MACROFLASH BOILOFF CALORIMETRY INSTRUMENT FOR THE MEASUREMENT OF **HEAT TRANSMISSION** THROUGH MATERIALS

2019 Cryogenic Engineering Conference and
International Cryogenic Materials Conference (CEC-ICMC)

Hartford, Connecticut USA July 21-25, 2019

James E. Fesmire

NASA Kennedy Space Center
Cryogenics Test Laboratory
KSC, FL 32899 USA

Cody D. Bateman

Cryotek LLC
1425 White Drive
Titusville, FL 32780 USA

Julien L. Thomas

Solution One Industries
Cryogenics Test Laboratory (LASSO)
KSC, FL 32899 USA

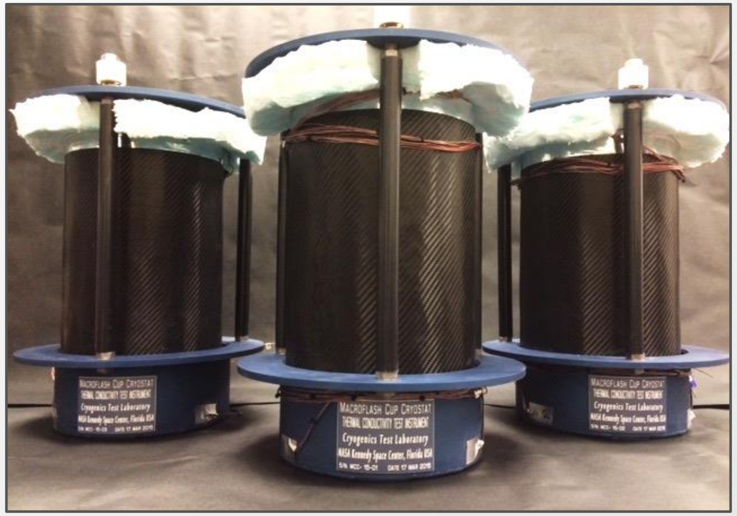
HIGHLIGHTS

- Macroflash: flat plate, comparative boiloff calorimeter providing thermal data for materials from thermal insulation to structural composites to ceramics
- Measurement principle is boiloff calorimetry covering under real-world conditions
  - Steady-state liquid nitrogen vaporization provides a direct calculation of heat flow rate
- Practical way to measure heat transmission through materials under steady-state conditions at below-ambient temperatures and under different compressive loads
- Provides test data at both large ΔT and/or small ΔT; ranging from 77 K (LN_2) to 403 K
- Test specimens may be isotropic or non-isotropic; homogeneous or non-homogeneous

DESIGN OBJECTIVES

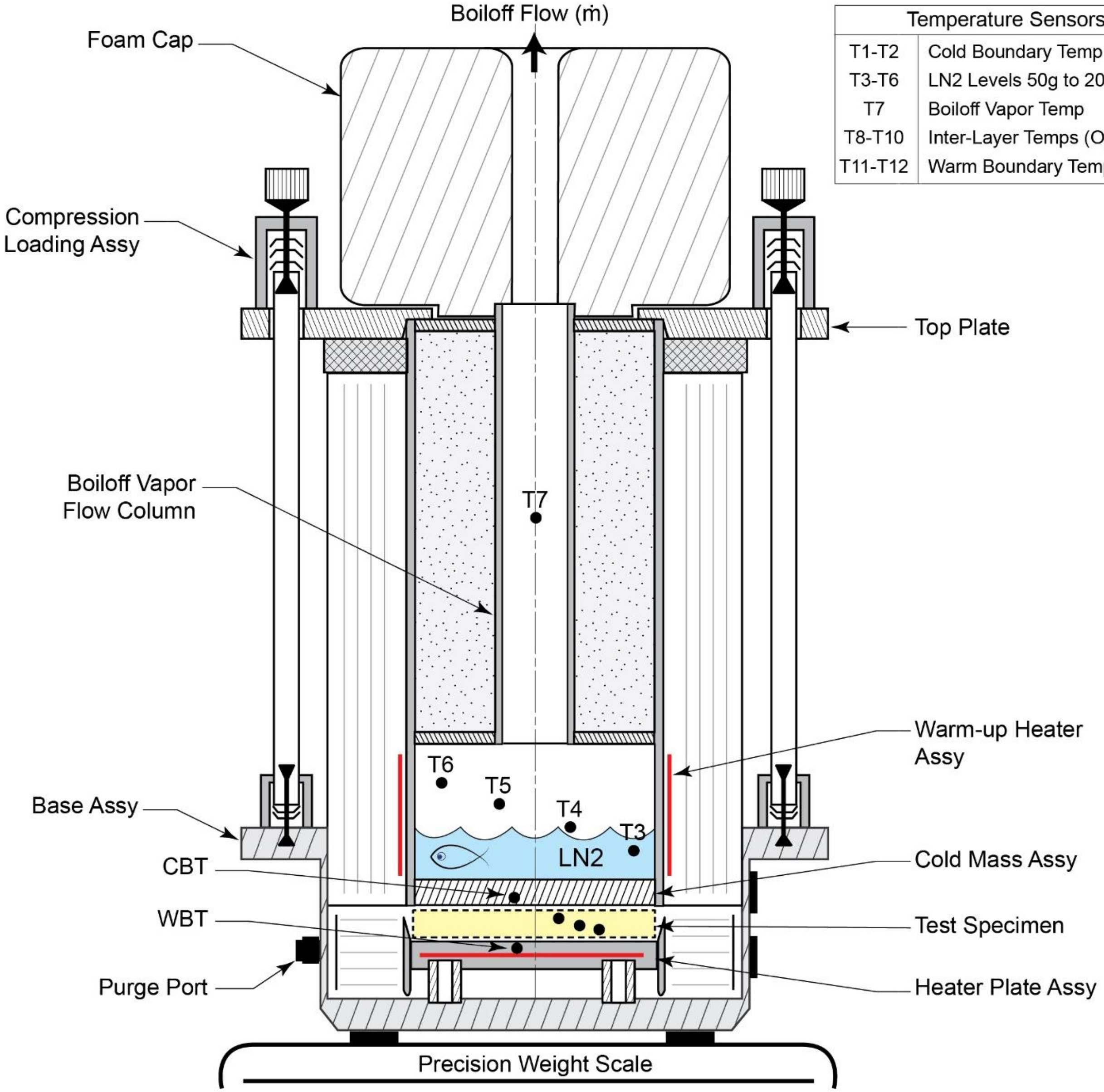
- Calibration standard: Cold Boundary Temperature (CBT) of 77 K; Warm Boundary Temperature (WBT) of 293 K
  - Temperature range: from 77 K (LN_2) to 403 K
- Directly calculate: heat flow rate (Q)
  - Heat flux (q) and effective thermal conductivity (k_e)
- Test specimen dimensions: 76-mm diameter by 6.4-mm thickness (calibration standard)
  - Any thickness from 1 mm to 10 mm can be tested for comparative data
- Specimen material/design type: non-isotropic, layered, asymmetrical, inhomogeneous types are all acceptable; solids or powders; foams, aerogels, wood, metals, ceramics, glass, composites
- Compression loading of test specimen: 0, 2, or 5 psi [standard settings]

