Effect of annealing temperature and time on the microstructure, mechanical properties and conductivity of cold-rolled explosive Cu/Al composite sheets

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Abstract

The 1.5 mm thick Cu/Al composite sheet was obtained after explosive-welding and 6 passes cold rolling. The effects of annealing temperature and time on the microstructure, mechanical properties, and conductivity of cold-rolled Cu/Al composite sheets were investigated. The results show that the thickness of the interface diffusion layer with compounds of Al2Cu, AlCu, and Al4Cu9 increases with the increase in annealing temperature and holding time, and the total activation energy is 108 kJ mol−1. The tensile strength of composite sheets decreases and the fracture elongation increases with the increase of annealing temperature. Especially when the annealing temperature increased from 300 °C to 350 °C, the tensile yield strength decreased from 225 MPa to 77 MPa, and the elongation increased from 16% to 37.5%. The tensile-shear strength of composite sheets reaches to 117 MPa after annealing at 400 °C for 2 h. The conductivity of composite sheet is greatly affected by the annealing time. At the annealing temperature of 350 °C and time of 4 h, the composite sheets show a better conductivity of 96.4% IACS.

1. Introduction

Cu/Al bimetal composite materials not only have the advantages of the inexpensiveness and lightweight of Al but also have remarkable thermal conductivity, high electrical conductivity, facile brazing ability and low contact resistance of Cu, so they are widely applied in metallurgical equipment, as well as in energy, aerospace, automotive, marine, power, electronics and electrical industries [1–3].

At present, the Cu/Al layered metal composite materials can be produced by cold rolling bonding [4–8], explosive welding [9–13], roll casting composite [14–18], extrusion bonding [19, 20], plasma activated sintering [21] and others. These studies mainly focus on the materials preparation, property, microstructure evolution, formation and effect of interfacial compound. In particular, the relationship between interfacial metallurgical bonding, control of interfacial compound and mechanical properties, electrical conductivity, heat conduction, corrosion resistance of composites is the important research content in the Cu/Al composites.

Kim et al [22] fabricated a Cu/Al/Cu clad-composite by rolling, and studied the interactive deformation and ductility of composite. They found that the interactive and constraint deformation of Cu and Al layers induced co-deformation, which had a positive influence on the ductility; however the thick brittle interfacial intermetallics have no beneficial effect on the deformability. Chen et al [23] studied the coupling effects of interface bonding strength and matrix strengths of Cu and Al layers on the plastic deformation behaviors of horizontal twin-roll casting Cu/Al composite, and found that both high interfacial bonding strength and low
strength difference between Cu and Al were beneficial for enhancing the cooperative deformation performance of Cu/Al laminated composite. Mao et al [24] reported that, due to the mechanical locking of Cu/Al interface, the peel strength of horizontal twin-roll caster Cu/Al clad sheet first slightly increased and then gradually decreased with the increase of the rolling pass, and after annealing, the peel strength improved remarkably. For Cu/Al composite, the explosive welding and subsequent cold rolling is a good method to fabrication high bonding strength sheet, due to the cold rolling can cause secondary tensile stresses in the plates and intensifies mechanical properties of bimetallic plates. Hoseini et al [25] investigated the effects of rolling reduction on the interface properties of the explosive Al/Cu/Al composite plates. Their research revealed that a more uniform hardness profile near the interface was obtained after the rolling process. Compared with the traditional rolled composite, the explosive composite maintains the bonding quality and surface quality at the interface of the multi-layer metal sheet. Asemabadi et al [26] also studied the effect of cold rolling on the mechanical properties and bond strength of Al/Cu/Al bimetal, and they found that the ultimate strength and hardness increased significantly but the elongation diminished with the increase of thickness reduction, which was due to the brittle intermetallic compounds at the interface and the nucleation and propagation of microcracks accelerated under tension and plastic deformation.

In this study, the combination of explosive welding and cold rolling was used to prepare Cu/Al composite sheets, considering the excellent metallurgical bonding interface produced by an explosive composite and the facile formation ability of the cold-rolling process to prepare thin sheet. The cold-rolled composite sheets were annealed at different temperatures and time in order to endow them with excellent mechanical properties and electrical conductivity.

2. Materials and methods

2.1. Materials
A 10 mm thick industrial pure copper T1 (Cu ≥ 99.95 wt%) plate and 10 mm thick industrial pure aluminum AA1060 plate were used as the starting materials for explosion welding. The explosion-welded 20 mm thick Cu/Al composite plate was prepared by Hunan Phohom New Material Technology Co. LTD (Changsha, Hunan, China). Before explosion welding, the surface of the plate is mechanically polished to ensure no ash layer and oxide film. Then the copper plate was placed above the aluminum plate, with an angle of 5° between them. The No. 2 rock ammonium nitrate explosive with 1.7 g cm⁻² was covered on the plate. The detonator is located at the edge of plate, and the dosage of explosive is 2.2 g cm⁻² around the detonator.

