. More flexible/formable materials, such as aluminum and aluminum alloys, can reduce plate restrictions on flow channel dimensions and decrease stamping time and costs. The corrosion issues, however, would require a perfect or near-perfect coating, which would be very difficult to achieve.

Manufacturing methods to allow rapid forming of steels and forming beyond stretch-forming limitations or corrosion-resistant coatings on aluminum plates would be beneficial. Advanced manufacturing techniques—such as AM or some photochemical etching techniques—might allow for improved flow-field designs; however, the high volume of plates needed for applications such as automotive use should be kept in mind. Plate joining by welding and application of gaskets to BPPs for sealing to the MEAs also are time consuming, and there are opportunities for other high-speed joining and sealing techniques. For carbon-composite BPPs, processing speed is a limiting issue, including processing time to seal the plate against permeability of hydrogen or air. High-volume manufacturing processes and additional capacity/throughput are needed. Manufacturing processes that allow for carbon-composite plates of comparable thickness to current state-of-the-art metal plates also would be beneficial.

2.2.5.4 Manufacturing of Other Advanced Storage Materials/Processes

In addition to chemical to electric energy storage highlighted in the previous sections, several types of thermal energy storage materials and processes are the subjects of active research (Dhar, Wijewarnasuriya, and Dutta 2018). For example, current commercial thermal energy storage technologies operate at two ends of the temperature spectrum: low-temperature (<100° C) storage mainly for building heating and cooling applications and high-temperature (~500° C)[47] storage for concentrating solar power plants.

2.2.5.4.1 Low-Temperature Thermal Energy Storage Materials

The cutting edge of thermal storage technology for building applications is at a very low temperature (<30° C). Manufacturing R&D for low-temperature thermal energy storage materials is centered in EERE's Building Technologies Office because it provides significant opportunities for reducing building energy consumption and the flexibility to time-shift demand on hourly time scales. The most

[47] https://www.nrel.gov/analysis/solar-industrial-process-heat.html.

widely used phase-change materials (PCMs) in building applications are paraffins, yet their high cost, low volumetric energy capacities, and high combustibility have prevented their widespread acceptance and suggest a search for alternatives (Bland, Khzouz, Statheros, and Gkanas 2017). Two classes of new low-temperature energy storage materials appear promising for such applications: (1) inorganic salt hydrate PCMs and (2) thermochemical energy storage materials. Under category 1, inorganic salt hydrates and their eutectics have features that show great promise as primary PCMs for building applications. These features include high volumetric energy densities and suitable transition temperatures that can be achieved at low cost (Xie et al. 2017). However, technical challenges remain that include excessive subcooling effect, incongruent melting and phase segregation during transition, corrosiveness, and difficulty in efficiently microencapsulating. Overcoming these barriers requires low-cost and stable (e.g., chemically, physically, thermally) salt hydrate PCMs. Under category 2, thermochemical energy storage technologies are promising because they have potentially high energy densities and display the possibility of storing energy for long periods with negligible self-discharge.[48] Advances are needed to optimize their operating requirements including, but not limited to, operating temperatures closer to 50° C, multicycling efficiency, material and reactor cost, and appropriate systems design for building applications.

2.2.5.4.2 Medium- and High-Temperature Thermal Energy Storage Material

Manufacturing R&D for medium- and high-temperature thermal storage materials is centered in EERE's Solar and Advanced Manufacturing Offices because of the higher temperatures found in concentrating solar power and industrial applications. Although research continues on improving concentrating solar power–related storage, higher-temperature thermal energy storage is nascent for many other temperature regimes, including medium-temperature applications in the geothermal and general industries (<300° C) and ultrahigh-temperature nuclear, fossil, and certain industrial (e.g., glass) applications (>1,000° C). In addition to being more cost-effective and robust for such applications, these materials also are challenged to be both environmentally friendly and safe. Advances in storage materials are needed for PCMs (encapsulated and unencapsulated), inorganics, molten salts, Ni-based alloys, and stainless steels. All forms of thermal energy storage (e.g., sensible, latent, thermochemical) require improvements to maximize thermal conductivity and durability (e.g., with aging and thermal cycling) and scale to industrially relevant volumes and masses (Teller et al. 2013).

2.2.6 Metal Processing and Manufacturing

The production of metals involves, in addition to mining, an energy-intensive extractive step, wherein the metal is reduced from the thermodynamically stable compounds in the ore, commonly oxides or sulfides. Considering that the embedded energy in coal used for carbothermic reduction of steel in an integrated steel plant amounts to 90% of the total energy (14 GJ/ton of steel) of steel manufacturing (Fruehan, Fortini, Paxton, and Brindle 2000) and nearly 30 million tonnes of pig iron are produced annually in the United States, this amounts to about 4×10^{17} joules, or 0.4 quads. The percentage is similar for other carbothermal reductions. Although there have been significant advances in recycling through electrically powered electric arc furnaces for primary metals, carbothermic smelting is still carried out at significant scales for value-added products. Further, when targeting smaller volumes of CMs, including rare earths and precious metals, electrolysis offers a competitive alternative to hydrometallurgical and pyrometallurgical routes when the price of electric power decreases.

[48] https://irena.org/documentdownloads/publications/irena-etsap%20tech%20brief%20e17%20thermal%20energy%20storage.pdf.

Recent breakthroughs in understanding the relation between micro- and nanoscale characteristics and bulk properties (e.g., grain-size-dependent yield strength in metals where strength as a function of grain size alone can vary by more than an order of magnitude) (Cordero, Knight, and Schuh 2016) suggest that advances in understanding atomic and molecular phenomena could speed progress in metal processing and manufacturing R&D.

2.2.6.1 Primary Manufacturing Processes

Drivers for the transition to hydrogen as an energy source include the ability to produce hydrogen from diverse, domestic resources—including from renewable energy sources and fossil fuels—as well as opportunities for developing technological innovations that rely on low-cost hydrogen and reduce carbon emissions. An opportunity also exists for developing technological innovations that enable increased use of electric power and reducing carbon in primary metals production.

The majority of steel in the United States (~68%) is produced by the electric arc furnace route through reuse of recycled metal scrap. The impurities in the scrap mandate diluting this scrap with virgin iron units to produce high–value-added steel products, offsetting some of the inherent energy savings in recycling scrap (Huellen 2006). Although the use of electric power to produce steel by electrolysis has been proven in the laboratory (Allanore, Yin, and Sadoway 2013), scale-up is challenging due to the inherent difficulties with high-temperature electrolytic cells, surface-area controlled processes, and the storage of electrons to avoid sensitivity to transient operation. Therefore, electrolysis is not a viable technology route for flexible steelmaking in the near term. The use of hydrogen—produced from renewable energy, even when intermittently available—offers an attractive alternative because hydrogen can be more readily stored than electricity. And it can be used as a reactive molecule (i.e., not just for energy) to reduce iron oxides in continuous shaft furnaces where the production rate scales with the *volume* of the reactor, rather than the electrode *area* (as in electrolysis). Several European steelmakers already are engaged in projects that use hydrogen in steelmaking. GrInHy (Salzgitter in Germany) and H_2FUTURE (voestalpine in Austria) focus on electrolyzer development. The Swedish consortium HYBRIT (SSAB, LKAB, and Vattenfall) is looking at the entire fossil-free value chain for primary steel. The basic concept is to use hydrogen produced by electrolysis to reduce iron into direct reduced iron, which is then converted to steel in an electric arc furnace at the pilot plant scale (~1 ton/h). ArcelorMittal has announced the construction of a demonstration-scale (~13 tons/h) hydrogen-based shaft furnace to produce direct reduced iron in Hamburg, Germany, where the “grey” hydrogen is taken from the existing MIDREX plant. POSCO, the world’s fourth-largest steelmaker (located in South Korea), is developing direct-reduction technologies to obtain hydrogen gas from small- or mid-sized nuclear reactors. Several commercial facilities exist for natural-gas–based direct reduction (e.g., voestalpine’s Texas hot briquetted iron plant).

Regardless of approach, there exists a knowledge gap: There is little new and relevant information on hydrogen and mixed natural gas direct reduction processes in the scientific literature. In particular, information is lacking on ways to enhance the reduction kinetics when using modern iron oxide pellets at temperatures and pressures relevant to the industrial furnaces. Because reduction kinetics is driven by temperature, pellet structure, and structure of the reaction product layer that depends on the reduction conditions, there are gaps in estimating process rates during operation in a flexible manner, where reduction through natural gas and hydrogen is switched or rebalanced.

2.2.6.2 Secondary Manufacturing Processes

Secondary processes in metallurgy generally are focused on attaining the correct melt chemistry for the desired product. The product after extraction often is obtained in a molten state and is further processed pyrometallurgically, requiring alloying additions to adjust the melt chemistries. Undesired impurities stemming from the ore, reactor walls, slags, or gas atmosphere are removed through heterogeneous reactions whereby products are separated

- into an immiscible molten phase, e.g., an oxide slag;
- to a gas phase or vacuum; or
- through electro-plating or during partition during solidification/crystallization.

A critical reactor for secondary metallurgy is the basic oxygen furnace (BOF), used for adjusting the composition of liquid steel from pig iron. Although its prime purpose is to adjust carbon, it also serves as a gatekeeper to adjust Si, manganese, and phosphorus; thus, the ability to control the reactor enables flexible operation with different raw material sources while maintaining product quality.

In the BOF, reactor carbon-rich pig iron is converted to steel, pure oxygen is injected though a water-cooled lance into the molten metal bath, and the bath is covered by a molten oxide (slag phase). The role of the oxygen is to selectively react with oxidizable elements and remove primarily carbon (from more than 4% to less than 0.1%) as a gas phase, but also other unwanted impurities stemming from the iron ore as oxides that separate and dissolve into the slag phase. The BOF process kinetics are very complex because they involve multiple simultaneous processes. Simultaneous multiphase interactions, heat and mass transfer, gas-slag-metal chemical reactions in multiple zones, and vigorous fluid flow caused by the impingement of oxygen jets occur in a BOF reactor at high temperatures. Additionally, it is a dynamic and transient process that makes the kinetics involved in a reactor more complex. Direct measurements of temperatures and chemistries are very difficult due to the nature of the process, which involves harsh conditions. That is why many researchers have been trying to address these difficulties through modeling the process. A multiphysics description of the converter process, with the ultimate aim of predicting carbon content in the melt, will have to involve several submodels at different scales. These models will have to capture various phenomenon occurring in the gas/metal, metal/slag, and metal/slag/gas mixtures, as well as transport processes in bulk metal and bulk slag.

Using ML, techniques could be developed that do not employ any simplifying assumptions. Algorithms could be trained on real data sets and could provide a ring road to all the complexities involved in a BOF reactor, including prediction of key parameters such as the decarburization rate precisely.

2.2.7 Cement Production

Cement is produced by 96 facilities in 34 states in the United States.[49] Cement production is an energy-intensive process resulting in significant CO_2 and other emissions from the combustion of fuel for process heat. Calcium carbonate (limestone) is a major material used to produce clinker for Portland cement. The calcination process by which calcium carbonate (limestone) is heated to produce calcium oxide (lime) is also a significant source of CO_2 because the process produces lime and CO_2 in a 1:1 molar ratio in the following reaction.

[49] https://doi.org/10.3133/mcs2020.

$$CaCO_3 \text{ (limestone)} \rightarrow CaO \text{ (clinker)} + CO_2 \text{ (byproduct)}.$$

In 2018, the United States produced ~88 million metric tons (MT) of Portland cement from clinker (Bernhardt and Reilly 2019). This production resulting in about 69 million MT of associated production-related CO_2 emissions. Of this, roughly 41 million MT or 60% were from calcination and the remainder from fuel combustion.

The primary inputs to cement manufacturing are limestone, clay, and gypsum that are quarried and transported to the facility via trucks or rail. These materials are stored on-site until they are crushed and ground before pyroprocessing. The raw meal is prepared from the combination of limestone, clay, gypsum, and other additives such as sand or iron ore to achieve the desired composition. The major components of Portland cement are Ca, Si, Al, Fe, and O; additional minor components include Mg, S, Na, and K, typically totaling less than 5% of the mixture.

The raw meal is fed to a rotary kiln, which heats it to about 2,700° F in a step referred to as "pyroprocessing." During pyroprocessing, a series of chemical reactions take place to convert the ingredients into clinker that exits the kiln. Heat is recovered from the clinker as it cools and is fed back to the kiln to improve energy efficiency.

Although newer cement kilns employ a dry process, some older kilns use a wet mixture that requires more energy to evaporate the water as the mixture is heated to the reaction temperatures. Variations of the newer, dry-kiln technology also can employ preheaters and precalciners that further improve the energy efficiency of the process. In the United States, the more energy-efficient systems incorporating precalciners account for about 80% of cement production, which reduces fuel energy consumption by ~45% compared with the wet process, and ~30% compared with the less-sophisticated dry process without a preheater.

The clinker then is cooled and passed through a series of grinders, at which point additional limestone and gypsum are added to improve the properties of the cement mix. The cement powder is screened and siloed for storage before shipment to ready-mix batch plants or another form of distribution. At ready-mix batch plants, cement is mixed with water and aggregate to make the concrete mixture provided to concrete trucks for transport to job sites. The aggregate is catered to the specific use case and can include sand, gravel, crushed stone, and iron blast furnace slag.

Argonne recently has created a new life cycle inventory for US average concrete. Figure 13 provides the process flow diagram and shows the subprocesses included within the boundaries of the cement production facility and the ready-mix batch plant. Each box represents a unit process in the model. The model includes all major inputs to the processes involved in the production of concrete; other detailed inputs to the processes are omitted from the diagram for simplicity. The life cycle GHG and criteria air pollutant emission results are presented in Figure 14. For most emitted substances, fuel combustion for the cement kiln represents the most significant source of emissions. In the case of particulate matter emissions (PM2.5 and PM10), however, the ready-mix facility is also a significant source. For CO_2, cement production is the most significant contributor because of significant contributions from both fuel combustion and the calcination reaction. Negative values for methane reflect the avoidance of landfill gas emissions associated with the combustion of municipal solid waste for process heat.

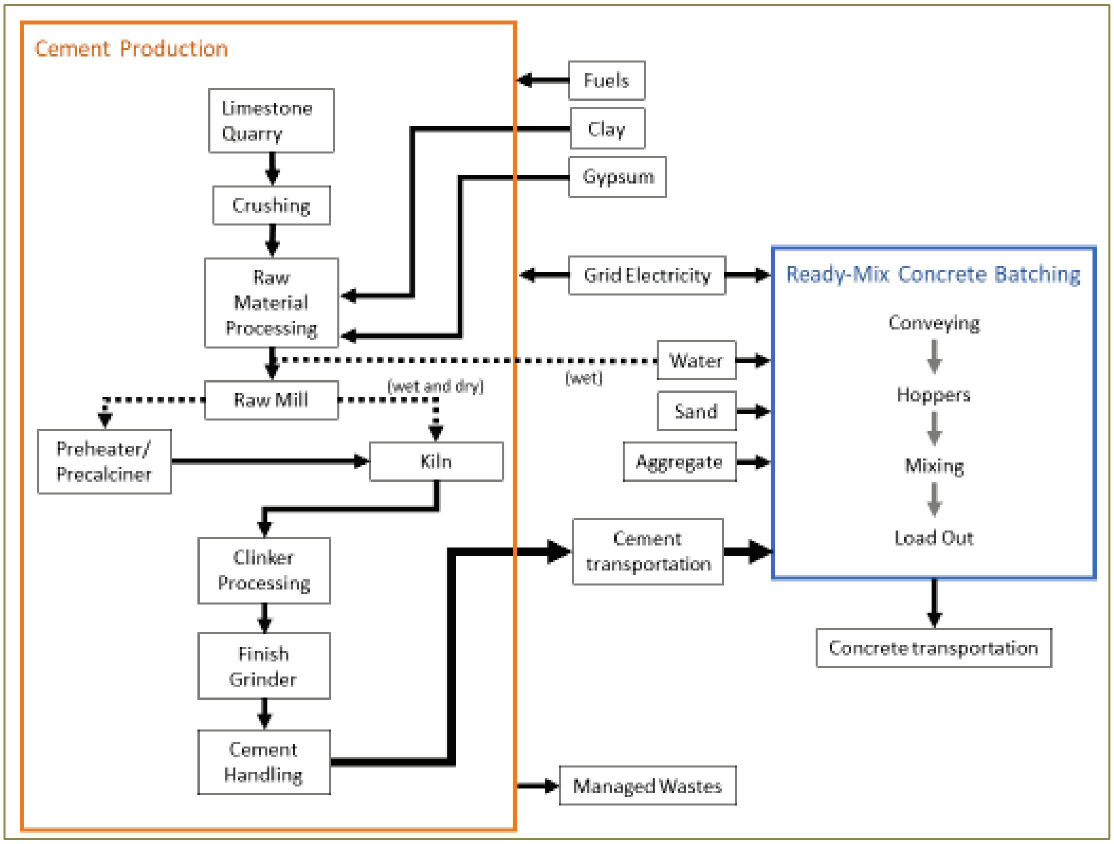


Figure 13. Process diagram for Argonne's life cycle inventory for concrete. The individual boxes represent unit processes in the model, and the orange and blue boxes represent the boundaries of the cement production facility and ready-mix batch facility (*Source*: Life cycle energy and environmental impacts of concrete: GREET update, T. Hawkins et al., May 19, 2020)

These results demonstrate a significant opportunity to reduce the impacts associated with cement production through new technologies to improve energy efficiency and to provide cleaner sources of process heat. The cement production process has been considered as a candidate for carbon capture and utilization or sequestration because of the concentrated emissions from fuel use and calcination. Improved technologies also could benefit human health by reducing criteria air pollutant emissions from cement production and ready-mix facilities.

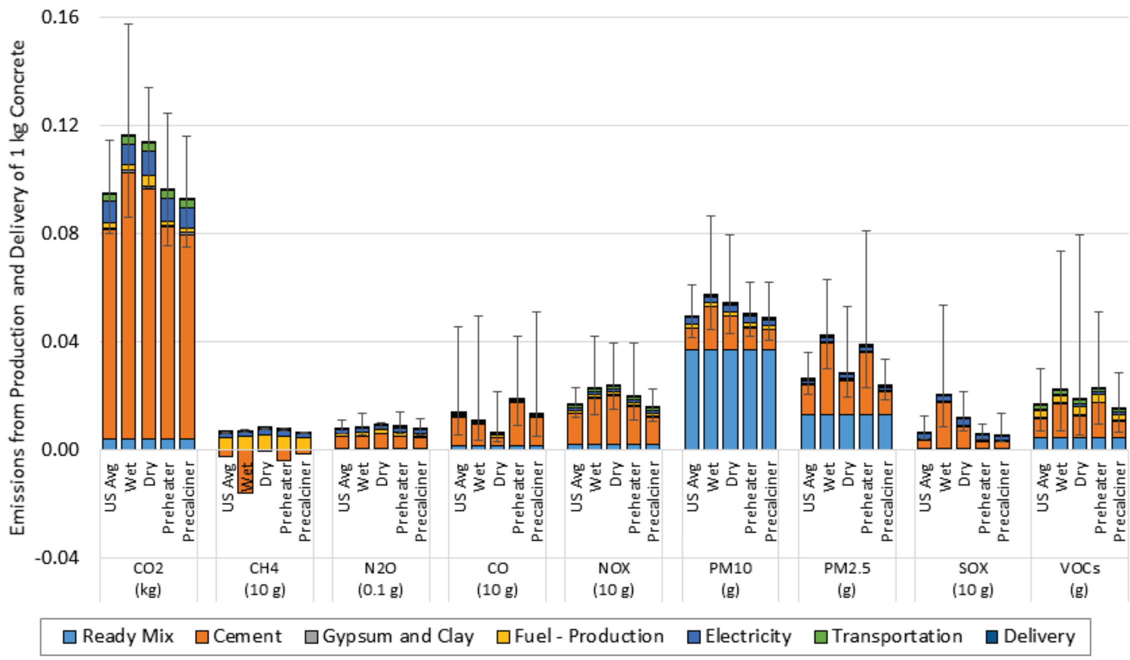


Figure 14. Greenhouse gas and criteria air pollutant emissions for concrete production for US average concrete and by technology. The vertical stacks indicate the contribution of each life cycle stage to the total emissions associated with the production and delivery of one kilogram of concrete; error bars depict the tenth and ninetieth percentile values based on Monte Carlo simulation of emissions using distributions estimated based on the variability in the underlying, facility-specific emissions data (*Source*: Life cycle energy and environmental impacts of concrete: GREET update, T. Hawkins et al., May 19, 2020)

2.2.7.1 Cement Clinker Chemistry

Cementitious materials have been used for construction applications for thousands of years. However, the current formulation of cement has been in existence only since the early 1800s (Biernacki et al. 2017). During this time, there have been few developments in the admixtures of cements. Cement usage worldwide was about 4.1 billion tons in 2019.[50] With the projected growth in population and increased need for infrastructure, cement usage is projected to be as high as more than 5 billon tons per year by 2050.[51] Given decreasing raw material supplies and CO_2 release as a result of cement production, it is imperative that new, more-efficient cement manufacturing approaches be developed.

Raw materials required to produce clinker are shown in Figure 15 (IEA 2010). The key components for clinker are calcium carbonate, silica, and alumina. Cement formulation is produced by mixing clinker with gypsum and admixtures. Subsequently, concrete used in construction is produced by adding aggregates and water in appropriate proportions. The cement proportion is about 20% of the weight in concrete.

[50] https://www.statista.com/statistics/219343/cement-production-worldwide.
[51] https://www.iea.org/news/cement-technology-roadmap-plots-path-to-cutting-co2-emissions-24-by-2050.

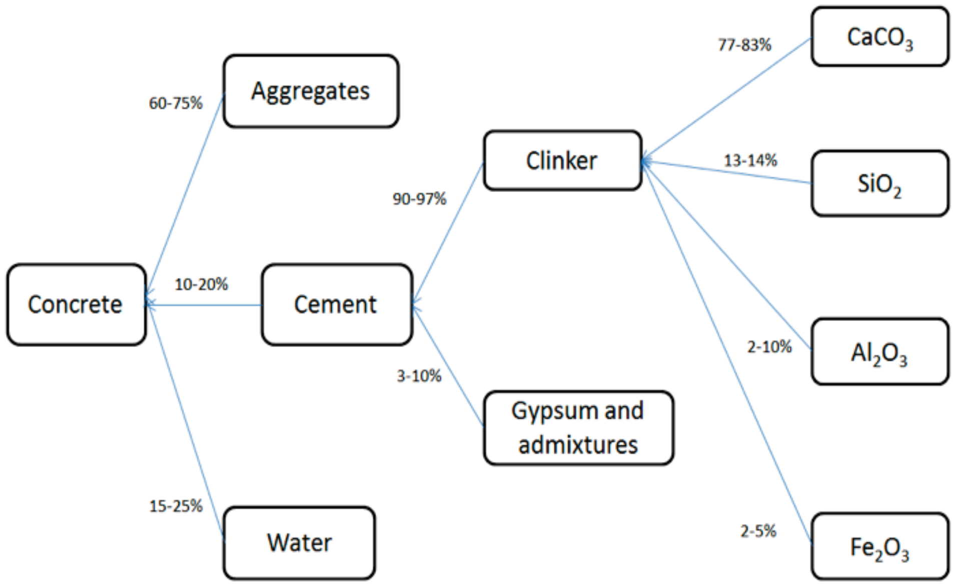


Figure 15. Typical compositions of clinker, cement, and concrete. (*Source*: C. Bonnet, S. Carcanague, E. Hache, A. Jabberi, G. Seck, and M. Simoën, *The Impact of Future Generation on Cement Demand: An Assessment based on Climate Scenarios*. Working Paper 2019-2, Energies nouvelles, Institue de Relations Internationales and Strategiques, and French National Research Agency.)

2.2.7.2 Cement Manufacturing Process

Figure 16 shows the various manufacturing processes for cement production (Huntzinger and Eatmon 2009) and the associated energy needs per ton of the product produced (IRENA 2018). Electrical energy is used for raw material extraction and crushing operations. However, thermal energy is used for the clinkering process in which calcium carbonate is heated up to 1,450°C and decomposed to produce calcium oxide and CO_2. To minimize energy consumption for manufacturing cement, more-efficient extraction and crushing processes are needed.

The second challenge that cement manufacturing faces is the release of CO_2 to the environment. For every ton of cement produced, 750 kg of CO_2 is released to the environment. In this regard, new pathways are needed for capture and sequestration of CO_2 resulting from the clinkering process.

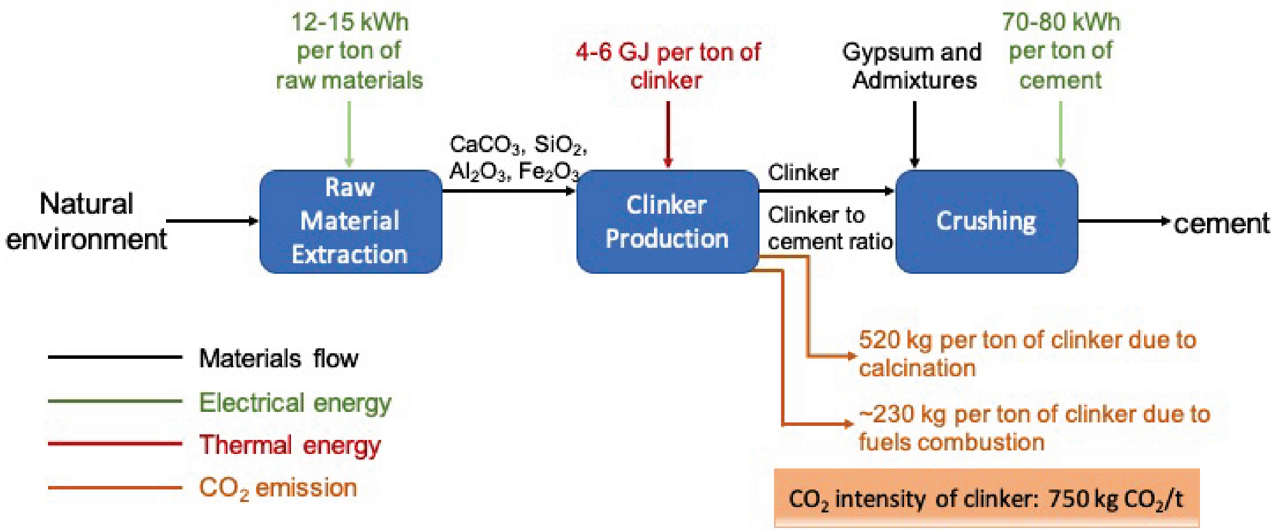


Figure 16. Processing steps and associated energy needs for cement manufacturing. (*Source*: C. Bonnet, S. Carcanague, E. Hache, A. Jabberi, G. Seck, and M. Simoën, *The Impact of Future Generation on Cement Demand: An Assessment Based on Climate Scenarios*, working paper 2019-2, Energies nouvelles, Institue de Relations Internationales and Strategiques, and French National Research Agency)

2.2.7.3 Cement Manufacturing R&D Challenges

2.2.7.3.1 Materials Modeling and Machine Learning

Fundamental mechanisms of the cementation process still are not fully understood. Cement structural modeling at various length and time scales is needed to predict the structure and properties of cements, which is a possibility with the HPC capabilities now available. ML of cementitious materials is already being used for predicting the performance of the admixtures (Biernacki et al. 2017). Using the imaging and x-ray diffraction data of the cement powder and admixtures, the data have been used to predict the strength of the set cement.

2.2.7.3.2 Efficient Cement Processing

Efficient processes for cement production are needed. These processes include improved thermal and electrical efficiencies of ore extraction, quarrying, crushing, clinkering, and transportation of materials. Linking cement production with other industrial pyroprocesses can result in improved manufacturing efficiencies and reduced costs.

2.2.7.3.3 Low-Carbon Cementation Solutions

To minimize the use of cement, alternate chemistries such as Mg-, Al-, and Fe-based cements are needed as a substitute in concrete. Supplementary cementitious materials (SCMs) such as fly ash or silica do not require thermal processing and do not release CO_2 into the environment. Therefore, SCMs can be used to partially replace cement and minimize CO_2 intensity for concrete production. Basic research into identifying and developing new SCMs is recommended.

2.2.7.3.4 Carbon Capture and Storage

Breakthroughs are needed in methods for carbon capture and storage (CCS). Most cement manufacturing plants are relatively small and operate in dispersed locations. Inexpensive and efficient CCS technologies are needed at various stages of cement production. CCS technologies are being developed as a part of other industries and could be applicable to cement manufacturing. However, CO_2 can be recycled back into cement and accelerate its setting behavior. Basic understanding of such phenomena and translating it into the manufacturing cycle is needed.

2.2.7.3.5 New Admixtures/Chemistries

Admixtures allow for controlling the rheology of the cement formulations, dispersants, air entrainment, and foaming. In typical usage scenarios, admixtures have a strong effect on the durability of the cement. Developing new synthetic admixtures and understanding their interactions with the cementation process will lead to new cement manufacturing approaches, use of recycled materials, and high-performance cements.

2.2.7.3.6 New Manufacturing Approaches

By reducing waste, cement usage can be minimized. In this regard, new manufacturing techniques such as AM could be developed. Research into how to change the structural design of buildings and infrastructure to use less concrete by using precast structures can also improve efficiency.

2.2.8 Atomically Precise Manufacturing

Research areas important to atomically precise manufacturing include device and other condensed matter physics; chemical sciences, including catalysis, separations and self-assembling chemical processes (e.g., atomic layer deposition, or ALD); and biomolecular research (e.g., DNA origami-based methods).

2.2.8.1 Molecular Self-Assembly

Molecular self-assembly relies on chemical and inter-molecular interactions for atomically precise positioning of nanoscale objects. As a principle for creating objects and patterns with high spatial resolution, self-assembly belongs to the class of bottom-up strategies that are alternatives to many conventional top-down technologies, such as CNC machining, 3D printing, lithographic patterning, and direct laser write processing. The key feature and unique advantage of bottom-up approaches is that they can achieve atomic precision due to the spatial scale of the intrinsic driving forces rather than by downscaling larger designs via some action of external stimuli or fields. Principles of molecular self-assembly have drawn considerable attention since the early 1970s, when charge transport through monolayers of organic molecules was demonstrated in the pioneering studies by Hans Kuhn's team (Mann and Kuhn 1971). In subsequent years, the idea of using individual molecules as a basis for future electronic devices led to the emergence of molecular electronics (Ratner 2013) and, in turn, accelerated implementation of various molecular self-assembled systems.

