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Du, Y.; Fan, G.H.; Yu, Tianbo; Hansen, Niels; Geng, L.; Huang, Xiaoxu

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Effects of interface roughness on the annealing behaviour of laminated Ti-Al composite deformed by hot rolling

Y Du^{1,2}, G H Fan¹, T Yu², N Hansen², L Geng¹ and X Huang²

¹School of Materials Science and Engineering, Harbin Institute of Technology, Harbin, 150001 Harbin, China

²Danish-Chinese Center for Nanometals, Section for Materials Science and Advanced Characterization, Department of Wind Energy, Risø Campus, Technical University of Denmark, DK-4000 Roskilde, Denmark

E-mail: yandua@dtu.dk

Abstract. A laminated Ti-Al composite has been fabricated by hot compaction and hot rolling of alternate layers of commercial purity Ti and Al sheets with a thickness of 200 μm . The hot compaction temperature was 500°C and in a following step the composite has been reduced 50% in thickness by hot rolling. The fully consolidated composite has been annealed at 300°C and 500°C for different length of time. As a result of the differences in crystal structure and mechanical properties between Ti and Al protrusions and retrusions formed at the interface. A heterogeneous interface has thereby been created. The heterogeneity affected the recovery kinetics of the aluminium phase which at 300°C was faster near the interface than in the middle of the Al layer. This effect of a heterogeneous interface is of relevance when optimizing the thermomechanical processing of the composite to obtain high strength and formability for application.

1. Introduction

Efforts have been and would continue to be made in order to develop strong and light metals in response to the energy shortage. Both lightweight aluminium and titanium alloys have been developed and applied in the automobile industry. Significant progress has also been made in the development of laminated metal composites (LMCs), which have excellent mechanical properties including strength, toughness, high impact and fracture resistance [1-2]. The interface plays an important role in LMCs and has been studied in recent years for example in roll bonded composites. In such studies experimental observations have been analyzed also including numerical simulation of evolving interfaces [3-5]. It has also been reported that recrystallization and abnormal grain growth can occur in an Al layer after hot deformation and during subsequent annealing [6]. It has also been observed that roughness can form during deformation of polycrystalline metallic materials with FCC structure or HCP structure due to heterogeneity of plastic deformation [7-9]. This phenomenon has also been observed at the interfaces of LMCs produced by rolling [4] and this kind of heterogeneity can cause structural differences during annealing of the LMC. Thus, an investigation of interface roughness effects on microstructure of LMCs during deformation and annealing is important, especially for



controlling the microstructure by interface design and optimizing deformation and annealing conditions.

In this study of a LMC, commercial purity Al (1060) sheets and commercial purity Ti (TA1) sheets are selected as materials to produce laminated Ti-Al composites by hot rolling. The interface roughness generated during hot rolling is analyzed and its effect on microstructural evolution of Al in the Ti-Al laminate both after hot rolling and during subsequent annealing is examined.

2. Experimental methods

2.1. Materials

Commercial purity titanium TA1 sheets with a purity of 99.4 wt% (0.25 wt% Fe, 0.2 wt% O, 0.1 wt% C) purchased from Tianrui Non-ferrous Metal Co., Ltd. (Baoji, China) and commercial purity aluminium 1060 O sheets with a purity of 99.2 wt% (0.35 wt% Fe, 0.25 wt% Si) purchased from Northeast Light Alloy Co., Ltd. (Harbin, China) were selected as starting materials. The as-received Ti sheets had a recrystallized structure (preferred orientation $\langle 0001 \rangle$ parallel to the ND) with an average grain size of 50 μm while the as-received Al sheets had a slightly deformed structure (random orientation) with an average grain size of 100 μm . Both the TA1 and 1060 O Al sheets had a thickness of 200 μm . Small pieces with a size of 100 mm \times 100 mm \times 200 μm were cut from the as-received Ti and Al sheets to fabricate laminated Ti-Al composites.

2.2. Processing and annealing treatment

The laminated Ti-Al composites were fabricated by three steps as shown in figure 1(a). i) Sheet preparation: the Ti and Al sheets were firstly treated by 10 vol.% HF liquor and 10 vol.% NaOH liquor, respectively, to remove contaminants and the oxide layer from the surface. Then, the sheets were roughened with a steel brush, cleaned in an ultrasonic bath of acetone for 5 min, dried and stored in air for later use. ii) Diffusion bonding: the sheets were stacked alternately like a sandwich with Ti sheets at both surface layers. The sandwich was put into a graphite die for confinement and pressed under a pressure of 40 MPa at 500°C for 1 h in a vacuum furnace to obtain LMCs. iii) Hot rolling: the compressed LMCs were held at 500°C for 10 minutes before each rolling pass and rolled through 5 passes with a total thickness reduction of 50% from 6 mm to 3 mm. The rolling geometry is shown in figure 1(b). Thickness reduction of each pass were controlled to ensure a macro homogeneous rolling condition, i.e. the ratio of contact length between the roll and the sheet over the average thickness L/h was between 0.5 and 5. This ratio can be expressed by the relationship:

