Polish Society of Composite Materials
Polskie Towarzystwo Materiałów Kompozytowych

13: 2 (2013) 102-106

COMPOSITES THEORY AND PRACTICE

Marek Konieczny

Kielce University of Technology, Faculty of Mechatronics and Machine Building, al. 1000-lecia PP 7, 25-314 Kielce, Poland
Corresponding author: E-mail: mkon@interia.pl

Received (Otrzymano) 28.10.2012

MECHANICAL PROPERTIES OF Ti-(Al_3Ti+Al) AND Ti-Al_3Ti LAMINATED COMPOSITES

Laminated Ti-(Al_3Ti+Al) and Ti-Al_3Ti composites have been synthesised with controlled temperature and treating time using 50, 100 and 150 μm thick titanium and 50 μm thick aluminium foils. Microstructural examinations showed that Al_3Ti was the only phase formed during the reaction between Ti and Al. After 20 minutes of treating at 650°C, not all the aluminium was consumed and the composites consisted of alternating layers of Ti, Al and Al_3Ti. After 60 minutes, the aluminium layers were completely consumed, resulting in microstructures with Ti residual layers alternating with the Al_3Ti layers. Tensile strength, flexural strength and impact toughness measurements were performed on the materials with different microstructures to establish the properties and fracture behaviour. After 60 minutes of treating, all the composites had a higher yield strength, higher ultimate tensile strength and higher flexural strength than those composites after 20 minutes of treating produced with the same thickness of starting Ti foil. On the other hand, the strain at fracture and impact toughness of the composites behaved conversely. The results of the investigations indicated that the mechanical properties of the composites strongly depend on the thickness of the individual Ti layers and the presence of residual Al layers at the intermetallic centrelines.

Keywords: laminated composites, Al_3Ti intermetallic phase, mechanical properties, fracture

WŁASNOŚCI MECHANICZNE KOMPOZYTÓW WARSTWOWYCH Ti-(Al_3Ti+Al) I Ti-Al_3Ti

Kompozyty warstwowe Ti-(Al_3Ti+Al) oraz Ti-Al_3Ti wytworzono w kontrolowanych warunkach, używając jako materiałów wyjściowych folii tytanowej o grubościach: 50, 100 i 150 μm oraz folii aluminiowej o grubości 50 μm. Mikroanaliza rentgenowska pozwoliła stwierdzić, że faza Al_3Ti była jedyną fazą międzymetaliczną powstałą podczas reakcji między tytanem i aluminium w temperaturze 650°C niezależnie od czasu reakcji. Badania wykazały, że po 20 minutach wygrzewania aluminium nie w pełni przereagowało i dlatego kompozyty składały się z warstw Ti, Al oraz Al_3Ti. Po 60 minutach wygrzewania kompozyty składały się już tylko z naprzemiennie ułożonych warstw Ti oraz Al_3Ti. Wszystkie wytworzone kompozyty poddano próbie statycznej rozciągania, zginania trójpunktowego oraz udarności. Kompozyty składające się tylko z warstw Ti oraz Al_3Ti charakteryzowały się większą granicą plastyczności, wytrzymałością na rozciąganie oraz zginanie w porównaniu do kompozytów Ti-(Al_3Ti+Al). Z drugiej jednak strony kompozyty zawierające warstwy Ti, Al i Al_3Ti charakteryzowały się większym wydłużeniem oraz wyższą udarnością. Rezultaty badań wykazały jednoznacznie, że właściwości mechaniczne kompozytów są silnie zależne od grubości indywidualnych warstw tytanu oraz od obecności warstw aluminium. Na podstawie analizy fraktograficznej dokonano oceny mechanizmów niszczenia tych materiałów ze szczególnym uwzględnieniem wpływu obecności warstw aluminium występujących pomiędzy warstwami fazy Al_3Ti na propagację pęknięć.