Ordering of amphiphilic molecules in close-packed molecular layers is one of the historically earliest and simplest examples of molecular self-assembly that can be used to create coatings and membranes with atomically precise thicknesses and well-defined molecular arrangements. Similar to fatty acid salts and lipids, a great variety of newly synthesized amphiphilic molecules that consist of hydrophobic chains and hydrophilic head groups can self-assemble into ordered monolayers at the gas-liquid or liquid-liquid interfaces. Although self-assembly of amphiphilic molecules into ordered mono- and multilayers relies on the action of relatively weak van der Waals forces, more robust systems with many potential applications can be realized by covalent self-assembly when chemically active molecular moieties are present. For instance, covalent self-assembly of thiols and silanes is a promising approach in creating ultrathin water-repellant and anticorrosion coatings.

Starting with synthesis of macromolecules that exhibit a combination of non-covalent and covalent interactions, a great variety of complex self-assembled systems can be implemented. Macrocycles with molecular cavities, such as cyclodextrins, calixarenes, and rotaxanes, can be used to self-assemble systems with molecular-scale porosity and highly specific molecular recognition functionalities that are promising for emerging chemical separation and sensing applications.

Systems with additional functionalities can be manufactured by taking advantage of electrostatic interactions between differently charged ionized groups. In particular, the layer-by-layer-electrostatic assembly pioneered by Decher (Decher 1997) subsequently was extended to a wide range of organic-inorganic nanocomposites.

Continued advances in macromolecular synthesis have led to many remarkable achievements in the area of molecular self-assembly. Some of the recent notable examples in this direction include artificial molecular motors, a nanocar demonstration (Peplow 2015), and a biomimetic protein-building molecular robot (Peplow 2013).

2.2.8.2 Biological Self-Assembly

Nature offers many examples of complex architectures that combine atomic precision with functionalities not yet possible in any synthetic systems. Biological self-assembly is centered around the idea of using biologically inspired and biologically derived building blocks and mimicking these complex architectures with the ultimate goal of creating nanomaterials and nanomachines capable of self-replicating, self-healing, and functioning in analogy to living organisms. Major efforts in the area of biological self-assembly have focused on using DNA molecules as building blocks or templates for assembly of nanomaterials with complex 3D shapes (Wei, Dai, and Yin 2012). The remarkable ability of DNA molecules to interact specifically through Watson–Crick base pairing has led to the emergence of the technological strategy commonly referred to as “DNA origami.” This strategy is extremely versatile and prolific for creating various 2D and 3D shapes with atomic-level precision (Castro et al. 2011). Promising breakthroughs have been demonstrated by combining the DNA origami strategy with various postprocessing techniques to create nanoscale shapes and patterns in inorganic materials (Surwade et al. 2013).

More recent advances in this field indicate that self-assembly of either natural or biologically derived DNA molecules is a very promising pathway toward new materials with applications in biosensing, drug delivery, and emerging biomedical devices (Ke, Castro, and Choi 2018). By taking advantage of biological molecular assembly, a reprogrammable molecular computer has also been demonstrated (Woods et al. 2019). In addition to the DNA origami, biological self-assembly encompasses a range of many other promising strategies applicable to creating complex structures with close to atomic precision. For instance, cell wall silicification in marine diatom microorganisms can be used as a strategy for creating inorganic nanomaterials with intricate nanoscale architectures (Dolatabadi and de la Guardia 2011). Biologically assembled systems mimicking photosynthesis are promising for energy applications, and kinesin molecular motors can be used to drive static and dynamic self-assembly (Hess 2006).

2.2.8.3 Scanning Probe and Beam Methods

Scanning probe (SP) and scanning beam (SB) methods enable the removal, addition, and transformation of materials with atomic precision and have tremendous potential in manufacturing. These methods rely on positioning an atomically sharp tip (SP) or a focused beam of charged particles (SB) at precise locations on a surface to create a desired pattern by manipulating the surface atoms. In most cases, the probe or beam position can be controlled with atomic precision, but the resulting feature size is limited by the dimensions of the probe or beam, and the size of the physically or chemically affected region. Consequently, these techniques are generally considered atomic-scale or close-to-atomic-scale manufacturing (ACSM). These techniques generally create patterns serially, similar to a pen on paper. Transitioning these techniques broadly to manufacturing will require developing instruments with arrays of probes or beams for parallel processing. All these methods originated as characterization tools for atomic-scale imaging, but researchers quickly harnessed them for synthesis.

Current AMO manufacturing research on improving myriad types of scanning probe microscopes[52] could greatly accelerate advances in scattering and instrumentation science, which in turn, through advances in understanding enabled by accelerators using beams of x-rays, neutrons, and electrons, could enhance progress in atomically precise manufacturing.

Scanning probe methods began 30 years ago with scanning tunneling microscopy (STM). In the STM, the tunneling current varies exponentially with the tip-sample distance, and a feedback loop based on this current enables atomically resolved imaging. Furthermore, by controlling the tip-sample voltage, atoms can be added to or removed from the surface. The classic example of SP synthesis was performed in 1990 when IBM researchers manipulated xenon atoms on a single-crystal Ni substrate to form the company logo (Eigler and Schweizer 1990). A more recent example of nanoscale manufacturing using SP methods is hydrogen depassivation lithography (HDL), in which a voltage applied to an STM tip removes surface Si-H with atomic-scale resolution. Hydrogen removal creates reactive dangling bonds that allow for subsequent chemical reactions at these sites to build a device (Randall et al. 2018). This method has been used recently to create nanoscale electronic devices including wires, transistors, qubits, and quantum dots. One potential issue for manufacturing is tip crashing, but this problem was recently overcome using a tip-tuning method whereby the proportional integral gains are continuously updated (Tajaddodianfar, Moheimani, Owen, and Randall 2018). In addition to STM, atomic force microscopy (AFM) methods for ACSM are under development. For instance, AFM can also be used to create nanoscale patterns on Si surfaces with atomic-layer thickness precision using shear-induced mechanochemical reactions. Dip pen nanolithography (DPN) is another scanning probe method for ACSM in which an AFM tip is used to spread an “ink” on a substrate surface with ~10 nm resolution. The ink is typically a self-assembled monolayer solution that reacts to and chemically bonds to the surface upon contact. Massively parallel DPN has been demonstrated with 55,000 pens (Salaita 2006).

In scanning beam methods, a focused beam of ions or electrons is rastered across a surface using electric fields to create patterns through physical or chemical processes. For instance, ion beam lithography typically uses a focused ion beam of liquid metal gallium that can be used to remove material through sputtering with ~10 nm resolution. Replacing the gallium with helium ions improves the resolution to ~1 nm, and neon ions have a theoretical resolution of about one atomic bond length (~0.25 nm). Similarly, electron beams can be used to remove or chemically modify material at the ~1 nm scale using

[52] For descriptions of examples of AMO’s atomically precise manufacturing projects, see Nanoday Fact Sheets at: https://www.energy.gov/eere/amo/nanotechnology-day-2019.

electron beam lithography (EBL). EBL typically is used to pattern a polymer resist layer that serves as a mask in subsequent etching steps that transfer the pattern into the underlying substrate. However, focused electron beams also are used to directly pattern materials such as transition-metal dichalcogenides, including molybdenum disulfide. Scanning beam methods can be used to deposit materials on a surface by introducing a vapor containing the element of interest while the beam is scanned. For instance, Pt features can be prepared using (methylcyclopentadienyl)trimethyl Pt vapor during focused ion beam exposure with gallium ions. One example of parallel processing for scanning beam manufacturing is the reflective EBL tool developed by KLA-Tencor (Petric et al. 2011). This device uses an array of micron-scale electrostatic lenses to pixelate an intense electron beam into 10^6 individual "beamlets" that can be controlled individually. This approach has the potential to advance EBL from prototyping to manufacturing.

2.2.8.4 Atomic Layer Processing

Atomic layer processing refers to a collection of related methods for adding and removing thin-film atomic layers on a substrate surface. Contrary to many thin-film methods, atomic layer processing methods are intrinsically scalable because they use chemical vapors that react on a substrate surface in a self-terminating fashion. Consequently, an atomic layer process developed on small substrates in the lab can readily be applied to manufacturing on large area substrates or large batches of parts. The most well-known atomic layer process is ALD. ALD uses a pair of reactive chemical vapors (A and B) to grow inorganic materials. Each ALD cycle (AB) deposits approximately one atomic layer; repeating the cycles (ABABAB . . .) grows the film to any desired thickness in a digital fashion. The ALD precursors react only with specific surface functional groups. Once these groups are consumed, the growth terminates even if excess precursor is supplied. Perhaps the most useful attribute of ALD for energy applications is the ability to coat nonplanar and porous materials with high precision. For instance, ALD coatings have been demonstrated to improve the performance of mesoporous catalysts, nanoporous and nanowire solar cells, and Li battery electrode powders. A diverse range of materials can be prepared by ALD, including oxides, nitrides, and metals, and this allows the technique to benefit a wide range of industries. For example, ALD is used commercially in the high-volume production of consumer electronics, crystalline Si solar cells, and displays. ALD is nearing manufacturing readiness in other areas, such as LIBs in which ALD coatings are deposited on cathode powders at the tons-per-day scale. Some of the emerging applications for ALD require nontraditional coating equipment for cost-effective manufacturing, such as R2R, fluidized bed, and spatial ALD tools. Although research-scale equipment is commercially available to implement these nontraditional ALD strategies, pilot- and manufacturing-scale tools are either proprietary or still under development. Molecular layer deposition (MLD) is a related thin-film coating method that also uses a pair of reactive chemical vapors to grow materials in an atomic layer-by-layer fashion. As the name implies, the coatings produced are either polymers or hybrid organic-inorganic materials. Compared with their inorganic counterparts, MLD films typically are softer and more compliant, and this makes MLD films attractive in applications requiring flexibility, such as moisture barrier layers on organic LED displays. In fact, some of the best-performing moisture barriers consist of alternating ALD and MLD layers that combine the flexibility of the polymer layers with the vapor impermeability of inorganic layers.

Recently, there has been intense interest and rapid development in atomic layer etching (ALE) (Nam and Kim 2020). This technique uses alternating exposures to chemical vapors or energized particles to remove material with atomic layer precision. This capability is vital to the continued downscaling of microelectronic components such as field effect transistors (FETs); 3D fin-FETs and gate-all-around FETs are being developed. Etching is one of the core technologies underpinning the microelectronics industry, in which plasma-based methods are principally employed for removing material from a masked substrate to create patterned thin-film layers. Consequently, the first examples of ALE used a plasma step in at least one of the exposures of the AB cycles. More recently, ALE methods have emerged that use molecular precursors for both the A and B exposures. For instance, Al_2O_3 thin films can be removed in an atomic layer-by-layer fashion using alternating exposures to trimethyl aluminum and hydrogen fluoride. The benefit of molecular precursors compared with plasma species is that they can diffuse deeply into a porous substrate or between closely spaced parts in a coating batch to access the necessary reactive functional groups to complete the etching chemistry. The greater "throwing power" of molecular precursors compared with plasma species is highly desirable for the energy applications discussed above that rely on porous or high-surface-area supports. One hypothetical example combining ALD and ALE for manufacturing is PEMFC catalysts. These catalysts are typically composed of Pt supported on carbon. ALD Al_2O_3 has been shown to stabilize supported Pt particles, but inevitable growth of the insulating Al_2O_3 on the carbon substrate reduces performance. Because Al_2O_3 ALD is inhibited on carbon surfaces, however, Al_2O_3 ALD on Pt and carbon, followed by Al_2O_3 ALE to remove the thinner coating on the carbon, might be a route to manufacture stabilized proton exchange membrane catalysts. Finally, note that the molecular-layer analog of ALE, molecular layer etching, recently has been demonstrated, thus completing the toolbox of atomic layer processing methods.

3 Resilient and Sustainable Manufacturing

The US Environmental Protection Agency defines sustainable manufacturing as “the creation of manufactured products through economically sound processes that minimize negative environmental impacts while conserving energy and natural resources. Sustainable manufacturing also enhances employee, community and product safety.”[53]

Resilient and sustainable manufacturing includes by-design integration of product and process design. It also requires attention to operational criteria of safety, business models, and economics across the entire life cycle of a product, including the end-of-intended-use phases. Sustainable product design includes attention to characteristics—in the final product—that enable benign end-of-life behavior, flexibility in repair, remanufacturing, and circularity. Sustainable process design requires attention to energy, water, and use of natural resources; minimization and optimization of processes regarding emissions and externalities; carbon balances; and waste generation. Sustainable operational criteria include workplace safety and ergonomics in automation, for example, as well as the engineering control of potentially hazardous materials. Additionally, operational criteria include the adoption of new business models that enable the implementation of circularity in society through the facilitation of recovery, recycle, repair, and the manufacturers’ ownership of the full product cycle (including different societal use models).

Life cycle assessment and material and energy flow analysis tools are critical to the design of sustainable manufacturing processes and materials. These tools need to become fully integrated with engineering models to deliver robust analytical outputs of alternate design and production scenarios in real time. Also, the life cycle and materials flow assessment should incorporate full environmental analysis from resource extraction to post-consumer impacts. Risk-assessment methods related to a product’s environmental hazards, mobility, likely use and reuse, and disposition need to be reconceived to become tools in the design phase.

Most current efforts in manufacturing are related to achieving sustainability by reducing environmental impacts via, for example, resource conservation and reduced waste emissions. Limited information is available, however, about the links among manufacturing (e.g., product and process sustainability, water and energy consumption, raw materials availability, waste generation), social factors (e.g., economics, jobs, wealth), and the environment (e.g., biotic, aquatic, soil, air). A framework needs to be developed that includes the flows of information, materials, energy, economics, jobs, and waste, as well as the characterization of the interdependencies throughout pre-manufacturing, manufacturing, and post-manufacturing activities. A better understanding of manufacturing processes to adapt and cope with disturbance is also crucial. Attributes of production system resiliency, persistence, adaptability, agility, redundancy, learning capability, and decentralization in the context of Industry 4.0 also should be considered during the framework development.

A comprehensive systems approach linking industrial, ecological, and social domains is crucial for effective decision making with regard to resilient and sustainable manufacturing. This requires development of frameworks and models aimed at guiding principles that describe the dynamic (mostly far from steady states and nonlinear) and adaptive behavior of complex systems and their resilience in the face of disruptions at the operational level. This framework will provide a foundation for redesigning and reconfiguring conventional product or process technologies, and therefore the development of

[53] https://archive.epa.gov/sustainablemanufacturing/web/html.

innovative technologies transforming current manufacturing practices. Challenges to achieve this framework require a comprehensive and consistent definition of system boundaries.

3.1 Integrated Manufacturing Processes

3.1.1 Hybrid or Integrated Manufacturing Processes

Hybrid manufacturing processes combine the best features of conventional subtractive machining with AM (3D printing) and provide new opportunities and applications that cannot be done technically and economically by one manufacturing process alone. Hybrid manufacturing offers many benefits, such as adding multiple materials to the same part, building and repairing damaged parts, applying expensive materials where needed, 3D printing, and finishing the part in a single setup. However, there are no standards or protocols that guide the manufacturing industry. Development of a process planning, simulation, and toolbox is crucial for the broad applications of hybrid manufacturing processes. This approach also will help visualization of fabrication before manufacturing, thus reducing errors and increasing the precision and accuracy.

3.1.2 Components, Modular Assemblies

Scalable modular processes are essential for manufacturing industries and provide opportunities for high-margin growth in decentralized, flexible production facilities because of their economic and safety benefits and fast response to changes in demand. The links between modularization and PI also should be further evaluated to determine challenges and future directions. The roles/applications of sensors and microsensor systems should be considered to provide flexibility to manufacturing, real-time controls, and increased precision.

3.1.3 Toward a Circular Economy

Central to sustainable and resilient manufacturing, the concept of a circular (or closed-loop) economy has risen in the last decade as an alternative to the “take-make-discard” linear approach that has been the staple of industrial development since the early days of industrialization. Dwindling resources and environmental challenges make concepts of industrial ecology not only appealing but also necessary for competitiveness and sustainable economic growth. Although full thermodynamic circularity cannot be achieved, efforts toward efficient resource use through multiple cycles of use-repair-deconstruction-remanufacturing and reuse offer the potential for significant resource sparing, energy efficiency, and minimization of waste dispersion in the environment.

Though the concept is simple, implementation is not. The circular economy concept has all the hallmarks of a complex system problem, with critical success factors relying on technology and innovation, as well as—equally important—alternative business models, behavior modification, and social acceptance.

The path toward a circular approach encompasses different levels of recovery of the embedded energy, value, and resources in manufactured products. Some strategies are shown in Table 5, ordered by decreased intensity of recovery of added value. Importantly, the potential for each of the strategies is largely dependent on the inclusion of the correct product properties in the integrated design phase.

Table 5. List of Potential Strategies in Decreased Intensity of Recovery of Added Value

Strategies	Desirable Product/Process Properties
Direct reuse	Robustness, repair potential, flexibility toward multiple types of uses, as applicable, are characteristics that need to be included in product design.
Remanufacturing	Design must include criteria that facilitate disassembling and reassembling, access to inspection points, use of durable and standardized materials, and must implement identifiers that can be easily traced and do not fade with use (bar codes or other).
Upcycling	The ability for a product to be transformed into another product of higher value requires that the product can be deconstructed to its building blocks or discrete parts and reorganized into higher-value products. Energy intensity and cost of this process must be accounted for.
Recycling	The ability of a product to be recycled into another of equal or lower value requires techniques for identification, sorting, and processing that are more carbon efficient and energy efficient than manufacturing a new product.
Energy recovery	This is not usually considered a true circular economy strategy as it does not maintain the intrinsic value of a product besides using its energy.
Disposal	In a circular economy context, disposal is an undesired strategy to be minimized.

The needs for the full development of a circular manufacturing economy can be summarized as follows:

- Robust multiobjective optimization algorithms need to be developed to import dynamic life cycle analyses of carbon, energy, environmental stressors, health, cost, and social acceptance into decision support systems that include different sustainability goals.
- Research should continue in the development of sensors, sensor systems, and spectroscopic techniques to guide material sorting and identification, and for the understanding and advancing the effectiveness of materials separation.
- Metallurgical and chemical sciences should develop trigger molecules or other components that allow the on-demand deconstruction of complex materials. This is particularly relevant for composite materials that currently are difficult to separate and repurpose. Advances that allow the design of post-use product separations (such as depolymerization, alloy separation, delamination, devulcanization, and de-coating) will be critical for the success of a circular economy.
- Atomic-level and molecular-level recycling will need to separate and reuse chemical building blocks and elements, such as metals.
- Manufacturing systems will need to be designed for modularity and for plug-and-play standardized components that will enable flexibility in production.
- Use of Industry 4.0 connectivity and robotics will need to enable precise and flexible manufacturing systems.
- Research challenges exist in assessing and overcoming concerns about impurities, and in building systems that have higher tolerance for impurities without sacrificing quality and safety (fit for purpose optimization).
- Use of renewable or harvested energy sources should be holistically designed into manufacturing systems.

3.2 Reduced Carbon Emissions in Manufacturing

Manufacturing-related research to reduce carbon emissions is informed by basic research in chemical transformations, including catalysis, separation, and geosciences, as well as computational and theoretical chemistry, photochemistry, biochemistry, solar photochemistry photosynthetic systems, and physical biosciences.

3.2.1 Manufacturing Energy Savings Potential

Energy efficiency improvements in manufacturing processes provide a straightforward pathway to reducing carbon emissions. DOE AMO has performed "bandwidth" studies tailored to 9 of the 15 individual manufacturing industries, representing 86% of on-site energy use and 89% of on-site CO_2 emissions, to ascertain the potential for energy savings. Although the bandwidth study analyses used 2010 Manufacturing Energy Consumption Survey data, the results are scalable. In Table 6, estimates of potential on-site energy savings (as percentages of the 2010 base year, applied to 2014 consumption) are shown for 15 US manufacturing industries. "Current" savings represent the best available technologies today (the difference between current typical technology and state-of-the-art best practices), whereas "R&D" represents what might be accomplished with state-of-the-art technology R&D (the difference between current best practices and the practical minimum energy required if the applied R&D technologies under development worldwide were deployed).

Table 6. Manufacturing Sector Estimated Energy Savings

Manufacturing Industry	Savings Opportunity (2010) (%)			Potential On-Site Energy Savings (2014) (Tbtu)		
	Current	R&D	Remainder	Current	R&D	Remainder
Alumina and Aluminum[a]	26%	23%	51%	63	55	124
Cement[b]	25%	3%	72%	75	9	215
Chemicals[c]	24%	38%	38%	839	1,337	1,351
Computers, Electronics and Electrical Equipment		N/A			N/A	
Fabricated Metals		N/A			N/A	
Food and Beverage[d]	27%	11%	62%	329	134	746
Forest Products (Pulp and Paper)[e]	22%	7%	71%	545	172	1756
Foundries		N/A			N/A	
Glass[f]	31%	8%	61%	64	17	127
Iron and Steel[g]	24%	15%	61%	260	163	661
Machinery	N/A	N/A				
Petroleum Refining[h]	13%	25%	62%	446	842	2,085
Plastics and Rubber Products[i]	32%	8%	60%	93	24	177
Textiles		N/A			N/A	
Transportation Equipment		N/A			N/A	
Subtotal				**2,714**	**2,753**	**7,241**

Table 6. Manufacturing Sector Estimated Energy Savings (continued)

Manufacturing Industry	Savings Opportunity (2010) (%)			Potential On-Site Energy Savings (2014) (Tbtu)		
	Current	R&D	Remainder	Current	R&D	Remainder
All Manufacturing (extrapolation)				**3,152**	**3,197**	**8,410**
% of All Manufacturing				**21%**	**22%**	**57%**

[a] https://www.energy.gov/sites/prod/files/2019/05/f62/Aluminum_bandwidth_study_2017.pdf.
[b] https://www.energy.gov/sites/prod/files/2017/12/f46/Cement_bandwidth_study_2017.pdf.
[c] https://www.energy.gov/sites/prod/files/2015/08/f26/chemical_bandwidth_report.pdf.
[d] https://www.energy.gov/sites/prod/files/2019/05/f62/Food_and_beverage_bandwidth_study_2017.pdf.
[e] https://www.energy.gov/sites/prod/files/2015/08/f26/pulp_and_paper_bandwidth_report.pdf.
[f] https://www.energy.gov/sites/prod/files/2019/05/f62/Glass_bandwidth_study_2017.pdf.
[g] https://www.energy.gov/sites/prod/files/2015/08/f26/iron_and_steel_bandwidth_report_0.pdf.
[h] https://www.energy.gov/sites/prod/files/2015/08/f26/petroleum_refining_bandwidth_report.pdf.
[i] https://www.energy.gov/sites/prod/files/2017/12/f46/Plastics_and_rubber_bandwidth_study_2017.pdf.

3.2.2 Reducing Greenhouse Gas Emissions

To go beyond the "practical minimums" identified in the bandwidth studies, the GHG emissions associated with the remaining 38% to 72% of energy consumption in each subsector must be eliminated through technology innovation and the identification of additional R&D opportunities. Examples include the following:

- Further increases in energy efficiency;
- Substitution of net zero- or negative-carbon fuels (such as hydrogen or biomass);
- Substitution of fuel combustion with net zero-carbon electricity;
- Substitution of existing feedstocks, processes, or products with those having lower net life cycle GHG emissions; and
- Capture and sequestration (or utilization) of CO_2 and other GHG (CH_4, N_2O) emissions from manufacturing processes.

3.2.2.1 Substitution of Fuels and Feedstocks

To reduce emissions for energy, electrification becomes critical as it can remove almost half of GHG emissions[54] through the availability of low-cost renewable electrons mainly through solar, wind, hydropower, and nuclear power. There are additional opportunities in the manufacturing sector focusing principally chemicals and fuels. Reducing emissions requires new technologies and processes that can replace traditional feedstocks with more sustainable feedstocks. This electrochemical refinery concept has been discussed at both the academic (Seh et al. 2017) and industrial levels.[55] The key is to use electrons, CO_2, and water to produce chemical feedstocks or perhaps fuels. Such initiatives have been studied in places like the Joint Center for Artificial Photosynthesis[56] and in practice (Opus 12 and Siemens)[57] by reducing CO_2 to CO. Improvements are necessary, however, to understand how to scale

[54] https://www.otherlab.com/blog-posts/us-energy-flow-super-sankey).
[55] https://www.shell.com/energy-and-innovation/the-energy-future/scenarios/shell-scenario-sky.html.
[56] https://www.lbl.gov/a_z_link/jcap-joint-center-for-artificial-photosynthesis/.
[57] https://www.opus-12.com/.

the technologies; there is a rich chemical space, but the exact mechanisms, interfaces, and reaction pathways are unknown (Yan, Kawamata, and Baran 2017). Finally, electrochemical technologies and pathways present a new type of scaling of processes. This is because each device is inherently small and scalable (e.g., stack and cell architectures), and thus provides new integration and colocation possibilities at the point of need compared with existing, large-scale refineries and chemical plants.

In particular, global chemical (and petrochemical) manufacturing accounts for more than 10% of global energy consumption and 30% of total industrial energy demand; the equivalent numbers for carbon footprint are 7% and 20% respectively (Brueske, Kramer, and Fisher 2015; Nimbalkar et al. 2014). The top 18 products by production volume account for 80% of energy use and 75% of GHG emissions in the industry, and the 2050 production volume is expected to increase by 200% from 2010 values (IEA 2013). The energy and carbon footprint versus production volume is illustrated in Figure 17 for the top ten products globally (Lanzafame et al. 2017; Perathoner et al. 2017). Heavy reliance on fossil fuels as both primary feedstocks and energy sources drives this footprint. Although the industry is primarily oil-centric, less than 10% of the crude oil–based raw materials go to chemical production; the rest provide energy to drive the production process (Brueske, Kramer, and Fisher 2015). Some oil-based raw materials drive endothermic reactions (e.g., for producing core intermediate chemicals such as syngas and olefins that support the bulk of current petrochemical manufacturing value chain) and separations (Lanzafame et al. 2017).

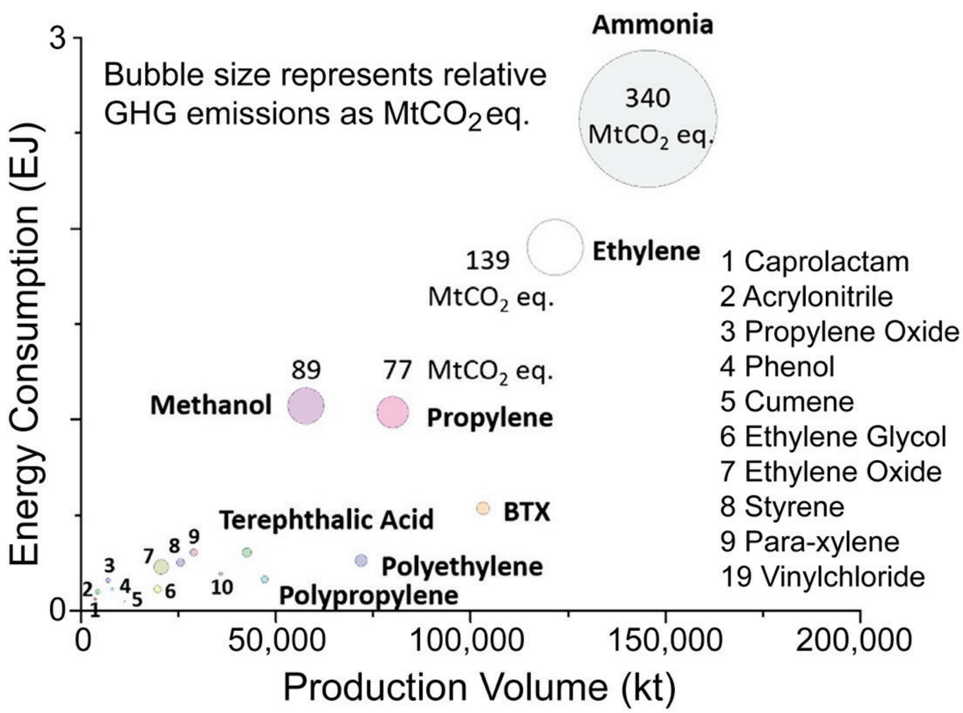


Figure 17. Global energy consumption versus production volumes and global greenhouse gas emissions (proportional to bubble size) of top-18 large-volume chemicals in 2010. (*Source*: reproduced from Perathoner et al., Looking at the future of chemical production through the European Roadmap on Science and Technology of Catalysis, the EU effort for a long-term vision, *ChemCatChem*, Wiley-VCH, © 2017 Wiley-VCH verlag GmBH & Co. KGaA, Weinheim)

Therefore, the challenge of drastically reducing overall emissions of the chemical sector must consider the strategy for providing energy to drive chemical transformations, going beyond traditional thermal energy to include alternative energy sources such as photons, electrons, and other forms of radiation (including direct solar-thermal). Another option is processes that store renewable energy in chemical vectors, from light gases such as hydrogen to high-value chemical raw materials, including olefins that form the staple feedstock for most chemical production. Additionally, these approaches can leverage process configurations that use renewable energy sources to simultaneously reduce process steps and increase process energy intensity (Lanzafame et al. 2017; Navarrete et al. 2017). Both solutions can leverage current energy, transport, and industrial infrastructure and do not require radical modifications. These are particularly suited to providing options for long-term energy storage required to complement emerging trends in renewables penetration, the growth of microgrids, and distributed-energy resources. Another approach is to trap or use the electrons in hydrogen and subsequently use that hydrogen for transportation fuel, energy storage, or industrial processes (e.g., direct reduction of iron for steel manufacturing, biomass upgrading, or sustainable ammonia synthesis). Such ubiquitous use of hydrogen would greatly reduce carbon emissions from the various energy sectors.[58] Although slowly reaching maturation, electrolysis technology is still unproven when coupled to intermittent supplies and remains cost prohibitive. The DOE EERE H_2@SCALE and HySteel initiatives aim to use hydrogen as a key chemical intermediate and reducing agent that can be generated from renewable or green electrons.[59]

Solutions that provide efficient ways of storing and re-dispatching renewable energy in chemical bonds and driving chemical reactions with unconventional energy sources form the basis for a successful transition to a lower emission industrial economy. This transition will be sustainable when coupled with simultaneous reductions of the energy intensity and the cost of chemical production processes.