Production of Macroflash instruments



GENERATION 2
MACROFLASH TEST INSTRUMENT

Measurement of heat transmission through materials by boiloff calorimetry



Temperature Sensors	
T1-T2	Cold Boundary Temp (CBT)
T3-T6	LN2 Levels 50g to 200g
T7	Boiloff Vapor Temp
T8-T10	Inter-Layer Temps (Optional)
T11-T12	Warm Boundary Temp (WBT)

CYROTEK NASA CryogenicsTestLab KENNEDY SPACE CENTER

HARDWARE / SOFTWARE

- Macroflash unit
- Weight scale
- Mass flow meter (option to weight scale)
- Heater controller
- Gaseous nitrogen (GN_2) purge supply
- National Instruments LabView with thermocouple module
- Custom software interface with input prompts, database codes, graphical summary, and final report with all calculations
- 10-liter LN_2 dewar and 350-ml pouring cup

METHODOLOGY

- Principle of heat flow measurement: boiloff calorimetry
  - Flat plate, comparative (ASTM C1774, Annex A4)
- Test specimen: single material or specialized combination of different materials
- Materials: monolithic, composites, blanket, layered blanket, MLI, or bulk-fill type
- Thickness: critical measurement for k_e calculation
  - Fit-up for good thermal contact is also crucial for rigid materials
- LN_2 Fill to 220 g (or more) for initial cooldown and stabilization
- Steady-state condition: between 50 g and 100 g LN_2

THERMAL CONDUCTIVITY (λ) AND MEAN TEMPERATURE (T_m)

- Bonus! Optional intermediate temperature sensors for calculating thermal conductivity (λ) versus temperature (T_m)
- Multiple data points can be obtained from a single test
  - Any two temperatures between 78 K and 403 K can be set up
- However! Steady-state heat transmission: heat flows according to the ΔT, not as a function of T_m

Interlayer temperature sensors installed for a flat disk test specimen

Macroflash in operation during cooldown

Macroflash software interface: preparations and set-up

PREPARATION

Test Specimen:

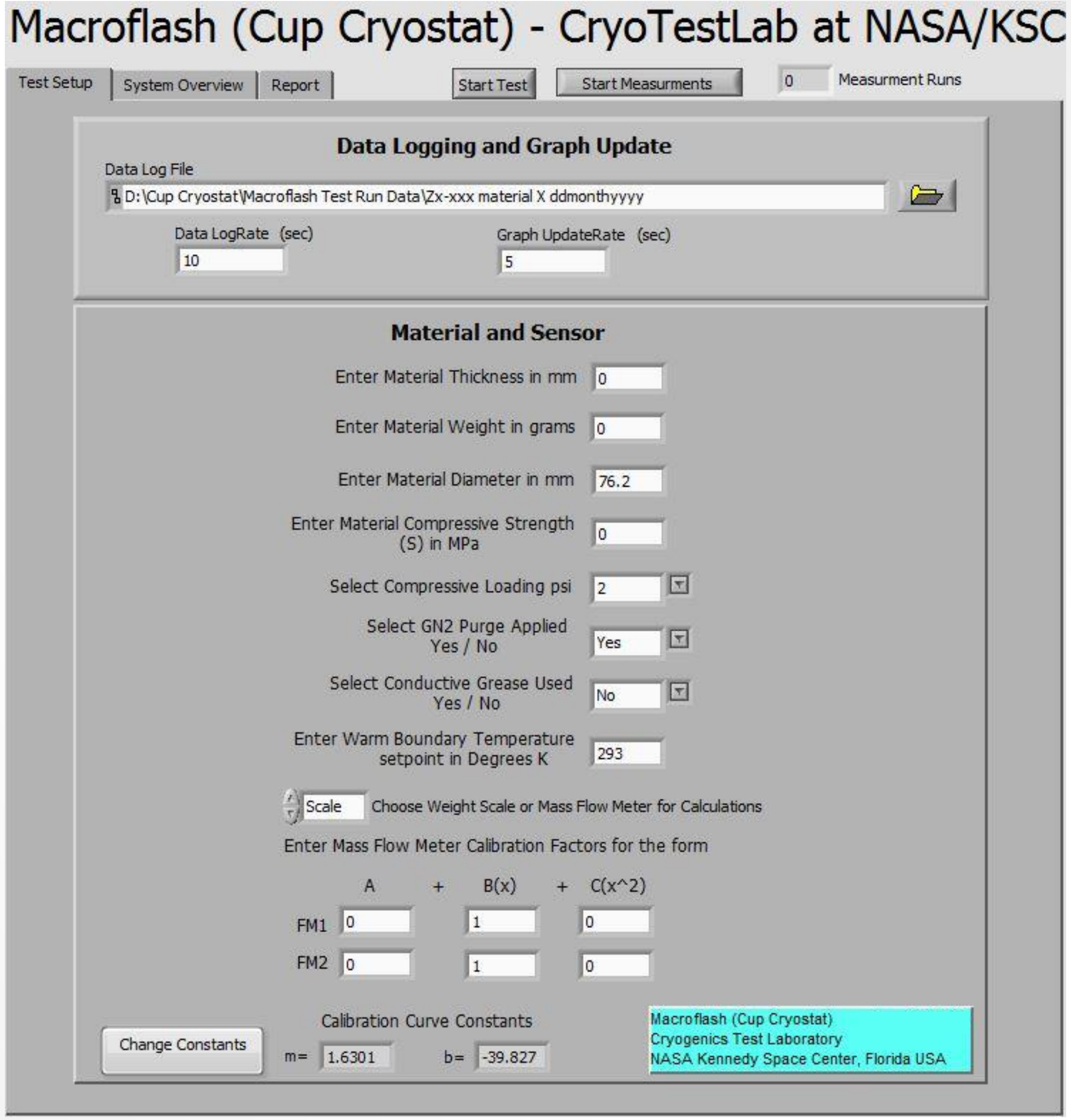
- 76.2-mm by 6.35-mm thick
- Flatness is important
- Material type
- Manufacturer
- Compressive strength (MPa)