Słowa kluczowe: kompozyty warstwowe, faza międzymetaliczna Al_3Ti, własności mechaniczne, pękanie

INTRODUCTION

Metal-intermetallic laminated (MIL) composites have the potential to perform various functions, such as ballistic protection, blast mitigation, thermal management, heat exchange and vibration damping [1]. Lamination can significantly improve many properties including fatigue behaviour, fracture toughness, wear, corrosion and damping capacity, or provide enhanced formability or ductility for brittle intermetallics [2]. The production techniques of MIL composites may be divided into deposition or bonding. Sputter or vapour deposition techniques involve the atomic scale transport of component materials. Such nano-engineered laminated materials are typically fabricated by depositing hundreds of alternate nanoscale layers and they have received significant interest due to their extremely high strength [3]. Unfortunately, deposition techniques require sophisticated manufacturing equipment and are too slow to be practical for making large-scale components. On the other hand, bonding techniques (diffusion bonding, transient liquid phase bonding and reaction bonding) between metal foils involve relatively simple processing [4]. Furthermore, the laminated structure of the composite allows for variations in the layer thickness and phase volume fractions of the components simply through the selection of initial foils thickness. A great number of laminated composites have been produced using Al and Ni [4-6], Nb [7], Fe [8] or Mg [9, 10] foils. Among laminate composites, Ti-intermetallic laminates have a great technological advantage and attract special attention for various applications [1]. Previous works reveal that titanium-intermetallic composites can be produced by a reaction that occurs at the interface of Ti and Nb [11], Ti and Cu [12] or Ti and Ni [4]. In particular, the Ti-Al system has a great practicable capability. The titanium aluminides Ti_3Al, TiAl and Al_3Ti offer the potential for an increased temperature range and enhanced high temperature strength, stiffness and oxidation resistance compared to conventional titanium alloys [13]. Specifically, Ti-Al_3Ti laminated composites can be considered for aerospace, automotive and other structural applications because of their lower density than monolithic titanium or other Ti-based laminates. Furthermore, the Ti-Al_3Ti system is economically more attractive than monolithic titanium because aluminium is relatively inexpensive. In the present study, the reaction synthesis process was employed to fabricate laminated composites in vacuum using Ti and Al foils. Mechanical tests were performed on materials with different microstructures to establish the properties of the composites and their fracture behaviour.

EXPERIMENTAL PROCEDURE

In the experiment, 50, 100 and 150 μm thick foils of titanium (99.14 at. % Ti) and 50 μm thick foil of aluminium (99.53 at. % Al) were used to produce laminated titanium-intermetallic composites with controlled treating time, temperature and pressure. The titanium and aluminium foils were cut into 50 mm x 10 mm rectangular pieces. Any contamination on the surface of foils was removed in a bath of 5 pct HF in water. After that, the foils were rinsed in water and then in ethanol. After drying rapidly, they were stacked into laminates in an alternating sequence. To obtain 5 mm thick laminates, 51, 34 or 26 pieces of Ti were used, depending on the thickness of the Ti foils. A pressure of 5 MPa was employed at room temperature in a specially constructed vacuum furnace to ensure good contact between the metals. The samples were heated in a vacuum of 0.01 Pa at 600°C for 2 h under applied 5 MPa pressure to allow diffusion bonding of the layers. After that, the foils were heated to 650°C and held at this temperature for 20 or 60 minutes. The pressure was reduced to 1 MPa during this processing sequence. The temperature was then decreased slowly to 600°C and the pressure of 5 MPa was applied again. The thermal ageing cycle at 600°C was employed for 2 hours to remove any residual porosity that might have formed as a result of solidification of the transient liquid phase. Finally, the samples were furnace-cooled to room temperature. The laminated composites prepared from 50, 100 and 150 μm thick Ti foils are denoted later as samples Ti50, Ti100 and Ti150, respectively. After fabrication, the specimens were cut, mounted in a cold setting resin, mechanically polished initially with a grade 800 abrasive paper and finally using a Struers polishing machine using a 1 μm diamond suspension. Microstructural observations were performed using a JEOL JMS 5400 scanning electron microscope. The volume fraction of the metals and intermetallics was determined experimentally from image analysis. The chemical composition of the phases was determined by energy dispersive spectroscopy by means of an ISIS 300, Oxford Instruments. X-ray diffraction using a D/max RAPID2 diffractiometer was employed to identify the intermetallic phases. Samples with dimensions of 50 mm x 8 mm x 4 mm, made from fabricated composites, were subjected to tension tests on an INSTRON screw machine at a constant crosshead speed of 0.1 mm/min. The flexural strength measurements were performed using a three-point bending test on un-notched specimens with dimensions of 40 mm x 4 mm x 4 mm where the loading span was 30 mm. The tests were carried out under displacement control at a rate of 0.2 mm/min. Charpy tests were conducted on a pendulum impact tester using identical rectangular samples as in the three-point bending test. Three-point bending and impact tests were performed in both the perpendicular and parallel directions to the metal-intermetallic interfaces. The reported data for all the mechanical tests are average values of three tested specimens.