Relevant to this transition are (1) direct renewable energy technologies (defined below) that use renewable energy sources to generate platform chemicals, providing an alternative feedstock for the chemical manufacturing industry; and (2) indirect renewable energy technologies (defined below) that use electricity from renewable energy sources to generate electromagnetic waves, providing alternative energy sources (and catalytic effects) for driving chemical transformations.

3.2.2.1.1 Direct Renewable Energy Technologies

Direct renewable energy technologies are the general class of solar fuel technologies, including the use of hybrid concentrated solar-thermochemical or photochemical reactors to generate hydrogen or other platform hydrocarbons. Concentrated solar thermochemical reactors are reasonably well established at the pilot scale and have been studied for applications such as steam methane reforming, coal gasification, methane cracking, high-temperature water splitting, and CO_2 hydrogenation (Mul, Schacht, van Swaaij, and Moulijn 2012; Sheu, Mokheimer, and Ghoniem 2015; Stolarczyk, Bhattacharyya, Polavarapu, and Feldmann 2018). Challenges that provide opportunities for scientific research include difficulty in maintaining catalyst activity and selectivity spatially across the reactor and over the use phase; technology-scaling challenges; and structural stability issues that arise from thermal cycling (Navarrete et al. 2017).

Solar photochemical reactors affect the transformation of sunlight to chemical fuels through organic or inorganic pathways ranging from biomimicry with artificial synthesis of critical photosynthetic molecules to the use of reengineered transition metal catalysts that can integrate advances in photovoltaics (PVs), electrochemistry, and chemical synthesis (Barber and Tran 2013; Smestad and

[58] https://www.energy.gov/eere/fuelcells/h2scale.
[59] https://www.energy.gov/eere/fuelcells/h2scale.

Steinfeld 2012; Yan, Q et al. 2017). Many scientific challenges exist at the material, process, and system integration levels. Discovery of substitutes will reduce expensive catalytic materials and light absorbers and maintain high conversion efficiency and stable operation. At the process level, efficient pathways would enable higher-value hydrocarbons, as well as highly selective membranes for efficient product separation. At the systems-integration level, the key challenge is ensuring compatibility across different components, robust operation across varying conditions, and resilience against minor fluctuations (Chabi et al. 2017; Fountaine, Lewerenz, and Atwater et al. 2016; Lewis 2019; Sun et al. 2017; Wu, Y. et al. 2013). Connected to all the above is the requirement to directly produce higher-value chemical industry feedstock (e.g., olefins, acids, alcohols), for which recent studies show promise (Dogutan and Nocera 2019).

Science challenges for solar-fuel technologies include finding inexpensive substitutes for catalytic materials and photo-absorbers, developing efficient pathways for directly producing high-value platform chemicals, and ensuring system-level material compatibility and operational stability.

3.2.2.1.2 Indirect Energy Technologies

Indirect energy technology includes chemical transformation processes driven or catalyzed by interaction with electromagnetic waves such as microwave and plasma technology and made possible by very low cost energy sources. Using plasma to activate chemical reactions—alone, or in synergistic concert with catalytic material—represents an emerging area of research with promise for realizing this through very low cost, preferably renewable, energy (Bogaerts and Neyts 2018; Tu, Whitehead, and Nozaki 2019). Comprising a highly ionized gas teeming with excited electrons and ions and dissociated species, plasma creates a highly reactive environment for promoting otherwise thermodynamically unfavorable chemical transformations (Tu, Whitehead, and Nozaki 2019). Similarly, the potential for enhanced reaction activity and selectivity also makes microwaves a promising technology for supporting this renewable energy transition. Studies suggest that microwave interactions with catalysts and molecules accelerate reaction rates by up to two orders of magnitude by reducing activation energy and increasing catalytic activity (Zhou et al. 2016; Horikoshi and Serpone 2014). These interactions enable chemical transformations to occur at lower temperatures and higher energy efficiency compared with requirements for conventional heating. Further, these interactions have unique signatures specific to molecules or surface characteristics, allowing for greater reaction selectivity in heterogeneous systems (Zhou et al. 2016; Horikoshi and Serpone 2014).

Beyond the challenges related to optimizing the synergistic interaction between catalytic materials and plasma/microwave systems, challenges exist at the level of fundamental modeling and process design. Currently, no models are capable of fully describing the plasma-catalyst interactions necessary to capture the influence of plasma effects on surface reactions and catalyst properties and the impact of catalyst activity and surface temperature on the evolution and characteristics of the plasma (Tu, Whitehead, and Nozaki 2019). The situation is similar for microwave-assisted catalysis, particularly with respect to the role of thermal versus non-thermal microwave effects (de la Hoz, Diaz-Ortiz, and Moreno 2005; Kappe, Pieber, and Dallinger et al. 2013). Part of the challenge is the wide range in time and length scales of key processes involved. Breakthrough advances require the development of integrated multiscale or multimethod models to comprehensively represent the scale and diversity of relevant phenomena, as illustrated in Figure 18.

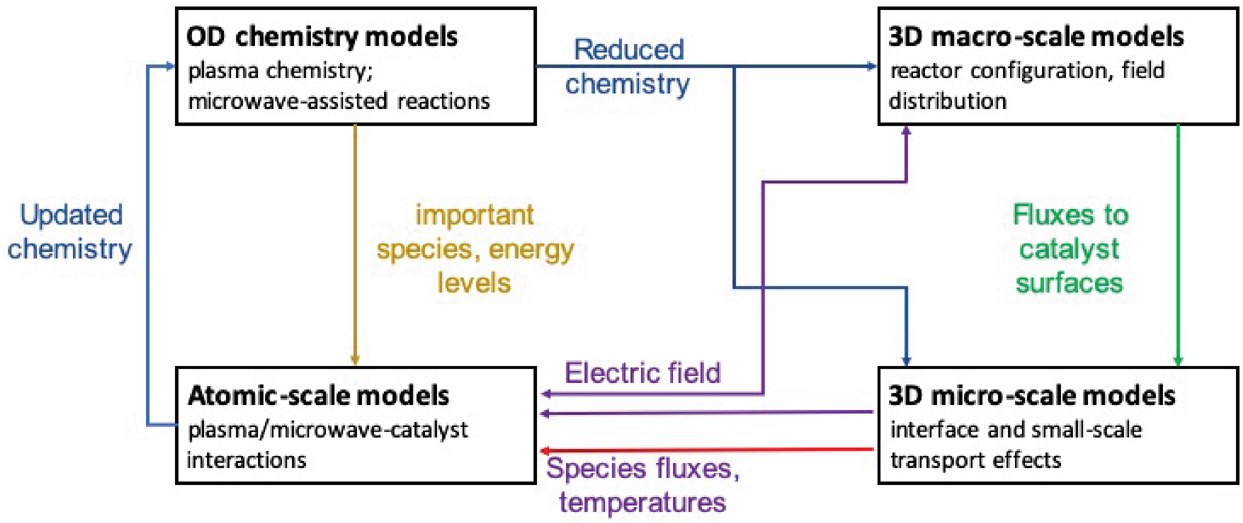


Figure 18. Generalized version of Integrated modeling approach. (*Source*: adapted by permission from CCC8: Springer, *Plasma Catalysis: Fundamentals and Applications*, Springer Series on Atomic, Optical, and Plasma Physics, by X. Tu, J. Whitehead and T. Nozaki, 2019)

At the process design level, the research opportunity remains to develop novel concepts for plasma or microwave-catalyzed, direct production of high-value chemical industry feedstock. Current attempts are promising (Li et al. 2019; Puliyalil, Jurković, Dasireddy, and Likozar 2018) but yield very low conversion (or energy) efficiencies, which limit the prospects of competing economically with conventional technology. This process requires rethinking catalyst design, focusing on synergistic processes (e.g., integrating plasma and photo-catalysis (Li et al. 2019), and rethinking reactor configurations to leverage nonequilibrium effects for improved energy and conversion efficiency, product selectivity, and separation.

Integrated multiscale, multiphysics modeling and simulation tools; new catalyst discovery; radically novel reactor designs; and optimized system integration are key ingredients to replace conventional thermal energy from hydrocarbon combustion with unconventional forms derived from renewable electricity.

3.2.2.2 Microgrids

The share of CO_2 emissions due to energy use[60] in the industrial sector was 1,462 million MT in 2018. One way to mitigate the indirect CO_2 emissions and decarbonize the manufacturing sector is using a renewable energy microgrid to generate electricity, as shown in Figure 19. Advantages of microgrids for the manufacturing sector include that they can increase reliability (through utilization of various types of energy sources), reduce waste gas emissions, and increase resilience in the case of energy supply chain disruptions. An example of a microgrid is shown in Figure 19.

[60] https://www.eia.gov/totalenergy/data/monthly/pdf/sec11_7.pdf.

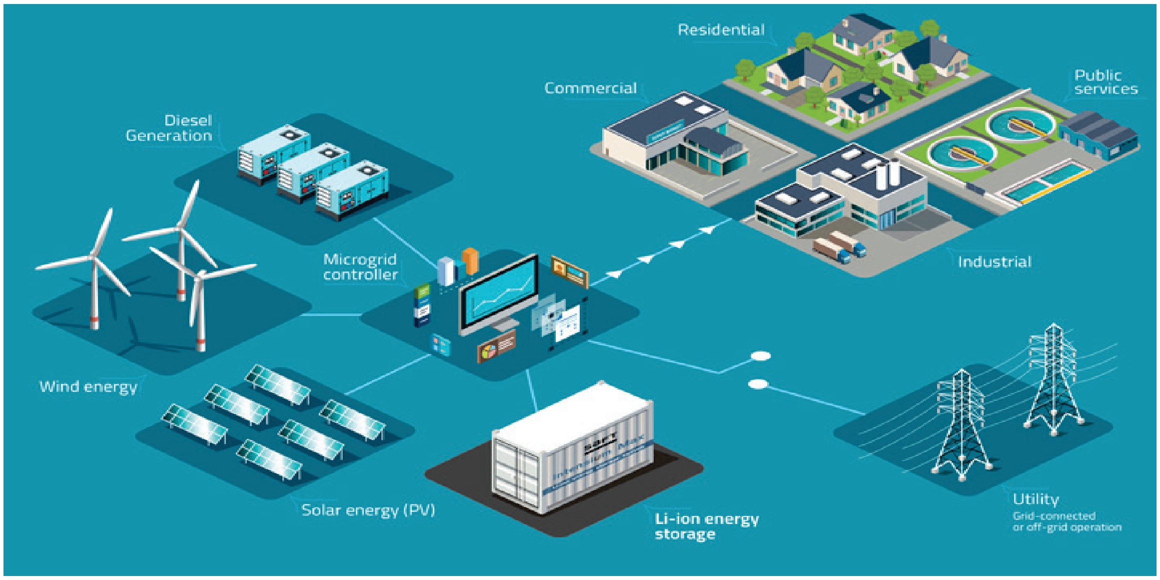


Figure 19. Schematic of a renewable energy microgrid providing power to an industrial, residential facility or commercial facility. (*Source*: Taking microgrids to the next level with Li-ion energy storage, J. McDowell, altenergymag.com, March 6, 2018. Used by permission of Saft America.)

Most manufacturers in the industry rely heavily on fossil fuel sources and purchased power to meet electricity demand. A few manufacturers, however, currently are using renewable power sources (microgrids) to meet their power demand and to reduce their own environmental footprint. For instance, Anheuser-Busch's brewery located in California generates 30% of its electricity via wind turbines and solar PV panels installed on its manufacturing site.[61] The company also recently announced that by 2022 it will meet 100% of its power demand through its 222-MW capacity solar-powered facility located in Texas.[62] Another manufacturing company—OPEX Corporation, located in New Jersey—produces automated material handling systems to meet its entire power demand through 2.77-MW capacity photovoltaic panels.[63] Few manufacturers have installed microgrids in their facilities. Broader adoption of sustainable-energy solutions would enable a global reduction in carbon emissions. Before a microgrid can be deployed on any industrial site, a detailed technoeconomic and environmental analysis should be conducted with an aim to tackle the following challenges.

- What is the optimum mix of renewable sources (e.g., capacity of solar panels, wind turbines) within a microgrid that would generate the power at the lowest cost?
- How many tons of CO_2 emissions would be offset by producing electrical power through such a microgrid instead of via a conventional electric grid?

Detailed technoeconomic and environmental impact analysis can quantify the environmental and economic impacts of a microgrid powering a manufacturing facility (see Figure 20). The results of such analyses and their comparison with corresponding economic and environmental metrics of a conventional grid would serve as a powerful decision-making tool for manufacturers, policymakers, and researchers. Such detailed analyses have been conducted by Nagapurkar and Smith (2019) in which a

[61] https://www.anheuser-busch.com/about/breweries-and-tours/fairfield-ca.html.

[62] https://www.theclimategroup.org/news/anheuser-busch-switches-renewable-electricity-us.

[63] https://www.opex.com/case-studies/achieving-net-zero-use-of-grid-energy-in-manufacturing.

renewable energy microgrid was designed for three US cities—Tucson, Arizona; Lubbock, Texas; and Dickinson, North Dakota (Nagapurkar and Smith 2019). Optimum microgrid configurations were determined, and they supplied uninterrupted power to residential communities of up to 50 homes. The cost of producing power (or levelized cost of energy) produced via the microgrid was determined to be between \$0.32/kWh and \$0.42/kWh.

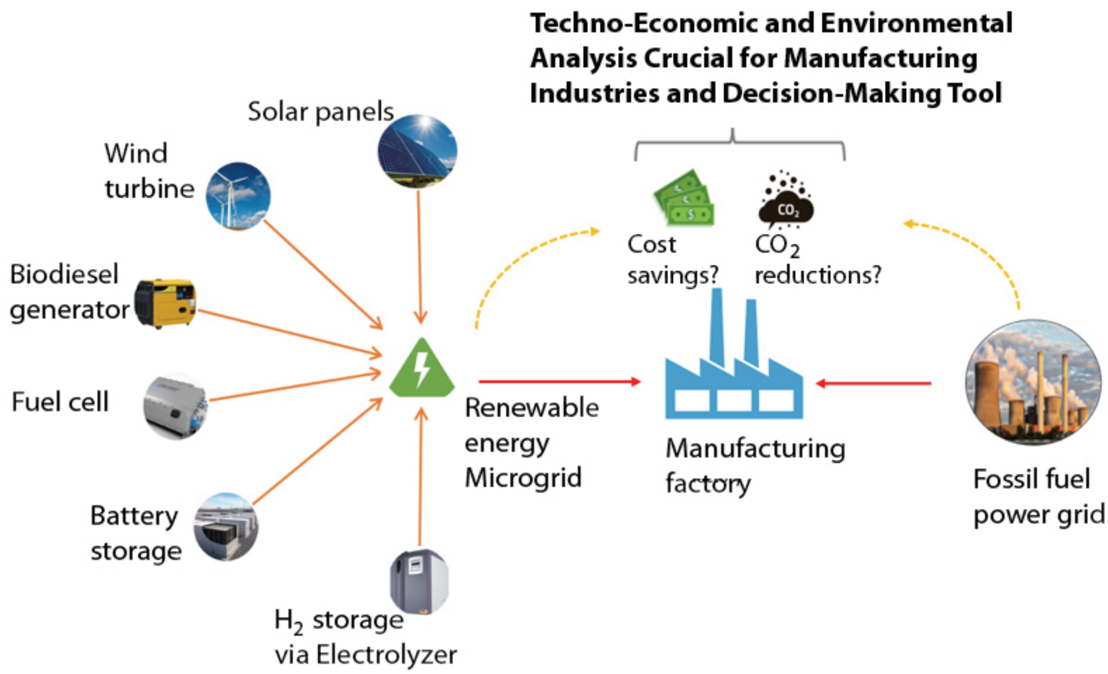


Figure 20. Using microgrids instead of conventional grid to power a factory—a comparison with regard to economic and environmental impact. (*Source*: Sujit Das, Oak Ridge National Laboratory)

Further, the analyses revealed that CO_2 emissions of the microgrid on a life cycle basis were 90% less than emissions of an equivalent conventional grid. Even though the amount of CO_2 emissions was captured in this work, their effects in terms of socioeconomic costs were not. The social cost of CO_2 in terms of economic metrics was quantified in Nagapurkar's subsequent work (Nagapurkar and Smith 2019). Such analyses conducted within the framework of economic, environmental, and social metrics applied within the context of energy used in the manufacturing sector would prove to be immensely beneficial.

3.3 Self-Configuration and Self-Optimization

Modern manufacturing is fast becoming increasingly technologically complex (Larik et al. 2020). The lack of design guidance to support self-configuration and flexibility within the architectural and engineered systems of manufacturing systems requires particular attention (Madson, Franz, Molenaar, and Kremer 2020). Flexible and self-configuration systems are key in high-mix, low-volume production. More recently, work has been done that addresses the impacts of various parameters on sustainability and its dependence on manufacturing flexibility (Ojstersek and Buchmeister 2020).

Flexible and reconfigurable manufacturing systems have already been suggested by Koren and colleagues (Koren et al. 1999) as a solution to address the needs for meeting the demands for changing products. Today's modular manufacturing systems are not typically designed for online flexibility and reconfiguration (Friedrich, Scheifele, Verl, and Lechler 2014). Reconfigurable manufacturing is important primarily because of future challenges, including volatility, uncertainty, complexity, and ambiguity (Kapoor et al. 2020). A flexible modular-production manufacturing system can support the trend toward individually produced products (Friedrich, Scheifele, Verl, and Lechler 2014).

Modular systems are crucial to customization and configuration and can provide an approach to implement functional requirements using minimal resources (Reuter, Kircher, and Verl 2010), as well as the possibility to optimize the operational capacity of the whole system by adding modules to slow production steps (Friedrich, Scheifele, Verl, and Lechler 2014; Wang, W. and Koren 2012). Further, self-configuration requires that the manufacturing process support reconfiguration. Reconfiguration can be at the level of the manufacturing process workflow or selection of individual manufacturing processes (Madureira, Pereira, and Sousa 2011). Adaptation and monitoring are integral features of the manufacturing process and greatly enhance the ability to self-reconfigure quickly and efficiently. Additionally, a reasoning engine will be required as part of the manufacturing process in a feedback loop (Madureira, Pereira, and Sousa 2011). Apart from that, a reasoning engine must make an adaptation decision based on feedback monitoring.

3.4 Full Life Cycle Manufacturing

The Rio Declaration, developed at the United Nations Conference on Environment and Development, came up with 27 principles, with three relevant to this assessment.[64]

1. Development today must not undermine the development and environment needs of present and future generations.
2. Nations shall use the precautionary approach to protect the environment. Where there are threats of serious or irreversible damage, scientific uncertainty shall not be used to postpone cost-effective measures to prevent environmental degradation.
3. To achieve sustainable development, environmental protection shall constitute an integral part of the development process and cannot be considered in isolation from it.

A life cycle approach provides a framework for product designers and service providers to be able to consider and incorporate these principles in their design and service choices, evaluating impacts to all environmental media and across all phases of the life cycle (cradle to grave or cradle to cradle). Life cycle approaches avoid shifting problems from one life cycle stage to another, from one geographic area to another, and from one environmental medium (e.g., air quality) to another (e.g., water, land). This is a multiple criteria analysis evaluating a range of environmental impacts across the scope of the full life cycle of a product or process.[65]

From the industrial perspective, by integrating life cycle thinking in overall management and bringing product and process development in a more sustainable direction, industry can harvest the benefits of environmental friendliness, occupational health and safety, and risk and quality management, as well as develop and apply cleaner process and product options.

[64] https://www.un.org/en/development/desa/population/migration/generalassembly/docs/globalcompact/A_CONF.151_26_Vol.I_Declaration.pdf.
[65] https://www.life cycleinitiative.org/starting-life cycle-thinking/benefits/

Life cycle thinking is about going beyond the traditional focus on production site and manufacturing processes to include environmental, societal, and economic impacts of a product over its entire life cycle. The main goals of life cycle thinking are to reduce a product's resource use and emissions to the environment as well as improve its socioeconomic performance through its life cycle. This could facilitate links among the economic, societal, and environmental dimensions within an organization and through its entire value chain.

In the industrial sector, a life cycle–thinking approach means going beyond the narrower traditional focus on an enterprise's production facility. A product life cycle can begin with the extraction of raw materials from natural resources in the earth and the energy generation. Materials and energy are then part of production, packaging, distribution, use, maintenance, and eventually recycling, reuse, recovery, or final disposal. In each life cycle stage, there is the potential to reduce resource consumption and improve the performance of products.[66]

Life cycle assessments take an inventory of all resource use and all emissions generated in a process[67] (see Figure 21). This inventory then is characterized into an impact category. Although many life cycle assessments evaluate just one or two impacts (e.g., energy use, GHG emissions), a wide range of impacts can be considered. The US Environmental Protection Agency has developed an impact assessment tool (Tool for Reduction and Assessment of Chemicals and Other Environmental Impacts) that translates the life cycle inventory data to higher-level impacts (ozone depletion, global warming, acidification, eutrophication, tropospheric ozone/smog formation, ecotoxicity, human health criteria–related effects, human health cancer effects, human health noncancer effects, fossil fuel depletion, water consumption, land-use effects).[68]

As an example (presented in the DOE quadrennial technology review[69]), Hawkins and colleagues (Hawkins, Singh, Majeau-Bettez, and Strømman 2013) evaluated the life cycle impacts of different conventional and electric vehicle scenarios (see Figure 22). The results of this study highlight how electric vehicles can provide improvements in some impact categories but will have negative impacts in other impact categories (e.g., human toxicity potential, mineral resource depletion) and how impacts can vary depending on the grid mix (global warming potential). The life cycle approach provides a unique perspective to understand the hot spots in a product life cycle or design and what trade-offs are associated with alternative pathways.

[66] https://www.life cycleinitiative.org/starting-life cycle-thinking/what-is-life cycle-thinking/.
[67] https://www.energy.gov/sites/prod/files/2016/05/f31/QTR2015-6L-Sustainable-Manufacturing.pdf
[68] https://www.epa.gov/chemical-research/tool-reduction-and-assessment-chemicals-and-other-environmental-impacts-traci) https://link.springer.com/article/10.1007/s10098-010-0338-9#page-1.
[69] Quadrennial Technology Review 2015, Chapter 6: Innovating Clean Energy Technologies in Advanced Manufacturing - Sustainable Manufacturing—Flow of Materials through Industry Technology Assessment. https://www.energy.gov/quadrennial-technology-review-2015.

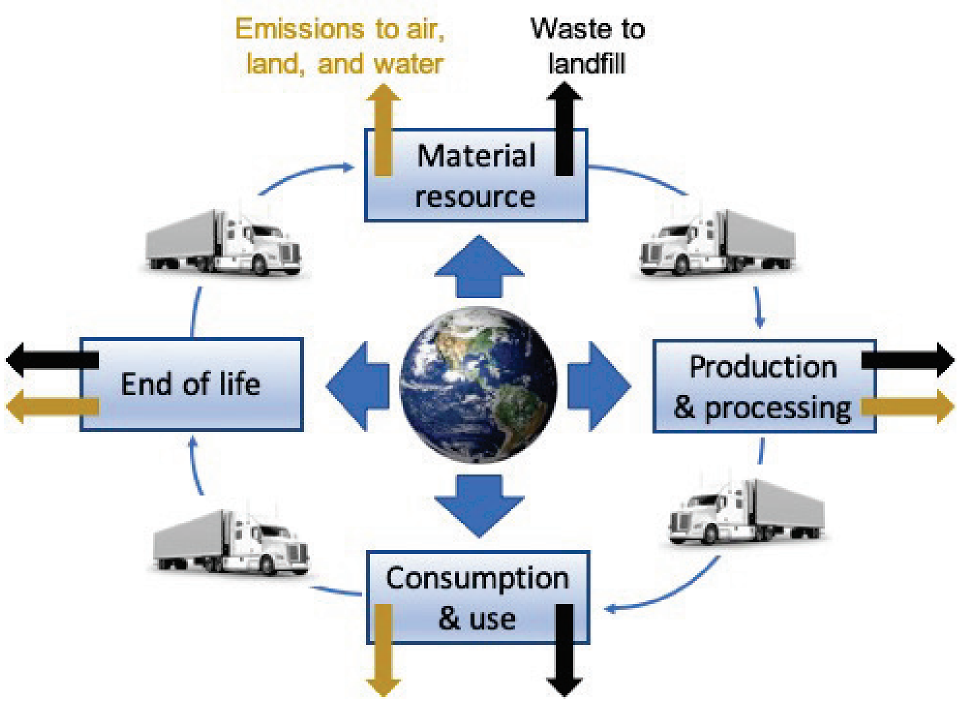


Figure 21. High-level schematic representing the accounting for life cycle assessments. The thin interior arrows represent movement of materials within the life cycle system; the thick orange arrows represent emissions to air, soil, and water, the thick black arrows represent waste products sent for disposal; the thick blue lines represent extracted resources going into each life cycle stage (energy, water, material); the orange, black, and blue arrows—which are associated with opportunities to reduce energy, resource use, and environmental impacts—represent the inputs and outputs for the system evaluated in life cycle approach (*Source*: *Quadrennial Technology Review 2015*, US Department of Energy)

The life cycle assessment and supply chain analysis methodologies can be used in evaluating technologies of interest to understand and minimize the externalized impacts and the material efficiency associated with the supply chain. Multicriteria analysis methods and system optimization can be used to incorporate this additional impact information into the decision-making process. The increasing focus on water scarcity due to drought impacts in the western United States and stressed aquifers from over-withdrawals indicates the pressing need to consider the connections between water and energy, and how life cycle assessment can help inform energy decisions by also considering water impacts. At a minimum, understanding the environmental impacts can minimize the risk of investing in a technology.[70]

[70] Quadrennial Technology Review 2015, Chapter 6: Innovating Clean Energy Technologies in Advanced Manufacturing - Sustainable Manufacturing—Flow of Materials through Industry Technology Assessment. https://www.energy.gov/quadrennial-technology-review-2015.

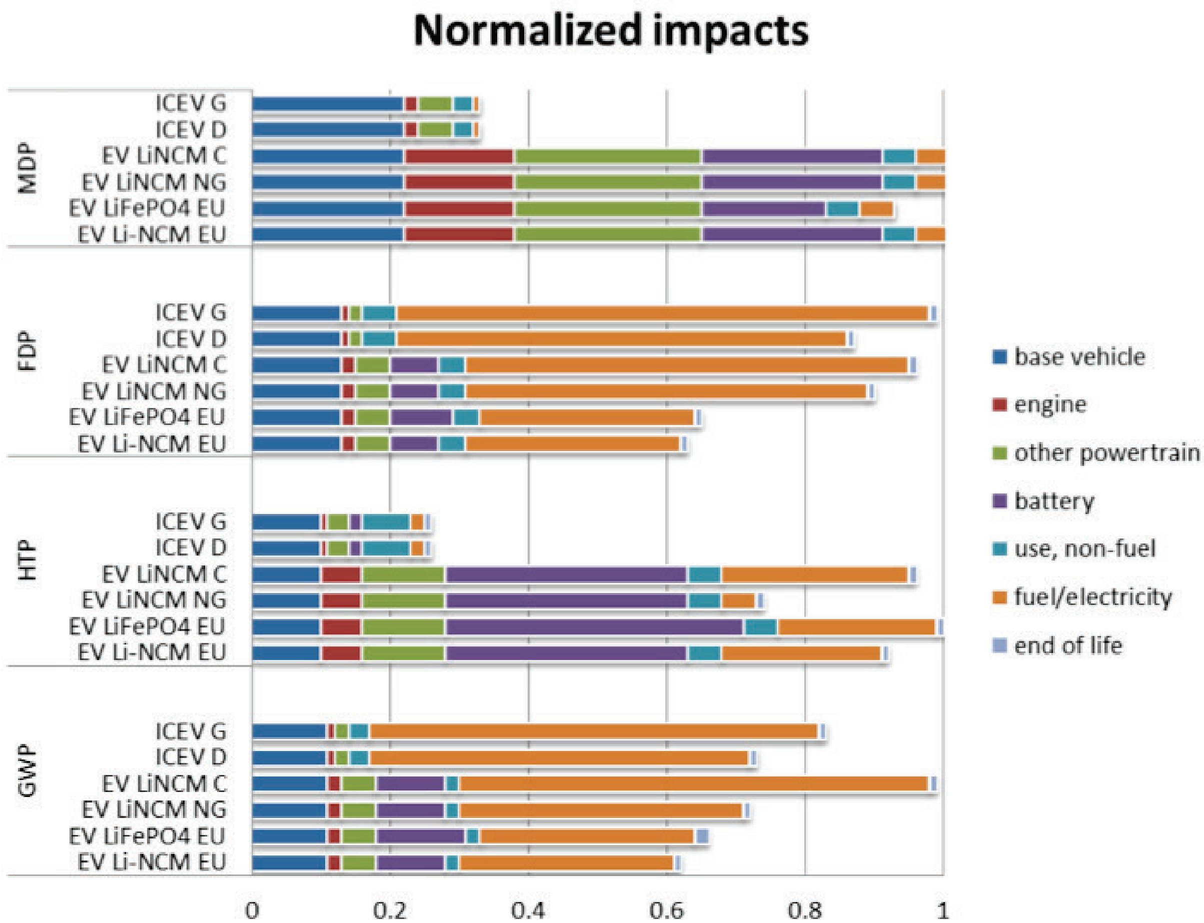


Figure 22. Life cycle impacts of first-generation electric versus conventional vehicles normalized to the largest total impacts attributed to life cycle stage or vehicle-component production. (*Source*: T. R. Hawkins, et al., Comparative environmental life cycle assessment of conventional and electric vehicles, *J. Ind. Ecol.* 17 (1), 53, 2013, ©2012 by Yale University

(Key: Global warming potential (GWP), human toxicity potential (HTP), mineral resource depletion potential (MDP), fossil resource depletion potential (FDP), internal combustion engine vehicle (ICEV), electric vehicle (EV), lithium iron phosphate (LiFePO4), lithium nickel cobalt manganese (LiNCM), natural gas sourced electricity (NG), coal-sourced electricity (CSE), European electricity mix (EU) (Hawkins, Singh, Majeau-Bettez, and Strømman 2013)

3.5 End-of-Life Issues

In the linear economy, as products reach their end of life, the intent is for the products to be disposed of in a landfill. This thinking is relatively new in the past 50 years. Access to materials and improvements in resource extraction and manufacturing technologies have made product manufacturing much cheaper and accessible to most parts of the US population. In earlier years, the concept of repair was built into products and allowed for products to stay in use for longer (and they were expensive to replace). Disposal thinking, however, does not account for the inherent value and expense associated with creating the product (embodied energy used to extract the materials and manufactured and assemble and transport the product) and the value of the materials themselves.

