Fabrication and Characterization of High-Bonding-Strength Al/Ti/Al-Laminated Composites via Cryorolling

Juan Liu^{1,2,3} · Yuze Wu^{1,2,3} · Lin Wang^{1,2,3} · Hui Wang⁴ · Charlie Kong⁵ · Alexander Pesin⁶ · Alexander P. Zhilyaev⁶ · Hailiang Yu^{1,2,3}

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Abstract

Sandwich-like Al/Ti/Al-laminated composites have many advantages such as low density and high specific strength with value in mechanical manufacturing and aerospace engineering. Here, Al/Ti/Al-laminated composites were fabricated by hot roll bonding and subsequent processes: cryorolling (− 190 °C and − 100 °C), cold rolling (25 °C), and hot rolling (300 °C). Their bonding strength and mechanical properties were then studied by an Autograph AGS-X universal electronic testing machine. The results show that cryorolling can improve the interface bonding strength and tensile strength of Al/Ti/Al-laminated composites. For the Al/Ti/Al-laminated composites subjected to cryorolling at − 100 °C, they have the highest strength near 260 MPa—this is 48 MPa and 41 MPa higher than the laminated composites subjected to cold and hot rolling, respectively. These results also show the strongest peeling strength. Finally, the mechanisms of the enhancement of bonding strength and mechanical properties of Al/Ti/Al-laminated composites subjected to cryorolling were mainly discussed.

Keywords Laminated composites · Cryorolling · Bonding strength · Peeling strength · Mechanical property · Bonding mechanism

1 Introduction

Modern technology requires materials with high strength and good ductility. However, strengthening can often decrease ductility. Fortunately, layered and shell–core structures can improve the strength without the ductility drop [1–3]. Laminated composites (LCs) fabricated by two or more materials may have superior properties of each component [4], and such composites are widely used in petroleum, chemical, automobile, and aerospace engineering. Theories such as recrystallization theory, metallic bond theory, and diffusion theory have promoted the manufacturing technologies for development of LC plates, including explosive cladding [5, 6], hot pressing [7, 8], ultrasonic consolidation [9, 10], and roll bonding [11–15]. For LC production, people focus on the microstructure, mechanical properties, fracture mechanism, and interface properties [16–24]. In addition, surface treatment methods [21], microelement distribution [22, 24], and thickness ratio between soft and hard layers [25, 26] may also affect the mechanical properties of LCs.

As an ultra-low-temperature deformation technique, cryorolling inhibits the dynamic recovery process and produces nanocrystalline materials with a smaller deformation

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✉ Hailiang Yu
yuhailiang1980@tom.com; yuhailiang@csu.edu.cn

- ¹ State Key Laboratory of High Performance Complex Manufacturing, Central South University, Changsha 410083, China
- ² College of Mechanical and Electrical Engineering, Central South University, Changsha 410083, China
- ³ Light Alloys Research Institute, Central South University, Changsha 410083, China
- ⁴ Research Center for Advanced Science and Technology, The University of Tokyo, Meguro, Tokyo 1530041, Japan
- ⁵ Mark Wainwright Analytical Centre, University of New South Wales, Sydney, NSW 2052, Australia
- ⁶ Laboratory of Mechanics of Gradient Nanomaterials, Nosov Magnitogorsk State Technical University, Magnitogorsk, Russia 455000

versus high-temperature deformation techniques [27–31]. Wang et al. [27] obtained AA1060 sheets with a strength up to 180 MPa by cryorolling, which is about 2.5 times higher than that before rolling. Yu et al. [28] reported that Cu sheets fabricated by asymmetrical cryorolling had finer grains, higher strength, and better thermal stability. Bhat-tacharjee et al. [31] reported that cryorolling resulted in better mechanical properties of an AlCoCrFeNi_{2.1} alloy versus a control sample subjected to cold rolling.

