Abstract
50, 100 and 150 μm thick titanium and 50 μm thick aluminum foils have been used to fabricate Ti-Al₃Ti and Ti-(Al₃Ti+Al) composites. These laminated materials were synthesized in vacuum with controlled treating time and temperature. Since the examinations showed that Al₃Ti was the only intermetallic phase formed during the reaction between titanium and aluminum, the initial foil thicknesses affected only the volume fraction of the resultant Ti, Al and Al₃Ti layers. All composites were synthesized at 650 °C. Treating time was a main factor determining the composition and tensile behavior of the composites. After 20 minutes of the reaction not all aluminum was yet consumed and the composites consisted of alternating layers of Ti, Al and Al₃Ti. Aluminum layers reacted completely after 60 minutes resulting in microstructures with Ti residual layers alternating with the Al₃Ti layers. Tensile tests were performed on the materials with different microstructures to establish their properties and fracture behavior. After 60 minutes of treating all composites had higher yield strength and higher ultimate tensile strength than composites after 20 minutes of treating produced with the same thickness of starting Ti foil. On the other hand, strain at fracture behaved conversely. The results of investigations indicated that tensile behavior of the composites depended strongly on the thickness of individual Ti layers and the presence of residual Al layers at the intermetallic centerlines.

Keywords: titanium, aluminum, Al₃Ti, tensile behavior, fracture

1. INTRODUCTION
In late years, there has been significant interest in the design, fabrication and mechanical behavior of a variety of laminated composites, such as metal-metal [1], metal-ceramic [2] and metal-intermetallic [3-11] systems. Especially, there is a great concern with metal-intermetallic laminated (MIL) composites, that have the potential to perform various functions, such as ballistic protection, blast mitigation, thermal management, heat exchange and vibration damping [5]. MIL composites allow for the possibility of combining the good ductility and toughness of metals with the higher elastic modulus, higher strength, and lower density of intermetallics. Lamination can significantly improve many properties including fatigue behavior, fracture toughness, wear, corrosion and damping capacity, or provide enhanced formability or ductility for brittle intermetallics [1]. Production techniques of MIL composites may be divided into deposition or bonding. Sputter or vapor deposition techniques involve atomic scale transport of the 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 [12]. Unfortunately, deposition techniques require sophisticated manufacturing equipment and are too slow to be practical for making large-scale components. These factors increase the cost of component production and make the application of these composites economically unattractive. On the other hand, bonding techniques (diffusion bonding, transient liquid phase bonding and reaction bonding) between metal foils have some advantages, e.g., they involve relatively simple processing and the size and the number of layers that can be produced are not limited [3]. 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 [3, 10, 11, 13], Nb [4], Fe [13] or Mg [14] foils. Among laminate composites, the Ti-intermetallic laminates have a great technological advantage and attracts special attention for various applications [5]. Previous works reveal that titanium-intermetallic composites can be produced by reaction that occurs at the interface of Ti and Nb [15], Ti and Cu [7, 9] or Ti and Ni [13]. In particular, the Ti-Al system has a great practicable potentiality. The titanium aluminides Ti₃Al, TiAl and Al₃Ti offer potential for increased temperature range and enhanced high temperature strength, stiffness and oxidation resistance compared with conventional titanium alloys [16]. Specifically, the Ti-Al₃Ti laminated composites can be considered for aerospace, automotive and other structural applications because of their lower density than monolithic titanium or other Ti-based laminate systems. Furthermore, the Ti-Al₃Ti system is economically more attractive than monolithic titanium because aluminum is relatively inexpensive. This kind of laminated composites has been extensively investigated by Alman et al. [3, 13], Peng et al. [17] and Vecchio et al. [5, 18]. In the present study, the reaction synthesis was employed to fabricate laminated composites in vacuum using Ti foils with different original thicknesses and Al foils with one constant thickness. As a result, the laminated Ti-Al₃Ti and Ti-(Al₃Ti+Al) composites were produced. Tensile tests were performed on the materials with different microstructures to establish their properties and fracture behavior.