Primary Inputs:

- Thickness & diameter (mm)
- Mass (g)
- WBT set point
- Interlayer temperatures (if used)

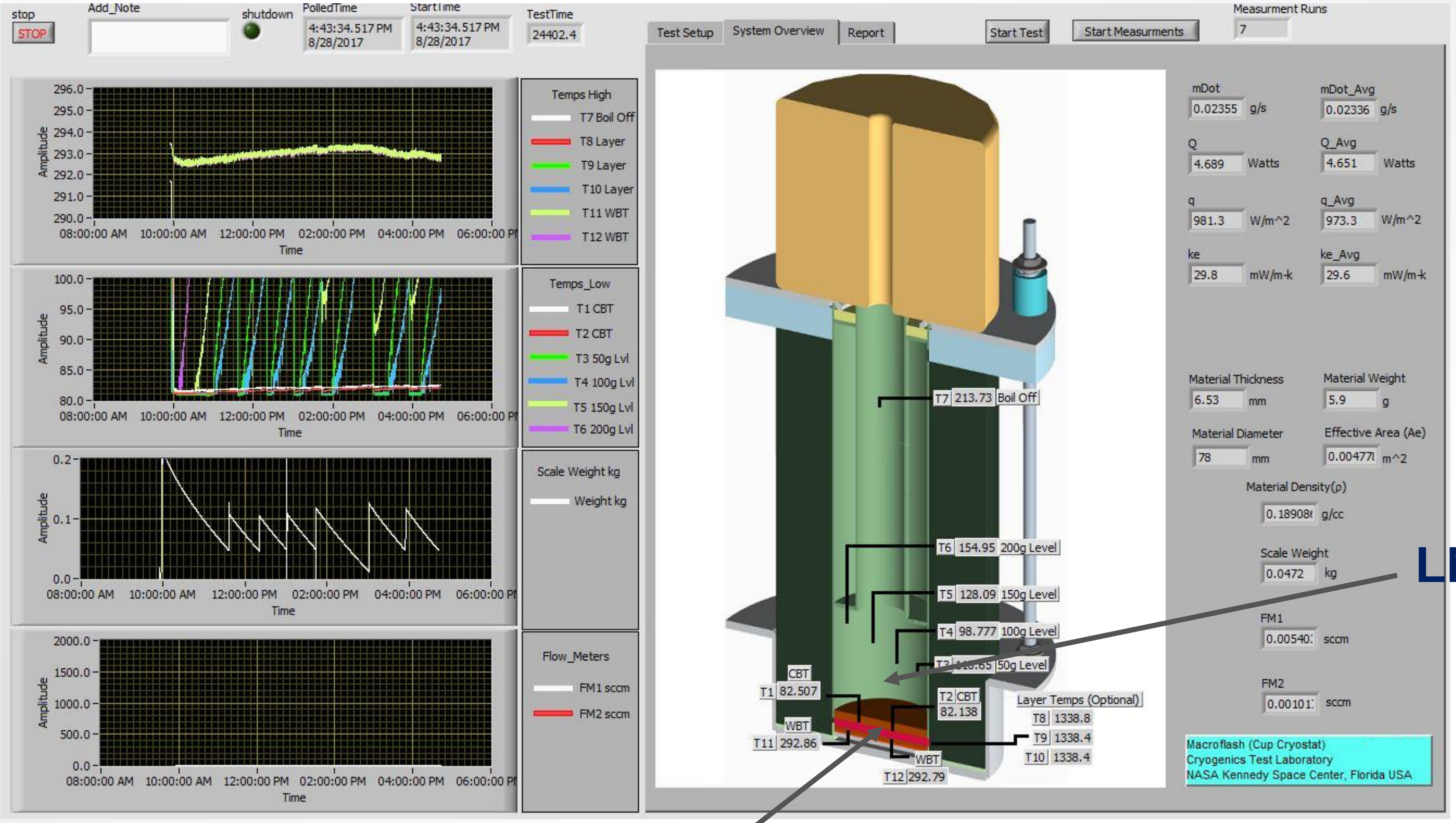
Test Parameters:

- Loading (0, 2, or 5 psi)
- Thermal greases (yes/no)
- Gaseous nitrogen (GN_2) purge (yes/no)
- Basis by weight scale or flow meter
- Flow meter constants (if used)
- Calibration range (LO, FULL, or HI)



PROCEDURE

Macroflash software interface: procedure



LN2

Test Specimen

Primary Inputs:

- Start: Cooldown and stabilization with >220 g LN_2
- Test Run: Steady-state measurement from >100 g to <50 g LN_2
- Repeat 3 times

Test Parameters:

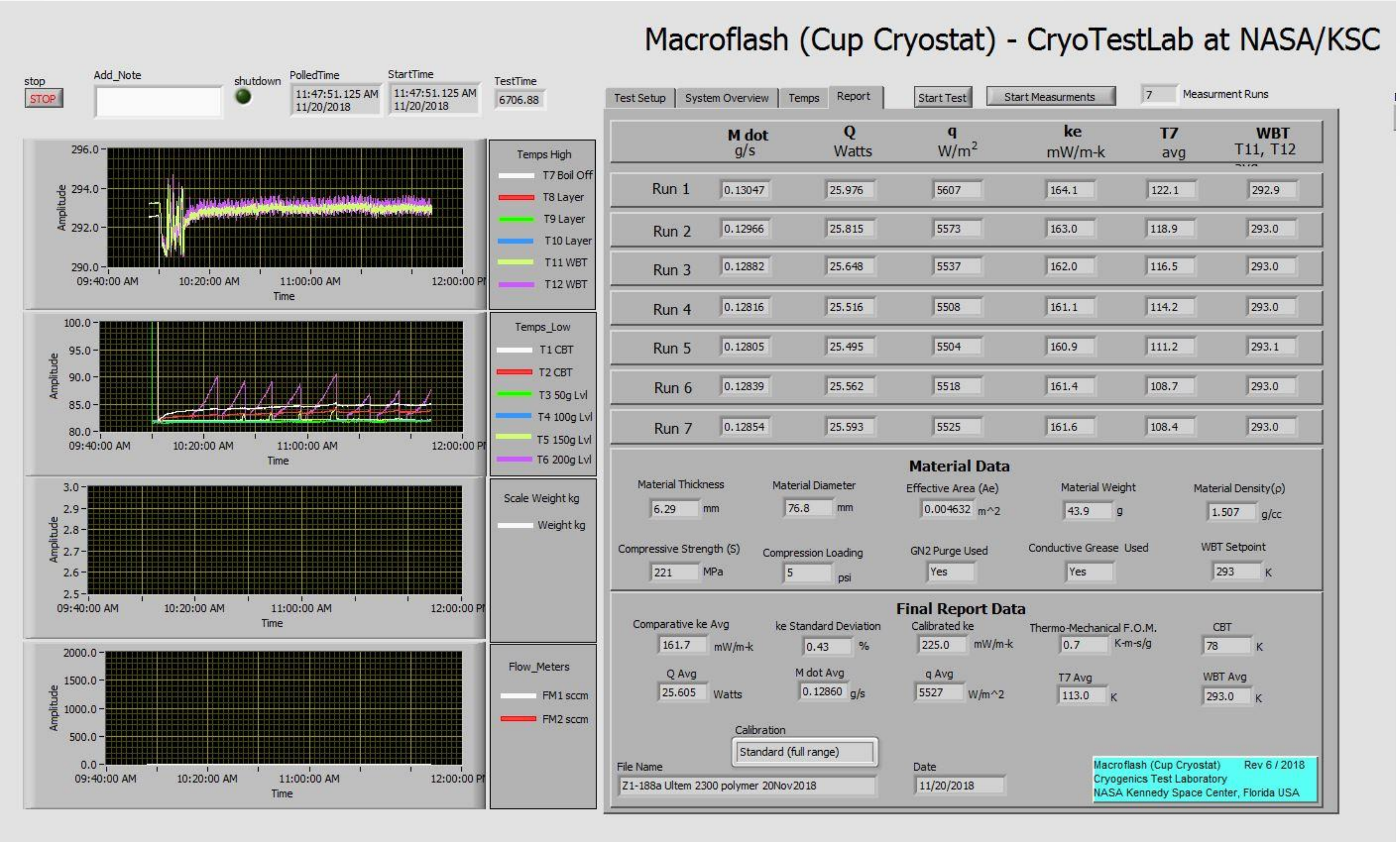
- WBT temp control

Specimen Inputs:

- Material type
- Manufacturer
- Compressive strength (MPa)

RESULTS

Macroflash software interface: test report



Primary Outputs:
- k_e calibrated
- Mdot (mass flow rate)
- q (heat flow rate)
- Q (heat flux)
- WBT and CBT

Test Parameters:
- Std dev
- Interlayer temps (optional)

Specimen Outputs:
- Structural-Thermal Figure-of-Merit (F_{ST})
- Bulk density

CYROTEK NASA CryogenicsTestLab KENNEDY SPACE CENTER

REPORTING OF RESULTS

Test Specimen:

- Test Series No.
- Material Code
- Material
- Test Date
- No. of test runs

Data Tabulation:

- Mdot
- Q
- q
- K_e-comp
- Std Dev
- K_e (calibrated)
- Comp Strength
- F_{ST}
- WBT & CBT (293 K & 78 K)

A	Aerogels
C	Composites
E	Epoxies
F	Foams
G	Glasses
L	Layered systems
M	Metals
P	Plastics
R	Reference Material (all kinds)
W	Woods
X	Exotica