2.2. Cool rolling and annealing
The explosion-welded plates with a size of 20 mm × 130 mm × 150 mm were rolled by a Φ 550 mm × 650 mm two-roll mill at room temperature for 6 passes, as shown in figure 1. As measured, the thicknesses of the composite sheets after each pass were 18 mm, 12 mm, 7 mm, 4.5 mm, 3 mm and 1.5 mm. The total reduction rate of the plate was 92.5%. Due to work hardening, the respective reduction of Cu and Al tended to be the same with the increase of the total reduction. Therefore, the thickness of Cu and Al was almost the same after the last pass.

The cold-rolled Cu/Al composite sheets with a size of 1.5 mm × 140 mm × 1850 mm are shown in figure 2(a). During the rolling process, Al and Cu maintained a good state of synergistic deformation. However, since the deformation of Al is higher than that of Cu along the transverse direction, the Al-side had a lateral overflow, due to which the edge cracked. The rolled sheet maintained good bonding according to macroscopic morphology observations, as shown in figures 2(b) and (c). Then, the cold-rolled Cu/Al composite sheets were annealed for different time at temperatures of 300 °C, 350 °C, 400 °C, and 450 °C, respectively.
2.3. Characterization of properties and microstructure

Room temperature tensile tests and interface tensile shear tests were performed on the MTS-810 test machine and the size of samples are shown in figure 3. The tensile speed was 1 mm min⁻¹, and the tensile direction was parallel to the rolling direction. A relatively small shearing area (20 mm²) was set to prevent the sheet from breaking on the Al side in advance during the tensile process. Conductivity was measured by an FD102 eddy current conductivity meter at room temperature. Quanta-200 scanning electron microscopy and JXA-8230 electron probe micro-analysis were performed for observing the microstructure of the Cu/Al interface.

3. Results and discussion

3.1. Interface microstructure

The interface microstructure of the original explosive-welded plate and cold-rolled sheet are shown in figure 4. The original explosive-welded interface shows a wavy structure composed of vortexes (figure 4(a)). After rolling, the vortex structures are destroyed, while the interface bonding is straight, and there are not any visible gaps and cracks (figure 4(b)).

Figure 5 shows the interface microstructure of a cold-rolled sheet annealed at different temperatures and time periods. After annealing heat treatment, the Cu/Al interface became clear with a canine-shaped joint. In addition, a three-layer structure is clearly observed due to elemental diffusion. At the same annealing time, the thickness of the diffusion layer increased significantly with the increase in annealing temperature, i.e., from 2 μm at 300 °C to 13 μm at 450 °C. At the same temperature, the thickness of the diffusion layer increased as the annealing time increased. For example, on holding the annealing temperature at 450 °C for 0.5 h to 4 h, the thickness raised from 6.6 μm to 17.6 μm. Table 1 lists the thickness of the diffusion layer under different annealing temperatures and time. According to the table, the temperature had a greater influence on the growth of the diffusion layer than the annealing time, which was similar to the results of some other researchers [4, 7, 27].
Table 1. The interface microstructure of a cold-rolled sheet annealed at different temperatures and time.

| Time (h) | 300 °C | 350 °C | 400 °C | 350 °C |
|---------|--------|--------|--------|--------|
| 0.5h    | Cu     | Al     | Cu     | Al     |
| 1h      | Cu     | Al     | Cu     | Al     |
| 2h      | Cu     | Al     | Cu     | Al     |
| 4h      | Cu     | Al     | Cu     | Al     |

Figure 4. The interface microstructure of explosive-welded plate (a) and cold-rolled sheet (b).