RESULTS AND DISCUSSION

The composition of the synthesised phases in the composites was determined by comparing the results of the EDX analysis with the data in the binary Ti-Al phase diagram [14]. The examinations showed that only an Al_3Ti intermetallic phase was formed in the samples irrespective of the treating time at 650°C. The XRD results confirmed that the composites treated for 20 minutes consisted of alternating Ti, Al_3Ti and Al layers. No unreacted Al was detected in any of the composites processed for 60 minutes, therefore the composites consisted of Ti and Al_3Ti layers. The microstructural investigations also indicated that the central zones of the formed Al_3Ti layers contained many Al_2O_3 inclusions. They are thought to originate from surface oxide films on the Al foils, which after breakdown were pushed aside towards the liquid Al side by the growing continuous Al_3Ti layers. A migration of oxide films from the surfaces of Al foils to the middle of the formed intermetallic layers was previously observed during the formation of other aluminides belonging to the binary Ni-Al [5, 6] and Mg-Al [10] systems. This is an obvious disadvantage because any oxides accumulated at the centreline of intermetallics can be weak points in the microstructure making the development of cracks possible. Table 1 summarises the tensile properties of all the synthesised composites.

TABLE 1. **Tensile properties and phase composition of Ti-(Al_3Ti+Al) and Ti-Al_3Ti composites**

TABELA 1. **Właściwości mechaniczne oraz skład fazowy kompozytów Ti-(Al_3Ti+Al) oraz Ti-Al_3Ti**

Treating time at 650°C [min]	Sample designation	Volume fraction [%]			Tensile properties		
		Ti	Al	Al_3Ti	σ_{YS} [MPa]	σ_{UTS} [MPa]	Elongation [%]
20	Ti50/20	44.4	28.3	27.3	155	216	16.4
	Ti100/20	63.8	18.2	18.0	167	224	16,8
	Ti150/20	73.1	13.8	13.1	172	230	17,3
60	Ti50/60	24.0	0	76.0	234	454	3.2
	Ti100/60	54.5	0	45.5	231	388	8.2
	Ti150/60	67.7	0	32.3	228	350	10.8

With an increase in the treating time at 650°C, the Al_3Ti layers grow, leading to an increase in volume fraction of the intermetallics. As a result, the yield strength and the tensile strength of all the investigated composites increased and the total strain at fracture decreased. During deformation, the crystal structures mismatch between the base metals and the Al_3Ti layers, making it almost impossible for the slip-bands to slide through the metal/intermetallic interfaces. Therefore, the formation of cracks in the Al_3Ti layers was a characteristic feature of the deformation. The observed serrations in the stress-strain curves corresponded to the formation of multiple cracks in the intermetallic layers (Fig. 1).

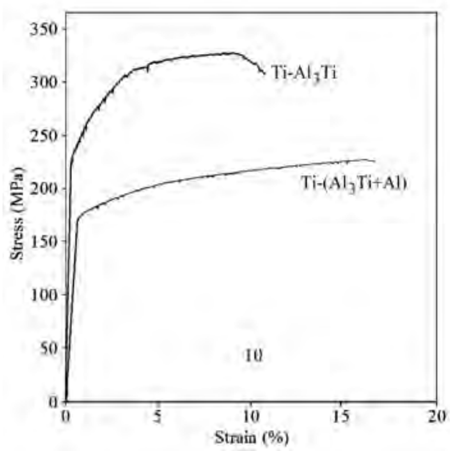

Fig. 1. Tensile curves for Ti-(Al_3Ti+Al) and Ti-Al_3Ti composites

Rys.1. Krzywe rozciągania kompozytów Ti-(Al_3Ti+Al) oraz Ti-Al_3Ti

The cracks developed at defect points within the brittle Al_3Ti layers and then propagated across them. The perpendicular cleavage cracks were blunted by the titanium and aluminium layers when the cracks reached the Ti/Al_3Ti or Al/Al_3Ti interfaces. The energy absorption capability of the metal layers allowed numerous cracks to develop within each intermetallic layer before failure. With a permanent increase in the number of cracks in the Al_3Ti layers, the titanium and aluminium layers gradually underwent total external load. As a result, the plastic flow that took place in the metals layers was restricted to small regions between opposite cracks in the neighbouring Al_3Ti layers. When the number and distribution of cracks in the intermetallic layers reached a critical limit, final failure occurred by shearing fracture of the metals layers (Fig. 2).