Recently, cryorolling has also been used to develop LCs with good mechanical properties. Takagawa et al. [32] adopted cryogenic accumulative roll bonding to obtain nanostructured Cu–2.0Ni–0.5Si–0.1Zr alloy with improved tensile properties without reduced electrical conductivity. Yu et al. [33] reported that cryorolling can further improve the ultrafine-grained AA1060 sheets subjected to accumulative roll bonding. It was worth noting that the cryogenic temperature was –196 °C in these studies [27–33], and there are no reports on the effects of intermediate temperatures between –196 °C and room temperature on the bonding strength and mechanical properties of LCs during cryorolling.

Al/Ti/Al LCs combine the advantages of Al and Ti such as low density, high specific strength, and good corrosion resistance. They have great application prospects in mechanical manufacturing and aerospace engineering [33–36]. There are many studies on the fabrication of Al/Ti/Al LCs by hot roll bonding [37–43]. During hot roll bonding, Ti and Al atoms form a multilayer Ti/Al diffusion system [37], and TiAl₃ forms in the interface between Ti and Al layers [40]. The grain size of the TiAl₃ in the Ti/TiAl₃ interface was much finer than that in the Al/TiAl₃ interface [38], and the diffusion rate of Al in TiAl₃ was faster than that of Ti [39]. The peeling strength and mechanical properties of the Ti/Al LCs are significantly affected by the width of the inter-metallic layer. Recently, Karfoul and Tatlock [44] found the possibility of seamless bonding of Ti/Al composite plates under non-vacuum conditions. To date, there have been few reports on the bonding strength and mechanical properties of Al/Ti/Al LCs subjected to cryorolling.

In this study, Al/Ti/Al LCs were fabricated by hot roll bonding combined with cryorolling (–190 °C and –100 °C, respectively), cold rolling, or hot rolling. Their mechanical properties and bonding strength were studied and discussed.

The results show that the cryorolling at –100 °C results in excellent bonding strength and mechanical properties of Al/Ti/Al LCs.

2 Materials and Methods

The sheets adopted here were commercially pure Ti (TA2) and commercially pure Al (AA1100) sheets. Their chemical compositions are listed in Tables 1 and 2, respectively. The thicknesses of the Ti and Al sheets were 0.2 mm and 0.4 mm, respectively. The Ti sheets were vacuum-annealed at 550 °C for 30 min, and the Al sheets were vacuum-annealed at 400 °C for 1 h before rolling. Annealing was performed in a SA2-6-12TP resistance heating furnace. After annealing, both Ti and Al sheets were surface-treated to remove the oxide film. The Ti and Al sheets were then stacked with a sandwich-like structure as Al/Ti/Al LCs. The sandwich-like Al/Ti/Al LCs were heat-treated at 300 °C for 10 min to improve the bonding quality between Al/Ti interface and then rolled to a total thickness of 0.5 mm (rolling reduction ratio 50%) after two rolling passes. The hot roll-bonded Al/Ti/Al LCs were then further processed to 0.42 mm (rolling reduction ratio 16%), by cryorolling (–190 °C and –100 °C), cold rolling (25 °C), or hot rolling (300 °C). The rolling speed was 2 m min^{–1} in a multifunction rolling mill with 80-mm-diameter work rolls.

After rolling, peeling tests and tensile tests were carried out by an Autograph AGS-X universal electronic testing machine to study the bonding strength and mechanical properties of the Al/Ti/Al LCs on an electronic testing machine. The specimens used for the peeling test were 6 mm in width and 120 mm in length. During the peeling tests, the cross-head speed was 50 mm min^{–1}. The peeling strength was the average peel force divided by the specimen width (Eq. (1)):

$$\sigma_p = F/W. \quad (1)$$

Here, σ_p (N mm^{–1}) is the peeling strength, F (N) is the average peeling force, and W (mm) is the width of the peeling specimen. The specimens for tensile testing were 36 mm long and 5 mm wide. The peeling tests and tensile tests were repeated three times. During tensile testing, the strain rate

Table 1 Chemical composition (wt%) of AA1100 sheets

Al	Fe	Si	Cu	Ni	Zn	Mg	Mn	Ti
Bal	0.46	0.18	0.11	0.01	0.01	<0.01	<0.01	0.005

Table 2 Chemical composition (wt%) of TA2 sheets

Ti	Fe	Si	O	C	N	H
Bal	0.30	0.15	0.15	0.10	0.05	0.015

was $1.0 \times 10^{-3} \text{ s}^{-1}$. In addition, a triboindenter was used to test the nanohardness of Al and Ti layers with 30 mN testing load.

