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Determination of the Young Modulus of Ti–$TiAl_3$ Metallic Intermetallic Laminate Composites by Nano-Indentation

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Nano-indentation is an important technique to determine the Young modulus of multiphase materials where normal tensile tests are not appropriate. In this work, Ti–$TiAl_3$ metallic–intermetallic laminate composites have been fabricated successfully in open atmosphere using commercial purity Al and Ti foils with 250 μm and 500 μm initial thicknesses, respectively. Sintering process was performed at 700 °C under 2 MPa pressure for 7.5 h. Mechanical properties including the Young modulus were determined after manufacturing. The Young moduli of metallic and intermetallic phases were determined as 89 GPa and 140 GPa, respectively. Microstructure analyses showed that aluminum foil was almost consumed by forming a titanium aluminide intermetallic compound. Titanium aluminides grow up through spherical shaped islands and metallic–intermetallic interface is a wavy form in Ti–Al system. Thus, the final microstructure consists of alternating layers of intermetallic compound and unreacted Ti metal. Microstructure and phase characterizations were performed by scanning electron microscopy, energy dispersive spectroscopy, and X-ray diffraction. Hardness of test samples was determined as 600 HV for intermetallic zone and 130 HV for metallic zone by the Vickers indentation method.

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1. Introduction

Nano-indentation is a particularly attractive technique for extracting the Young modulus of these intermetallics because of the relatively small volume being tested. Thus, any adverse effects of oxidation and porosity commonly obtained in bulk property measurements can be avoided. Indeed, the properties measured from nano-indentation are the true properties of the intermetallic layers [1]. During the indentation, the most interesting phenomenon is the indentation size effect (ISE) which is manifested as an increase in hardness H with decreasing indentation peak load $P_{\max}$ [2, 3]. The goal of the majority of nano-indentation tests is to extract the Young modulus and hardness of the specimen material from load-displacement measurements. Conventional indentation hardness tests involve the measurement of the size of plastic indentation in the specimen as a function of the indenter load. This provides a measure of the area of contact for a given indenter load [3].

It is well known that the widespread engineering application of ceramics and other highly brittle materials, e.g. intermetallic compounds, is severely limited by their low toughness [4]. One of the most effective toughening techniques is to introduce a ductile phase, which remains intact and bridges the crack faces in the wake of a growing crack which enhances the toughness. In this case very large crack bridging ligaments can be formed, which stabilize the crack [5–9]. Specifically, the titanium–titanium tri-aluminide (Ti–$TiAl_3$) metallic–intermetallic laminate (MIL) composite system has great potential for structural applications because of its combination of high strength, toughness and stiffness at a lower density than monolithic titanium or other laminate systems [10–13]. In the Ti–Al system there are various possible aluminides. Among this the formation of the intermetallic $TiAl_3$ is thermodynamically and kinetically favored over the formation of other aluminides when reacting Al directly with Ti. This preferential formation of $TiAl_3$ is fortuitous as its the Young modulus (216 GPa) and oxidation resistance are higher, and the density (3.3 g/cm^3) is lower than that of the other titanium aluminides such as Ti_3Al and TiAl [5–7, 14, 15].

The objective of the present research is to synthesize titanium–titanium tri-aluminide metallic–intermetallic composites and characterize some of the optical and mechanical properties such as the Young modulus via nano-indentation.

2. Materials and methods

The MIL process consists of stacking commercial purity aluminum and titanium aluminide foils in alternating layers. The foils were provided from Alfa Aesar company and their properties are shown in Table I.

The dimensions of the processed samples are in the shape of plates as 10 mm × 10 mm. 3% HF, 15% HNO_3 and H_2O was used to remove oxide layers on the surface. After chemical treatment, the samples were rinsed in alcohol and dried. Each stack consisted of 5 titanium

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TABLE I

Properties of foils used in experiment.

foil	thickness [μm]	purity [%]
Ti	500	99.5
Al	250	99.5

and 4 aluminum foils. An initial pressure of 20 MPa is applied at room temperature to ensure good contact between foils. Sintering process is applied in an open-air electrical resistance furnace at 70 °C for 7.5 and 10 h. The morphologies of the samples were examined by scanning electron microscopy (SEM-EDS) in terms of resulting phases. The microhardness of the investigated samples was measured employing a Vickers indentation technique with a load of 50 gf using Struers Duramin hardness instrument. For determination of the Young modulus of the samples, nano-indentation device DUH-211S by Shimadzu was used with 200 mN load and 25 s holding time with the Vickers type indenter (Fig. 1).

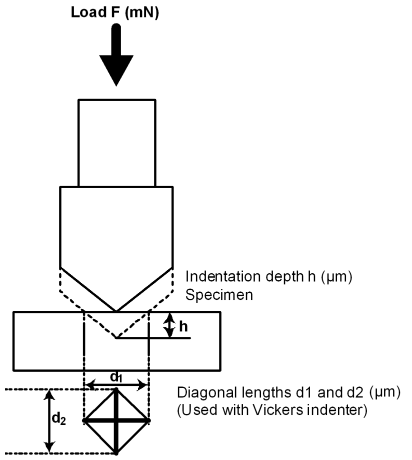

Fig. 1. Vickers type indenter.

3. Experimental results

3.1. Microstructure and SEM-EDS analyses

The typical microstructures of the MIL composites are shown in Fig. 2. The presence of different regions indicates the different phases in the composites. In relatively

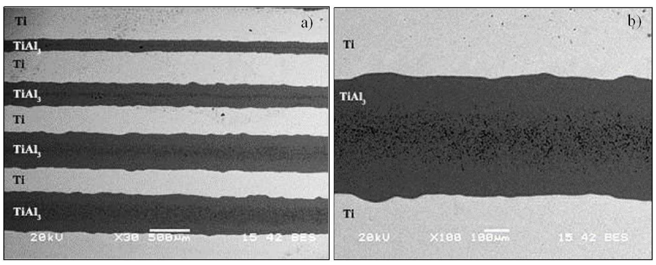

Fig. 2. SEM micrograph of laminated composites produced at 700 °C and 7.5h. (a) 30X, (b) 100X magnification.