In the past decade, there has been increasing interest in understanding the potential to improve material efficiency in manufacturing processes. The World Economic Forum published an estimate of economic loss due to single-use plastics waste at $80 to $120 billion annually.[71] Municipal solid waste is well tracked (Figure 23) in the United States.[72] It does not include industrial waste, which is significantly greater and is not tracked. Recycling rates have stagnated for a number of reasons—new types of products that cannot be adequately recovered in the recycling systems, contamination in recycling streams, lack of adequate education about what can be recycled and what cannot, trash going into recycle bins, recyclables going into trash bins, and reduced quality of recovered materials (and thus lower market value).

For clean energy technologies, the growth in technology deployment is projected to have significant impacts in future years. Global PV growth is projected to result in significant PV waste (Figure 24) as the systems come to their end of life.[73]

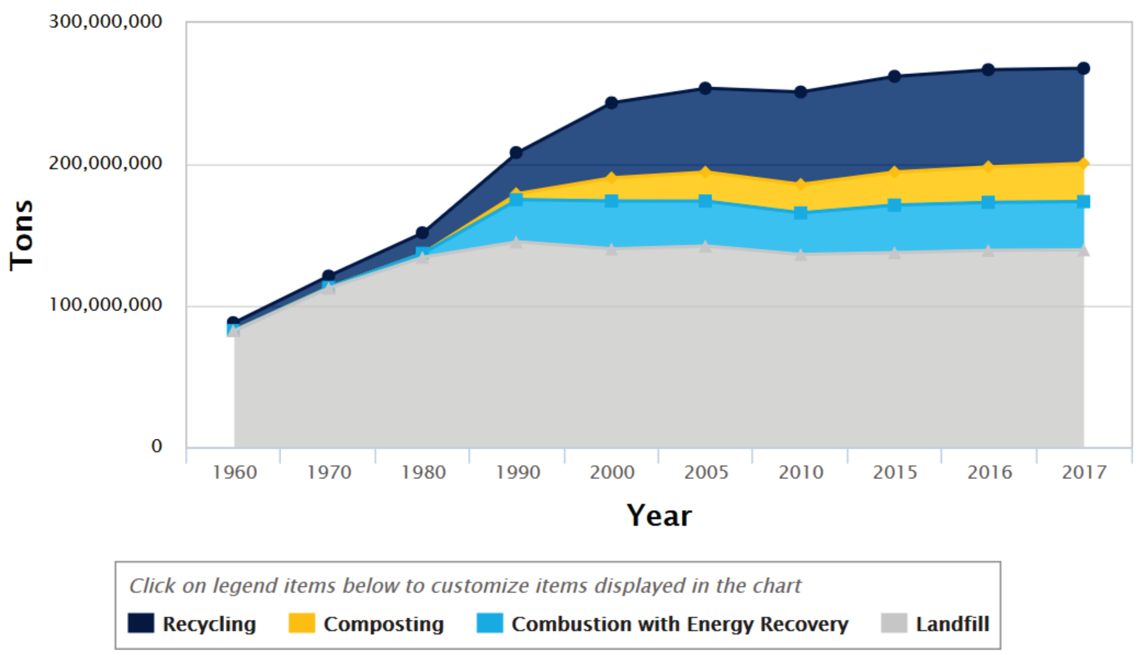


Figure 23. Management of municipal solid waste continues to be a high priority for states and local stakeholders and is well tracked in the United States. (*Source:* *National Overview: Facts and Figures on Materials, Wastes and Recycling*, US Environmental Protection Agency)

[71] WEF, 2016, The New Plastics Economy (www3.weforum.org/docs/WEF_The_New_Plastics_Economy.pdf).
[72] https://www.epa.gov/facts-and-figures-about-materials-waste-and-recycling/national-overview-facts-and-figures-materials.
[73] IEA/IRENA 2016 (https://www.irena.org/publications/2016/Jun/End-of-life-management-Solar-Photovoltaic-Panels).

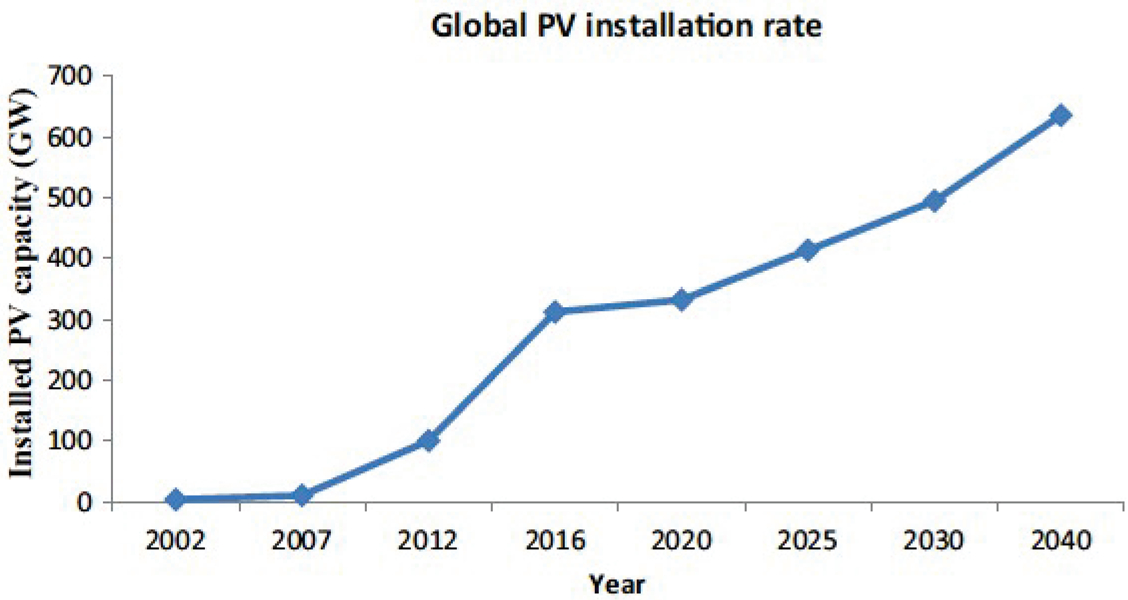


Figure 24. Installed Capacity of PV.
(*Source*: S. Diwania et al. *IJEE* 11, 33–54, 2020, doi: https://link.springer.com/article/10.1007/s40095-019-00327-y. This article is distributed under the terms of the Creative Commons Attribution 4.0 International License (creativecommons.org/licenses/by/4.0)

The use of wind turbines has grown over the past few decades so that disposal of the blades alone is logistically challenging. Although most of a turbine can be recycled or find a second life on another wind farm, researchers estimate the United States will have more than 720,000 tons of blade material to dispose of over the next 20 years, a figure that does not include newer, taller, higher-capacity versions.[74] The blade material is evolving from thermoset plastics that are difficult to recycle to new thermoplastics that are more amendable to repair and recycling.[75] However, there is a need to find a viable recovery mechanism for the large volume of thermoset plastic blades currently in the field that will eventually come to end of life and need to be managed.

The market for LIBs is continuing to grow, which poses a challenge and an opportunity. LIBs are not governed by the same regulations as lead acid batteries (which require that all lead acid batteries be recovered and managed—providing incentive for a recycling market). The recovery cost incentive for LIBs is currently driven by the cobalt content, but future LIBs (Figure 25) might have either reduced or no cobalt, reducing the cost driver that is required to make recovery and recycling cost effective.[76]

News media have extensively covered the problem of environmental leakage of plastics. For 2010, Jambeck and colleagues estimated that 275 million MT of plastic waste were generated in 192 coastal countries and that between 4.8 and 12.7 million MT of the waste entered the ocean (Jambeck et al. 2015). This issue can be attributed to at least a couple of factors: (1) inadequate waste management systems around the world and (2) plastic materials that do not easily degrade being used in single-use products that are highly dispersed and harder to control.

[74] https://www.sciencefriday.com/segments/wind-turbine-waste/.
[75] https://iacmi.org/2017/04/10/the-manufacturing-evolution-of-wind-turbine-blades/.
[76] http://www3.weforum.org/docs/GBA_EOL_baseline_Circular_Energy_Storage.pdf.

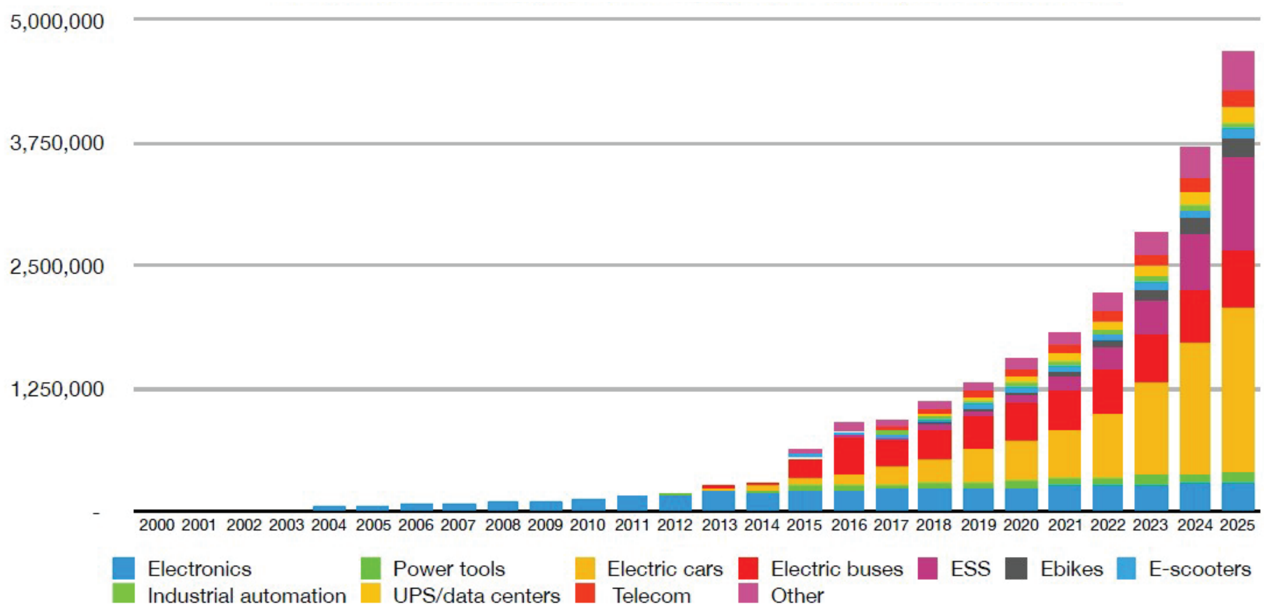


Figure 25. Lithium-ion batteries placed on the global market (cell level, tonnes). (*Source:* "The lithium-ion battery end-of-life market—A baseline study," Hans Eric Melin, Circular Energy Storage)

Additional research has found that this leakage in the form of microplastics is found in the most remote locations[77] and in food.[78] Researchers are also finding that even biodegradable plastics are degrading only into microplastics.[79] Plastic contamination is also found in composting streams because of plastic coatings on packaging that end up in compost systems.[80]

3.5.1 Resource Conservation

The value in resource conservation and material efficiency comes from several paths. Products and the materials used in them require some level of cost and energy to extract and transport and produce. Disposing of products made of energy-intensive materials means that all the cost and energy to produce the materials is lost. Critical materials are also a strong driver. Materials with limited availability and with supply chains at risk of disruption from political upheaval or market forces need to be retained in the economy rather than being disposed of.[81] When materials end up in the landfill or lost to the environment or stockpiled, the economy is not deriving any value from them. Value can be derived from their limited and restricted availability, the cost and energy to extract and produce them, or the service they provide in their functional form. Maintaining high material efficiency (Allwood et al. 2011) means that this value is being retained as well as possible and comes through a range of different strategies and actors (Figure 26).[82]

[77] https://www.bbc.com/news/science-environment-48230157.
[78] https://www.bbc.com/news/science-environment-42270729.
[79] https://www.sciencealert.com/those-eco-friendly-biodegradable-bags-don-t-degrade-as-fast-as-you-might-think.
[80] https://www.ecocycle.org/files/pdfs/microplastics_in_compost_white_paper.pdf.
[81] U.S. DOE Critical Materials Strategy (https://digital.library.unt.edu/ark:/67531/metadc834802/).
[82] DOE Quadrennial Technology Review, 2015, Sustainable Manufacturing Tech Assessment (https://www.energy.gov/sites/prod/files/2016/05/f31/QTR2015-6L-Sustainable-Manufacturing.pdf).

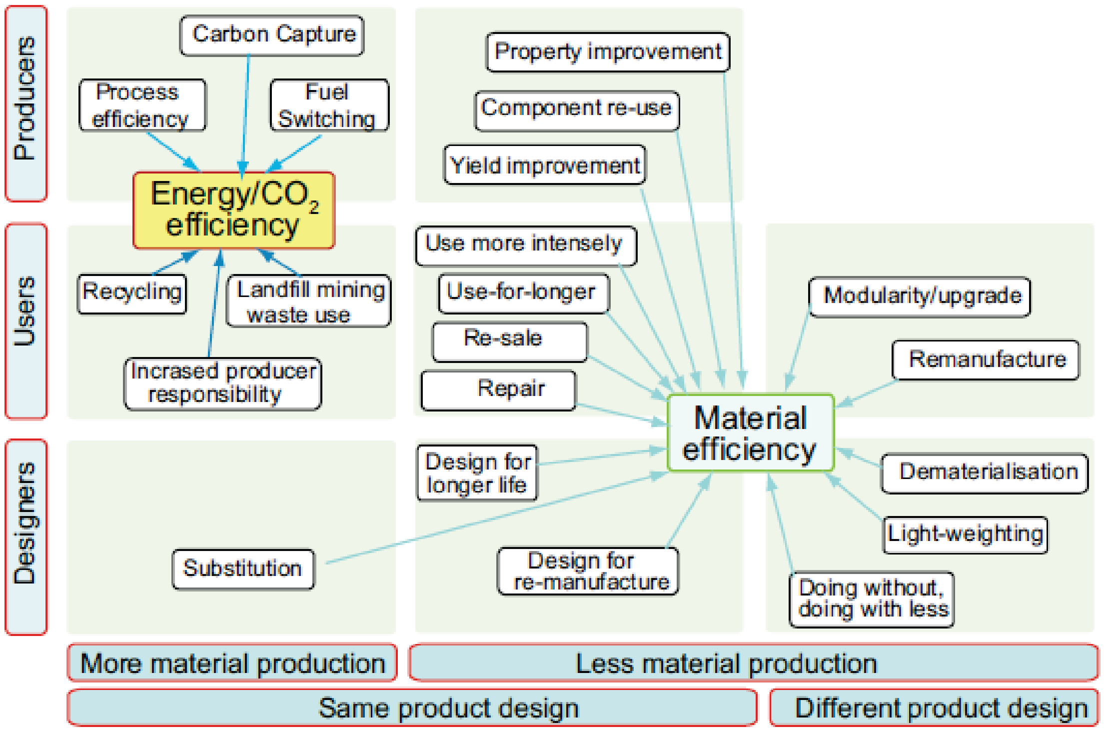


Figure 26. Categories and actors for implementing material efficiency.
(*Source*: Reprinted from *Resour. Conserv. Recy*. 55, J. M. Allwood et al., Material efficiency: A white paper, 362, copyright 2011, with permission from Elsevier)

A number of strategies can help reduce end-of-life waste. To the three "Rs" of old (reduce, reuse, recycle), new Rs have been added: revaluation, redistribution, relations, resilience, reassessment, and restructuring[83]; and in the context of the circular economy, other Rs have been identified, as shown in Figure 27.

The waste-management hierarchy starts with eliminating or reducing use of materials, especially hazardous materials that are heavily regulated.[84] Following that, the strategy should be to keep products in use for as long as possible through reuse, upgrading (e.g., modularization, software updates), repair, refurbishment, or remanufacturing. Products in their functional form have a certain amount of value that comes from the energy and expense required to create the products. Moving to the next option, recycling, would result in a loss in the functional form. Trade-offs to consider involve how efficient a product is and whether a new version would have overall lower environmental impacts. Once no value remains in the functional form of a product, or it is not repairable or usable in any way, the next option is to recycle the product down into its different separate materials. If no value remains in the recovered materials, a last option before landfilling would be energy recovery, using the waste material as a fuel for energy generation. Energy recovery means that no new value will ever be extractable from the material.

[83] https://www.activesustainability.com/sustainable-life/learnsustainability-the-3rs-6rs-and-9rs/.
[84] www.solarwaste.eu.

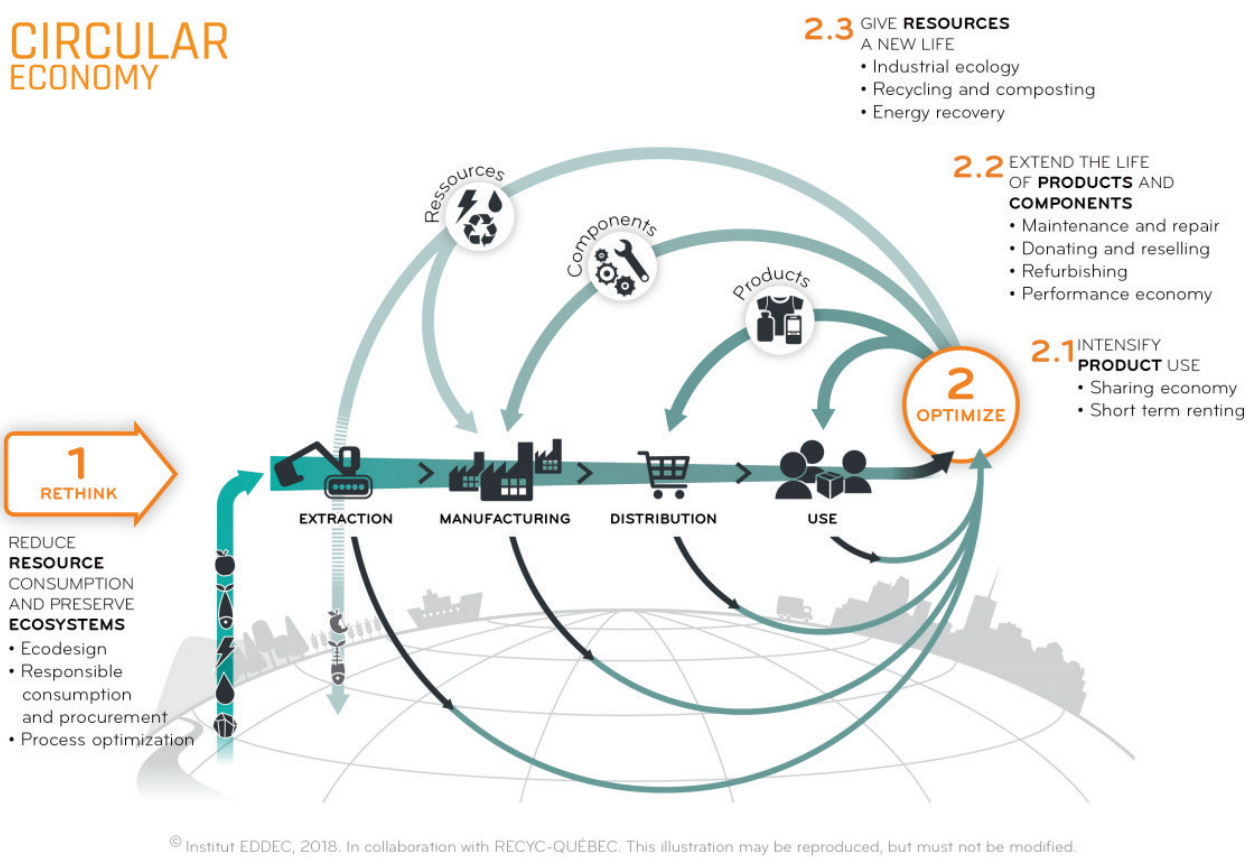


Figure 27. Circular economy strategies that look to narrow, slow, and close the loops in the economy. (https://www.activesustainability.com/sustainable-life/learnsustainability-the-3rs-6rs-and-9rs/) (*Source*: Institut EDDED 2018, in collaboration with RECYC-QUEBEC)

Several different types of challenges must be considered in designing products that are compatible with the circular economy.

- Materials are used that exceed the need of the product—plastics that last for 100+ years for a product that is single use and has a lifetime of perhaps a month or so.
- Technology turnover makes older versions obsolete (e.g., smart phone updates that work only up to a certain point, making old smart phones function poorly). Longer-lasting products end up not having the same performance and functionality as new products (e.g., old PV panels do not have the same efficiency as newer panels, old cars and appliances are more energy-intensive than newer products).
- Design of products for repair, reuse, or remanufacture conflicts with design for durability, resilience, or longevity—a product that can come apart more easily has more vulnerabilities and is more likely to expose sensitive components to the elements.
- The dispersed nature of products makes reverse logistics challenging (e.g., single-use packaging, smart phones, and consumer electronics).
- The use of small amounts of valuable materials in dispersed products makes it difficult to collect and separate them (e.g., gold, silver, or copper in consumer electronics).

- The use of any type of hazardous material in a product may cause the product to fall under hazardous waste regulations and thus make reverse logistics difficult (e.g., PV panels, treated wood).
- Degradation of products over their lifetimes results in microplastics being released into the environment (e.g., all single-use plastics, residue from textiles during laundering).
- New material types and alloys are used that have no end-of-life mechanisms for extracting the original materials. (e.g., recycled steel and recycled aluminum have value, but the value is reduced because they cannot be returned to pure iron or aluminum steel).
- Alloys are used that at end of life can contaminate recovered materials (e.g., small amounts of copper in a steel recycle stream can result in reduced performance of newly made steel) (Daehn, Cabrera Serrenho, and Allwood 2017).
- Understanding of the limits of the existing material recovery systems is needed to ensure that products are best able to be recovered at end of life.
- Impacts from processing at end of life can be nontrivial (Figure 28) (Oliveira et al. 2015).

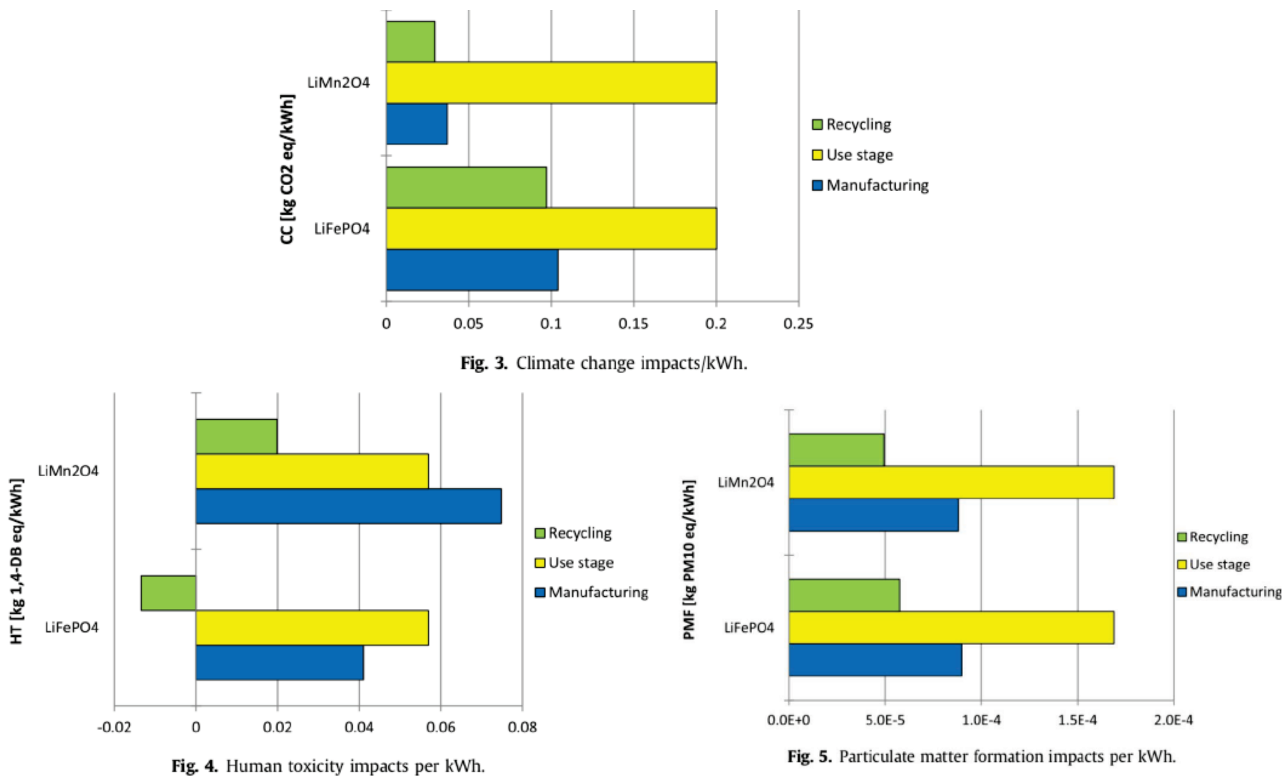


Figure 28. Climate change, human toxicity, and particulate matter life cycle impacts of LIBs. Impacts are evaluated for the manufacturing, use, and recycling stages; recycling processes for LIBs are not insignificant (*Source*: Reprinted from *J. Clean. Prod*. 108, L. Oliveria, et al., Key issues of lithium-ion batteries–From resource depletion to environmental performance indicators, 354, copyright 2015, with permission from Elsevier)

3.6 Critical Materials

Over the past decade, material criticality has gained increasing attention. Governments, academia, and independent organizations have developed methods for assessing material criticality and identifying CMs (also referred to as critical minerals and critical elements). In 2018, the US Department of the Interior published a "final" list of CMs based on an in-depth, interagency study (see Table 7).[85] The list of 35 CMs is intended to be updated to reflect future data on demand, supply, concentration of production, and policy priorities. This CM list was derived based on the materials' geographic concentration of their production, their US net import reliance, and their recovery as byproducts from mining of other minerals. Applications considered in the analysis span important technologies in aerospace, defense, energy, telecommunications and electronics, and non-aerospace transportation. Information on the CMs and the methodology for their selection is published in the literature (Fortier et al. 2018).

Table 7. US Department of the Interior's Final List of Critical Materials

US Critical Materials (*Federal Register* 2018)

Aluminum (bauxite)	Antimony	Arsenic	Barite	Beryllium
Bismuth	Cesium	Chromium	Cobalt	Fluorspar
Gallium	Germanium	Graphite (natural)	Hafnium	Helium
Indium	Lithium	Magnesium	Manganese	Niobium
Platinum group metals	Potash	**Rare earth elements group**	Rhenium	Rubidium
Scandium	Strontium	Tantalum	Tellurium	Tin
Titanium	Tungsten	Uranium	Vanadium	Zirconium

The EU issued a list of 24 individual and grouped CMs based on their economic importance to the EU economy and the risk of disruption in EU supply.[86] The EU CM list includes a few materials not in the US list, namely borate, coking coal, natural rubber, phosphate rock, phosphorous, and Si metal. DOE issued an energy-focused CM strategy in 2011 (DOE 2011). Materials assessed were those important to energy technologies, wind turbines, solar PV cells, grid storage batteries, electric vehicles, vehicle lightweighting, catalytic converters, and LEDs.

Based on results from surveys of the literature and of 169 European scientists, Hoffman et al. (2018) found a lack of CM awareness and concern within the materials science community. Helbig et al. (2017) called for consideration of supply and environmental risks associated with resources in the early stages of basic research. Figure 29 illustrates their proposed multidisciplinary research approach for material scientists.

Rare earth elements (REE) provide an instructive example of CM basic science opportunities. In a comprehensive assessment of REE markets, Nassar et al. (Nassar, Du, and Graedel 2015) identified

[85] *Federal Register*/83(97), Friday, May 18, 2018, "Notices," https://www.govinfo.gov/content/pkg/FR-2018-05-18/pdf/2018-10667.pdf

[86] https://ec.europa.eu/growth/sectors/raw-materials/specific-interest/critical_en

supply risk, low substitution potential, environmental implications of supply, and vulnerability to supply disruption as key indicators of their criticality. REE materials provide important functionalities in applications that span energy, transportation, catalysis, defense, and electronics. Demand growth is particularly strong for neodymium-iron-boron (NdFeB) magnets used in high-efficiency motors, including those for electric and hybrid vehicles and wind turbines. Supply risk stems from China's dominance in the market. China supplies more than 80% of global REE demand and is the largest global producer of REE-containing downstream products, including metals, magnets, glass polish, alloys, and phosphors. Although specific examples of basic science needs described in the following paragraphs are from the REE literature, the research needs are applicable to all CMs.