The fracture surface of the specimens after tensile testing and peeling testing was observed by TESCAN MIRA3 LMU scanning electron microscopy (SEM). The interface morphology of Ti/Al was examined via an optical microscope (OM). The elemental distribution near Ti/Al interface was analyzed by energy-dispersive spectrometry (EDS).

3 Results

Figure 1 shows the morphology of the Al/Ti/Al LCs interface after rolling. In this figure, the Ti/Al interface is serrated due to Al squeezing into the Ti layer. Figure 1a, b shows the interface morphology of the Al/Ti/Al LCs by cryorolling. The interface is rough and jagged, and the interface is wide and deep in the Ti layer. The interface becomes smooth and straight as the rolling temperature rises (Fig. 1c). However, the interface becomes wider with the further temperature increases, and the flatness of the interface decreases (Fig. 1d). Ma et al. [45] reported that the interface became

rougher when the rolling temperature ranged from 300 to 550 °C.

Figure 2a illustrates the EDS line scan across the Ti/Al interface. Figure 2b–e shows the element distribution of Al and Ti atoms near the Ti/Al interface for different rolling processes. During rolling, the elemental interdiffusion occurs near the Ti/Al interface forming an intermetallic diffusion layer. The width of the intermetallic diffusion layer increases with rolling temperature. For the Al/Ti/Al LCs subjected to cryorolling, the width of the intermetallic diffusion layer ranges from 2.2 to 2.4 μm , but it increases to 2.7 μm for the Al/Ti/Al LCs subjected to hot rolling.

Figure 3a shows the peeling strength curves between the Ti layer and Al layer. The interface bonding strength improves with higher reduction regardless of the rolling temperature. The peeling strength curves gave the average peeling strength of the Al/Ti/Al LCs with different processing (Table 3). The variation in the average peeling strength with rolling temperature is shown in Fig. 3b. The bonding strength of the Al/Ti/Al LC subjected to cold rolling has the lowest value of 6.4 N mm^{-1} . This is slightly lower than the sample subjected to hot rolling. The bonding strength of the Al/Ti/Al LCs sample prepared by cryorolling increases

Fig. 1 Morphologies of interface of an Al/Ti/Al-laminated composite subjected to **a** cryorolling at -190°C , **b** cryorolling at -100°C , **c** cold rolling, **d** hot rolling

Fig. 2 **a** Illustration of the EDS analysis of the Al/Ti/Al LCs. Element distribution near the interface in the rolled specimens: **b** cryorolling at -190°C , **c** cryorolling at -100°C , **d** cold rolling, **e** hot rolling

Fig. 3 **a** Peeling strength–distance curves of Al/Ti/Al LCs, **b** peeling strength versus the rolling temperature of the Al/Ti/Al LCs with the thickness of 0.42 mm. The illustration in **b** shows the schematic diagram of the peeling test

Table 3 Peeling strength and mechanical tensile properties of Al/Ti/Al LCs with the thickness of 0.42 mm

Rolling temperature ($^{\circ}\text{C}$)	σ_p (N mm^{-1})	σ_b (MPa)	δ_s (%)	Nanohardness (GPa)	
				Ti layer	Al layer
-190	6.7 ± 0.2	234.8 ± 8.5	10.4 ± 0.1	2.8 ± 0.1	0.50 ± 0.04
-100	7.2 ± 0.7	261.4 ± 1.6	12.4 ± 0.4	2.9 ± 0.1	0.55 ± 0.01
25	6.4 ± 0.3	215.2 ± 2.9	9.4 ± 1.1	$2.7 \pm .02$	0.48 ± 0.02
300	6.6 ± 0.1	219.4 ± 0.3	13.2 ± 0.2	2.6 ± 0.1	0.43 ± 0.03

versus the samples subjected to cold rolling. The bonding strength is highest in the $-100\text{ }^{\circ}\text{C}$ samples—this strength is 7.2 N mm^{-1} and is 12.5% higher than the sample subjected to cold rolling.