$$\frac{L}{h} = \frac{\sqrt{R(h_0 - h_1)}}{(h_0 + h_1)/2} \quad (1)$$

where h_0 is the thickness of sheet before each rolling pass, and h_1 is the thickness of sheet after each rolling pass. The average thickness of sheet h is the average of h_0 and h_1 and R is the radius of the roll.

The hot rolled LMCs were annealed at 300 °C for 10 min, 30 min and 1 hour, and at 500°C for 1 hour to examine the thermal behaviour of the LMCs.

2.3. Characterization

The microstructure and the texture of hot-rolled Ti-Al LMCs were characterized in a scanning electron microscope (SEM, Zeiss Supra 35) equipped with an automated HKL electron backscatter diffraction (EBSD) facility. The samples were prepared by electropolishing and ion etching using a cross section polisher (JEOL IB-09020CP) to reveal various aspects of microstructures of the LMCs. Two step sizes of 2 μm and 0.1 μm were used for coarse and fine EBSD scans, respectively. Hardness of the Al layers in different conditions were measured to analyze the recovery and recrystallization behaviour of the aluminium phase in the LMCs. All tests were done in the ND-TD section where ND and TD stand for normal and transverse directions, respectively. The hardness measurement points were centered to the

region 20 μm near the interface in the Al and in the middle of the Al layers. For each test, 5 selected points were measured.

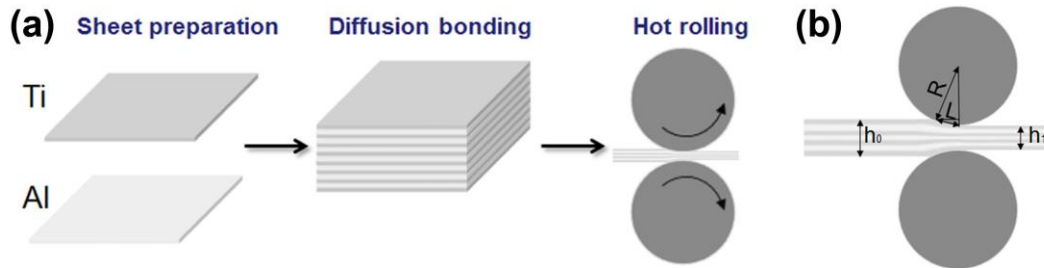


Figure 1. (a) Schematic illustration of three step processing of laminated Ti-Al composite; (b) rolling geometry.

3. Results and Discussion

3.1. Microstructure of deformed laminated Ti-Al composite

The microstructure of the LMCs before and after hot rolling were examined by SEM as shown in figure 2(a) and figure 2(b). It is clear that the interface of the LMC is flat before hot rolling and it turns rough after hot rolling. The microstructure of hot-rolled laminated Ti-Al was also examined by EBSD as shown in figure 3(a). It shows interface roughening and the formation of protrusions and retrusions at interfaces. The Al layers showed a hot deformed structure, which was confirmed by TEM observations. The Ti layer contains deformed grains with an average diameter of about 10 μm . Distributions of grain/subgrain sizes in Al and grain sizes in Ti layers are presented in figure 3(b) and 3(c), respectively. Since laminated Ti-Al composites were held at 500 $^{\circ}\text{C}$ for 10 min before each pass of rolling, the microstructure in the Al layers before rolling may be recrystallized. After the final pass, the hot-rolled Ti-Al was still kept at high temperatures, which provided energy for static recrystallization in Al. Several recrystallized Al grains with an average diameter of 20 μm were found near the interfaces as marked by arrows in figure 3(a). Most of these recrystallized grains are near the interface characterized by large protrusions. Similar microstructures were observed by EBSD in other Al layers in the ND-TD section of the hot-rolled laminated Ti-Al composite. For the Ti layers, the temperature of 500 $^{\circ}\text{C}$ did not reach the recrystallization temperature. Thus, as shown in figure 3(c), the Ti layers contain deformed grains with a diameter ranging from 5 μm to 22 μm .