Example of Macroflash Test Data Summary

Test Series	Matl Code	Test Specimen Description Material Type	Manufacturer	Test Runs	Density g/cm^3	Load psi	Grease Y or N	Q W	q W/m^2	Std Dev %	k_e mW/m-K calibrated	*Cal* L, S, H	Comp. Strength MPa	F_{ST} K-m-s/g
Z1-155	A	Ultra Low Density (ULD) Aerogel composite	Aspen Aerogels	7	0.118	0	N	4.651	1020	1.0	18.6	L	0.8	365
Z1-149	A	Cryogel x201 #3 (1 Layer)	Aspen Aerogels	5	0.166	0	N	5.970	1309	0.6	12.4	L		
Z1-118	A	X-aerogel Yellow Disk GRC (ref. Z315)	GRC	4	0.150	5	N	6.760	1482	0.8	25.0	L	1.6	426
Z1-115	A	X-aerogel FXP Tan Disk #1 (Flexcon)	Blueshift	3	0.148	5	N	7.299	1601	0.5	28.1	L	1.6	385
Z1-094a	A	Primaloft Thin Aerogel	Primaloft	4	0.247	2	N	4.933	1082	0.3	12.8	L		
Z1-158b	B	Glass Bubbles, nominal tap density	3M	5	0.073	0	N	7.070	1550	0.1	29.7	L	1.72	799
Z1-021	B	Perlite Powder		5	0.080	0	N	7.622	1671	0.1	33.4	L		
Z1-039	B	JSC Mars 1A regolith	JSC	4	0.783	2	N	15.411	3379	0.1	125	S		
Z1-188a	C	Ultem 2300 #1 (after sanding flat)	Sabic	7	1.507	5	Y	25.591	5612	0.4	225	S	221	651
Z1-162e	C	G-10 CR composite (cal)		7	1.939	5	Y	47.959	10516	0.4	464	S	448	498
Z1-017	C	G-10CR (warp direction)		7	1.929	5	Y	47.850	10492	0.2	624	S	448	372
Z1-130	E	DGEBF-DDS #8 Epoxy Resin	South Dakota	7	1.256	5	Y	20.117	4411	0.8	187	S	105	448
Z1-095	E	Epon862-04 (ref. Z325)	South Dakota	6	1.260	5	Y	22.666	4970	0.8	212	S	105	393
Z1-157a	F	FoamGlass #3	Pittsburgh Corning	6	0.118	5	N	7.455	1635	0.4	32.3	L	0.8	210
Z1-153	F	Polyimide Foam Solimide AC-550	Evonik	5	0.008	0	N	8.119	1780	0.4	36.6	L	0.01	34
Z1-150a	F	SOFI BX-265 #2	NCFI	3	0.037	2	N	5.916	1297	0.2	22.6	L	0.4	483
Z1-129	F	Rohacell WF-300 stack #3/#4	Evonik	4	0.319	2	N	8.592	1884	0.3	42.4	L	17.8	1317
Z1-125	F	Divinycell Foam H45 #5	Evonik	4	0.051	2	N	6.024	1321	0.1	23.9	L	0.6	493
Z1-018	F	Certifoam-25 Extruded Polystyrene	Diversifoam	4	0.032	2	N	5.620	1232	0.1	22.0	L	0.3	392
Z1-177	G	Plain Glass Sample #3		7	2.475	5	N	57.696	12652	0.5	872	S	50	23
Z1-160	L	Cabot Corp. Thermal Wrap	Cabot	5	0.074	2	N	5.604	1229	0.2	16.3	L		
Z1-032	L	Reflectix RSB3 Single Bubble	Reflectix	5	0.066	0	N	8.026	1760	0.1	33.3	L		
Z1-145	P	Aeroplastic Versify Sample-A 22-stack	KSC	5	0.896	5	N	18.883	4141	0.2	153	S		
Z1-015	P	Ultem PEI #1 (w/ grease)	Sabic	7	1.027	5	Y	18.889	4142	0.4	157	S	151	935
Z1-002	P	Teflon TFE	DuPont	7	2.120	5	Y	28.375	6222	0.5	253	S	24	45
Z1-151	W	Balsa (in-plane) 7.3mm		5	0.166	5	N	8.264	1812	0.6	46.0	L	7	916
Z1-020	W	Balsa Wood (with the grain; warp direction)		4	0.160	5	N	15.548	3409	0.4	133	S	7	328
Z1-014	W	Pine Wood #1		4	0.515	5	N	15.695	3442	0.0	128	S	7	106
Z1-182	X	Ceramic resin post-fire (U Mich)	Formlabs	7	1.412	5	Y	79.335	17397		370	S	72.2	138

CALCULATION & ANALYSIS

- Total uncertainty in k_e is calculated to be 4.8 % for the Macroflash
- Overall repeatability for most test series is demonstrated to be within 0.5%
- Direct calculation of heat flow rate (Q) by boiloff calorimetry

$$Q = mdot * hfg$$

$$k_e = \frac{Qx}{A_e \Delta T} = \frac{4Qx}{\pi d_e^2 \Delta T}$$

$$q = \frac{Q}{A_e}$$

Where:

mdot = mass flow rate (g/s)

h_{fg} = heat of vaporization (J/g)

k_e = effective thermal conductivity (mW/m-K)

x = thickness (mm)