Figure 4 shows the SEM morphology of the Ti and Al surface after peeling tests for the specimens prepared after hot roll bonding. There are many cracks on the surface of the Ti side perpendicular to the rolling direction. In addition, there are few residual Al chips on the Ti surface: These are characterized by the bonding mode studied via mechanical locking, and the results are similar to Hwang et al. [46].

The morphologies of the Ti surfaces and Al surfaces after further rolling processing are shown in Fig. 5. The cracks on the Ti surface are wider than those on hot roll bonded. The residual Al chips increase like a “ridge”—the ridge width is much wider for the specimen subjected to cryorolling. The formation of this “Al ridge” is because the bonding strength between the Ti and Al layers is higher than the tensile strength of the Al layer. The Al is necked and fractured, and the “Al ridge” forms during peeling [47]. The width of the cracks on the Ti surface first decreases and reaches the minimum value at $25\text{ }^{\circ}\text{C}$ as rolling temperature increases (Fig. 5a, c, e, g). It then widens with increasing rolling temperature. The area fraction of the bond-well area between Ti and Al after peeling tests is shown in Fig. 6.

The tensile curves and mechanical properties of the Al/Ti/Al LCs are shown in Fig. 7 and Table 3, respectively. The results indicate that both cryorolling and hot rolling can improve the tensile strength and ductility of the Al/Ti/Al LCs versus samples subjected to cold rolling. In addition, the Al/Ti/Al LCs subjected to cryorolling at $-100\text{ }^{\circ}\text{C}$ have the best properties with a tensile strength of 260 MPa, and the failure strain is 12.4%.

Figure 8 shows the SEM morphology of the tensile fracture surface. The interface delamination of the Al/Ti/Al LCs can be seen in the tensile fracture. The Al/Ti/Al LCs subjected to cryorolling at $-100\text{ }^{\circ}\text{C}$ have obvious dimples in both Al and Ti layers, and the dimples in the Ti layer are nearly the same size. The hot-rolled Al/Ti/Al LCs have

dimples that appear in the Ti layer and the Al layer, and the Al has a ridge-like fracture due to the strong necking. Both the Al/Ti/Al LCs subjected to cryorolling at $-100\text{ }^{\circ}\text{C}$ and hot rolling show a plastic fracture with good ductility. However, the Al/Ti/Al LCs subjected to cryorolling at $-190\text{ }^{\circ}\text{C}$ or cold rolling has very few dimples on the Al layers and only a few shallow dimples on the Ti layer. There are also a few flat areas on the fracture surface, and thus, the ductility of the Al/Ti/Al LCs decreases.

The nanohardness values of the Ti layer and Al layer of Al/Ti/Al LCs are listed in Table 3. The change in the nanohardness of the Al layer and the Ti layer with temperature is shown in Fig. 9. The nanohardness value of the Ti layer by cryorolling is lower than 3.25 GPa reported by Fomenko—this is likely because their test load was much smaller than 30 N [48]. In Fig. 9, the Al layer and Ti layer have the maximum nanohardness values for the Al/Ti/Al LCs subjected to cryorolling at $-100\text{ }^{\circ}\text{C}$, and the nanohardness decreases with increasing rolling temperature.