2. EXPERIMENTAL PROCEDURE

In the work, 50, 100 and 150 μm thick foils of titanium (99.14 at. % Ti, 0.35 at. % Fe, 0.24 at. % Al, 0.21 at. % O, 0.04 at. % N, 0.02 at. % C) and 50 μm thick foil of aluminum (99.53 at. % Al, 0.21 at. % Fe, 0.16 at. % Si, 0.05 at. % Zn, 0.03 at. % Cu, 0.02 at. % Ti) were used to produce laminated titanium-intermetallic composites with controlled treating time, temperature and pressure. Titanium and aluminum foils were cut into 50 mm x 10 mm rectangular pieces. No special surface preparation treatment was applied to the foils prior to processing. Any contamination on the surface of foils was removed with cotton swabs in a bath of 5 pct HF in water. After that 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, there were used 51, 34 or 26 pieces of Ti dependently 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 temperature was increased from 20 to 600 °C at a heating rate of 0.25 °C/s. The samples were heated in vacuum of 0.01 Pa at 600 °C for 2h 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 with the purpose of eliminating possible expulsion of liquid phases. The temperature was then decreased slowly (cooling rate of 0.16 °C/s) to 600 °C and the pressure of 5 MPa was applied again. The thermal aging 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 samples were polished using standard metallographic techniques. Microstructural observations were performed using a JEOL JMS 5400 scanning electron microscope and a Carl Zeiss NEOPHOT 2 optical microscope. The chemical composition of the phases was determined by an energy dispersive spectroscopy utilizing a ISIS 300 Oxford Instruments. An X-ray diffraction using a D/max RAPID2 diffractometer was employed to identify the intermetallic phases. Before the samples were examined with the optical microscope they had been etched using an aqueous HF solution (2%) to reveal any titanium grain boundaries and the structure of the intermetallic layers. Samples with dimensions of 50 mm x 8 mm x 4 mm, made from fabricated composites, were subjected to tension test on an INSTRON screw machine at a constant crosshead speed of 0.1 mm/min. Fracture surfaces of specimens were examined by macroscopic observations and scanning electron microscopy to evaluate deformation mechanisms.
3. RESULTS AND DISCUSSION

3.1 Microstructural characterization

After fabrication, the composition of the phases in the composites was determined. The examinations showed that only $\text{Al}_3\text{Ti}$ phase was formed in the samples irrespectively of treating time. The XRD results confirmed that the composites treated at 650 °C for 20 minutes consisted of alternating Ti, $\text{Al}_3\text{Ti}$ and Al layers. No unreacted Al was detected in any of the composites processed at 650 °C for 60 minutes, so the composites consisted of Ti and $\text{Al}_3\text{Ti}$ layers. The laminated materials were well-bonded and nearly fully dense. The thickness of the $\text{Al}_3\text{Ti}$ layers formed during processing was independent of the starting thickness ratio of the Ti and Al foils, what indicated that only certain quantity of titanium was consumed by the aluminum during the synthesis reaction. Microstructural investigations indicated also that the central zones of the formed $\text{Al}_3\text{Ti}$ layers contained many $\text{Al}_2\text{O}_3$ 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 $\text{Al}_3\text{Ti}$ layers. A migration of oxide films from the surfaces of the Al foils to the middle of the formed intermetallic layers was previously observed during formation of another aluminides belonging to the binary Ni-Al [10, 11] and Mg-Al [14] systems. Any oxides accumulated at the centerline of intermetallics can be a weak point in the microstructure because cracks are usually developed at the defect points.

3.2 Tensile testing

Fig. 1 shows the typical tensile stress-strain curves for the as-processed Ti100/20 and Ti100/60 laminated materials. Table 1 summarizes the tensile properties of all synthesized composites. With an increase of the treating time at 650 °C, the $\text{Al}_3\text{Ti}$ layers grow, leading to an increase in volume fraction of the intermetallics. As a result, the yield strength and the tensile strength of all investigated composites increased and the total strain at fracture decreased. The tensile behavior of the Ti-$\text{Al}_3\text{Ti}$ and Ti-$\text{Al}_3\text{Ti}$ composites was majorly dependent on the laminates microstructure, particularly, on the volume fraction of the layers. The failure mechanism in the course of straining of both types of the composites was carefully investigated. At the early stage of plastic straining the Ti and Al grains were deformed by slip. No traces of plastic deformation were noticed in the intermetallic layers. Unfortunately, during deformation the crystal structures mismatch between the base metals (HCP crystal structure for Ti and FCC for Al) and $\text{Al}_3\text{Ti}$ layers crystallizing in the tetragonal $\text{DO}_{22}$ unit cell [17] makes almost impossible slip-bands slide through a metal/intermetallic interfaces.