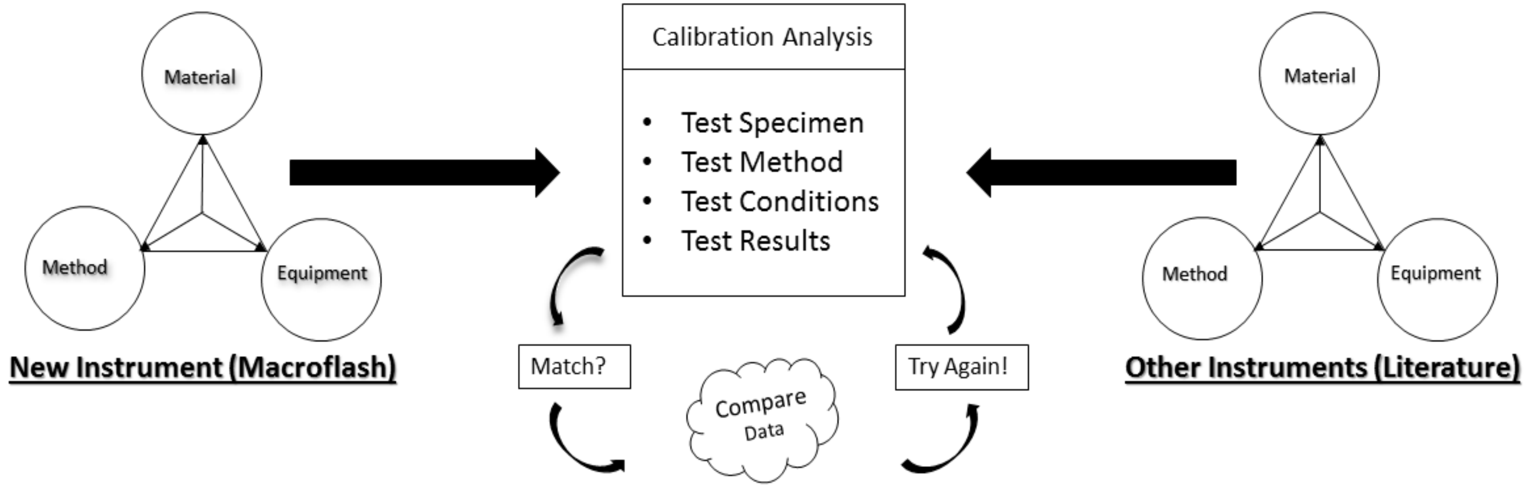
ΔT = temperature difference

d_e = effective diameter

A_e = effective area

CALIBRATION APPROACH

- Produce and compare thermal conductivity test data of reference materials under similar conditions:
  - With data from Cryostat-100 or Cryostat-500 (absolute instruments)
  - With literature data for similar materials
- Compare with "next best" related cryogenic data (the hard reality, in most cases)
- Connect back to ambient test data (and perform these tests also!)



1) Test Specimen: material, density, size, shape, surface finish, etc.
2) Test Method: type of apparatus, principal of heat flow calculation, steady state or transient, etc.
3) Test Conditions: WBT, CBT, ΔT, calculated T_m, thermal paste, compression load, gas environment, etc.
4) Test Results: type of thermal conductivity, large ΔT or small ΔT, T_m, method of calculating thermal conductivity (λ, λ_e, k-value, k_a, k_e, etc.)

CALIBRATION

- Macroflash has three ranges of calibration: low end (LO), full range (FULL), and high end (HI)
- The default is FULL but the user can select LO (<50 mW/m-K) or HI (>500 mW/m-K)

LO END Calibration Materials

Reference Material	Density	Macroflash Test Data	Absolute Value	Ref.
		comparative-Ke	k_e	
	kg/m^3	mW/m-K	mW/m-K	
Aerogel Blanket (Cryogel)	177	30.0	12.3	1
Polyiso Spray Foam (SOFI BX-265)	36	39.6	23.2	2
Divinycell Foam H45	50	40.3	24.2	3
Cellular Glass	118	48.2	32.0	4
Balsa wood (in plane)	166	60.1	45.0	5

1. Fesmire J, Ancipink J, Swanger A, White S, and Yarbrough D, "Thermal conductivity of aerogel blanket insulation under cryogenic-vacuum conditions in different gas environments," Advances in Cryogenic Engineering, IOP 278 (2017) 012198.

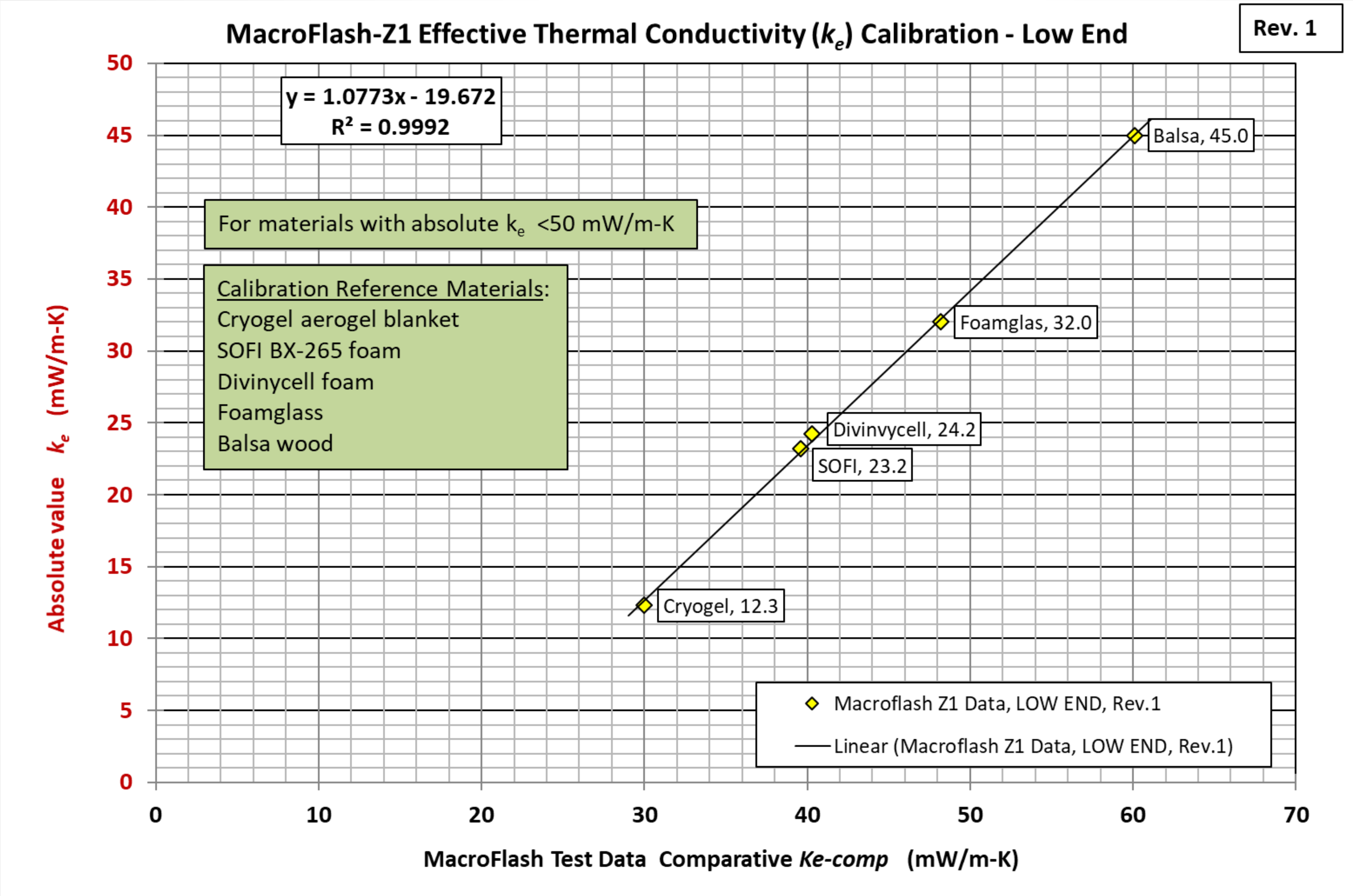
2. Fesmire, J. E., Coffman, B. E., Meneghelli, B. J., Heckle, K. W., "Spray-On Foam Insulations for Launch Vehicle Cryogenic Tanks," Cryogenics, doi:10.1016/j.cryogenics.2012.01.018.