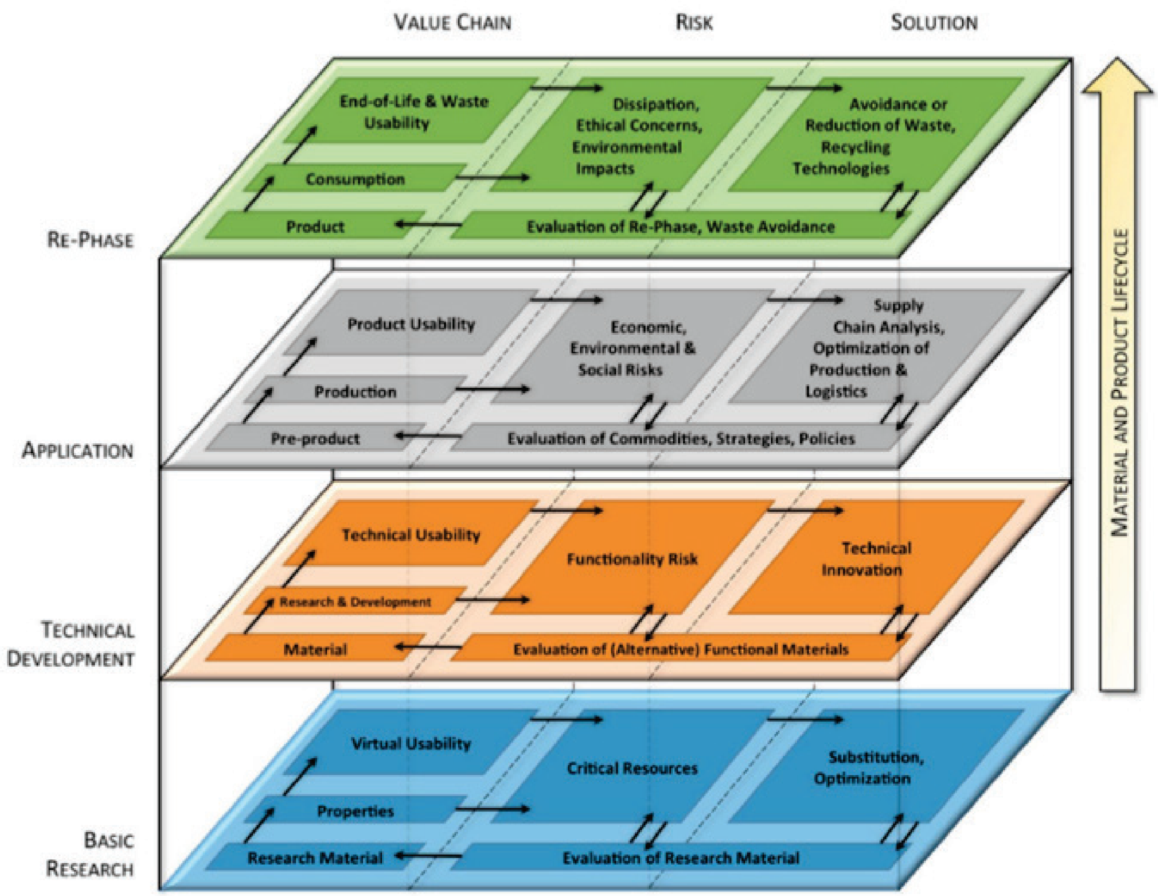


Figure 29. Guideline for material scientists: Elements of multidisciplinary research approach to identify and mitigate CM risks. (*Source*: Reprinted from *Sustain. Mater. Techno*., 12 /1 C. Helbig et al., Benefits of resource strategy for sustainable materials research and development, 1, copyright 2017, with permission from Elsevier)

Opportunities to mitigate the criticality of REEs span their life cycles. Geoscience advances in analytic methods for accurate measurements of individual REE content in diverse primary, secondary, and unconventional resources can improve quantification of their economically viable recovery (Balaram 2019; Hartzler, Bhatt, Jain, and McIntyre 2019). Processing steps from mine to REE products (see Figure 30) are capital and energy intensive and environmentally burdensome (Navarro and Zhao 2014; Zaimes, Hubler, Wang, and Khanna 2015). Element-specific separation technologies could improve the viability of developing mines outside of China or recovering REEs from unconventional sources, such as coal ash, red mud, mine tailings, and geothermal fluids. Novel separation agents being studied for REE recovery applications include metal-selective ligands (Izatt et al. 2016) ordered mesoporous materials (Hu et al. 2018), bacteria (Bonificio and Clarke 2016), and functional ionic liquids (Khodakarami and Alagha 2020). Advancements in modularization and PI technologies could be particularly important for unconventional sources. These sources are geographically dispersed and contain relatively small amounts of REE materials (Borra et al. 2015; Jin et al. 2017; Lo et al. 2014; Peelman, Sun, Sietsma, and Yang 2016).

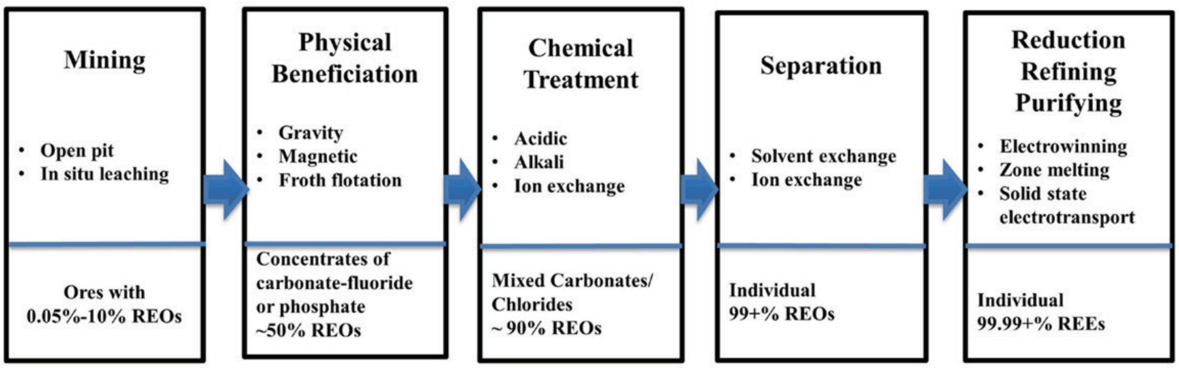


Figure 30. Process steps for recovery of REE from mines. REO = rare earth oxide (*Source*: J. Navarro and F. Zhao, *Front. Energy Res.* 2, 45, 2014)

Beyond resource recovery, strategies for mitigating criticality include improving materials efficiency in manufacturing (e.g., reducing losses and improving quality control); minimizing CM consumption via material or product substitution, process innovation, and product design; and recovering CMs from products after their useful life. REE examples follow. AM has been demonstrated to produce bonded NdFeB magnets with less material loss (Li et al. 2016). The development of grain boundary diffusion technologies has allowed a reduction in dysprosium content, while improving the performance of NdFeB magnets (Hirota, Nakamura, Minowa, and Honshima 2006). The invention of LED technology has reduced the demand for compact fluorescent lighting—a trend that has significantly reduced the consumption of REEs used in phosphors, specifically, terbium, europium, and yttrium.

Basic science advances are also needed to develop approaches for product design in the circular economy (den Hollander, Bakker, and Hultink 2017) and recycling technologies that enable economic recovery of CMs. A few REE recycling approaches under study include plasma separation (Gueroult, Rax, and Fisch 2018), selective dissolution with functionalized ionic liquids (Dupont and Binnemans 2015), and adsorption with mesoporous silica nanoparticles (Zheng et al. 2015). Finally, technological change emerging from basic science has affected and will likely continue to affect the criticality of specific materials (Langkau and Espinoza 2018). In the research process, scientists need to maintain cognizance of potential supply risks associated with materials and technology innovation. Research opportunities in CMs include the following:

- Geoscience research to improve characterization of CM resources;
- Process and chemistry innovation for economically viable, environmentally benign, and resource-efficient recovery of CMs from mined ores, unconventional sources, end-of-life products, and landfills;

- Application of advanced theory, modeling, synthesis, fabrication, and characterization techniques to design CM substitutes that meet or surpass required functionality;
- Innovative product design to enable reuse, remanufacturing, and recycling.

3.7 Substitutes or Alternative Approaches

Electrochemical refinery approaches use novel processes, renewable and conventional energy sources, and green feedstocks, including renewable low-cost electrons, to form chemical compounds that can be organic or inorganic. Key to the success of these approaches is the use of CO_2 feedstocks as well, which can potentially open up new avenues for synthesis, including metamaterials and metastable materials.

3.8 Designing for Recycle, Remanufacture, and Reuse

Designing a product with the end of life in mind frequently offers untapped potential that can provide an opportunity to more cost effectively reuse, remanufacture or recycle a product. The priority in product design is manufacturing a product that meets its performance requirements. In many instances, adding features that aid in end-of-life handling inherently reduce the performance or increase the cost of a product. However, these negative aspects do not always exist. Effort must be taken to maximize the potential of this opportunity. A challenge that manufacturers face is that there is no benefit to them in designing end-of-life handling into their products. To change this mindset, product designers and manufacturers need to have tools available to demonstrate the benefit of designing products for end-of-life handling. This can be done with models that show the cost and environmental impacts of the product at the various stages of life. These tools help show that an added cost may enable a larger cost savings to the manufacturer or other stakeholders at end of life. It is also valuable for product designers and manufacturers to have a list of examples or other case studies to aid in concept realization.

A list of examples for easing the disassembly of products for remanufacture or recycling is provided by East West[87] ("How to Design Sustainable Products for Recycling by the End User") with the following suggestions.

- Use fewer parts;
- Use common parts;
- Reduce the types and number of fasteners used in an assembly;
- Use common fasteners that do not require specialty tools for removal;
- Avoid using glue or other adhesives;
- If glue is required, consider soluble adhesives for easier disassembly; and
- Include disassembly instructions with product.

[87] https://news.ewmfg.com/blog/how-to-design-sustainable-products-for-recycling-by-the-end-user

4 Cross-Cutting Topics in Manufacturing

Many cross-cutting topics in manufacturing—from understanding defects to improving characterization tools—would benefit from basic research in areas such as condensed phase and interfacial molecular science, scattering and instrumentation science, and physical and mechanical behavior of materials.

4.1 Manufacturing Scale-Up

The goal of basic research is an in-depth, fuller understanding of the fundamental aspects of a concept or phenomenon. This understanding is usually the first step and a necessary foundation for further R&D. These activities typically do not necessarily have a direct application or particular product in mind. Complementary to basic research, applied research involves activities to acquire specific additional knowledge about a particular process or product beyond the basic concept needed to envision such a process. Applied activities may be the determination and development of a new product or process or simply an improvement of an existing one.

In materials and chemical manufacturing, process R&D is the necessary and essential step to innovate and introduce new products to the market. It is the first stage in the development process, preceding even a pilot run or further scale-up to manufacturing operation. Process R&D bridges the gap between the laboratory synthesis of a new material and industrial manufacturing at a large scale. Although process R&D needs to address several different aspects, the common goal is to develop a safe, reliable, scalable, and cost-effective process that produces materials, chemicals, or components with the desired specification and cost in a commercial environment.

4.1.1 Manufacturing Requirements

The Industrial Revolution in the nineteenth century developed the foundation for modern manufacturing. Emerging technologies like the steam engine led to advancements in many fields such as mining, transportation, and chemical manufacturing. International trade allowed global sourcing of feedstock materials and other goods. Over the centuries, several aspects of manufacturing needed to be considered for their effect on materials and chemical processing in the manufacturing cycle and their specific impact on the economy, health and environment.

Two related main factors made modern manufacturing successful (Schulz 2005): the quality of goods and the reliability of deliveries. Modern manufacturing operations rely increasingly on automation technology and data collection and processing in real time. Although manufacturing operations are becoming more and more autonomous, requiring less operator intervention in normal operation, they also require a more sophisticated, well-educated, and experienced workforce. It is not uncommon to have equipment on the plant floor integrated and connected by a computer network to a central system that has real-time decision-making ability. This approach involves the use of computer-controlled semi-autonomous supervision of production systems, yet these systems still need to be overseen by skilled workers. The system relies on distributed, customized software developed for specific manufacturing tasks, which is yet another duty for a skilled-in-the-art domestic workforce.

Production automation has posed its own problem. To address it, Six Sigma (6σ)[88] was introduced as a set of techniques and tools for process improvement. It initially was implemented by Bill Smith (Motorola, early 1980s), but Jack Welch made it essential for modern manufacturing at General Electric

[88] https://en.wikipedia.org/wiki/Six_Sigma American Society for Quality, https://asq.org/quality-resources/six-sigma.

in 1995. In the Six Sigma process, 99.99966% of all products are expected to meet the requirement to be free of defects.

In the modern fast-paced environment, yet another restriction is that industries tend to change products frequently because of demand, trends, and competition (Bereznoy 2019). Production setups for these types of products that rely on demand-driven markets require high quality and low cycle times, because industries must maintain high production rates and reliably deliver high-quality products to remain competitive.

High-throughput, robotic quality control/quality assurance instrumentation and software are key necessities for modern manufacturing. There is also an opportunity to implement robotics and automation to allow an intelligent machine to carry out highly hazardous work that may be too precise or too tedious for humans. The application of AI and ML will push the boundary of intelligence and capability for many forms of autonomous or semiautonomous manufacturing operations.

4.1.2 Challenges of Scaling

Bringing a new material to market quickly and profitably requires efficient and cost-effective processes for preparing many intermediates that lead to the final target material or chemical. The key to rapid and successful process development for material/chemical manufacturing is avoiding scale-up problems. An understanding of scale-up issues at early stages of development is vital for developing more efficient, high-yielding, safe, and environmentally friendly processes in the future.

The objectives of some approaches to discovery science are to create and evaluate new materials/chemicals quickly or to evaluate the synthesis techniques in more detail. Researchers will make a material/chemical by expedient routes on the laboratory scale and characterize (in detail) the synthesis and properties of the product. This may involve several techniques that may not be economical or even possible on the manufacturing scale. Designing the route to a new material/chemical at the discovery stage usually does not consider factors like feedstock material costs, capital equipment cost or availability, safety (at larger scales), and the amount and nature of production waste streams. Direct translation of the lab's original route to implementation in large-volume manufacturing is rarely possible.

Hazards associated with the process and waste streams generated are usually not considered at the discovery stage. The level of hazard linked to the process can be directly translated to the cost because higher hazards require more engineering countermeasures. Handling and disposal of waste streams is an integral part of the manufacturing process. Depending on the volume and type of waste, waste handling can add substantial cost to the overall expense of manufacturing.

Optimum process development is a complex task that requires multidisciplinary teams with skills ranging from chemistry and materials science to chemical engineering, computer modeling and simulation, and technoeconomic analysis. Assembling a team with the required expertise to solve demanding problems associated with bringing a new material/chemical from the discovery lab to market is yet another challenge. Several challenging aspects of materials/chemical manufacturing scale-up need to be carefully considered to develop a robust and safe process that delivers products according to specifications.

- The reaction vessel itself must be carefully considered. Lab-scale work uses glass equipment that is chemically inert to most reagents. However, glass reaction vessels have limitations due to mechanical strength and other properties. Most industrial equipment is made of metal or glass-lined metal that poses its own challenges (e.g., corrosion and contamination, different thermal expansion between glass and metal that limits the usable temperature range). Maintaining the integrity of the manufacturing vessels for long-term use is a challenge.
- The process environmental impact needs to be assessed in the context of regulatory constraints. This includes not only waste generation and utilization, but also chemical transportation, storage, and assessment of accidental release. Stability and toxicity of feedstock material, intermediates, and final product need to be considered.
- Time versus energy consumption versus mass of product per volume of unit of the reactor is one of the critical factors in the technoeconomical evaluation of the process. Because heat transfer is proportional to the surface-volume ratio of the reaction vessel, operation on a large scale inevitably takes longer than in a small-volume discovery lab glass reaction flask, particularly if the process is run in a batch mode. To help address this concern, new processes for manufacturing should be evaluated for suitability in either batch or continuous flow. Continuous-flow chemistry offers many advantages that should be considered as a first choice to reduce cost and improve quality and safety. (Gutmann, Cantillo, and Kappe 2015)
- Assessment of the thermal behavior of the process (exothermic events) is a common problem for scale-up that can lead to a catastrophic event if not addressed and understood in the early stage of R&D. It is essential that the thermochemistry is fully evaluated and understood before any large-scale manufacturing is undertaken. Unfavorable surface-to-volume ratios in an industrial reaction vessel need to be considered for an exothermic process scale-up. A thermal runaway reaction in a discovery-scale lab will have a relatively insignificant impact on operation or safety of personnel compared with an undesirable event on a plant scale.

Process parameter screening requires accurate models or the systematic exploration of a broad set of relevant reaction variables to achieve optimization (which also are the foundation for developing accurate models). Although most researchers follow standard R&D processes to scale up manufacturing of materials, developing more science-based, data-driven systematic, holistic approaches to the modern manufacturing of advanced materials and chemical is needed. A well-planned approach using high-throughput screening in combination with computer modeling provides researchers with an expedited path to quickly see the big picture and abandon disadvantaged routes, and rapidly pinpoints advantageous and effective processing conditions to focus on in the next stages of optimization.

4.2 Characterization Tools

Modern manufacturing typically incorporates complex equipment and processes with real-time monitoring, intelligent feedback and control, robotics and automation, enhanced sustainability, and energy efficiency. Many of these features can be supported by rapid in situ characterization.

4.2.1 Manufacturing-Specific Phenomena and Systems

Manufacturing activities encompass large-scale, high-throughput production of products through multiple processing steps. Depending on the product, R2R, sheet-to-sheet, or inline systems often are used. R2R processing enables high-speed and continuous production; inline processes eliminate the waiting time between steps. These and similar approaches improve manufacturing efficiency and speed.

For high-speed, continuous production processes like R2R, compatibility between the upstream and downstream processing steps is important. For example, with an in-line, thin-film deposition system, substrate heating and film deposition are designed to occur in two subsequent chambers. The substrates first travel through the heating chamber to reach the deposition temperature and then move to the deposition chamber for film growth. To achieve a continuous flow of substrates, the heating and deposition conditions need to be designed in a way that ensures compatible heating and deposition time. Another example involves R2R slot die coating, where the coating speed and web moving speed need to be well matched. In a scenario in which coating films need to be dried or cured, the thermal treatment should be made fast enough to catch the R2R coating speed. In this case, rapid infrared or flash lamp annealing is often helpful. Otherwise, a long oven zone and a product transfer conveyor would be required. In addition to compatibility, process precision and repeatability is also critical for a high-speed process. Taking the R2R coating process as an example again, servo-driven rollers, speed control, and tension control units are needed to improve web stability and accuracy.

Operational conditions can be optimized by using rigorous global, multiobjective optimization algorithms that maximize economic performance and minimize environmental and social impacts (Asadi and Farahani 2018). Multistage batch processes have additional challenges that increase the complexity of monitoring (Undey and Cinar 2002). Data collected from each processing step and each phase tend to possess different variable correlation structures. Multiblock partial least-squares techniques for modeling large chemical processes (Gavidel, Lu, and Rickli 2019) have been applied to monitoring polymer processing (Yuan, You, and Ricardez-Sandoval 2019). Several techniques have been developed for modeling and monitoring multiphase processes and have been applied to the development of regression models between stages (Undey and Cinar 2002).

4.2.2 Rapid In Situ Characterization

In modern manufacturing, in-line and in situ sensing and characterization are used for process monitoring and product quality diagnostics and control. In-line and in situ characterization within a manufacturing process provides (1) transient and intermediate information, leading to a better understanding of the physics and chemistry involved in the process; (2) real-time product information, monitoring of product quality, and minimization of defects and error rates; and (3) a feedback control loop that improves process automation.

Depending on the specific product, different attributes can be used to evaluate and monitor product qualities, such as shape and dimension, surface roughness, mechanical deformation, composition, chemistry, temperature, and defects. Correspondingly, there is also a wide choice of in-line and in situ metrologies that are applicable to investigate product properties and status. High-resolution, high-speed cameras, x-ray, ultrasound, and lasers are often used to detect product defects and surface conditions. Raman and Fourier transform infrared spectrometers are sensitive to product chemistry and composition. Gas sensors can monitor solvent evaporation rates in chemical reactions, and infrared thermography and spectroscopy are reported to be highly capable of detecting inner, buried defects (Tofail, Mani, Bauer, and Silien 2018).

Recently, use of large analytical facilities such as synchrotron and neutron sources has become popular. These analytical facilities provide brighter x-ray or neutron sources, which provide a temporal and spatial resolution that is unobtainable with other lab tools. A physical and chemical process can be characterized in situ at such a facility through a small model system. These studies deepen scientific understanding, thus accelerating technology advancement as well as process optimization. The knowledge achieved from the model system is then connected to real production through parameter correlation with the in situ sensing and characterization equipment on the manufacturing floor. Figure 31 shows small angle x-ray scattering (SAXS) of nanofibers synthesized by electrospinning techniques at Argonne's Advanced Photon Source (APS). The SAXS experiments were performed in situ during nanofiber fabrication and demonstrated high-throughput characterization capabilities by testing more than 90 processing conditions/recipes within 24 hours (Zhang, Y. et al. 2019). From the SAXS 2D images (e.g., intensity I(q) versus wave vector q), nanofiber diameter and diameter distribution can be extracted. If the above quantitative information is not required, the SAXS pattern can be analyzed to understand the fiber quality directly; for example, a more scattered pattern corresponds to smaller variations in fiber diameter and thus more uniform fiber morphology. This information can be linked to manufacturing-level R2R electrospinning by monitoring and comparing the microjet shape and size near the spinning nozzles observed by high-resolution cameras during R2R electrospinning and during in situ experiments at APS. ML models that are capable of image identification and validation can help extract additional information and perhaps increase the value of the data using fewer measurements on the manufacturing floor.

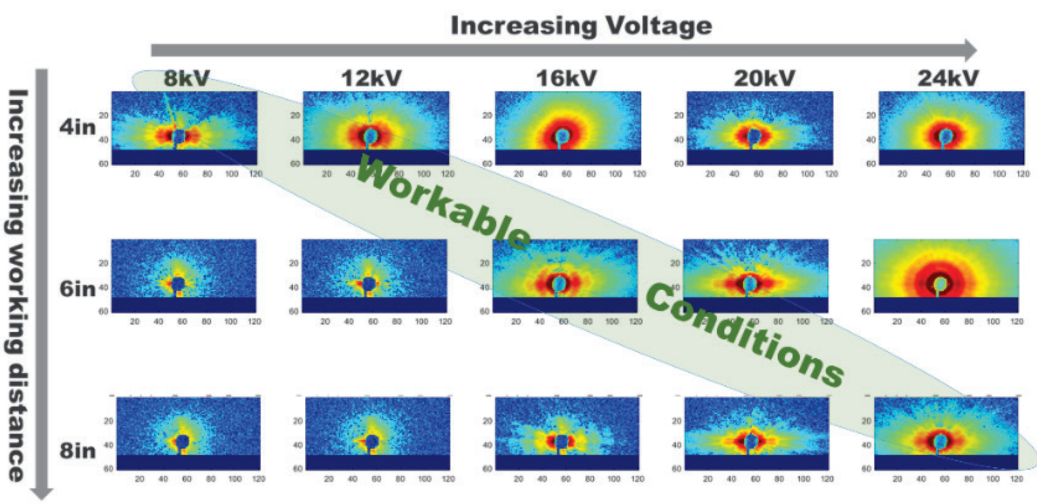


Figure 31. SAXS 2-dimensional images of electrospinning nanofibers fabricated with different process voltages and working distances. The data were collected at the Advanced Photon Source and in situ. Workable electrospinning conditions can be identified from the SAXS intensity scattering level (*Source*: Y. Zhang, et al., In situ synchrotron characterization of nanofiber synthesis for solid state battery applications, 31st Annual Electronic Packaging Symposium, 2019. Used with permission from Argonne National Laboratory)

4.2.3 Sensing and Correcting Problems in Manufacturing

With the development of 5G mobile and wireless communication technology and ML, the (big) data obtained from in situ characterization can be streamed and analyzed more rapidly. With 5G, a combination of sensors can be installed on a single machine, yielding more comprehensive information and higher sensing resolution. Figure 32 shows ML-enabled, real-time material synthesis optimization for a frame spray system at Argonne. Research in progress is characterizing the particle chemistry, size, and size distribution in situ by laser imaging and a particle analyzer, and this characterization will be entered into an ML model together with computational fluid dynamics and thermodynamics simulations.

A version of this overall approach to data integration focused on the particle size distribution has enabled further and improved correlation with flame spray process parameters (Paulson et al. 2020). In the end, the entire process significantly reduced the number of experiments required for process optimization.

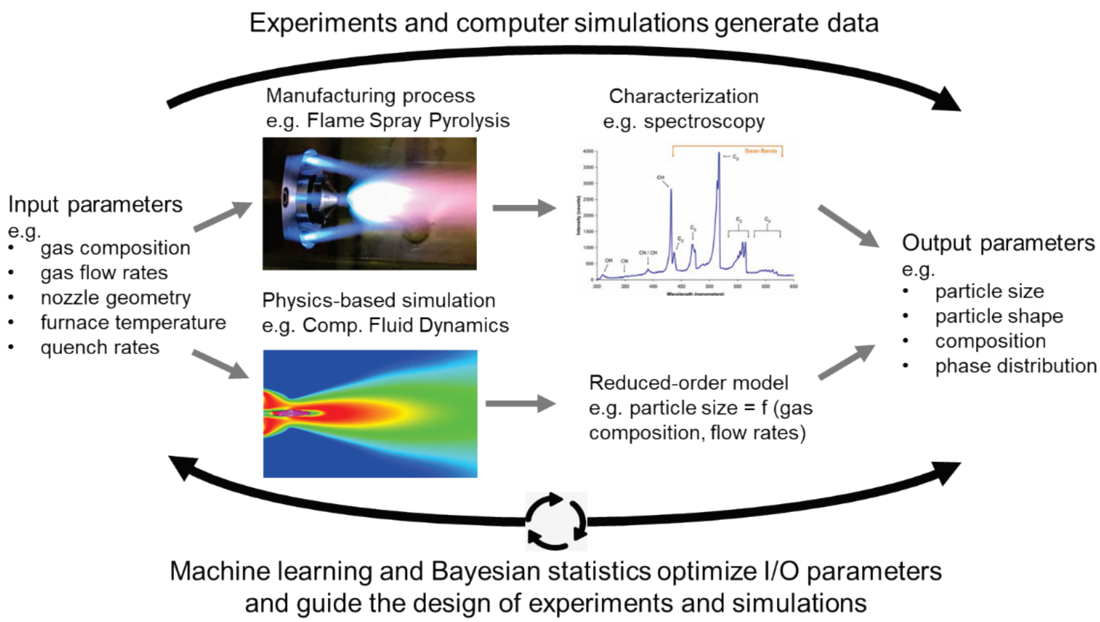


Figure 32. Schematic of the real-time optimization of a complex manufacturing process, with application to flame spray pyrolysis. (*Source*: Noah Paulson and Marius Stan, Argonne National Laboratory)

Recently, a new electronics fabrication technique was proposed based on printing technology. Sensors fabricated by this method have reduced size, weight, and cost. They can also be printed on or underneath the surface of machines, thus minimizing interference with machine operation. These sensors are suitable for massive implementation on the factory floor and are expected to enhance real-time monitoring and promote self-diagnostics and production process corrections.

4.3 Multiscale Predictive Theory and Modeling

Basic research underpinning advances in physics-based, ML and other optimization approaches in manufacturing include computational and theoretical chemistry, condensed matter and materials physics, and other chemical transformation sciences.

4.3.1 Physics-Based Models Across Multiple Scales

To understand, predict, and ultimately control matter and energy at the electronic, atomic, molecular, microstructural, and continuum levels, scientists need to investigate materials and chemistry at a combination of length and time scales that are characteristic to relevant physical and chemical phenomena. Therefore, experimental, theoretical, and computational methods must cover a wide range of space and time scales, starting with the nucleus and the electronic structure of individual or clustered atoms (Ångstrom scale), to molecular and nano/microstructural features, all the way to continuum properties of the sample (cm/ml). Along the time scale, the investigation domain ranges from excitations (ps) to nucleation of new phases and molecules (ns), all the way to diffusion (minutes, hours) and aging characteristic times (months, years).

Figure 33 shows examples of applicability domains of several theoretical and computational methods that operate at various time and length scales (Stan and Sarrao 2018). Some applicability limits are rooted in the physics of the associated phenomena. For example, density functional theory—a quantum mechanical method—is best suited for investigations at short times and in small volumes, where quantum effects are prominent. Other limitations result from computational aspects such as bandwidth (speed of communication) or available computer memory. For example, molecular dynamics simulations can account for all atoms in a *mole* of matter, but the time necessary to converge to solution is unrealistic, at present. In the next decades, some methods most likely will expand their investigation domains and other methods will disappear. With the accelerated advancement of theoretical and computational methodologies and capabilities, it is conceivable that quantum mechanical calculations will soon predict properties of polycrystalline, multicomponent materials at room and higher temperatures. In recent years, considerable progress has been made in bridging mesoscale to neighboring scales by either downscaling to atomistic simulation in materials science and chemistry or upscaling, for example, to the finite element simulations in structural engineering (Geers, Kouznetsova, and Brekelmans 2010). More detailed discussion of multiscale models and simulations for soft matter characterization can be found in the literature (Praprotnik, Site, and Kremer 2008).