![Fig. 1 Tensile curves for the Ti-$\text{Al}_3\text{Ti}$ and Ti-$\text{Al}_3\text{Ti}$+Al composites](image-url)
Table 1 Summary of tensile properties of Ti-Al₃Ti and Ti-(Al₃Ti+Al) laminate composites

<table>
<thead>
<tr>
<th>Treating time at 650 °C (min)</th>
<th>Sample designation</th>
<th>Volume fraction (%)</th>
<th>Tensile properties</th>
<th>Strain at fracture (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Ti</td>
<td>Al</td>
<td>Al₃Ti</td>
</tr>
<tr>
<td>20</td>
<td>Ti50/20</td>
<td>44.4</td>
<td>28.3</td>
<td>27.3</td>
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<tr>
<td></td>
<td>Ti100/20</td>
<td>63.8</td>
<td>18.2</td>
<td>18.0</td>
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<td></td>
<td>Ti150/20</td>
<td>73.1</td>
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<td>13.1</td>
</tr>
<tr>
<td>60</td>
<td>Ti50/60</td>
<td>24.0</td>
<td>0</td>
<td>76.0</td>
</tr>
<tr>
<td></td>
<td>Ti100/60</td>
<td>54.5</td>
<td>0</td>
<td>45.5</td>
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<tr>
<td></td>
<td>Ti150/60</td>
<td>67.7</td>
<td>0</td>
<td>32.3</td>
</tr>
</tbody>
</table>

Therefore, formation of cracks in the Al₃Ti layers was the characteristic feature of the prolonged deformation. The observed serrations in the stress-strain curves in Fig. 1 correspond to the formation of multiple cracks in the intermetallic layers. The cracks developed at defect points within the brittle Al₃Ti layers and then propagated across them. The perpendicular cleavage cracks were blunted by the titanium and aluminum layers when the cracks reached the Ti/Al₃Ti or Al/Al₃Ti interfaces. The energy absorption capability of the metals layers allowed numerous cracks to develop within each intermetallic layer before failure (Fig. 2a). With permanent increase of the crack number in the Al₃Ti layers the titanium and aluminum layers gradually underwent the total external load. As a result the plastic flow that took place in the metals layers was restricted to the small regions between opposite cracks in the neighboring Al₃Ti layers. When the number and distribution of cracks in the intermetallic layers reached a critical limit, the final failure occurred by shearing fracture of the metals layers. It is obvious that strain hardening of the tensile specimens (Fig. 1) was produced due to plastic deformation of the titanium and aluminum layers. The only exception to the deformation model was the Ti50/60 composite having the thinnest titanium layers (only 17.6 µm). Since there was not sufficient material with the metal layers to absorb the cracks release energy, only one single crack propagated. As a result the Ti50/60 composite failed in a brittle manner (Fig. 2b). The obtained data are consistent with the results previously reported by Alman et al. [3, 13] and Vecchio et al. [5, 18]. The authors demonstrated that fracture mechanism and failure energy in metal-intermetallic laminated composites were controlled by varying the intermetallic-to-metal volume ratio. Their considerable body of work was also devoted to the mechanical behavior aspects and fracture behavior of the Ti-aluminide laminated composites, but their composites were fabricated using much thicker titanium foils than in the present investigations.
The fracture behavior of the Ti-(Al₃Ti+Al) and Ti-Al₃Ti laminated composites was typical of ductile-phase-toughened matrix composites. The failure characteristic described for the Ti-(Al₃Ti+Al) and Ti-Al₃Ti composites is strictly consistent with previous studies of cracking and damage mechanisms in Ti-Ti₂Ni [13], Ti-(TiCu+Ti₂Cu) [7], Nb-Nb₃Al [4], Ni-Ni₂Al₃ [3, 13], Fe-intermetallics [8] and Cu-intermetallics [6] laminated composites. The damage mechanisms of the unlike laminated composites during tensile testing are alike because different intermetallics formed from different constituent metals behave very similar. They are ordinarily brittle at room temperature due to the limited mobility of dislocations, have insufficient number of slip or twinning systems, and very low surface energy resulting in little or no plastic deformation at crack tips.

4. CONCLUSION

The laminated Ti-(Al₃Ti+Al) and Ti-Al₃Ti composites have been successfully produced by interlayer reaction process using Ti and Al foils. Microstructural examinations show that the Al₃Ti phase is the only phase formed during the reaction between Ti and Al at 650 °C independently on the treating time. The microstructural characterization indicates that after 20 minutes not all aluminum is consumed and therefore the formed composites consist of alternating layers of Ti, Al and Al₃Ti. After 60 minutes aluminum is completely consumed resulting in microstructures with Ti residual layers alternating with the Al₃Ti layers. The central areas of the intermetallic layers contain many Al₂O₃ inclusions which originate from surface oxide films on the Al foils. The mechanical response and fracture behavior of both types of composites were examined by tensile test. After 60 minutes of treating at 650 °C all investigated composites have higher yield strength, higher ultimate tensile strength than composites after 20 minutes of treating produced with the same thickness of starting Ti foil. On the other hand, strain at fracture of the composites behave conversely. It happens because an amount of residual aluminum at the intermetallic centerlines increases ductility of the laminated composites. The results also show that tensile properties of the composites depend strongly on the thickness of individual Ti layers.
LITERATURE