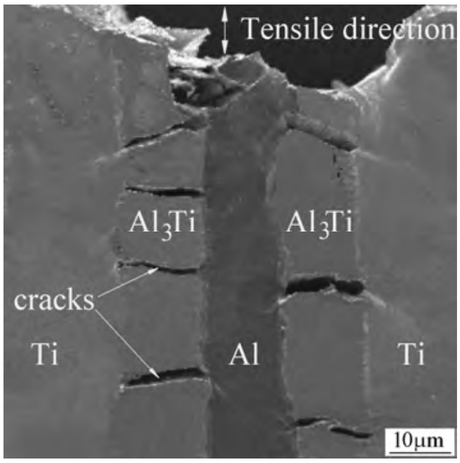

Fig. 2. Cross-section of fractured Ti-(Al_3Ti+Al) tensile specimen showing metals layers bridging many cracks in aluminide layers

Rys. 2. Miejsce zerwania próbki z kompozytu Ti-(Al_3Ti+Al) w próbie rozciągania z widocznymi wieloma pęknięciami w warstwach Al_3Ti

The only exception to the deformation model was the Ti50/60 composite having the thinnest titanium layers (only 17.6 μm). Since there was insufficient material in the metal layers to absorb the cracks energy release, only one single crack propagated. As a result, the Ti50/60 composite failed in a brittle manner. The obtained results are stringently compatible with the results previously reported by Vecchio [1] and Alman et al. [4]. The fracture behaviour of the Ti-(Al_3Ti+Al) and Ti-Al_3Ti laminated composites was typical of ductile-phase-toughened matrix composites. The failure characteristic described for the Ti-(Al_3Ti+Al) and Ti-Al_3Ti composites is strictly consistent with previous studies of cracking and damage mechanisms in Ti-Ti_2Ni [4], Nb-Nb_3Al [7], Ni-Ni_2Al_3 [5] and Cu-(Cu_4Ti+CuTi) [15] laminated composites. The damage mechanisms of the unlike laminated composites during tensile testing are alike because different intermetallics formed from different constituent metals behave in a very similar manner. They are ordinarily brittle at room temperature due to the limited mobility of dislocations, have an insufficient number of slip or twinning systems, and very low surface energy resulting in little or no plastic deformation at the crack tips [16].

The Ti-(Al_3Ti+Al) and Ti-Al_3Ti laminated composites were also investigated at parallel and perpendicular load directions during three-point bending tests. The bend curves for both types of specimens showed a similar pattern when a load perpendicular to the laminates was applied. They contained several load drops due to cracking of the different intermetallic layers and flat regions that corresponded to plastic deformation of the metal layers. Progressive cracking in the intermetallic layers created stress concentration points at the metal-intermetallic interfaces, which formed shear bands that propagated from the crack tip in the titanium and aluminium layers to successive Al_3Ti layers. The following load drop occurred at the critical strain needed for crack renucleation. This process was repeated until all the layers cracked, resulting in a step-like load-displacement response (Fig. 3).

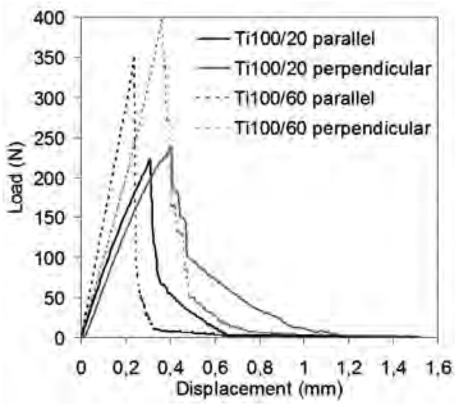

Fig. 3. Load-displacement curves of Ti100/20 and Ti100/60 specimens at different load directions during three-point bending test

Rys. 3. Krzywe obciążenie-przemieszczenie uzyskane podczas prób zginania trójpunktowego kompozytów Ti100/20 oraz Ti100/60 w kierunku prostopadłym i równoległym do warstw kompozytów

The curve corresponding to the Ti-Al_3Ti laminate remains nearly linear up to the peak load, at which a crack initiates, giving the first load drop. In the Ti-Al_3Ti specimens, a main crack travelled across the titanium layer but then was arrested and deflected by the adjacent Al_3Ti layer causing a prevalent intermetallic centreline fracture. It could happen because as was mentioned before, the centrelines of the intermetallic layers were weakened by the presence of Al_2O_3 inclusions. Further loading caused transverse cracking of the Al_3Ti layer and the formation of some new cracks in the next metal layer (Fig. 4).