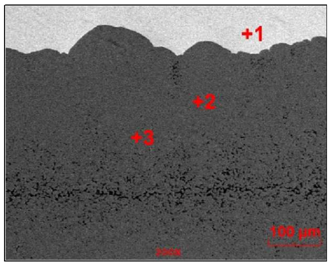

Fig. 3. SEM-EDS analyses of laminated composites produced at 700 °C and 7.5 h.

low magnification (Fig. 2a), it appears that the foils are stacked properly. Furthermore, the laminated composites are well-bonded. There is a symmetric change from a light colored phase to another. Following the light colored phase, there is a grey colored formation, subsequently, darker grey colored phase and in the center line a black colored phase. The light colored layers consist of unreacted Ti, which are separated by the apparently darker TiAl$_3$ layers, as was identified by quantitative SEM-EDS analysis in Fig. 3 and Table II.

TABLE II

EDS analyses of laminated composites produced at 700 °C and 7.5h.

Analytic points	Elements [wt.%]			Total
	Ti	Al	O	
1	100	–	–	100
2	30.89	69.11	–	100
3	30.36	69.64	–	100

3.2. Nano-indentation and hardness

A typical nano-indentation curve used for calculation of the elastic unloading modulus is shown in Fig. 4. Here: F = load, h = penetration depth, S = elastic unloading stiffness.

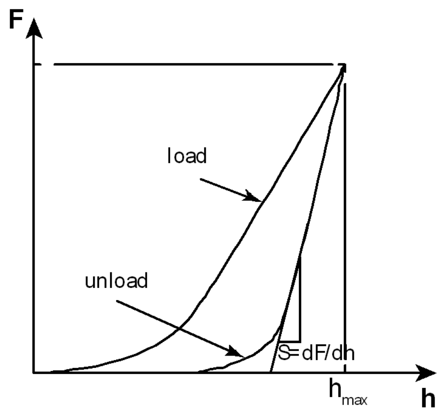

Fig. 4. Typical load-displacement curve used for calculation of the Young modulus [16].

The indentation procedure consisted of pressing an indenter onto the surface of a sample by applying an increasing normal load. The Young modulus of the specimen can be determined from the slope of the unloading of the load-displacement response. The modulus measured in this way is formally called the "indentation modulus" E_i of the specimen material. Ideally, the indentation modulus has precisely the same meaning as the term "the Young modulus" [1, 3]. The value of the Young modulus of the indented material is obtained from expression (1) where E_i and υ_i are the elastic modulus and Poisson's ratio of the indenter, and υ_{it} is Poisson's ratio of the indentation zone [17] (set at 0.37 for the whole sample). In this study, E_i was taken as 1.141 GPa and υ_i as 0.07.

$$\frac{1}{E_r} = \frac{1-\nu_{it}^2}{E_{it}} + \frac{1-\nu_i^2}{E_i}. \tag{1}$$

the Young modulus and hardness of metallic Ti phase are: 87 ± 12 GPa and 128 ± 9 HV, whereas intermetallic region is 141 ± 7 GPa, 600 ± 25 HV. The value of indentation modulus can be affected greatly by material behavior (e.g. piling-up) that is not accounted for in the analysis of load-displacement data. For this reason, care has to be taken when comparing the modulus for materials generated by different testing techniques and on different types of specimens [3]. Having some porosity in the metallic and intermetallic region (residual aluminum), elastic modulus values are detected lower than the literature value of 216–220 GPa [18]. Besides this 7.5 h sintering time was not enough for a fully dense microstructure. Nano-indentation curve for this type of MIL composite can be seen in Fig. 5.

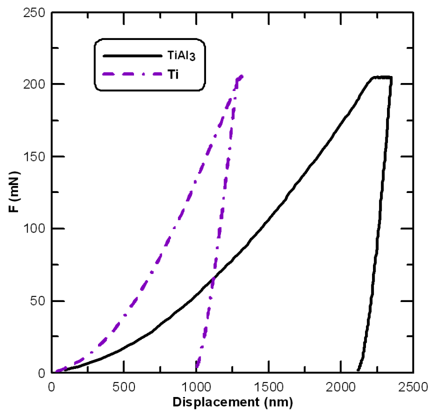

Fig. 5. Nano-indentation curve for MIL composite sintered at 700 °C and 7.5 h.

4. Conclusions

Ti–$TiAl_3$ metallic–intermetallic laminate (MIL) composites have been successfully synthesized by reactive foil sintering technique in open air at 700 °C for 7.5 h under 2 MPa pressure. The laminated structure is well-bonded, nearly fully dense. A tailored amount of residual aluminum remains at the intermetallic centerline.

Microstructural characterization by SEM and EDS indicates that Ti and $TiAl_3$ are intermetallic phases in the composite. Titanium aluminide phase occurs due to the thermodynamic reaction between Ti and Al. The existence of liquid Al phase plays important roles in the nucleation and growth of $TiAl_3$ particles and the eventual formation of continuous alternative intermetallic layers.

The hardness of fabricated laminated composite was dramatically changed. Whereas the hardness of metallic aluminum and Ti respectively is about 45 and 110 HV, hardness of intermetallic zone is approximately 600 HV.

the Young modulus of the samples are detected via nano-indentation technique. the Young modulus value of 87 GPa was obtained for the metallic phase; whereas it increased to 141 GPa for intermetallic phase.

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