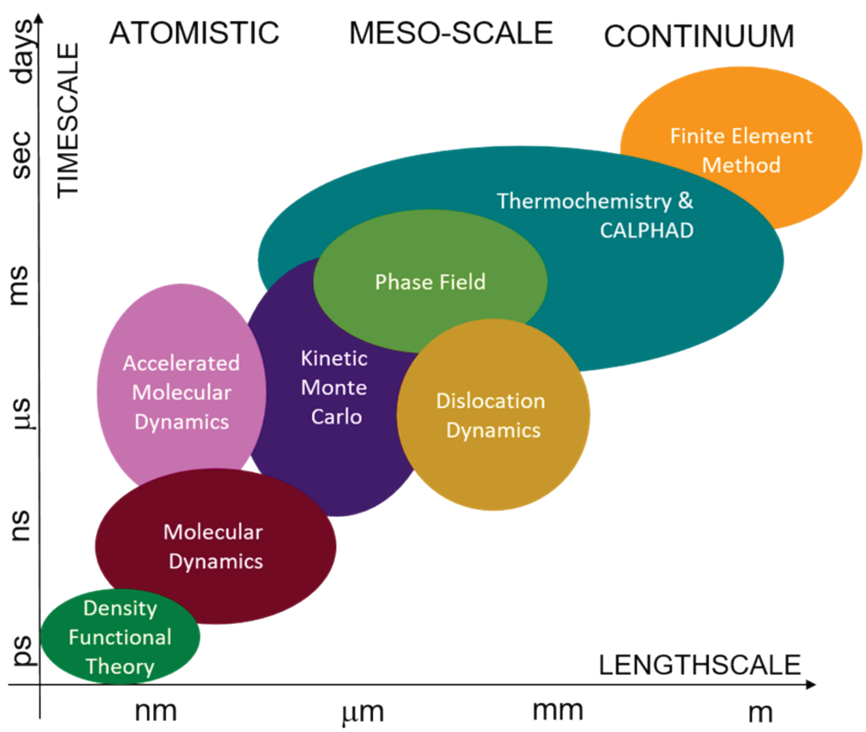


Figure 33. Multiscale theoretical and computational methods.
(*Source*: M. Stan, Discovery and design of nuclear fuels, *Mater. Today* 12(11), 20, 2009)

Mesoscale interactions yield complex architectures and phenomena that serve as the building blocks of macroscopic behavior. Science at the mesoscale builds on dramatic advances at the atomistic and nanoscale that the research community has produced in recent years and continues to produce. The mesoscale brings profound changes, replacing the atomic granularity of matter and the quantization of energy with continuous matter and energy. This length scale includes the onset of collective behavior, the interaction of coupled and competing degrees of freedom and the appearance of defects and fluctuations that alter the behavior of perfect structures. These emergent mesoscale phenomena represent a profound challenge for multiscale models. A series of articles exemplify the excitement and challenges that exist at the various scales (Jonušauskas, Juodkazis, and Malinauskas 2018; Short, M. P. and Yip 2015).

Even acknowledging the successes of multiscale methods in predicting the evolution of matter and the impact on properties, a number of questions remain (Stan and Sarrao 2018). For example, is 3D representation always necessary? There is no doubt that spatial distribution of features, especially at the mesoscale, is key to many phenomena such as heterogeneous microstructure evolution. Furthermore, computational methods will interface with in situ characterization methods such as 3D material tomography to collect input data and validation information. Therefore, the answer is yes, multidimensionality is important.

Another critical issue is the treatment of nonequilibrium processes that require the system to overcome energy barriers. Do multiscale computational methods capture nonequilibrium? The laws of physics and chemistry are universal and—when applied correctly—describe well both equilibrium and nonequilibrium processes. The success of multiscale methods, however, depends upon their ability to account for nonequilibrium mechanisms of microstructure and molecular evolution, such as nucleation and growth of new phases. Capturing nucleation of secondary phases is challenging because of the small characteristic length and time scales. Often, mesoscale methods advance the phases using as input critical nucleus information and nucleation rates from atomistic simulations such as density functional theory or molecular dynamics. Therefore, coupling scales may provide the optimal path forward.

Simulated microstructures are not replicas of experimentally observed microstructures but rather representations of reality. Similarly, experimental samples are not often replicas but “experimental models” of real materials or molecular systems. Therefore, the quantitative aspect of multiscale models is difficult to evaluate. In some instances, scientists qualitatively evaluate the dominant mechanisms behind complex phenomena as a preliminary step toward more rigorous, quantitative models and predictions. The long-term goal of modeling is to attain a high level of precision and accuracy in quantitatively representing real materials.

The computational efficiency of mesoscale methods is also under debate (Stan and Sarrao 2020). As with any computational method, going beyond the limits of applicability requires coupling with approaches that are valid at adjacent length or time scales. For example, the spatial and temporal correlations in mesoscale simulations require information exchange with atomistic and continuum methods. Additionally, adaptive time and mesh refinement can improve the precision but decrease the numerical efficiency of solving probability density function equations. Furthermore, the smallest time step or grid size is the limiting factor. In conclusion, multiscale simulations require highly scalable methods.

4.3.2 Uncertainties in Models and Physical Systems

Prediction of material properties in chemical, mechanical, and thermodynamic equilibrium is a fundamental part of the design of multicomponent materials. Answering the question of what phases are present, along with their percentages and their compositions at a particular overall composition, temperature, and pressure (X-T-P), provides basic information regarding the suitability of a material for a desired application (Geers 2010). Even in the development of metastable materials, it is critical to have an accurate understanding of the material properties over X-T-P space to identify and refine optimal processing routes. Furthermore, these basic quantities serve as building blocks for more complex predictive methods—for example, predicting the evolution of microstructures during processing (including diffusion and precipitation simulations) (Jonušauskas 2018).

Uncertainties in property data and model predictions derive from a number of sources, including both random and systematic errors in the measurement or simulation of quantities of interest used to calibrate the CALculation of PHAse Diagrams (CALPHAD models), as well as the choice of specific model forms used to describe the thermodynamic properties of the phases (Paulson and Bocklund 2019; Paulson and Jennings 2019). Although rarely addressed in a rigorous manner, data and model uncertainty can benefit or hinder materials design efforts.

Over the past several decades, a number of authors have presented frameworks for uncertainty quantification of material models through both frequentist paradigms (Praprotnik, Site, and Kremer 2008) and Bayesian paradigms (Paulson and Jennings 2019; Short, M. P. and Yip 2015; Stan 2009; Stan and Sarrao 2018). In most published work, uncertainty in the model parameters has been analytically propagated across scales through the moments of the parameter distributions, or numerically through samples of the distributions. The power of this representation of the uncertainty is limited. First, the parametric space is designed to quantify the uncertainty due to variation in one of three areas, temperature, composition, or pressure, but not a combination of these independent variables. It is not always clear which variables are selected, or if they will reasonably capture the uncertainty. Along similar lines, this approach does not explicitly address coupled phenomena, nor the potential for various phases to fall out of equilibrium for a certain subset of the distribution of model parameters.

Recent studies emphasized the use of Bayesian statistics to estimate and assess elemental material properties and models from published data (Bartók et al. 2017). This has significant importance and can be generalized to multicomponent materials. The example application to hafnium resulted in an uncertainty quantification that emphasized the challenges of using legacy experimental data and multiscale calculations. The optimized models compared well with other assessments, such as the HSC Chemistry software and handbook.[89] These models involve the effect of the inclusion or exclusion of individual data sets and the discussion of outliers. Figure 34 shows the uncertainty associated with the specific heat of hafnium across a large temperature domain.

[89] https://www.hsc-chemistry.com/

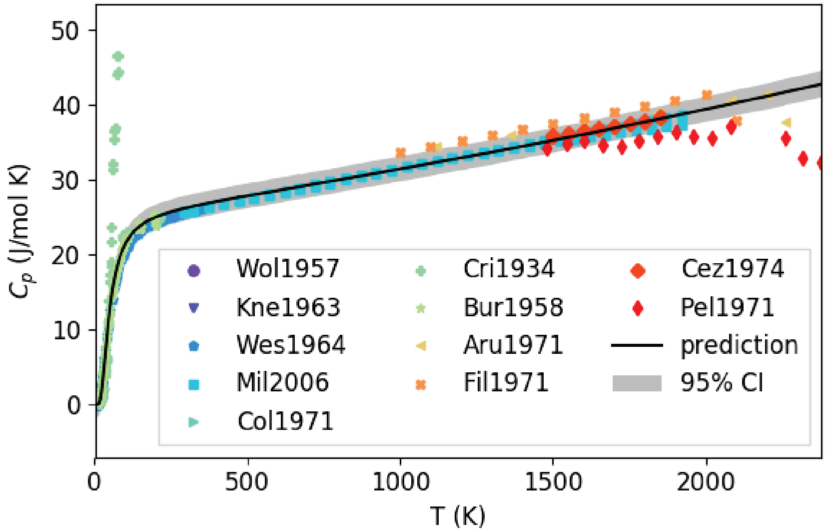


Figure 34. Uncertainty of hafnium specific heat calculated using Bayesian statistics and symbolic regression. The symbols represent experimental data sets described in Paulson and Jennings 2019. (*Source*: N. H. Paulson et al., *Int. J. Eng. Sci*. 142, 74, 2019; N. H. Paulson et al., *Acta Mater*. 174, 9, 2019)

Additional studies in this area resulted in several methods that extend beyond quantifying the uncertainty of individual properties in directions well suited to address materials design challenges. For example, Paulson et al. have developed an approach to gain a qualitative understanding of the uncertainty in multicomponent phase diagrams (Paulson and Bocklund 2019).

The most important result of that work for design applications is a method that extracts the phase stability of an X-T-P point, irrespective of the number of components under consideration, and returns the probabilities that each phase is stable. This same approach results in probability distributions for phase fractions, compositions, activities, sublattice site fractions, Gibbs energies, and their derivatives. Furthermore, the techniques are trivially extended to metastable equilibria where uncertainty quantification is critical. These methodologies and their relevance to materials design are demonstrated through a case study with the Cu-Mg binary system using CALPHAD model parameter samples obtained from Monte Carlo Markov Chain optimization in the ESPEI software.[90] Figure 35 shows the calculated uncertainty of Cu-Mg phases as function of composition at 800 K.

[90] https://espei.org/en/latest/

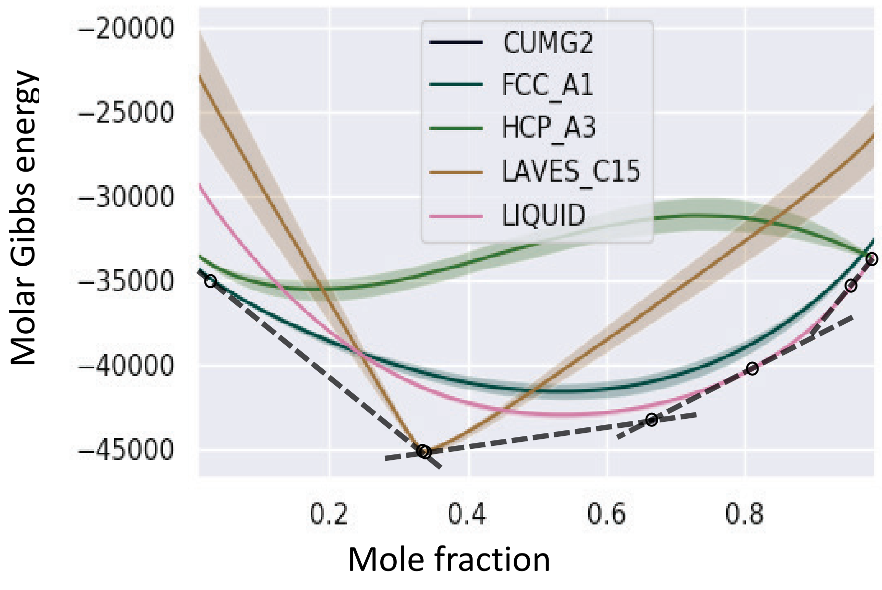


Figure 35. Gibbs free energy of several phases in the Cu-Mg system at 800 K and their uncertainty as a function of composition. (*Source*: N. H. Paulson et al., *Acta Mater*. 174, 9, 2019)

4.3.3 Machine Learning and Use of Data in Manufacturing

The digital thread of modern advanced manufacturing technologies is a data-rich environment. Driven by the Industry 4.0 revolution, manufacturing facilities are rapidly morphing into digital factories with the growing deployment of smart sensors (Zheng et al. 2018). Today, the amount of data produced at every step of the manufacturing process can retrace the entire history of every component at a spatiotemporal resolution never seen before. This data package—referred to as the digital clone or digital twin of the physical part—contains invaluable information that can be analyzed to gain a better understanding and ultimately control the manufacturing process. However, at this stage, the digital clone is data rich but information poor, as extracting knowledge systematically out of the sheer amount of data available is not common. Data analytics tools and techniques are being developed to explore such data, focusing on answering scientific questions that can help improve the quality, efficiency, and reliability of manufacturing processes. Additional focus is needed on big data challenges related to data quality, storage, and computing and the development of advanced analytics tools to process the data.

4.3.3.1 Application of Machine Learning to Manufacturing

The manufacturing digital thread captures information and data at each step of the manufacturing process. These steps include the material feedstock, geometrical design, manufacturing strategy optimization, process parameters optimization, development of new material, in situ quality control, and ex situ product characterization. Each data stream provides information regarding machine and product behavior. Recent research activities focus on leveraging ML techniques to better understand and improve each step. The following subsections provide some examples.

4.3.3.1.1 Feedstock Development

Depending on the final application of the manufactured component or product, new types of materials and chemicals will have to be developed to ensure performance. For either polymers or metals, the optimization of new material/chemical formulations is an intensive data exploration process that requires experimental data coupled with modeling and simulation techniques. More recently, ML has been employed to accelerate new discoveries (Liu, Y. et al. 2017).

4.3.3.1.2 Design

Component geometries have been created using established computer-aided design (CAD) software tools for decades, but the possibilities offered by advanced manufacturing technologies are breaking most of the historical design rules. Today, geometries are not regular and symmetrical. Instead they can look like bioinspired components, with designs that are counterintuitive to traditional, human-operator–driven designs (Libonati and Buehler 2017). The CAD software companies had to rely on generative design approaches to go beyond design limitations, bridging among materials science, finite element method analysis, and optimization and manufacturing technologies to produce optimal designs for the intended use.

The design and engineering of chemical processes involves stepwise stages of increasing complexity. Typically, designing a new chemical process involves the process design itself as well as the simulation of existing processes (Haydary 2019). Chemical process design involves both a series of steps and integration of the steps to achieve the complete manufacturing system (Smith 2005). In many cases, the design is a retrofit of an existing process as well as a design for new processes. The production scale, life cycle of the product, and product itself influence the chemical process design. For chemical processing, processes that efficiently use raw materials, energy, and water help prevent environmentally damaging waste and preserve resources (Smith 2005).

4.3.3.1.3 Manufacturing Process Optimization

Modern machines are operating on multiple axes: the tool and the manufactured object moves in three dimensions, and the addition of one or more robotized tools increase the complexity linearly. The optimization of the manufacturing process requires solving a multidimensional traveling salesman problem with a multiobjective target. In this scenario, the path planning for all tools and the orientation of the part must be considered. Humans operate at best in four dimensions; therefore, ML is preferred to fully automate the manufacturing process.

4.3.3.1.4 Quality Control

Manufacturing processes are observable using sensing modalities. It is therefore possible to detect flaws in situ, assess them rapidly, and reach conclusions on the viability of the product. For example, with imaging sensors, geometrical deviations compared with the intended CAD model or the presence of porosity or cracks can be detected. The amount of imagery is too large for an operator to review. In addition, the variability of data interpretation by different operators makes this an unreliable option. Instead, ML techniques are used to analyze the image data, highlighting detected features that can then be reviewed by the operator. Bringing augmented intelligence to the machine could help technicians be better operators of their machines.

4.3.3.1.5 Machine Behavior and Anomaly Detection

Manufacturing systems are subject to external and internal interactions. For example, aging elements of the machine degrade the performance; the overall temperature increase of a machine during operation could induce a physical change to the performance or product; or power fluctuation of the grid could interfere with the system. All these can be measured in situ using heterogeneous-sensing modalities. Coupling these data with ML can allow the performance of pattern-matching and pattern recognition tasks as an input to process control.

4.3.3.1.6 Surrogate for Modeling and Simulation Techniques

Physics-based modeling is computationally expensive, and it is not a realistic solution to simulate every product or part produced. However, portions of those models might be replaced by AI models to reduce the computation time. Learning from diverse scenarios, the AI models can capture the underlying physics rules and link a set of inputs and outputs. This requires a training campaign using multiple data streams, including modeling and simulation, experimental data, and domain knowledge.

4.3.3.1.7 Properties Prediction

The digital manufacturing data thread links feedstock, manufacturing process, and product performance. Relying on rigorous manufacturing campaigns to generate data to learn from, ML models can be trained to allow prediction of ultimate product performance to the feedstock and details of the actual manufacturing process.

4.3.3.1.8 Transfer Learning

Machines are constantly evolving in terms of build volume, technology, and speed, for example. For a manufacturing company, acquiring a new system is often seen as a major learning experience. However, all systems have commonalities in the way the feedstock behaves during the manufacturing process or how the machine operates in general. These similarities allow AI model architecture to transfer to the new system. In use, this corresponds to transferring the encoded domain knowledge to the new machine, with minimal retraining of the AI model with the data from the new system to quickly be fully operational.

4.3.3.2 Big Data in Manufacturing

Traditionally, modeling and simulation activities were the path forward toward understanding or planning manufacturing processes. With the complexity of modern systems, this approach is reaching its limits. The symbiotic combination of experiments, modeling/simulation, and data-driven techniques offer a higher prospect of success. All three categories are, in general, large producers of data. Modeling and simulation techniques provide data that can be recalculated by rerunning the models, whereas sensor-equipped manufacturing systems and material characterization platforms produce terabytes of data in short periods of time that cannot be regenerated and require storage. For advanced simulations, there may also drivers to retain data for future use/reference.

Running modeling and simulation algorithms can require an HPC system. Pushed to the extreme in a manufacturing setup, those systems will need to produce results while the manufacturing process is ongoing, such as for feedback loop control where the simulation calculation is the correction mechanism for a manufacturing failure detected in situ. Computing power is paramount to achieve such a level of control, but it does not necessarily mean that an HPC platform would handle the entire workload. An option is to use a distributed computing configuration, where independent tasks can be performed locally and then solutions are transferred to the HPC system for final simulation. Currently, it is envisioned that distributed computing with embedded systems running AI at the edge (e.g., within the sensor system) will become part of the standard machine offering for real-time[108] quality control tasks (Deng, S. et al. 2020; Wang, X. et al. 2020). Future development may include non-AI-based tasks running at the machine and as part of the HPC setup. This configuration will require standards for verification and validation as well as protocols for software upgrades.

For other data-driven techniques, attention is needed to data storage, curation, and assessment. Preserving the integrity of data packages for each component over multiple decades is critical for applications for the aerospace or nuclear industries. This need introduces additional challenges for data storage, lossless data compression, and eventually domain-driven data selection to balance data preservation with related infrastructure costs. The solution includes a hardware component for storage and network infrastructures, and a software component focusing on data manipulation and selection, as well as multiple cybersecurity aspects. Data storage is a mix between local architectures and cloud-based platforms, but emerging cybersecurity issues and the required bandwidth to transfer such data place the cloud as a secondary solution. Network infrastructures, both wired and wireless, are needed with the broader use of the internet (e.g., the Internet of Things concept), and therefore the risk of network intrusion is increased. Novel cybersecurity countermeasures will be required to protect intellectual property and the digital clone's integrity.

The quality and relevance of the data are also important. Nondestructive evaluation and property characterization need to reach high levels of precision and accuracy to inform the process. Imaging systems and image reconstruction techniques (such as those used in computed tomography) will need to be adapted to detect all critical defects and reduce noise in the data. These goals result in an increased need for standardization of testing and uncertainty quantification.

4.4 Safety and Workflow Considerations in Manufacturing

4.4.1 Environmental Health and Safety

Advances in manufacturing require recognition of potential environmental, safety, and health (ESH) issues. For example, ESH for AM is challenging because of the variety of AM processes, the increase of innovative materials, and postprocessing applications. The Occupational Safety and Health Administration (OSHA) does not currently publish material concerning rapid prototyping or AM. These types of manufacturing resemble chemical laboratories because materials used include flammable, toxic chemicals, and thus include the issues associated with chemical waste (Short, D. B., Sirinterlikci, Badger, and Artieri 2015). In many instances, existing standards and regulations for the hazards that are well understood can be translated to advanced manufacturing approaches. However, partially or completely new hazards will likely pose greater challenges for hazard characterization, exposure assessment, and consequently risk analysis (Roth et al. 2019).

Many well-characterized hazards already exist in manufacturing. They are well understood, with guidance from OSHA, the National Fire Protection Association (NFPA), the American National Standards Institute (ANSI), and the American Conference of Governmental Industrial Hygienists (ACGIH) on how to assess and control these hazards. However, as the use of AM expands, additional development is needed in the areas of exposure assessment techniques, emissions, effectiveness of controls, and worker exposure to hazards. The development of knowledge and assessment techniques from the nanotechnology occupational safety and health field may be a model for ESH developments and assessment in AM (Roth et al. 2019).

As nanotechnologies were introduced, a mixture of old and new processes, novel environments, and a change of pace made characterizing hazards and assessing risk ongoing challenges (Schulte et al. 2016). Exposure assessments in nanotechnology manufacturing processes today combine traditional industrial hygiene methods and new sampling techniques. The Nanotechnology Research Center, through the National Institute for Occupational Safety and Health, outlines exposure assessment strategies based on Nanomaterial Exposure Assessment Technique 2.0. This strategy consists of the following:

- Full state and task-based monitoring;
- Personal and area air sampling;
- Chemical and/or gravimetric analysis;
- Electron microscopy (if unbound nanomaterial is present), including transmission electron microscopy or scanning electron microscopy sampling for identification, sizing, and morphology; and
- Data logging with real-time aerosol instruments (Roth 2018).

In addition to using the nanotechnology field (Engeman et al. 2013; Hull and Bowman 2018) for a model to develop new guidance on AM, additional elements need to be considered, including the following.[91]

- Review of current codes and regulations applicable to the manufacturing process.
    - Codes to consider include (not an exhaustive list):
        - International Building Code for Occupancy classifications and chemical storage limits, OSHA 29 CFR 1910.1450 Exposure to Hazardous Chemicals in Laboratories standard, Prudent Practices in the Laboratory, Handling and Management of Chemical Hazards, NFPA 30 Flammable and Combustible Liquids, NFPA 45 Fire Protection for laboratories using chemicals, NFPA 101 Life Safety Code, NFPA 484 Standard for Combustible Metals, Metal Powders, and Metal Dusts, NEC 70 Articles 500-506, ANSI, Z136.1 Safe use of Lasers, and NEC 70 Articles 500-506.
- Risk assessment of the specific manufacturing technologies.
- Review of the environmental, chemical, and health hazards of the materials used in all facets of the manufacturing processes.
    - Review of the equipment manuals, Safety Data Sheets, research studies, and toxicological studies.

Many of the ESH concerns that occur in AM are due to the use of fine metal powder feedstocks (usually 15 to 100 micrometers in diameter) because they have a high surface-area-to-volume ratio that increases their reactivity relative to another feedstock that is larger in diameter. This increased reactivity can make many of these feedstocks flammable or even explosive when aerosolized and can make inhalation of

[91] https://www.cdc.gov/niosh/topics/advancedmnf/default.html

powder particles dangerous for operators (Graff et al. 2017). At least one case of an explosion related to AM of aluminum has been documented.[92]

Manufacturing and AM have become a blend of old and new technologies. The hazards that need to be considered when beginning a new manufacturing process must be evaluated during facility design or when changes occur. During the design, it is important to also include and discuss feedstocks, operation, and postprocessing hazards. The prominent hazards associated with manufacturing and AM are listed in Table 8 (Scime et al. 2018).

Table 8. General Hazards to Consider for Manufacturing and Additive Manufacturing Process (not an exhaustive list) (*Source:* Amy Harris, Argonne National Laboratory)

Chemical Hazards	Routes of Exposure	Physical Hazards	Environmental Hazards	Work Environment
Physical form of material, solid, liquid, gas, powder	Inhalation/ absorption/ingestion	Electrical—static/shock	Air emissions-volatile organic compounds (VOCs)	Transportation—movement of equipment, axillary equipment, samples, etc. Adequate egress and aisleways needed
Flammable/combustible	Inhalation/ absorption/ingestion	Ergonomics	Wastewater discharges	Storage—designated storage areas and powder processing rooms
Corrosives acid/bases	Inhalation/ absorption/ingestion	Material handling	EPCRA reportable chemicals	Ventilation recommended
Toxic	Inhalation/ absorption/ingestion	Pinch points/moving/rotating equipment.	Fire particles and ultra-fine particle emissions	Lighting
Reactive—air, water sensitive (pyrophoric, peroxide former)	Inhalation/ absorption/ingestion	Temperature hazards—thermal and cryogenic	VOC emissions	Temperature/ humidity/worker comfort
Carcinogens	Inhalation/ absorption/ingestion	Noise	Fires	Scheduling/stress
Unbound nanomaterials	Inhalation/ingestion	Non-ionizing radiation/Lasers /UV	Waste/spills	Contamination
Asphyxiants—compressed gases, cryogens-LN2 or argon	Inhalation/absorption	Confined spaces		Workflow
Dusts/particulates	Inhalation/ingestion			Co-located hazards

EPCRA = Emergency Planning and Community Right-to-Know Act

4.4.2 Workflow and Facility Design

Workflow analysis is an increasingly important area of investigation for advanced manufacturing facility design. For example, Carnegie Mellon University's NextManufacturing Center recently completed a

[92] https://www.osha.gov/news/newsreleases/region1/05202014

new metal AM laboratory as part of a major renovation.[93] They found a lack of both community-wide best safety practices and public institutional knowledge regarding workflow considerations. For these reasons, proper laboratory facility design emerged as a major component of their research success, showing why safety and workflow considerations for manufacturing begin with the design of the facility (Scime et al. 2018).

Many times, these issues are not just encountered in building a new facility but must be considered in retrofitting an existing facility tailored to accommodate different types of manufacturing. The operators, ESH department, engineers/architects, and building infrastructure staff must work together to understand the relevant national, state, and local safety codes and regulations to ensure compliance and worker safety. This allows each individual group's expertise to be considered in the design of the facility. The ESH department can identify the hazards and relevant codes, and the design engineers and architects can identify related ventilation, heating/cooling, temperature/humidity, electrical power, emergency power, and water temperature needs or constraints. The operators will know the equipment specifications and can identify workflow patterns and issues that could arise to help lay out the facility efficiently. These approaches will maximize innovation for the facility design. Effective and efficient layout of a facility will improve output and reduce overall costs. It will improve the safety culture as a result of the operators providing stakeholder input throughout the whole process.

In conclusion, efficient setup and operation of advanced manufacturing, including AM equipment/processes, requires identification of both well-characterized hazards (Table 8) and those that are novel. If the appropriate people are involved throughout the design and build process/or renovation, this positively affects the safety culture because of operator input, in addition to increasing the workflow of the facility. This in turn increases production, effectiveness, and efficiency of the research conducted in the facility, pushing the boundaries to the next level of manufacturing technology.

[93] https://engineering.cmu.edu/next/

References

Allanore, A.; Yin, L.; and Sadoway, D. R. (2013). “A New Anode Material for Oxygen Evolution in Molten Oxide Electrolysis.” *Nature*, 497, 353.

Allwood, J.; Gutowski, T.; Ashby, M.; and Worrell, E. (2011. “Material Efficiency: A White Paper.” *Resources Conservation and Recycling* 55(3), 362.

Ansari, F.; Erol, S.; and Sihn, W. (2018). “Rethinking Human-Machine Learning in Industry 4.0: How Does the Paradigm Shift Treat the Role of Human Learning?” *Procedia Manufacturing*, 23, 117.

Antolini, E. (2018). “The Oxygen Reduction on Pt-Ni and Pt-Ni-M Catalysts for Low-Temperature Acidic Fuel Cells: A Review.” *International Journal of Energy Research*, 42, 3747.

Asadi, J., and Farahani, F. J. (2018). “Optimization of Dimethyl Ether Production Process Based on Sustainability Criteria Using a Homotopy Continuation Method.” *Computers & Chemical Engineering*, 115, 161.

Balaram, V. 2019. “Rare Earth Elements: A Review of Applications, Occurrence, Exploration, Analysis, Recycling, and Environmental Impact.” *Geoscience Frontiers*, 10, 1285.

Barber, J.; and Tran, P. D. (2013). “From Natural to Artificial Photosynthesis,” *Journal of the Royal Society Interface*, 10, 20120984.

Bartók, A. P.; De, S.; Poelking, C.; Bernstein, N.; Kermode, J. R.; Csányi, G.; and Ceriotti, M. (2017). “Machine Learning Unifies the Modeling of Materials and Molecules.” *Science Advances*, 3, e1701816.

Bauernhansl, T. (2016). “WGP-Standpunkt Industrie 4.0.” WGP, Wissenschaftliche Gesellschaft für Produktionstechnik.

Becker, R. A. (2016). “Water Use and Conservation in Manufacturing: Evidence from US Microdata.” *Water Resources Management,* 30, 4185.

Beekman, G. B. (1998). “Water Conservation, Recycling and Reuse.” *International Journal of Water Resources Development,* 14, 353.

Bereznoy, A. 2019. “Changing Competitive Landscape Through Business Model Innovation: The New Imperative for Corporate Market Strategy.” *Journal of the Knowledge Economy* 10, 1362.

Bernhardt, D.; and Reilly, I. I. (2019). “J. Mineral Commodity Summaries 2019,” US Geological Survey, Reston, VA.

Biernacki, J. J.; Bullard, J. W.; Sant, G.; Brown, K.; Glasser, F. P.; Jones, S.; Ley, T.; Livingston, R.; Nicoleau, L.; and Olek, J. (2017). “Cements in the 21st Century: Challenges, Perspectives, and Opportunities.” *Journal of the American Ceramic Society,* 100, 2746.

Bland, A.; Khzouz, M.; Statheros, T.; and Gkanas, E. I. (2017). “PCMs for Residential Building Applications: A Short Review Focused on Disadvantages and Proposals for Future Development.” *Buildings*, 7, 78.

Bogaerts, A.; and Neyts, E. C. (2018). "Plasma Technology: An Emerging Technology for Energy Storage." *ACS Energy Letters*, 3, 1013.

Boisse, P.; Hamila, N.; and Madeo, A. (2017). *Analysis of Defect Developments in Composite Forming. The Structural Integrity of Carbon Fiber Composites* (P. Beaumont, C. Souti and A. Hodzic (ed.), Springer, Cham).

Bonificio, W. D.; and Clarke, D. R. (2016). "Rare-Earth Separation Using Bacteria." *Environmental Science & Technology Letters*, 3, 180–84.

Borangiu, T.; Trentesaux, D.; Thomas, A.; Leitão, P.; and Barata, J. (2019). "Digital Transformation of Manufacturing Through Cloud Services and Resource Virtualization." *Computers in Industry*, 108, 150.

Borra, C. R.; Pontikes, Y.; Binnemans, K.; and Van Gerven, T. (2015). "Leaching of Rare Earths from Bauxite Residue (Red Mud)." *Minerals Engineering*, 76, 20.