On the other hand, the bend curve for the Ti-(Al_3Ti+Al) laminate is not linear and serrated load deflections were noticed before the maximum load. The load peaks are thought to be the critical stresses needed for crack nucleation and the load minima points correspond to cracks arresting at the Al_3Ti/Al interfaces. Furthermore, any centreline fracture of the Al_3Ti layers was noticed in the fractured Ti-(Al_3Ti+Al) composites. When the load direction was parallel to the laminates, the curves for both types of composites grew continuously up to the peak load, at which point a crack initiated at the surface. The failure occurred by the cleavage mode showing limited plastic deformation. The investigations showed that only one main crack grew gradually in the through-thickness direction and finally travelled across the specimens. This failure mechanism has been shown in the literature to be typical of laminated composites [7]. Table 2 summarises the flexural strength of all the synthesised composites.

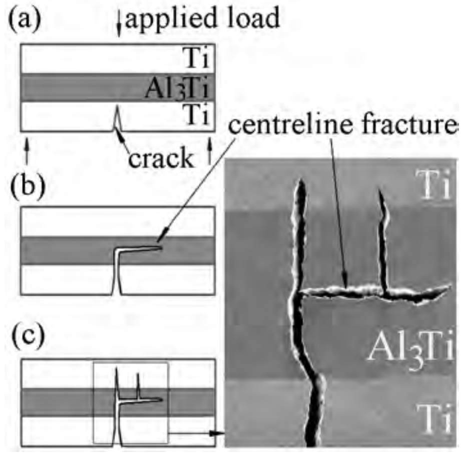

Fig. 4. Crack propagation in Ti-Al_3Ti specimen loaded perpendicular to layers

Rys. 4. Propagacja pęknięć w kompozycie Ti-Al_3Ti obciążanym prostopadle do warstw

TABLE 2. **Mechanical properties of investigated composites under different testing conditions**

TABELA 2. **Właściwości mechaniczne badanych kompozytów uzyskane w próbie zginania trójpunktowego oraz udarności**

Treating time at 650°C [min]	Sample desig-nation	Flexural strength [MPa]		Impact toughness [J/cm^2]	
		Perpen-dicular	Parallel	Perpen-dicular	Parallel
20	Ti50/20	184	171	38	16
	Ti100/20	168	153	53	22
	Ti150/20	153	144	62	27
60	Ti50/60	316	262	6	4
	Ti100/60	279	245	18	11
	Ti150/60	258	234	25	18

The flexural strength of the composites is dependent on the number and thickness of the individual layers. It increases with a decreasing remaining Ti metal thickness in the Ti-Al_3Ti composites. Furthermore, the Ti-(Al_3Ti+Al) composites having thinner layers of intermetallic and additionally aluminium layers show the same dependence - and their flexural strength also increases with decreasing Ti thickness. Nevertheless, the flexural strength of the Ti-Al_3Ti composites is about 70% higher in the perpendicular and about 50% higher in the parallel direction than for the Ti-(Al_3Ti+Al) composites. The Charpy-impact tests confirmed the discussion regarding the bending test. The Charpy-tested samples showed the same fracture behaviour as the bend-tested samples, which indicated that the same mechanisms operated at both the high impact rate and low bending-test rate. The results of impact toughness measurements are listed in Table 2. The Ti-(Al_3Ti+Al) laminated composites loaded perpendicular to the layers, especially the Ti150/20 composite, showed a toughness, which was higher than for the pure titanium (41 J/cm^2) that was used as the base material. When the load direction was parallel to the laminates, both composites behaved similarly and the failure occurred by the cleavage mode. The impact toughness of the composites is strictly dependent on the remaining Ti metal thickness. It increases with an increasing Ti content in both types of composites. The presence of aluminium layers in the Ti-(Al_3Ti+Al) composites also increases their impact toughness because the layers can absorb a greater amount energy than the titanium and Al_3Ti phase. Therefore, the Ti-(Al_3Ti+Al) composites have much higher impact properties in the perpendicular as well as in parallel load direction than the Ti-Al_3Ti composites.