3. Fesmire, J., et al., "Thermal Performance Testing of Candidate Tank Insulation Materials," Space Launch Initiative / 2nd Generation Launch Vehicles, Final Report to NASA-LaRC, Boeing, Northrup Grumman, May 30, 2002.

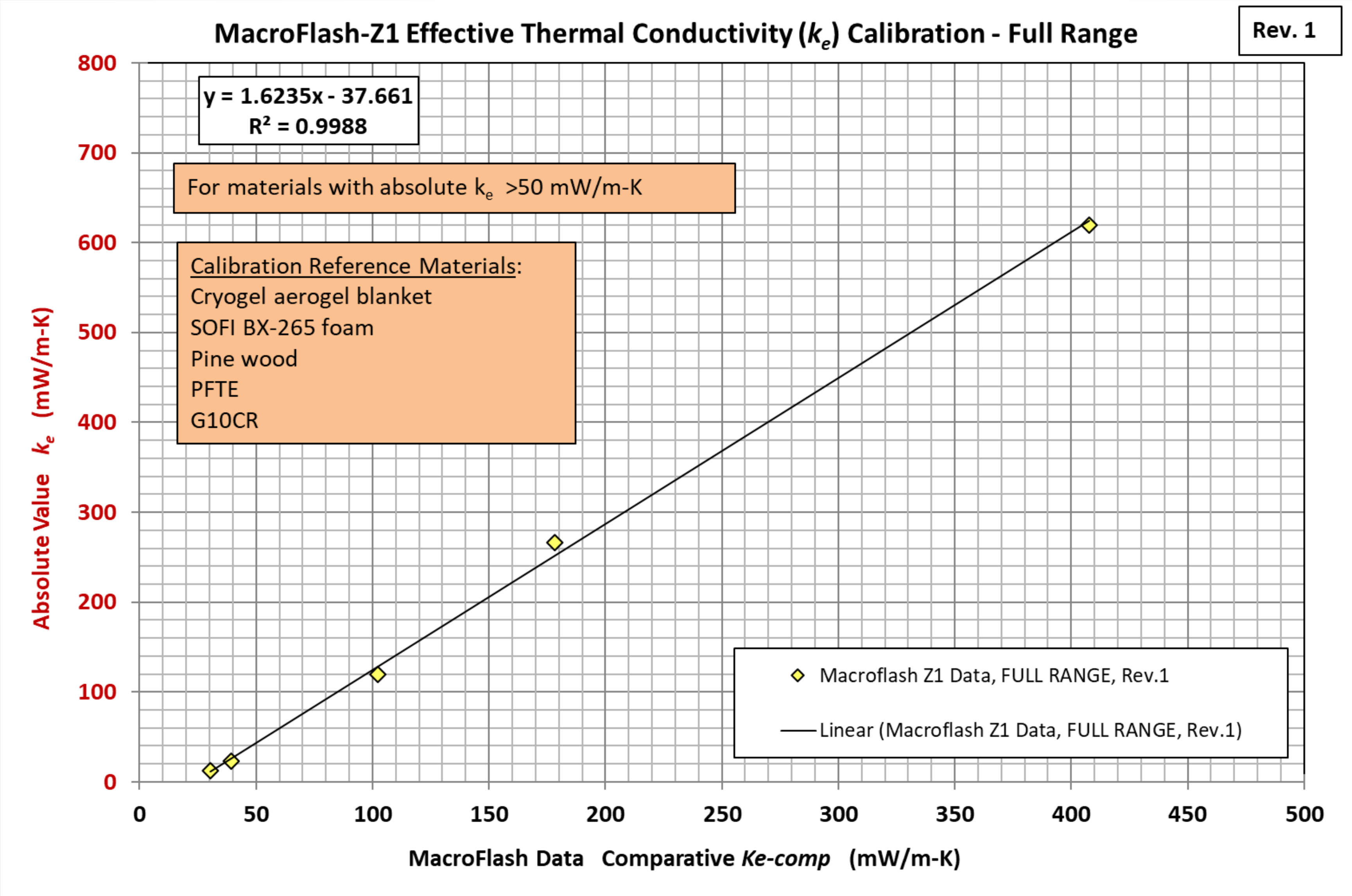
4. Flynn, Thomas, Cryogenic Engineering, 2nd edition, Marcel Dekker, New York, 2005.