Brueske, S.; Kramer, C.; and Fisher, A. (2015). "Bandwidth Study on Energy Use and Potential Energy Saving Opportunities in US Chemical Manufacturing." *Energetics*. No. DOE/EE-1229.

Castro, C. E.; Kilchherr, F.; Kim, D.-N.; Shiao, E. L.; Wauer, T.; Wortmann, P.; Bathe, M.; and Dietz, H. (2011). "A Primer to Scaffolded DNA Origami." *Nature Methods*, 8, 221.

Chabi, S.; Papadantonakis, K. M.; Lewis, N. S.; and Freund, M. S. (2017). "Membranes for Artificial Photosynthesis." *Energy & Environmental Science*, 10, 1320–38.

Chatterjee, T.; Mitchell, C. A.; Hadjiev, V. G.; and Krishnamoorti, R. (2007). "Hierarchical Polymer–Nanotube Composites." *Advanced Materials*, 19, 3850.

Chavarría-Barrientos, D.; Batres, R.; Wright, P. K.; and Molina, A. (2018). "A Methodology to Create a Sensing, Smart and Sustainable Manufacturing Enterprise." *International Journal of Production Research*, 56, 584.

Chen, D.; Heyer, S.; Ibbotson, S.; Salonitis, K.; Steingrímsson, J. G.; and Thiede, S. (2015). "Direct Digital Manufacturing: Definition, Evolution, and Sustainability Implications." *Journal of Cleaner Production*, 107, pp. 615.

Cheng, X.-B.; Zhang, R.; Zhao, C.-Z.; and Zhang, Q. (2017). "Toward Safe Lithium Metal Anode in Rechargeable Batteries: A Review." *Chemical Reviews*, 117, 10403.

Cherupalli, H.; Duwe, H.; Ye, W.; Kumar, R.; and Sartori, J. (2017). "Determining Application-Specific Peak Power and Energy Requirements for Ultra-Low-Power Processors." *ACM Transactions on Computer Systems*, 35, 1.

Cimini, C.; Pirola, F.; Pinto, R.; and Cavalieri, S. (2020). "A Human-in-the-Loop Manufacturing Control Architecture for the Next Generation of Production Systems." *Journal of Manufacturing Systems,* 54, 258.

Compton, B. G.; and Lewis, J. A. (2014). "3D-Printing of Lightweight Cellular Composites." *Advanced Materials*, 26, 5930.

Cooper, S. J.; Jannik Giesekam, G.; Hammond, G. P.; Norman, J. B.; Owen, A.; Rogers, J. G.; and Scott, K. (2017). "Thermodynamic Insights and Assessment of the 'Circular Economy'," *Journal of Cleaner Production*, 162, 1356.

Cordero, Z. C.; Knight, B. E.; and Schuh, C. A. (2016). "Six Decades of the Hall–Petch Effect—a Survey of Grain-Size Strengthening Studies on Pure Metals." *International Materials Reviews,* 61, 495.

Cresko, J. (2017). "An Overview of AMO Clean Water Analysis." Presentation at AMO Clean Water Workshop, Cleveland, OH, August 23.

Cresko, J. (2019). "Opportunities for a More Productive and Circular Economy." Lehigh University, Horizon Seminar November 12.

Daehn, K. E.; Cabrera Serrenho, A.; and Allwood, J. M. (2017). "How Will Copper Contamination Constrain Future Global Steel Recycling?" *Environmental Science & Technology*, 51, 6599.

Daniel, C.; Crowson, F.; Krumdick, G. K.; Prasher, R.; Ulsh, M.; and Wood III, D. L. (2018). "FY2017 Annual Report: Roll-to-Roll Advanced Materials Manufacturing DOE Laboratory Consortium Project." Oak Ridge National Laboratory (ORNL), Oak Ridge, TN.

Daniel, C.; Wood III, D. L.; Krumdick, G.; Ulsh, M.; Battaglia, V.; and Crowson, F. (2019). "Roll-to-Roll Advanced Materials Manufacturing DOE Laboratory Collaboration-FY2018 Final Report." Oak Ridge, TN: Oak Ridge National Laboratory (ORNL).

Daniel, C.; Wood III, D. L.; Krumdick, G.; Ulsh, M.; and Srinivasan, V. (2016). "Roll-to-Roll Advanced Materials Manufacturing DOE Lab Consortium-FY16 Annual Report." Oak Ridge, TN: Oak Ridge National Laboratory (ORNL).

Dano, S. 1966. *Industrial Production Models: A Theoretical Study*, Springer-Verlag: Wien.

Das, S.; Warren, J.; Wes, D. S.; and Schexnayder, S. M. (2016). *Global Carbon Fiber Composites Supply Chain Competitiveness Analysis.* Technical Report ORNL/SR-2016/100 | NREL/TP-6A50-66071).

de la Hoz, A.; Diaz-Ortiz, A.; and Moreno, A. (2005). "Microwaves in Organic Synthesis. Thermal and Non-Thermal Microwave Effects." *Chemical Society Reviews*, 34, 164.

Decher, G. (1997). "Fuzzy Nanoassemblies: Toward Layered Polymeric Multicomposites," *Science*, 277, 1232.

den Hollander, M. C.; Bakker, C. A.; and Hultink, E. J. (2017). "Product Design in a Circular Economy: Development of a Typology of Key Concepts and Terms." *Journal of Industrial Ecology*, 21, 517.

Deng, R.; Xia, Z.; Sun, R.; Wang, S.; and Sun, G. (2020). "Nanostructured Ultrathin Catalyst Layer with Ordered Platinum Nanotube Arrays for Polymer Electrolyte Membrane Fuel Cells." *Journal of Energy Chemistry*, 43, 33.

Deng, S.; Zhao, H.; Fang, W.; Yin, J.; Dustdar, S.; and Zomaya, A. Y. (2020). "Edge Intelligence: The Confluence of Edge Computing and Artificial Intelligence." *IEEE Internet of Things Journal*.

DOE (Department of Energy) (2011). "Critical Materials Strategy." Washington, D.C. https://www.energy.gov/sites/prod/files/DOE_CMS2011_FINAL_Full.pdf.

DOE (Department of Energy) (2006). "Energy Demands on Water Resources: Report to Congress on the Interdependency of Energy and Water." Washington, D.C. https://www.circleofblue.org/wp-content/uploads/2010/09/121-RptToCongress-EWwEIAcomments-FINAL2.pdf.

DOE (Department of Energy) (2014a). "Manufacturing Energy and Carbon Footprint." Washington, D.C. https://energy.gov/eere/amo/manufacturing-energy-and-carbon-footprints-2014-mecs.

DOE (Department of Energy) (2014b). "The Water-Energy Nexus: Challenges and Opportunities." Washington, D.C. https://www.energy.gov/sites/prod/files/2014/07/f17/Water%20Energy%20Nexus%20Full%20Report%20July%202014.pdf.

Dhar, N. K.; Wijewarnasuriya, P. S.; and Dutta, A. (2018). "Energy Harvesting and Storage: Materials, Devices, and Applications VIII." *International Society for Optics and Photonics* 10663.

Dieter, C. A.; Maupin, M. A.; Caldwell, R. R.; Harris, M. A.; Ivahnenko, T. I.; Lovelace, J. K.; Barber, N. L.; and Linsey, K. S. (2018a). "Estimated Use of Water in the United States in 2015." U.S. Geological Survey; Circular.

Dieter, C. A.; Maupin, M. A.; Caldwell, M. A.; Harris, T. I.; Ivahnenko, J. K.; Lovelace, N. L.; Barber; and Linsey, K.S. (2018b). "Estimated Use of Water in the United States in 2015." U.S. Geological Survey, No. 1441.

Dogutan, D. K.; and Nocera, D. G. (2019). "Artificial Photosynthesis at Efficiencies Greatly Exceeding that of Natural Photosynthesis." *Accounts of Chemical Research*, 52, 3143.

Dolatabadi, J. E. N.; and de la Guardia, M. (2011). "Applications of Diatoms and Silica Nanotechnology in Biosensing, Drug and Gene Delivery, and Formation of Complex Metal Nanostructures." *TrAC Trends in Analytical Chemistry*, 30, 1538.

Dupont, D.; and Binnemans, K. (2015). "Rare-Earth Recycling Using a Functionalized Ionic Liquid For the Selective Dissolution and Revalorization of Y2O3:Eu3+ from Lamp Phosphor Waste." *Green Chemistry*, 17, 856.

EIA (Energy Information Administration) (2019). "Annual Energy Outlook 2019 with Projections to 2050." Washington, D.C. https://www.eia.gov/outlooks/aeo/pdf/aeo2019.pdf.

EIA (Energy Information Administration) (2013). "Number of Establishments by Usage of General Energy-Saving Technologies, 2010." Washington, D.C. https://www.eia.gov/consumption/manufacturing/data/2010/pdf/Table8_2.pdf.

Eigler, D. M.; and Schweizer, E. K. (1990). "Positioning Single Atoms with a Scanning Tunnelling Microscope." *Nature*, 344, 524.

Endo, M.; Takeuchi, K.; Igarashi, S.; Kobori, K.; Shiraishi, M.; and Kroto, H. W. (1993). "The Production and Structure of Pyrolytic Carbon Nanotubes (PCNTs)." *Journal of Physics and Chemistry of Solids,* 54, 1841.

Engeman, C. D.; Baumgartner, L.; Carr, B. M.; Fish, A. M.; Meyerhofer, J. D.; Satterfield, T. A.; Holden, P. A.; and Harthorn, B. H. (2013). "The Hierarchy of Environmental Health and Safety Practices in the US Nanotechnology Workplace." *Journal of Occupational and Environmental Hygiene*, 10, 487.

EPA (Environmental Protection Agency) (2015). "What Is Green Infrastructure?" Washington, D.C. https://www.epa.gov/green-infrastructure/what-green-infrastructure.

Fortier, S. M; Nassar, N. T.; Lederer, G. W.; Brainard, J.; Gambogi, J.; and McCullough, E. A. (2018). "Draft Critical Mineral List—Summary of Methodology and Background Information." U.S. Geological Survey Technical Input Document in Response to Secretarial Order No. 3359 (No. 2018-1021), https://pubs.er.usgs.gov/publication/ofr20181021.

Fountaine, K. T.; Lewerenz, H. J.; and Atwater, H. A. (2016). "Efficiency Limits for Photoelectrochemical Water-Splitting." *Nature Communications*, 7, 1.

Friedrich, J.; Scheifele, S.; Verl, A.; and Lechler, A. (2014). "Flexible and Modular Control and Manufacturing System," *Procedia Cirp*, 33, 115.

Fruehan, R. J.; Fortini, O.; Paxton, H. W.; and Brindle, R. (2000). "Theoretical Minimum Energies to Produce Steel." Report to the U.S. Department of Energy, Office of Industrial Technologies. Washington, D.C.

Gavidel, S. Z.; Lu, S.; and Rickli, J. L. (2019). "Performance Analysis and Comparison of Machine Learning Algorithms for Predicting Nugget Width of Resistance Spot Welding Joints." *The International Journal of Advanced Manufacturing Technology*, 105, 3779.

Gedye, R. N.; Smith, F. E.; and Westaway, K. C. (1988). "The Rapid Synthesis of Organic Compounds in Microwave Ovens." *Canadian Journal of Chemistry*, 66, 17.

Geers, M. G. D.; Kouznetsova, V. G.; and Brekelmans, W. A. M. (2010). "Multi-Scale Computational Homogenization: Trends and Challenges," *Journal of Computational and Applied Mathematics*, 234, 2175.

Ghobakhloo, M. (2018). "The Future of Manufacturing Industry: A Strategic Roadmap Toward Industry 4.0." *Journal of Manufacturing Technology Management*, 29, pp. 910.

Govignon, Q.; Kazmi, S.; Hickey, C.; and Bickerton, S. (2011). "Control of Laminate Quality for Parts Manufactured Using the Resin Infusion Process." *18th International Conference on Composite Materials,* August 2011, Jeju, South Korea.

Graff, P.; Ståhlbom, B.; Nordenberg, E.; Graichen, A.; Johansson, P.; and Karlsson, H. (2017). "Evaluating Measuring Techniques for Occupational Exposure During Additive Manufacturing of Metals: A Pilot Study." *Journal of Industrial Ecology*, 21, S120.

Granade, H. C.; Creyts, J.; Derkach, A.; Farese, P.; Nyquist, S.; and Ostrowski, K. (2009). *Unlocking Energy Efficiency in the US Economy.* McKinsey & Company.

Gueroult, R.; Rax, J.-M.; and Fisch, N. J. (2018). "Opportunities for Plasma Separation Techniques In Rare Earth Elements Recycling." *Journal of Cleaner Production*, 182, 1060.

Guinard, D.; Trifa, V.; Mattern, F.; and Wilde, E. (2011). "From the Internet of Things to the Web of Things: Resource-Oriented Architecture and Best Practices." *Architecting the Internet of Things*. Springer 97–129.

Gutmann, B.; Cantillo, D.; and Kappe, C. O. (2015). "Continuous-Flow Technology—A Tool for the Safe Manufacturing of Active Pharmaceutical Ingredients," Angewandte Chemie International Edition, 54, 6688.

Hanft, D.; Glosse, P.; Denneler, S.; Berthold, T.; Oomen, M.; Kauffmann-Weiss, S.; Weis, F.; Häßler, W.; Holzapfel, B.; and Moos, R. (2018). "The Aerosol Deposition Method: A Modified Aerosol Generation Unit to Improve Coating Quality," *Materials*, 11, 1572.

Harlow, J. E; Ma, X.; Li, J.; Logan, E.; Liu, Y.; Zhang, N.; Ma, L.; Glazier, S. L.; Cormier, M. M. E.; and Genovese, M. (2019). "A Wide Range of Testing Results on an Excellent Lithium-Ion Cell Chemistry to be Used as Benchmarks for New Battery Technologies." *Journal of The Electrochemical Society,* 166, A3031.

Hartzler, D. A.; Bhatt, C. R.; Jain, J. C.; and McIntyre, D. L. (2019). "Evaluating Laser-Induced Breakdown Spectroscopy Sensor Technology for Rapid Source Characterization of Rare Earth Elements." *Journal of Energy Resources Technology*, 141, 070704.

Hawkins, T. R.; Singh, B.; Majeau-Bettez, G.; and Strømman, A. H. 2013. "Comparative Environmental Life Cycle Assessment of Conventional and Electric Vehicles." *Journal of Industrial Ecology,* 17, 53.

Hawley, W. B.; and Li, J. (2019). "Electrode Manufacturing for Lithium–Ion Batteries—Analysis of Current and Next Generation Processing." *Journal of Energy Storage*, 25, 100862.

Haydary, J. (2019). *Chemical Process Design and Simulation: Aspen Plus and Aspen Hysys Applications*. John Wiley & Sons.

Helbig, C.; Kolotzek, C.; Thorenz, A.; Reller, A.; Tuma, A.; Schafnitzel, M.; and Krohns, S. (2017). "Benefits of Resource Strategy for Sustainable Materials Research and Development." *Sustainable Materials and Technologies*, 12, 1.

Hess, H. 2006. “Self-Assembly Driven by Molecular Motors.” *Soft Matter*, 2, 669.

Hirota, K.; Nakamura, H.; Minowa, T.; and Honshima, M. (2006). “Coercivity Enhancement by the Grain Boundary Diffusion Process to Nd–Fe–B Sintered Magnets.” IEEE Transactions on Magnetics, 42, 2909.

Hofmann, M.; Hofmann, H.; Hagelüken, C.; and Hool, A. (2018). “Critical Raw Materials: A Perspective from the Materials Science Community.” *Sustainable Materials and Technologies*, 17, e00074.

Horikoshi, S.; and Serpone, N. (2014). “Role of Microwaves in Heterogeneous Catalytic Systems.” *Catalysis Science & Technology*, 4, 1197.

Hu, L.; Kim, H. S.; Lee, J.-Y.; Peumans, P.; and Cui, Y. (2010). “Scalable Coating and Properties of Transparent, Flexible, Silver Nanowire Electrodes.” *ACS Nano,* 4, 2955.

Hu, Y.; Florek, J.; Larivière, D.; Fontaine, F.-G.; and Kleitz, F. (2018). “Recent Advances in the Separation of Rare Earth Elements Using Mesoporous Hybrid Materials.” *The Chemical Record*, 18, 1261.

Huellen, M.; Schrade, C.; Wilhelm, U.; and Zulhan, Z. (2006). “EAF-Based Flat-Steel Production Applying Secondary Metallurgical Processes.” Ironmaking Steelmaking Conference, Linz, Austria.

Hull, M.; and Bowman, D. (eds.) (2018). *Nanotechnology Environmental Health and Safety: Risks, Regulation, and Management*. Elsevier, 2nd edition.

Huntzinger, D. N.; and Eatmon, T. D. (2009). “A Life cycle Assessment of Portland Cement Manufacturing: Comparing the Traditional Process with Alternative Technologies.” *Journal of Cleaner Production*, 17, 668.

IEA (International Energy Agency) (2010). “Energy Technology Perspectives: Scenarios and Strategies to 2050.” OECD Publishing, Paris, France. https://doi.org/10.1787/energy_tech-2010-en.

IEA (International Energy Agency) (2013). “International Council of Chemical Associations, and DECHEMA. Technology Roadmap—Energy and GHG Reductions in the Chemical Industry via Catalytic Processes,” Technical Report. Paris: IEA Publications. https://www.iea.org/reports/technology-roadmap-energy-and-ghg-reductions-in-the-chemical-industry-via-catalytic-processes.

IRENA (2018). “Renewable Capacity Statistics.” https://www.irena.org/-/media/Files/IRENA/Agency/Publication/2018/Mar/IRENA_RE_Capacity_Statistics_2018.pdf.

Ivanov, D.; and Dolgui, A. (2020). “A Digital Supply Chain Twin for Managing the Disruption Risks and Resilience in the Era of Industry 4.0.” *Production Planning & Control*, 1.

Izatt, S. R.; McKenzie, J. S.; Izatt, N. E.; Bruening, R. L.; Krakowiak, K. E.; and Izatt, R. M. (2016). “Molecular Recognition Technology: A Green Chemistry Process for Separation of Individual Rare Earth Metals.” White Paper on Separation of Rare Earth Elements, February 20, 1.

Jackson, C. D.; and Naskar, A. K. (2019). “Method of producing carbon fibers from multipurpose commercial fibers,” U.S. Patent 10,407,802.

Jambeck, J. R.; Geyer, R.; Wilcox, C.; Siegler, T. R.; Perryman, M.; Andrady, A.; Narayan, R.; and Law, K. L. (2015). “Plastic Waste Inputs from Land into the Ocean.” I, 347, 768.

Jazdi, N. (2014). “Cyber Physical Systems in the Context of Industry 4.0.” *IEEE International Conference on Automation, Quality and Testing, Robotics,* 1.

Jiang, X.; Ouyang, Q.; Liu, D.; Huang, J.; Ma, H.; Chen, Y.; Wang, X.; and Sun, W. (2018). “Preparation of Low-Cost Carbon Fiber Precursors from Blends of Wheat Straw Lignin and Commercial Textile-Grade Polyacrylonitrile (PAN).” *Holzforschung*, 72, 727.

Jilani, A.; Abdel-Wahab, M. S.; and Hammad, A. H. (2017). “Advance Deposition Techniques for Thin Film and Coating.” *Modern Technologies for Creating the Thin-Film Systems and Coatings*, 2, 137.

Jin, H.; Park, D. M.; Gupta, M.; Brewer, A. W.; Ho, L.; Singer, S. L.; Bourcier, W. L.; Woods, S.; Reed, D. W.; and Lammers, L. N. (2017). “Techno-Economic Assessment for Integrating Biosorption into Rare Earth Recovery Process.” *ACS Sustainable Chemistry & Engineering*, 5, 10148.

Jonušauskas, L.; Juodkazis, S.; and Malinauskas, M. (2018). “Optical 3D Printing: Bridging the Gaps in the Mesoscale.” *Journal of Optics*, 20, 053001.

Kang, S. H.; Pokroy, B.; Mahadevan, L.; and Aizenberg, J. (2010). “Control of Shape and Size of Nanopillar Assembly by Adhesion-Mediated Elastocapillary Interaction.” *ACS NANO*, 4, 6323.

Kapoor, Y.; Meyer, R. F.; Meyer, B. K.; DiNunzio, J. C.; Bhambhani, A.; Stanbro, J. K.; Ploeger, J. M.; Guidry, E. N.; Troup, G. M.; Procopio, A. T.; and Templeton, A. C. (2020). “Flexible Manufacturing: The Future State of Drug Product Development and Commercialization in the Pharmaceutical Industry.” *Journal of Pharmaceutical Innovation*, 1.

Kappe, C. O.; Pieber, B.; and Dallinger, D. (2013). “Microwave Effects in Organic Synthesis: Myth or Reality?” *Angewandte Chemie International Edition*, 52, 1088.

Ke, Y.; Castro, C.; and Choi, J. H. (2018). “Structural DNA Nanotechnology: Artificial Nanostructures for Biomedical Research.” *Annual Review of Biomedical Engineering*, 20, 375.

Kenny, J. F.; Barber, N. L.; Hutson, S. S.; Linsey, K. S.; Lovelace, J. K.; and Maupin, M. A. (2017). “Estimated Use of Water in the United States in 2005.” Circular, 1344. U.S. Geological Survey.

Khandavalli, S.; Park, J. H.; Kariuki, N. N.; Zaccarine, S. F.; Pylypenko, S.; Myers, D. J.; Ulsh, M.; and Mauger, S. A. (2019). “Investigation of the Microstructure and Rheology of Iridium Oxide Catalyst Inks for Low-Temperature Polymer Electrolyte Membrane Water Wlectrolyzers.” *ACS Applied Materials & Interfaces*, 11, 45068.

Khodakarami, M.; and Alagha, L. (2020). “Separation and Recovery of Rare Earth Elements Using Novel Ammonium-Based Task-Specific Ionic Liquids with Bidentate and Tridentate O-Donor Functional Groups.” *Separation and Purification Technology*, 232, 115952.

Kim, Y.-Y.; Ganesan, K.; Yang, P.; Kulak, A. N.; Borukhin, S.; Pechook, S.; Ribeiro, L.; Kröger, R.; Eichhorn, S. J.; and Armes, S. P. (2011). "An Artificial Biomineral Formed by Incorporation of Copolymer Micelles in Calcite Crystals." *Nature Materials*, 10, 890.

Kis, A.; Mihailovic, D.; Remskar, M.; Mrzel, A.; Jesih, A.; Piwonski, I.; Kulik, A. J.; Benoît, W.; and Forró, L. (2003). "Shear and Young's Moduli of MoS2 Nanotube Ropes." *Advanced Materials*, 15, 733.

Kopasz, J. P.; Benjamin, T. G.; and Schenck, D. (2017). "2017 Bipolar Plate Workshop Summary Report." Report No. ANL-17/12. Argonne, IL: Argonne National Laboratory.

Koren, Y.; Heisel, U.; Jovane, F.; Moriwaki, T.; Pritschow, G.; Ulsoy, G.; and Van Brussel, H. (1999). "Reconfigurable Manufacturing Systems." *Annals of the CIRP*, 48, 2.

Koumoulos, E. P.; Trompeta, A.-F.; Santos, R.-M.; Martins, M.; Santos, C. M. D.; Iglesias, V.; Böhm, R.; Gong, G.; Chiminelli, A.; and Verpoest, I. (2019). "Research and Development in Carbon Fibers and Advanced High-Performance Composites Supply Chain in Europe: A Roadmap for Challenges and the Industrial Uptake." *Journal of Composites Science*, 3, 86.

Kroposki, B.; Johnson, B.; Zhang, Y.; Gevorgian, V.; Denholm, P.; Hodge, B.-M.; and Hannegan, B. (2017). "Achieving a 100% Renewable Grid: Operating Electric Power Systems with Extremely High Levels of Variable Renewable Energy." *IEEE Power and Energy Magazine*, 15, 61.

Kumar, S. K.; Benicewicz, B. C.; Vaia, Ri. A.; and Winey, K. I. (2017). "50th Anniversary Perspective: Are Polymer Nanocomposites Practical for Applications?" *Macromolecules*, 50, 714.

Kusiak, A. (2018). "Smart Manufacturing." *International Journal of Production Research,* 56, p. 508.

Laitner, J. A.; Nadel, S.; Elliott, R. N.; Sachs, H.; and Khan, A. S. (2012). "The Long-Term Energy Efficiency Potential: What the Evidence Suggests, American Council for an Energy-Efficient Economy." Report Number E121.

Langkau, S.; and Espinoza, L. A. T. (2018). "Technological Change and Metal Demand Over Time: What Can We Learn from the Past?" *Sustainable Materials and Technologies*, 16, 54.

Lanzafame, P.; Abate, S.; Ampelli, C.; Genovese, C.; Passalacqua, R.; Centi, G.; and Perathoner, S. (2017). "Beyond Solar Fuels: Renewable Energy-Driven Chemistry." *ChemSusChem,* 10, 4409.

Larik, J. R.; Shaikh, S. A.; Hussain, T.; Jamro, L. A.; and Ali, Z. (2020). "Performance Evaluation of Flexible Manufacturing Systems: A Case Study." *Performance Evaluation* 3, 30.

Lewis, N. S. (2019). "A Prospective on Energy and Environmental Science," *Energy & Environmental Science*, 12, 16.

Li, L.; Tirado, A.; Nlebedim, I. C.; Rios, O.; Post, B.; Kunc, V.; Lowden, R. R.; Lara-Curzio, E.; Fredette, R.; and J. Ormerod. 2016. "Big Area Additive Manufacturing of High Performance Bonded NdFeB Magnets," Scientific reports, 6, 36212.

Li, N.; Jiang, R.; Li, Y.; Zhou, J.; Ma, Q.; Shen, S.; and Liu, M. (2019). "Plasma-Assisted Photocatalysis of CH4 and CO2 into Ethylene." *ACS Sustainable Chemistry & Engineering*, 7, 11455.

Li, S.; Zhang, S.-Q.; Shen, L.; Liu, Q.; Ma, J.-B.; Lv, W.; He, Y.-B.; and Yang, Q.-H. (2020). "Progress and Perspective of Ceramic/Polymer Composite Solid Electrolytes for Lithium Batteries." *Advanced Science*, 1903088.

Libonati, F.; and Buehler, M. J. (2017). "Advanced Structural Materials by Bioinspiration." *Advanced Engineering Materials*, 19, 1600787.

Lim, S.; Cho, B.; Bae, J.; Kim, A. R.; Lee, K. H.; Kim, S. H.; Hahm, M. G.; and Nam, J. (2016). "Electrohydrodynamic Printing for Scalable MoS2 Flake Coating: Application to Gas Sensing Device." *Nanotechnology*, 27, 435501.

Liu, L.; Sun, H.; Li, C.; Li, T.; Xin, J.; and Zheng, N. (2017). "Managing Battery Aging for High Energy Availability in Green Datacenters." *IEEE Transactions on Parallel and Distributed Systems*, 28, 3521.

Liu, Y., T. Zhao, W. Ju; and S. Shi. 2017. "Materials Discovery and Design Using Machine Learning," Journal of Materiomics, 3, 159.

Lo, Y.-C.; Cheng, C.-L.; Han, Y.-L.; Chen, B.-Y.; and Chang, J.-S. (2014). "Recovery of High-Value Metals from Geothermal Sites by Biosorption and Bioaccumulation." *Bioresource Technology*, 160, 182.

Lobe, S.; Dellen, C. H.-G. Gehrke, H.-G.; Sebold, D.; Tsai, C.-L.; Uhlenbruck, S.; and Guillon, O. (2016). "Radio Frequency Magnetron Sputtering of Li7La3Zr2O12 Thin Films for Solid-State Batteries." *Journal of Power Sources*, 307, 684.

Macknick, J.; Newmark, R.; Heath, G.; and Hallett, K. C. (2011). "Review of Operational Water Consumption and Withdrawal Factors for Electricity Generating Technologies." Golden, CO: National Renewable Energy Laboratory (NREL).

Madson, K. M.; Franz, B.; Molenaar, K. R.; and Kremer, G. O. (2020). "Strategic Development of Flexible Manufacturing Facilities." *Engineering, Construction and Architectural Management*.

Madureira, A.; Pereira, I.; and Sousa, N. (2011). "Self-Organization for Scheduling in Agile Manufacturing" (2011). IEEE 10th International Conference on Cybernetic Intelligent Systems (CIS), London, 38.

Maeda, T.; Zeng, S. M.; Tokumitsu, K.; and Mochida, I. (1993). "Preparation of Isotropic Pitch Precursors for General Purpose Carbon Fibers (GPCF) By Air Blowing—I. Preparation of Spinnable Isotropic Pitch Precursor from Coal Tar by Air Blowing." *Carbon*, 31, 407.

Mann, B.; and Kuhn, H. (1971). "Tunneling Through Fatty Acid Salt Monolayers." *Journal of Applied Physics*, 42, 4398.

Matsuda, W.; Fujimoto, M.; Aoyama, T.; and Mitsunaga, T. (2019). "Cyber Security Risk Assessment on Industry 4.0 Using ICS Testbed with AI and Cloud." November 2019 IEEE Conference on Application, Information and Network Security (AINS), 54.

Maupin, M. A.; Kenny, J. F.; Hutson, S. S.; Lovelace, J. K.; Barber, N. L.; and Linsey, K. S. (2014). "Estimated Use of Water in the United States in 2010." Circular 1405. U.S. Geological Survey, 56.

Maupin, M. A.; Kenny, J. F.; Hutson, S. S.; Lovelace, J. K.; Barber, N. L.; and Linsey, K. S. (2017). "Estimated Use of Water in the United States in 2010." Circular 1405. U.S. Geological Survey, 56.

Mazumdar, S. (2020). "2020 State of the Industry Report." *Composites Manufacturing*, January 3.

McIlvaine, R. (2014). "Green Infrastructure for Industrial Water and Wastewater." *Water and Wastes Digest*.

McMillan, P. F. (2003). "Chemistry of Materials under Extreme High-Pressure–High-Temperature Conditions." *Chemical Communications*, 8, 919.