4 Discussion

4.1 Bonding Mechanism and Bonding Strength

Severe shear deformation occurs on the surface of Ti and Al layers during Al/Ti/Al LCs rolling due to the different deformation capacity of Ti and Al layers and the difference in friction between the contact surfaces. The cracks first appear on the surface of Ti layer due to its poor deformation ability (Fig. 4). The Al will be squeezed into those cracks under the action of the rolling force, which leads to mechanical locking [49]. Generally, the LC bonding mode in the early rolling stage is mechanical locking [46]. Here, the deformation capacity of the Ti layer might decrease with decreasing rolling temperature, the number of cracks increases, and the crack size becomes wider (Fig. 5a, c, e, g). More Al squeeze into the cracks, which leads to wider cracks, and the mechanical bonding strength is then improved with

Fig. 4 SEM images of surfaces after peeling test for hot roll-bonded specimen: **a** Ti layer; **b** Al layer

Fig. 5 SEM images of Ti surface and Al surface after peeling tests for the specimens subjected to cryorolling at **a, b** – 190 °C and **c, d** – 100 °C, **e, f** cold rolling, **g, h** hot rolling

increasing contact area between the two metals [50]. In addition, cryorolling results in grain refinement [51], which can improve the strength of the materials (Fig. 6). The peeling strength is dependent on the strength of Al layer, when the bonding strength is greater than the tensile strength of the Al layer. Thus, the higher tensile strength of the Al layer can

increase the bearing capacity of the transverse load at the interface and improves the peeling strength due to improved mechanical locking strength.

In addition, with the formation of the Ti/Al interface, the Ti and Al layers form a solid diffusion system during rolling. Under the action of different atomic concentration, the

Fig. 6 Area fraction of the combined area between Ti and Al after peeling tests

Fig. 7 Engineering stress–strain curves of Al/Ti/Al LCs subjected to different rolling processes

active Ti (Al) atoms in Ti(Al) layer diffuse into the Al (Ti) layer leading to an intermetallic diffusion layer. The rearrangement of the atoms near the interface results in solution strengthening, and the interface bonding mode changes to metallurgical bonding. Over a certain range, the interfacial bonding strength increases as the intermetallic diffusion layer widens. The width of the interfacial diffusion layer is related to the atomic diffusion rate during rolling. The diffusion modes of the Ti and Al during rolling included atomic displacement, pipe diffusion along dislocations, and plastic deformation-induced vacancies. The energy inside an atom determines whether it can be diffused or not, and the diffusion rate is related to the number of atoms in the activated state. The diffusion rate of atoms in the diffusion system can be characterized by the diffusion coefficient [51] as shown in Eq. (2):

$$D = D_0 \exp(-\Delta E_a/RT). \quad (2)$$

Here, D is the diffusion coefficient, D_0 is the diffusion constant, ΔE_a is the diffusion activation energy (J mol^{-1}), R is the gas constant with a value of $8.31 \text{ J mol}^{-1} \text{ K}^{-1}$, and T is the absolute temperature (K). Temperature is the main factor affecting the diffusion coefficient. A higher temperature leads to more energetic atoms, higher frequency of their vibrations, and a greater chance that the energy of the atoms will exceed the barrier. Therefore, the atomic diffusion rate and the width of the diffusion layer at the Ti/Al interface will significantly increase as the temperature increases. The width of the interfacial diffusion layer increases with higher rolling temperature (Fig. 2). However, the high temperature may result in Kirkendall voids near the Al/Ti interface, which also affect the bonding strength [52]. Thus, the high metallurgical bonding quality will directly affect the peeling strength.

In conclusion, both mechanical locking and metallurgical bonding determine the bonding strength (σ_B), as expressed in Eq. (3):

$$\sigma_B = \alpha \sigma_C + \beta \sigma_M. \quad (3)$$

Here, the contribution of mechanical locking strength (σ_C) and metallurgical bonding strength (σ_M) is controlled, respectively, by α and β parameter. On the other hand, σ_C is related to the combined area, and σ_M is related to the width of the interfacial diffusion layer. According to the results in Figs. 2, 3, and 6, it can be known that the mechanical locking plays a key role in the enhancement of bonding strength. And the highest bonding strength was earned by cryorolling (Fig. 3).