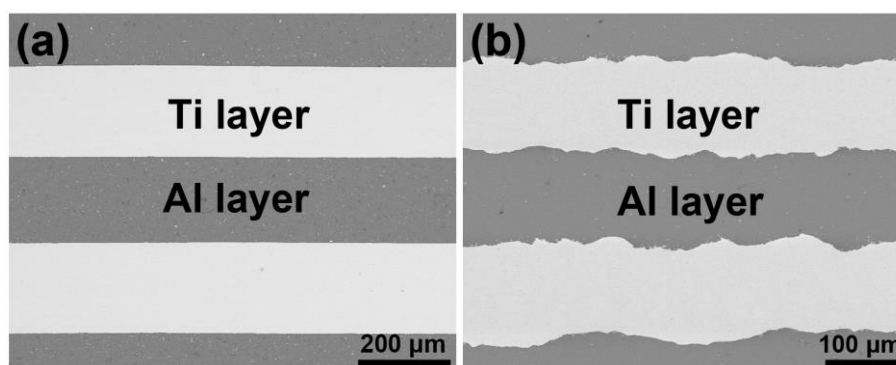


Figure 2. Microstructure in the ND-TD section of a LMC sample before and after hot rolling. (a) flat interface before hot rolling; (b) rough interface after hot rolling.

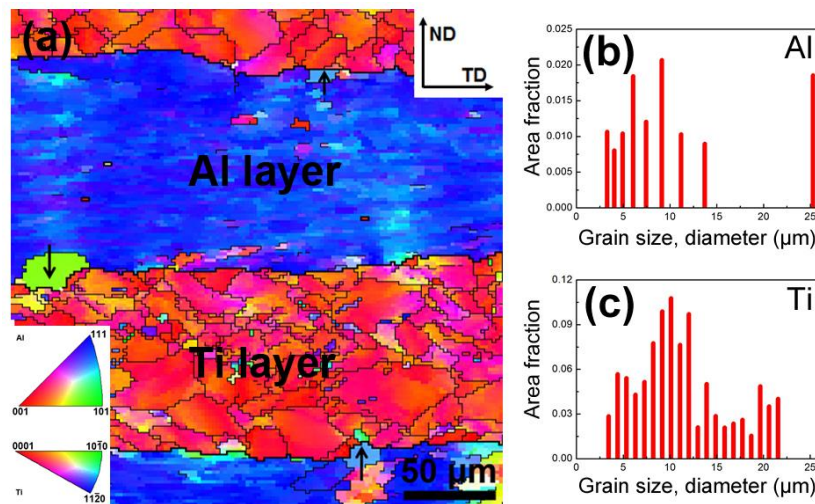


Figure 3. Microstructure in the ND-TD section of hot-rolled laminated Ti-Al. (a) EBSD map (inverse pole figure of the ND) of the LMC, showing recrystallized Al grains marked by arrows formed at the interface; (b) and (c) grain size distribution of Al and Ti, respectively.

During hot rolling both Al and Ti deformed by dislocation glide but their plastic deformation differ. Al with an FCC crystal structure can deform on many slip systems and thereby accommodate the rolling strain homogeneously. In contrast, Ti with an HCP crystal structure has fewer available slip systems and the plastic deformation may be fairly heterogeneous. As a result protrusions and retrusions can form at the Ti-Al interfaces. The protrusions/retrusions create variations in plastic strain and stored energy along the interface and thereby in the potential for formation of recrystallization nuclei where a few are marked by arrows in figure 3(a).

3.2. Microstructure of annealed laminated Ti-Al composite

The microstructure of as-annealed laminated Ti-Al composites in the ND-TD section was examined by EBSD as shown in figure 4, where the colours are based on the crystallographic orientation of the ND as indicated in the inset. After annealing at 500 °C for 60 min as shown in figure 4(a), Ti still shows very strong preferred orientations parallel to the ND which is similar to the hot-rolled structure. The roughness of the interface between Ti and Al layers still exists. The Al layers contain big recrystallized grains with an average diameter of about 100 μm. Distribution of recrystallized Al grains compared to the hot-rolled state is shown in table 1. Both grain size and area fraction of recrystallized Al grains show very large increase after annealing at 500 °C for 60 min. The Ti layers still contain recovered grains with an average diameter of about 20 μm.

Table 1. Distribution of recrystallized grains in Al layers.

	Position	Area fraction (%)	Average grain size (μm)
Hot rolled	interface	0.74	28.5
500 °C, 60 min	whole layer	100	109

Figures 4(b)-(d) show examples of EBSD maps viewed from the ND-TD section of Al layers in LMCs annealed at 300 °C for 10 min, 30min and 60 min, respectively. As shown in figure 4(b)-(d), protrusions and retrusions are seen very clearly at the interfaces and more subgrains can be found at the protrusions and retrusions than at the flat interface. Also, there are more subgrains forming near the interface than in the middle of the Al layers. Similar deformed structures as in the hot-rolled state can still be found in the middle of the Al layers after annealing at 300 °C for 10 min and 30min as

shown in figures 4(b) and 4(c), respectively. Subgrain coarsening is also very clear when comparing the microstructure between samples annealed at 300 °C for different times.