Mekonnen, M. M.; and Hoekstra, A. Y. (2010). *The Green, Blue and Grey Water Footprint of Farm Animals and Animal Products.* UNESCO-IHE Institute for Water Education Delft.

Menon, S.; Shah, S.; and Coutroubis, A. (2018). "An Overview of Smart Manufacturing for Competitive and Digital Global Supply Chains." 2018 IEEE International Conference on Technology Management, Operations and Decisions (ICTMOD), 178.

Milbrandt, A.; and Booth, S. (2016). "Carbon Fiber from Biomass." Golden, CO: National Renewable Energy Laboratory (NREL).

Mosconi, F. (2015). "The New European Industrial Policy: Global Competitiveness and the Manufacturing Renaissance." Routledge: London, UK.

Mul, G.; Schacht, C.; van Swaaij, W. P. M.; and Moulijn, J. A. (2012). "Functioning Devices for Solar to Fuel Conversion." *Chemical Engineering and Processing: Process Intensification*, 51, 137.

Nagapurkar, P.; and Smith, J. D. (2019). "Techno-Economic Optimization and Environmental Life Cycle Assessment (LCA) of Microgrids Located in the US Using Genetic Algorithm." *Energy Conversion and Management*, 181, 272.

Nam, T.; and Kim, H. S. (2020). "Atomic Layer Deposition for Nonconventional Nanomaterials and Their Applications." *Journal of Materials Research*, 35, 656.

Napporn, T. W.; Karpenko-Jereb, L.; Pichler, B.; and Hacker, V. (2018). "Polymer Electrolyte Fuel Cells." *Fuel Cells and Hydrogen*, 63.

Nardelli, P.; Papadias, C.; Kalalas, C.; Alves, H.; Christou, I. T.; Macaluso, I.; Marchetti, N.; Palacios, R.; and Alonso-Zarate, J. (2019). "Framework for the Identification of Rare Events via Machine Learning an IoT network." *2019 16th International Symposium on Wireless Communication Systems (ISWCS),* 656. IEEE.

Naskar, A. K.; Keum, J. K.; and Boeman, R. G. (2016). "Polymer Matrix Nanocomposites for Automotive Structural Components." *Nature Nanotechnology*, 11, 1026.

Nassar, N. T., Du, X.; and Graedel, T. E. 2015. "Criticality of the Rare Earth Elements," *Journal of Industrial Ecology*, 19, 1044.

National Academies of Sciences, Engineering, and Medicine (2018). *Direct Air Capture and Mineral PCarbonation Approaches for Carbon Dioxide Removal and Reliable Sequestration: Proceedings of a Workshop–in Brief*. Washington, DC: The National Academies Press.

NRC (National Research Council) (2012). *Application of Lightweighting Technology to Military Aircraft, Vessels, and Vehicles*. Washington, DC: National Academies Press.

NRC (National Research Council) (2011). *Assessment of Fuel Economy Technologies for Light-Duty Vehicles.* Division & Systems, Board & Economy, Committee, Washington DC: National Academies Press.

Navarrete, A.; Centi, G.; Bogaerts, A.; Martin, A.; York, A.; and Stefanidis, G. D. (2017). "Harvesting Renewable Energy for Carbon Dioxide Catalysis." *Energy Technology*, 5, 796.

Navarro, J.; and Zhao, F. (2014). "Life cycle Assessment of the Production of Rare-Earth Elements For Energy Applications: A Review." *Frontiers in Energy Research*, 2, 45.

Needell, Z. A.; McNerney, J.; Chang, M. T.; and Trancik, J. E. (2016). "Potential for Widespread Electrification of Personal Vehicle Travel in the United States." *Nature Energy*, 1, 16112.

Ng, T. C.; and Ghobakhloo, M. (2020). "Energy Sustainability and Industry 4.0," *Earth and Environmental Science*, 46, 012090.

Nguyen, N. A.; Barnes, S. H.; Bowland, C. C.; Meek, K. M.; Littrell, K. C.; Keum, J. K.; and Naskar, A. K. (2018). "A Path for Lignin Valorization via Additive Manufacturing of High-Performance Sustainable Composites with Enhanced 3D Printability," *Science Advances*, 4, 4967.

Nhan, N. T.; Obunai, K.; Okubo, K.; Shibata, O.; Tomokuni, H.; and Fujita, Y. (2019). "Mechanical Properties of Submicron Glass Diber Reinforced Vinyl Ester Composite." *Open Journal of Composite Materials*, 9, 365.

Nikolic, B.; Ignjatic, J.; Suzic, N.; Stevanov, B.; and Rikalovic, A. (2017). "Predictive Manufacturing Systems in Industry 4.0: Trends, Benefits and Challenges." *Annals of DAAAM & Proceedings*, 28.

Nimbalkar, S. U.; Thekdi, A. C.; Rogers, B. M.; Kafka, O. L.; and Wenning, T. J. (2014). "Technologies and Materials for Recovering Waste Heat in Harsh Environments." Oak Ridge National Laboratory Oak Ridge, TN.

Ojstersek, R.; and Buchmeister, B. (2020). "The Impact of Manufacturing Flexibility and Multi-criteria Optimization on the Sustainability of Manufacturing Systems." *Symmetry*, 12, 157.

Oliveira, L.; Messagie, M.; Rangaraju, S.; Sanfelix, J.; Rivas, M. H.; and Van Mierlo, J. (2015). "Key Issues of Lithium-Ion Batteries–from Resource Depletion to Environmental Performance Indicators," *Journal of Cleaner Production,* 108, 354.

Oztemel, E.; and Gursev, S. (2020). "Literature Review of Industry 4.0 and Related Technologies." *Journal of Intelligent Manufacturing*, 31, 127.

Papageorgopoulos, D. (April 29, 2019). "Fuel Cell R&D Overview." Presented at the Fuel Cell Technologies Office 2019 Annual Merit Review and Peer Evaluation Meeting, Washington, D.C.

Park, T.; Chang, I.; Lee, Y. H.; Ji, S.; and Cha, S. W. (2014). "Analysis of Operational Characteristics of Polymer Electrolyte Fuel Cell with Expanded Graphite Flow-field Plates via Electrochemical Impedance Investigation." *Energy*, 66, 77.

Paulauskas, F. L.; Warren, C. D.; Eberle, C. C.; Naskar, A. K.; Ozcan, S.; Fagundes, A.; Dias, R. B.; and de Magalhães, P. (2009). "Novel Precursor Materials and Approaches for Producing Lower Cost Carbon Fiber for High Volume Industries." *Automotive Applications & Recycling*, Edimburgh, Scotland.

Paulson, N. H.; Libera, J. A.; and Stan, M. (2020) "Flame spray pyrolysis optimization via statistics and machine learning," *Materials & Design* 196 108972. https://doi.org/10.1016/j.matdes.2020.108972.

Paulson, N. H., Libera, J. A.; and Stan, M. (2019) "Machine Learning Exploration and Optimization of Flame Spray Pyrolysis," *Technology & Maintenance Council (TMC*) Annual Meeting.

Paulson, N. H.; Bocklund, B. J.; Otis, R. A.; Liu, Z.-K.; and Stan, M. (2019). "Quantified Uncertainty In Thermodynamic Modeling for Materials Design." *Acta Materialia*, 174, 9.

Paulson, N. H.; Jennings, E.; and Stan, M. (2019). "Bayesian Strategies for Uncertainty Quantification of the Thermodynamic Properties of Materials." *International Journal of Engineering Science*, 142, 74.

Peelman, S.; Sun, Z.; Sietsma, J.; and Yang, Y. (eds.) (2016). "Hydrometallurgical Extraction of Rare Earth Elements From Low Grade Mine Tailings." *Rare Metal Technology* 2016, Springer, Cham.

Peng, B.; Locascio, M.; Zapol, P.; Li, S.; Mielke, S. L.; Schatz, G. C.; and Espinosa, H. D. (2008). "Measurements of Near-Ultimate Strength for Multiwalled Carbon Nanotubes and Irradiation-Induced Crosslinking Improvements." *Nature Nanotechnology*, 3, 626.

Peplow, M. (2013). "Molecular Robot Mimics Life's Protein-Builder." *Nature News*, http://www.nature.com/news/molecular-robot-mimics-life-s-protein-builder-1.12190.

Peplow, M. (2015). "March of the Machines," *Nature*, 525, 18.

Perathoner, S.; Gross, S.; Hensen, E. J. M.; Wessel, H.; Chraye, H.; and Centi, G. (2017). "Looking at the Future of Chemical Production Through the European Roadmap on Science and Technology of Catalysis the EU Effort for a Long-Term Vision." *ChemCatChem*, 9, 904.

Petric, P.; Bevis, C.; McCord, M.; Carroll, A.; Brodie, A.; Ummethala, U.; Grella, L.; Cheung A.; and Freed, R. 2011. "New advances with REBL for maskless high-throughput EBDW lithography," Alternative Lithographic Technologies III, SPIE International Society for Optics and Photonics, 7970, 797018.

Philippot, M.; Ayerbe, E.; Hoedemaekers, E.; Mierlo, J. V.; and Messagie, M. (2019). "Water Footprint of the Manufacturing of a Traction Lithium Ion Battery Pack." 32nd International Electric Vehicle Symposium, EVS32; Lyon, France; May 19–22.

Pollet, B. G.; Franco, A.; Su, H.; Liang, H.; and Pasupathi, S. (2016) "Proton exchange membrane fuel cells," *Compendium of Hydrogen Energy* 3, 3.

Praprotnik, M.; Site, L. D.; and Kremer, K. (2008). "Multiscale Simulation of Soft Matter: From Scale Bridging to Adaptive Resolution." *Annual Review Physical Chemistry*, 59, 545.

Puliyalil, H.; Jurković, D. L.; Dasireddy, V. D. B. C.; and Likozar, B. (2018). "A Review of Plasma-Assisted Catalytic Conversion of Gaseous Carbon Dioxide and Methane into Value-Added Platform Chemicals and Fuels." *RSC Advances*, 8, 27481.

Raileanu, S.; Borangiu, T.; Morariu, O.; and Iacob, I. (2018). "Edge Computing in Industrial IoT Framework for Cloud-Based Manufacturing Control," 2018 22nd International Conference on System Theory, Control and Computing, 261.

Randall, J. N.; Owen, J. H. G.; Lake, J.; Saini, R.; Fuchs, E.; Mahdavi, M.; Moheimani, S. O. R.; and Schaefer, B. C. (2018). "Highly parallel Scanning Tunneling Microscope Based Hydrogen Depassivation Lithography." *Journal of Vacuum Science & Technology B, Nanotechnology and Microelectronics: Materials, Processing, Measurement, and Phenomena*, 36, 06JL05.

Raney, J. R.; Compton, B. G.; Mueller, J.; Ober, T. J.; Shea, K.; and Lewis, J. A. (2018). "Rotational 3D Printing of Damage-Tolerant Composites with Programmable Mechanics." *Proceedings of the National Academy of Sciences*, 115, 1198.

Rao, P.; Sholes, D.; Morrow, W. R.; and Cresko, J. (2017). "Estimating US Manufacturing Water Use." Presented at the 2017 ACEEE Summer Study on Energy Efficiency in Industry, Denver, CO.

Rao, P.; Morrow, W. R.; and Cresko, J. (2017). "Estimating U.S. Manufacturing Water Use." *ACEEE Summer Study on Energy Efficiency in Industry*, 104.

Ratner, M. (2013). "A Brief History of Molecular Electronics." *Nature Nanotechnology*, 8, 378.

Reuter, A.; Kircher, C.; and Verl, A. (2010). "Manufacturer-Independent Mechatronic Information Model for Control Systems." *Production Engineering*, 4, 165.

Rissman, J.; Bataille, C.; Masanet, E.; Aden, N.; Morrow III, W. R.; Zhou, N.; Elliott, N.; Dell, R.; Heeren, N.; Huckestein, B.; Cresko, J.; Miller, S. A.; Roy, J.; Fennell, P.; Cremmins, B.; Blank, T. K.; Hone, D.; Williams, E. D.; and Helseth, J. (2020). "Technologies and Policies to Decarbonize Global Industry: Review and Assessment of Mitigation Drivers Through 2070." *Applied Energy*, 266, 114848.

Roth, G. (2018). "Additive Manufacturing: Methods, Hazards and Safety Culture." 2018 Indiana Safety and Health Conference and Expo.

Roth, G. A.; Geraci, C. L.; Stefaniak, A.; Murashov, V.; and Howard, J. (2019). "Potential Occupational Hazards of Additive Manufacturing." *Journal of Occupational and Environmental Hygiene*, 16, 321.

Salaita, K.; Wang, Y.; Fragala, J.; Vega, R. A.; Liu, C.; and Mirkin, C. A. (2006). "Massively Parallel Dip–Pen Nanolithography With 55 000-Pen Two-Dimensional Arrays." *Angewandte Chemie International Edition*, 45, 7220.

Schaefer, D. W.; and Justice, R. S. (2007). "How Nano Are Nanocomposites?" *Macromolecules*, 40, 8501.

Schmatz, B.; Lang, A. W.; and Reynolds, J. R. (2019). "Fully Printed Organic Electrochemical Transistors from Green Solvents." *Advanced Functional Materials* 29, 1905266.

Schmitt, M.; Baunach, M.; Wengeler, L.; Peters, K.; Junges, P.; Scharfer, P.; and Schabel, W. (2013). "Slot-Die Processing of Lithium-Ion Battery Electrodes—Coating Window Characterization." *Chemical Engineering and Processing: Process Intensification*, 68, 32.

Schnell, J.; Tietz, F.; Singer, C.; Hofer, A.; Billot, N.; and Reinhart, G. (2019). "Prospects of Production Technologies and Manufacturing Costs of Oxide-Based All-Solid-State Lithium Batteries." *Energy & Environmental Science*, 12, 1818.

Schoch, T. P. (2016). "EU Privacy Regulations' Impact on Information Governance," *Information Management* 50, 20.

Schulte, P. A.; Roth, G.; Hodson, L. L.; Murashov, V.; Hoover, M. D.; Zumwalde, R.; Kuempel, E. D.; Geraci, C. L.; Stefaniak, A. B.; and Castranova, V. (2016). "Taking Stock of the Occupational Safety and Health Challenges of Nanotechnology: 2000–2015." *Journal of Nanoparticle Research*, 18, 159.

Schulz, H. (2005). "Characteristics of Modern Manufacturing Techniques." AMST'05 Advanced Manufacturing Systems and Technology (pp. 5-15). Springer, Vienna.

Scime, L.; Wolf, S. D.; Beuth, J.; Mrdjenovich, S.; and Kelley, M. 2018. "Safety and workflow considerations for modern metal additive manufacturing facilities," Jom, 70, 1830.

Seh, Z. W.; Kibsgaard, J.; Dickens, C. F.; Chorkendorff, I. B.; Nørskov, J. K.; and Jaramillo, T. F. (2017). "Combining Theory and Experiment in Electrocatalysis: Insights into Materials Design." *Science*, 355, 4998.

Shen, F.; Dixit, M. B.; Zaman, W.; Hortance, N.; Rogers, B. M.; and Hatzell, K. B. (2019). "Composite Electrode Ink Formulation for All Solid-State Batteries." *Journal of The Electrochemical Society,* 166, A3182.

Sheu, E. J.; Mokheimer, E. M. A.; and Ghoniem, A. F. (2015). "A Review of Methane Reforming Systems." *International Journal of Hydrogen Energy*, 40, 12929.

Shobana, M. K. (2019). "Metal Oxide Coated Cathode Materials for Li Ion Batteries—A Review." *Journal of Alloys and Compounds*, 802, 477.

Short, D. B.; Sirinterlikci, A.; Badger, P.; and Artieri, B. (2015). "Environmental, Health, and Safety Issues in Rapid Prototyping." *Rapid Prototyping Journal*, 21, 105.

Short, M. P.: and Yip, S. (2015). "Materials Aging At Mesoscale: Kinetics of Thermal, Stress, Radiation Activations." *Current Opinion in Solid State & Materials Science*, 19, 245.

Singh, V.; and Willcox, K. E. (2018). "Engineering Design with Digital Thread." *AIAA Journal*, 56, 4515.

Smestad, G. P.; and Steinfeld, A. (2012). "Photochemical and Thermochemical Production of Solar Fuels from H2O and CO2 Using Metal Oxide Catalysts." *Industrial & Engineering Chemistry Research*, 51, 11828.

Smil, V. (ed.) (2003). *The Earth's Biosphere: Evolution, Dynamics, and Change.* MIT Press.

Smith, R. (2005). *Chemical Process: Design and Integration.* John Wiley & Sons.

Solley, W. B.; Pierce, R. R.; and Perlman, H. A. (1998). "Estimated Use of Water in the United States in 1995," Circular 1200. U.S. Geological Survey.

Stacy, J.: Regmi, Y. N.; Leonard, B.; and Fan, M. (2017). "The Recent Progress and Future of Oxygen Reduction Reaction Catalysis: A Review." *Renewable and Sustainable Energy Reviews*, 69, 401.

Stan, M. (2009). "Discovery and Design of Nuclear Fuels," *Materials Today*, 12, 20.

Stan, M.; Paulson, N. H.; Libera, J. A.; Dasgupta, D.; Torelli, R.; and Pinaki, P. (2020). "Real-Time Optimization of Manufacturing Processes via Machine Learning and Bayesian Analysis." *NPJ-Comp. Mater*.

Stan, M.; and Sarrao, J. L. (2018). *Modeling of Microstructure Evolution: Mesoscale Challenges, Handbook of Materials Modeling*, 2nd Edition, Springer, 1–10.

Stan, M.; and Sarrao, J. L. (2020). "Modeling of Microstructure Evolution: Mesoscale Challenges," Handbook of Materials Modeling: Methods: Theory and Modeling, 1039.

Stankiewicz, A. I.; and Moulijn, J. A. (2000). "Process Intensification: Transforming Chemical Engineering." *Chemical Engineering Progress,* 96, 22.

Stock, T.; and Seliger, G. (2016). "Opportunities of Sustainable Manufacturing in Industry 4.0." *Procedia CIRP*, 40, 536.

Stolarczyk, J. K.; Bhattacharyya, S.; Polavarapu, L.; and Feldmann, J. (2018). "Challenges and Prospects in Solar Water Splitting and CO2 Reduction with Inorganic and Hybrid Nanostructures." Acs *Catalysis*, 8, 3602.

Sun, K.; Moreno-Hernandez, I. A.; Schmidt, W. C.; Zhou, X.; Crompton, J. C.; Liu, R.; Saadi, F. H.; Chen, Y.; Papadantonakis, K. M.; and Lewis N. S. (2017). "A Comparison of the Chemical, Optical and

Electrocatalytic Properties of Water-Oxidation Catalysts for Use in Integrated Solar-Fuel Generators." *Energy & Environmental Science*, 10, 987.

Sundararajan, P. R.; Tyrer, N.; and Bluhm, T. (1982). "Solvent-Dependent Conformations in Gels of Isotactic Polystyrene." *Macromolecules*, 15, 286.

Surwade, S. P.; Zhou, F.; Wei, B.; Sun, W.; Powell, A.; O'Donnell, C.; Yin, P.; and Liu, H. (2013). "Nanoscale Growth and Patterning of Inorganic Oxides Using DNA Nanostructure Templates." *Journal of the American Chemical Society*, 135, 6778.

Tajaddodianfar, F.; Moheimani, S. O. R.; Owen, J.; and Randall, J. N. (2018). "On the Effect of Local Barrier Height in Scanning Tunneling Microscopy: Measurement Methods and Control Implications." *Review of Scientific Instruments,* 89, 013701.

Tan, R.; Wang, A.; Malpass-Evans, R.; Williams, R.; Zhao, E. W.; Liu, T.; Ye, C.; Zhou, X.; Darwich, B. P.; and Fan, Z. (2020). "Hydrophilic Microporous Membranes for Selective Ion Separation and Flow-Battery Energy Storage." *Nature Materials*, 19, 195.

Teller, O.; Nicolai, J. P.; Lafoz, M.; Laing, D.; Tamme, R.; Pederson, A. S.; Andersson, M.; Folke, C.; Bourdil, C.; and Conte, M. (2013). "Joint EASE/EERA Recommendations for a European Energy Storage Technology Development Roadmap Towards 2030." Brussels, Belgium: European Association for Storage of Energy (EASE) and European Energy Research Alliance (EERA).

Tenne, R.; Margulis, L.; Genut, M. E.; and Hodes, G. (1992). "Polyhedral and Cylindrical Structures of Tungsten Disulphide." *Nature*, 360, 444.

Thompson, M. K.; Moroni, G.; Vaneker, T.; Fadel, G.; Campbell, R. I.; Gibson, I.; Bernard, A.; Schulz, J.; Graf, P.; and Ahuja, B. (2016). "Design for Additive Manufacturing: Trends, Opportunities, Considerations, and Constraints." *CIRP Annals*, 65, 737.

Tian, Y.; Demirel, S. E.; Hasan, M. F.; and Pistikopoulos, E. N. (2016). "An Overview of Process Systems Engineering Approaches for Process Intensification: State of the Art." *Chemical Engineering and Processing-Process Intensification,* 133, 160.

Tjahjono, B.; Esplugues, C.; Ares, E.; and Pelaez, G. (2017). "What Does Industry 4.0 Mean to Supply Chain?" *Procedia Manufacturing*, 13, 1175.

Tofail, S. A. M.; Mani, A.; Bauer, J.; and Silien, C. (2018). "In Situ, Real-Time Infrared (IR) Imaging for Metrology in Advanced Manufacturing." *Advanced Engineering Materials*, 20, 1800061.

Tu, X; Whitehead, J. C.; and Nozaki, T. (2019). *Plasma Catalysis: Fundamentals and Applications.* Springer Series on Atomic, Optical, and Plasma Physics. Springer International Publishing.

Ue, M.; and Uosaki, K. (2019). "Recent Progress in Liquid Electrolytes for Lithium Metal Batteries." *Current Opinion in Electrochemistry*, 17, 106.

Undey, C.; and A. Cinar. 2002. "Statistical monitoring of multistage, multiphase batch processes," IEEE Control, magazine, 22, 40.

Vaia, R. A.; and Giannelis, E. P. (2001). "Polymer Nanocomposites: Status and Opportunities." *MRS Bulletin,* 26, 394.

Vaia, R. A.; and Maguire, J. F. (2007). "Polymer Nanocomposites with Prescribed Morphology: Going Beyond Nanoparticle-Filled Polymers." *Chemistry of Materials*, 19, 2736.

Van Gerven, T.; and Stankiewicz, A. I. (2009). "Structure, Energy, Synergy, Time—The Fundamentals of Process Intensification." *Industrial & Engineering Chemistry Research,* 48, 2465.

Vance, D.; Nimbalkar, S.; Thekdi, A.; Armstrong, K.; Wenning, T.; Cresko, J.; and Jin, M. (2019). "Estimation of and Barriers to Waste Heat Recovery from Harsh Environments in Industrial Processes." *Journal of Cleaner Production*, 222, 539.

Vaux Jr, H. (2011). "Water Conservation, Efficiency, and Reuse." *Elements*, 7, 187.

Vickery, J. L.; Patil, A. J.; and Mann, S. (2009). "Fabrication of Graphene–Polymer Nanocomposites with Higher-Order Three-Dimensional Architectures." *Advanced Materials* 21, 2180.

Wan, C.; and Chen, B. (2012). "Reinforcement and Interphase of Polymer/Graphene Oxide Nanocomposites." *Journal of Materials Chemistry*, 22, 3637.

Wang, W.; and Koren, Y. (2012). "Scalability Planning for Reconfigurable Manufacturing Systems." *Journal of Manufacturing Systems,* 31, 83.

Wang, X.; Han, Y.; Leung, V. C. M.; Niyato, D.; Yan, X.; and Chen, X. (2020). "Convergence of Edge Computing and Deep Learning: A Comprehensive Survey." *IEEE Communications Surveys & Tutorials*, 22, 869.

Wei, B.; Dai, M.; and Yin, P. (2012). "Complex Shapes Self-Assembled From Single-Stranded DNA Tiles." *Nature*, 485, 623.

Wood III, D. L.; Li, J.; and Daniel, C. (2015). "Prospects for Reducing the Processing Cost of Lithium Ion Batteries." *Journal of Power Sources*, 275, 234.

Woods, D.; Doty, D.; Myhrvold, C.; Hui, J.; Zhou, F.; Yin, P.; and Winfree, E. (2019). "Diverse and Robust Molecular Algorithms Using Reprogrammable DNA Self-Assembly." *Nature*, 567, 366.

Wu, M.; Mintz, M.; Wang, M.; and Arora, S. (2009). "Water Consumption in the Production of Ethanol and Petroleum Gasoline." *Environmental Management*, 44, 981.

Wu, W. (2017). "Inorganic Nanomaterials for Printed Electronics: A Review." *Nanoscale*, 9, 7342.

Wu, Y.; Lazic, P.; Hautier, G.; Persson, K.; and Ceder, G. (2013). "First Principles High Throughput Screening of Oxynitrides for Water-Splitting Photocatalysts." *Energy & Environmental Science*, 6, 157.

Xie, N.; Huang, Z.; Luo, Z.; Gao, X.; Fang, Y.; and Zhang, Z. (2017). "Inorganic Salt Hydrate for Thermal Energy Storage." *Applied Sciences*, 7, 1317.

Yan, M.; Kawamata, Y.; and Baran, P. S. (2017). “Synthetic Organic Electrochemical Methods Since 2000: on the Verge of a Renaissance.” *Chemical Reviews*, 117, 13230.

Yan, Q.: Yu, J.; Suram, S. K.; Zhou, L.; Shinde, A.; Newhouse, P. F.; Chen, W.; Li, G.; Persson, K. A.; and Gregoire, J. M. (2017). “Solar Fuels Photoanode Materials Discovery By Integrating High-Throughput Theory and Experiment.” Proceedings of the National Academy of Sciences, 114, 3040.

Yang, S.; Choi, Y.-W.; Choi, J.; Jeong, N.; Kim, H.; Jeong, H.; Byeon, S. Y., Yoon, H.; and Kim, Y. H. (2019). “Green Fabrication of Pore-Filling Anion Exchange Membranes Using R2R Processing.” *Journal of Membrane Science* 584, 181.

Yang, X.; Ding, D.; Xu, Y.; Zhang, W.; Gao, Y.; Wu, Z.; Chen, G.; Chen, R.; Huang, Y.; and Tang, J. (2019). “Tensile Properties and Corrosion Resistance of Al-xFe-La Alloys for Aluminium Current Collector of Lithium-Ion Batteries.” *Metals*, 9, 706.

Yilmaz, G.; Ellingham, T.; and Turng, L. S. (2019). “Injection and Injection Compression Molding of Ultra-High-Molecular Weight Polyethylene Powder.” *Polymer Engineering & Science,* 59, E170.

Yoonessi, M.; Shi, Y.; Scheiman, D. A.; Lebron-Colon, M.; Tigelaar, D. M.; Weiss, R. A.; and Meador, M. A. (2012). “Graphene Polyimide Nanocomposites; Thermal, Mechanical, and High-Temperature Shape Memory Effects.” *ACS Nano,* 6, 7644.

Yuan, Y.; You, H.; and Ricardez-Sandoval, L. (2019). “Recent Advances on First-Principles Modeling for the Design of Materials in CO_2 Capture Technologies.” *Chinese Journal of Chemical Engineering*, 27, 1554.

Zaimes, G. G.; Hubler, B. J.; Wang, S.; and Khanna, V. (2015). “Environmental Life Cycle Perspective on Rare Earth Oxide Production.” *ACS Sustainable Chemistry & Engineering*, 3, 237.

Zhang, C.; Shen, X.; Pan, Y.; and Peng, Z. (2017). “A Review of Pt-Based Electrocatalysts for Oxygen Reduction Reaction.” *Frontiers in Energy*, 11, 268.

Zhang, S. S. (2007). “A Review on the Separators of Liquid Electrolyte Li-Ion Batteries.” *Journal of Power Sources*, 164, 351.

Zhang, Y.; Dahl, E.; Yang, P.; Lee, B.; Seong, H. J.; and Krumdick, G. (2019). “In-Situ Synchrotron Characterization of Nanofiber Synthesis for Solid State Battery Applications.” 31stst Annual Electronics Packaging Symposium, Niskayuna, NY.

Zhao, Q.; Stalin, S.; Zhao, C.-Z.; and Archer, L. A. (2020). “Designing Solid-State Electrolytes for Safe, Energy-Dense Batteries.” *Nature Reviews Materials*, 1.

Zheng, J. M.; Wu, X. B.; and Yang, Y. (2011). “A Comparison of Preparation Method on the Electrochemical Performance of Cathode Material Li [Li0.2Mn0 54Ni0.13Co0.13] O2 for Lithium Ion Battery.” *Electrochimica Acta*, 56, 3071.

Zheng, P.; Wang, H.; Zhiqian, S.; Ray, Y. Z.; Yongkui, L.; Chao, L.; Khamdi, M.; Shiqiang, Y.; and Xun, X. (2018). "Smart Manufacturing Systems for Industry 4.0: Conceptual Framework, Scenarios, and Future Perspectives." *Frontiers of Mechanical Engineering*, 13, 37.

Zheng, X., Wang, C.; Dai, J.; Shi, W.; and Yan, Y. 2015. "Design of Mesoporous Silica Hybrid Materials as Sorbents for the Selective Recovery of Rare Earth Metals," *Journal of Materials Chemistry A*, 3, 10327.

Zhou, J.; Xu, W.; You, Z.; Wang, Z.; Luo, Y.; Gao, L.; Yin, C.; Peng, R.; and Lan, L. (2016). "A New Type of Power Energy for Accelerating Chemical Reactions: The Nature of a Microwave-Driving Force for Accelerating Chemical Reactions." *Scientific Reports*, 6, 25149.

Zhu, Y. Q.; Sekine, T.; Brigatti, K.; Firth, S. S.; Tenne, R.; Rosentsveig, R.; Kroto, H. W.; and Walton, D. R. M. (2003). "Shock-Wave Resistance of WS2 Nanotubes" *Journal of the American Chemical Society,* 125, 1329.

DOI: 10.2172/1618267

U.S. DEPARTMENT OF ENERGY | Office of Science