4.2 Mechanical Properties

In Fig. 6, the tensile curves of Al/Ti/Al LCs vary in stages, which are similar to the shape of Ti/Al/Mg/Al/Ti LCs prepared by Mi et al. [53]. The tensile curves show that the tensile progress of the Al/Ti/Al LCs can be divided into three stages. First, the Ti and Al undergo elastic–plastic deformation, and the stress is maximized. Strain localization and the strain redistribution appear in the specimens due to the change of deformation coordination between Al and Ti. Here, Ti is the main bearing layer, and the existence of the interface on both sides of Ti restricts the deformation behavior of Ti and transfers the strain to the Al layer—this strain transfer improves the bearing capacity of Ti [2]. Sun et al. [54] described how the LCs have good mechanical properties due to the stress redistribution and the crack front bridging. The constraining effect of the interface changes the deformation behavior of the Ti so that the higher bonding strength can enhance the tensile strength of the LCs [45].

Fig. 8 SEM images of fracture surfaces of Al/Ti/Al LCs subjected to cryorolling at $-190\text{ }^{\circ}\text{C}$ **a–c** and $-100\text{ }^{\circ}\text{C}$ **d–f**, cold rolling **g–i**, hot rolling **j–l**

The second step is a drop in stress, crack initiation and propagation, and interface delamination. When the local stress at the interface exceeds the bonding strength, micro-cracks will be generated at the weak interface [55, 56]. There will be more cracks as the deformation continues. However, the constraining effect prevents lateral extension of cracks. These can only expand along the interface and

will eventually delaminate (Fig. 8a, d, g, j). Furthermore, the constraining effect of the interface disappears due to delamination and the rapid decrease in strain.

Third, the Ti layer and Al layer successively fracture. The disappearance of the interfacial constraining effect forms cracks at the interface, the Ti and Al layers can then expand rapidly, and the LCs then fracture. Coarse grains

Fig. 9 Nanohardness of **a** Al layer; **b** Ti layer subjected to different rolling processes

can stop the propagation speed of cracks [57], and thus the third stage of the LCs subjected to hot rolling is more obvious than those cryorolled.

In addition to the plastic deformation characteristics and fracture modes, the grain size characteristics of the specimens are another important factor affecting the properties of the composite plates. Cryorolling inhibits the occurrence of the dynamic recovery process—it enhances the dislocation accumulation and grain refinement to improve the strength of the Al layer and the Ti layer [58]. For example, the tensile strength and elongation of commercially pure Al were improved by cryorolling of Rangaraju et al. [59]. In another example, the grain size of commercial pure Ti cryorolled at $-196\text{ }^{\circ}\text{C}$ reached 80 nm with improved microhardness [60]. In addition, some researchers [27–31] validated the cryorolled metals have finer grains compared to hot-rolled sheets with the same rolling reduction ratio. Figure 9 shows higher microhardness of Ti and Al prepared by cryorolling. According to the relationship between hardness and yield strength, the strength of single-layer Ti or Al by cryorolling is higher than that subjected to cold rolling and hot rolling. Data on the rule of mixture can be expressed by Eq. (4) [11]:

$$\sigma = \sigma_{\text{Ti}} \cdot V_{\text{Ti}} + \sigma_{\text{Al}} \cdot V_{\text{Al}} \quad (4)$$

Here, σ is the strength of the composite, σ_{Ti} and σ_{Al} are the strength of the Ti and the Al layers, and V_{Ti} and V_{Al} are the volume ratio of the Ti and the Al layers. When the volume ratio of each component remains the same, the hardness of single layer increases, its strength increases, and the strength of

composite plate will increase. Thus, cryorolling can improve the strength and ductility of Al/Ti/Al LCs.

5 Conclusions

1. Al/Ti/Al LCs were fabricated by cryorolling, cold rolling, or hot rolling. Cryorolling can significantly improve the bonding strength, tensile strength, and ductility of Al/Ti/Al-LCs. Excellent mechanical properties can be obtained with $-100\text{ }^{\circ}\text{C}$ cryorolling.
2. The bonding strength of the Al/Ti/Al LCs subjected to cold rolling is lower than that subjected to hot rolling or cryorolling. The bonding strength is highest for the specimens cryorolled at $-100\text{ }^{\circ}\text{C}$.
3. The bonding strength of Al/Ti/Al LCs depends on the mechanical locking strength, metallurgical bonding strength, and tensile strength of the Al layer.

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