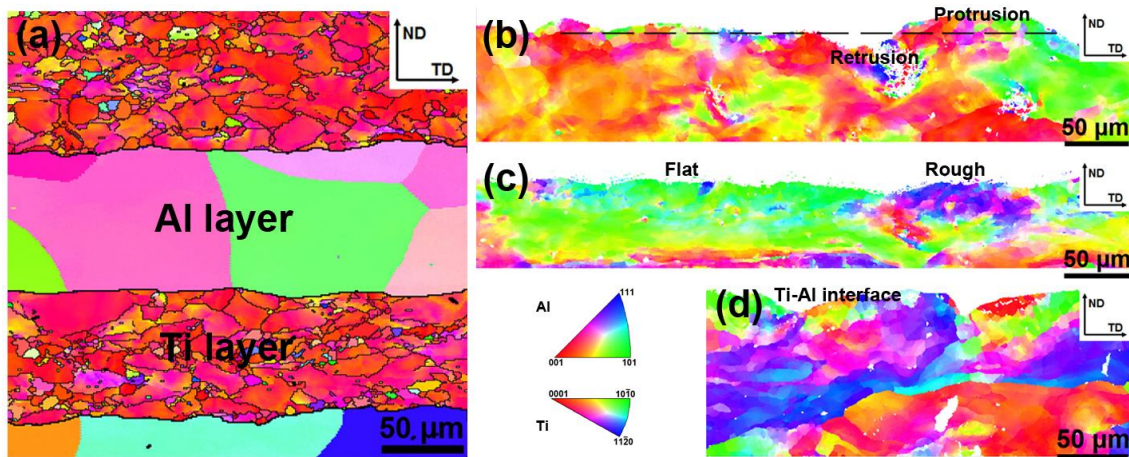


Figure 4. Microstructure in the ND-TD section of annealed laminated Ti-Al by EBSD (inverse pole figure of the ND). (a) EBSD map of the LMC annealed at 500 °C for 60 min, showing large fully recrystallized Al grains; (b), (c) and (d) are EBSD maps of Al layers in LMCs annealed at 300 °C for 10 min, 30 min and 60 min, respectively, showing that more subgrains had formed at the protrusions and retrusions than in the middle of the Al layer.

Statistical results of Al subgrains in the Al layer of laminated Ti-Al annealed at 300 °C for 10 min, 30 min and 60 min are shown in figure 5. For each annealing treatment state, 4-5 EBSD maps including 8-10 Al layers were measured. Both the average grain size and number of equiaxed Al subgrains forming in every 100 μm length during annealing corresponding to the distance between interfaces in laminated Ti-Al were analyzed. Since there are protrusions and retrusions at the interfaces, the average interface line is defined as the average level of highest protrusion and lowest retrusion in each map scanned by EBSD. In general, the number of equiaxed Al subgrains in every 100 μm length of the interface increases with increasing annealing time. It is also observed that many subgrains expands from the interfaces to the middle of the Al layer with increasing time and for each annealing condition the density of subgrains decreases as the distance from the interface increases. For the 10 min and 30 min conditions, the average size of subgrains at the interfaces is bigger than in the middle of the Al layers. Nevertheless, the average size of subgrains from the interface to the middle of the Al layer is nearly the same after annealing at 300 °C for 60 min.

Isothermal annealing treatments at 300 °C lead to microstructural coarsening and softening. The softening is by change in hardness as illustrated in figure 6 and it also shows the hardness of Al annealed at 500 °C for 60 min and in the deformed state (annealing time of 0 min) for comparison. Randomly selected points were measured at each state and both areas near the interface and in the middle of Al layers were tested. Hardness of both hot deformed Al and Al annealed at 500 °C for 60 min does not change much from the interface to the middle of the Al layers due to uniform hot deformation and fully recrystallized microstructure as shown in figure 3(a) and figure 4(a), respectively. Hardness of deformed Al shows highest value of 29 HV, and it decrease to the lowest value of 22 after annealing at 500 °C for 60 min. For annealing treatments at 300 °C, hardness of Al near the interface is lower than that in the middle of Al layers generally. Hardness difference between interface and middle areas of Al annealed at 300 °C first increases and then decreases with increasing annealing time. This is because Al starts recovery from the interface in hot deformed laminated Ti-Al structure and the effect of recovery homogenizes gradually the structure with increasing annealing time.