CONCLUSIONS

Laminated Ti-(Al_3Ti+Al) and Ti-Al_3Ti composites can be successfully produced by the interlayer reaction process using Ti and Al foils. The Al_3Ti phase is the only phase formed during the reaction between Ti and Al at 650^{o}C independent of the treating time. The microstructural characterisation indicates that after 20 minutes not all the aluminium is consumed and therefore, the formed composites consist of alternating layers of Ti, Al and Al_3Ti. After 60 minutes, the aluminium is completely consumed, resulting in microstructures with Ti residual layers alternating with the Al_3Ti layers. The Ti-Al_3Ti laminated composites have a higher yield strength, higher ultimate tensile strength and higher flexural strength than the Ti-(Al_3Ti+Al) composites produced with the same thickness of starting Ti foil. On the other hand, the strain at fracture and impact toughness of the composites behave conversely. It happens because the amount of residual aluminium at the intermetallic centrelines increases the ductility of the composites. The results also show that the mechanical properties of the composites depend strongly on the thickness of the individual Ti layers.

REFERENCES

[1] Vecchio K.S., Synthetic multifunctional metallic-intermetallic laminate composites, JOM 2005, 57, 25-31.

[2] Wadsworth J., Lesuer D.R., Ancient and modern laminated composites - from the Great Pyramid of Gizeh to Y2K, Mater. Charact. 2000, 45, 289-313.

[3] Misra A., Demkowicz M.J., Zhang X., Hoagland R.G., The radiation damage tolerance of ultra-high strength nanolayered composites, JOM 2007, 9, 62-65.

[4] Alman D.E., Hawk J.A., Petty A.V., Rawers J.C., Processing intermetallic composites by self-propagating high temperature synthesis, JOM 1994, 46, 31-35.

[5] Konieczny M., Mola R., Thomas P., Kopciał M., Processing, microstructure and properties of laminated Ni-intermetallic composites synthesised using Ni sheets and Al foils, Arch. Metall. Mater. 2011, 56, 693-702.

[6] Konieczny M., Microstructural characterisation and mechanical response of laminated Ni-intermetallic composites synthesised using Ni sheets and Al foils, Mater. Charact. 2012, 70, 117-124.

[7] Bloyer D.R., Venkateswara Rao K.T., Ritchie R.O., Laminated Nb/Nb_3Al composites: effect of layer thickness on fatigue and fracture behaviour, Mater. Sci. Eng. A 1997, 239-240, 393-398.

[8] Takuda H., Fujimoto H., Hatta N., Formabilities of steel/aluminium alloy laminated composite sheets, J. Mater. Sci. 1998, 33, 91-97.

[9] Dziadoń A., Mola R., Kompozyt warstwowy magnez-eutektyka odkształcany w próbie ściskania, Kompozyty 2008, 4, 364-368.

[10] Dziadoń A., Mola R., Błaż L., Formation of layered Mg/eutectic composite using diffusional process at the Al-Mg interface, Arch. Metall. Mater. 2011, 56, 677-684.

[11] Rudnitskii N.P., Studies on the strength and plasticity of Ti-Nb-based laminated composites in a temperature range of 290-1700 K, Strength Mater. 2002, 34, 612-616.

[12] Konieczny M., Zmiany struktury podczas formowania kompozytu warstwowego tytan - fazy międzymetaliczne, Kompozyty 2008, 2, 168-171.

[13] Ward-Close C.M., Minor R., Doorbar P.J., Intermetallic-matrix composites - a review, Intermetallics 1996, 4, 217--229.

[14] Ohnuma I., Fujita Y., Mitsui H., Ishikawa K., Kainuma R., Ishida K., Phase equilibria in the Ti-Al binary system, Acta Mater. 2000, 48, 3113-3123.

[15] Konieczny M., Dziadoń A., Strain behaviour of copper-intermetallic layered composite, Mater. Sci. Eng. A 2007, 460-461, 238-242.

[16] Li T., Jiang F.C., Olevsky E.A., Vecchio K.S., Meyers M.A., Damage evolution in Ti6Al4V-Al_3Ti metal-intermetallic laminate composites, Mater. Sci. Eng. A 2007, 443, 1-15.