5. National Institute of Standards, NIST Cryogenic Materials Property Database Index, https://www.nist.gov/mml/acmd/nist-cryogenic-materials-property-database-index

CALIBRATION (LO RANGE)



CALIBRATION (FULL RANGE)



TEST DATA

- Macroflash instruments have tested 500+ material test specimens representing thousands of test runs
  - Materials include composite panels, foams, ceramics, glasses, aerogels, layered composites, hybrid composites, and many others (insulators to conductors)
  - Extensive library and database of both “new” and “standard” materials
- Combined properties: Thermal Conductivity + Density + Strength

Structural-Thermal Figure-of-Merit (F_{ST})

where:

σ = compressive strength [MPa]
k_e = effective thermal conductivity [mW/mK]
ρ = bulk density [kg/m^3]

$$F_{ST} = \frac{\sigma}{\rho k_e} \text{ x } 10^6 \qquad \left[\frac{\mathrm{K \cdot m \cdot s}}{\mathrm{g}}\right]$$

CYROTEK NASA CryogenicsTestLab KENNEDY SPACE CENTER

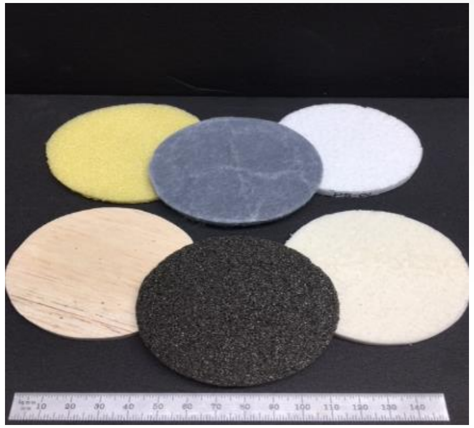
TEST DATA

- Example data for a range of insulation materials, polymers, and structural composites

Thermophysical data for structural-thermal materials used in cryogenic systems

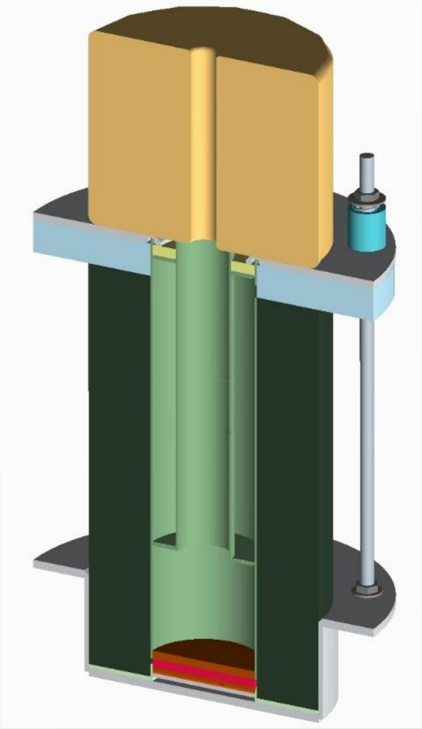
Material	$^{\dagger}\sigma$	ρ	$^{*}k_e$	F_{ST}
	MPa	kg·m^{-3}	mW·m^{-1}·K^{-1}	K·m·s·g^{-1}
G-10 (transverse direction)	448	1,939	467	495
Ultem® 2300 Glass Filled PEI	221	1,500	212	695
Ultem® 9185 PEI (3-D printed)	100	1,199	145	575
Teflon™ PTFE	24.1	2,120	253	45
Rohacell® WF-300 PMI foam (14 kPa)	17.8	324	42.1	1,305
Balsa Wood (transverse direction)	7.0	166	45.9	919
AeroZero® polyimide aerogel	1.6	150	28.1	380
Foamglas® Cellular Glass Foam	0.8	118	32.3	210
Divinycell® H45 PVC Foam (14 kPa)	0.6	50	23.8	504
Spray Foam Polyiso BX-265 (14 kPa)	0.4	37	22.6	483

†At ambient temperature *Boundary temperatures 293 K / 78 K; compressive load 5 psi or as noted.



CONCLUSION

- The easy to use Macroflash instrument provides effective thermal conductivity (k_e) data for a wide range of materials from thermal insulation to structural composites to ceramics
- The Macroflash follows the guidelines of standard ASTM C1774 (Annex A4), providing a cost-effective, real-world methodology to test any material for below-ambient temperature applications to moderately elevated temperature conditions
- Materials include solids, foams, powders, composites, and multi-layered systems and may be isotropic or non-isotropic; homogeneous or non-homogeneous; layered or engineered systems
- From materials development, to research testing, to quality control in manufacturing, the standardized device provides utility for the fields of energy, transportation, aerospace, construction, medical, and environment



CONCLUSION

Macroflash boiloff calorimetry instrument for the measurement of heat transmission through materials

- Calibration in work for HI range (>1,000 mW/m-K) materials
- Macroflash enables the testing of complex materials such as layered composites and other highly non-isotropic materials/systems
- Advancements in polymers, ceramics, carbon composites are opportunity for thermal characterization and materials science support
- Additive manufacturing technologies, for both metals and polymers, are opportunity to provide useful thermal data for engineered designs

CYROTEK NASA CryogenicsTestLab KENNEDY SPACE CENTER

THANK YOU

for your attention

Questions?

James E. Fesmire
James.e.fesmire@nasa.gov
1.321.867.7557

CYROTEK NASA CryogenicsTestLab KENNEDY SPACE CENTER