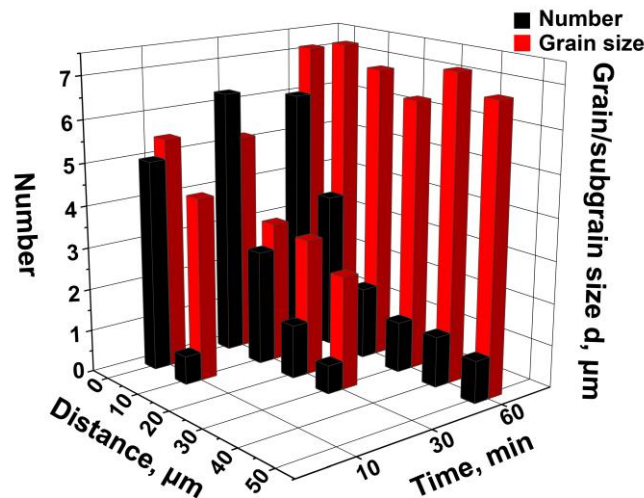


Figure 5. Statistical results of average grain/subgrain size and number in every 100 μm length of Al subgrains as a function of the distance from the interface in the LMC annealed at 10 min, 30min and 60 min at 300 °C according to the EBSD maps and measured in the ND-TD section of the samples.

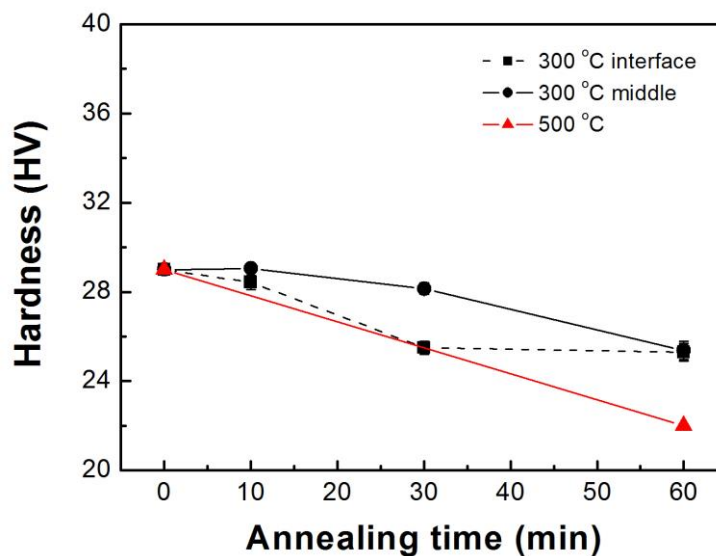


Figure 6. Change in hardness of Al with annealing condition in laminated Ti-Al composites (hardness of hot deformed Al is presented as annealing time of 0 min), showing that hardness in Al near the interface (black solid square) is lower than in the middle of the Al layer (black solid circle).

In an analysis of the thermal and mechanical behaviour of the laminated Ti-Al composite it must be taken into consideration the differences between Al and Ti when it comes to crystal structure and physical/mechanical/chemical properties. Some of these differences are illustrated in table 2. The difference in crystal structure between Al and Ti may give rise to the formation of protrusion/retrusion which is discussed in section 3.1. The difference in thermal expansion between Ti and Al may give rise to a thermal stress at the interface when the composite is cooled down from the hot compaction

temperature. These may cause some elastic/plastic deformation near the interface. This effect may not be large due to the relatively low flow stress of aluminium. The difference in Young modulus between Al and Ti leads to differences in the elastic/plastic behaviour of the two phases and a formation of compressive stresses in Al. Also this effect is expected to be small. Finally there may be a chemical reaction between Ti and Al during processing and post process annealing as has been observed. The interaction between the different reactions at or near the interface is part of ongoing research.

Table 2. Materials parameters for the Ti and Al layers.

Layer	Elastic Modulus (GPa)	Shear Modulus (GPa)	Thickness (μm)	CTE ($10^{-6}/^{\circ}\text{C}$)
Al	70	25	100	23
Ti	102	39	100	10.8

4. Conclusions

At present we conclude that the difference in plastic behaviour between Al and Ti due to their different crystal structures is the main cause of the observed heterogeneous deformation in Al near the interface. This difference results in a faster recovery in Al in the region near the interface than further away from the interface.

Our observations also show that the chosen Ti-Al system offers excellent possibilities for new research of scientific relevance. Such research will also guide a continued optimization of LMCs for future application for example in the car and aerospace industry.

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