Figure 5. The interface microstructure of a cold-rolled sheet annealed at different temperatures and time.
Table 1. The thickness of the diffusion layer at different annealing conditions (μm).

|        | 300 °C | 350 °C | 400 °C | 450 °C |
|--------|--------|--------|--------|--------|
| 0.5 h  | 1.3    | 1.5    | 3.2    | 6.6    |
| 1 h    | 1.5    | 2.0    | 5.2    | 9      |
| 2 h    | 1.9    | 2.5    | 7.4    | 13.1   |
| 4 h    | 2.6    | 3.6    | 9.8    | 17.6   |

Figure 6. The distribution of Cu and Al elements at the interface after annealing at (a)–(c) 300 °C, (d)–(f) 350 °C, (g)–(i) 400 °C, and (j)–(l) 450 °C for 1 h.
Figure 6 shows the distribution of Cu and Al elements at the interface after the cold-rolled sheets were annealed at 300 °C, 350 °C, 400 °C and 450 °C for 1 h. It can be observed that new phases were generated with distinct growth, and the copper atoms diffused into the aluminum side more than aluminum atoms diffused into the copper side. Copper atoms can diffuse deep into the aluminum side, while aluminum atoms diffuse only up to the interface, and the transition layer is mainly on the Al side. Moreover, the elemental diffusion rate of Al in Cu is much smaller than that of Cu in Al at the same temperature. Simultaneously, due to the relatively high thermal conductivity of Cu, the Cu-side component was evidently heavier than the Al side component.

Figure 7 shows the EPMA line analysis of interface in the sample annealed at 450 °C for 0.5 h. It can be observed that there were three layers of component compounds, reflecting the changes in the composition of Cu and Al. A relatively obvious stage in each of the three diffusion layers was formed, indicating that the content of intermetallic compounds in a certain diffusion layer does not change significantly. The point analysis of the three compounds is illustrated in table 2. From the proportions of element compositions, it can be inferred that the three compounds from the Cu layer to the Al layer are Al₄Cu₉, AlCu and Al₂Cu.

According to Al–Cu binary phase diagram, there are five kinds of intermetallic compounds of AlCu, Al₂Cu, Al₃Cu₉, Al₂Cu₅, and Al₄Cu₉. However, only three kinds of intermetallic compounds were detected in the interface, and the formation sequence was Al₂Cu, Al₄Cu₉ and AlCu inferred from the thickness of the compound in figure 7, which is corresponded with the literatures [28–30]. It is mainly caused by thermodynamic and atomic diffusion dynamics. In the initial stage of annealing, Cu and Al atoms diffuse to each other, leading to saturation of Cu and Al solid solutions on both sides of the interface. According to Al–Cu phase diagram, the maximum solid solubility of Cu in Al and Al in Cu are 2.48 at% and 19.7 at%, respectively. Therefore, the solid solution Cu (Al) reaches saturation first, and the supersaturated copper reacts with the aluminum base. Al₂Cu nucleated at the interface. After the formation of the Al₂Cu phase, the diffusion of Al and Cu atoms in the Al₂Cu layer becomes difficult. At the interface between Al₂Cu and Cu, Al was poorer and its effective concentration was lower in comparison with Cu. Al₄Cu₉ was generated at the interface of Al₂Cu and Cu. After the formation of Al₂Cu and Al₄Cu₉ layers, Al atoms passing through the Al₂Cu barrier and Cu atoms passing through the Al₄Cu₉ barrier are approximately the effective concentration at the Al₂Cu/Al₄Cu₉ interface, which caused the form of AlCu [30].
3.2. Diffusion kinetics

Generally, the solid-state diffusion reaction follows an empirical equation for the growth of bimetallic compounds [31]:

\[
y = k_1 t^n
\]

where \( y \) is the thickness of the diffusion layer, \( t \) is the reaction time, \( n \) is the time index, and \( k \) is the growth rate constant. Usually, \( n \) has two values. If \( n \) equals 1, the thickness of the compound is linear with time and the growth law of the compound is controlled by the reaction rate. If \( n \) equals 0.5, the thickness of the compound is parabolic with time and the growth law of the compound is controlled by volume diffusion [32].

Taking the logarithm on both sides of equation (1), it can be obtained:

\[
\ln y \propto \ln t
\]

According to equation (2), \( \ln y \) and \( \ln t \) have a linear relationship, and the slope of the straight line gives the value of \( n \). Substitute the thicknesses of the interface compounds (table 1) into equation (2), the relationship diagram is obtained, as shown in figure 8. The slopes at different temperatures are almost the same, so calculating the average of each slope, and the value of \( n \) (\( n = 0.46 \)) can be obtained, which is approximately 0.5. Hence, \( n \) in equation (1) is taken as 0.5, thus indicating that the growth of the compound is affected by volume diffusion.

So the growth equation can be written as:

\[
y = k_2 t^{0.5}
\]

Also,

Usually, \( k_2 \) is known as the velocity constant. By linearly fitting the thickness of the intermetallic compound between the interfacial layer and the annealing time values, the \( k_2 \) values at 300 °C, 350 °C, 400 °C, and 450 °C can be obtained. Under the same temperature, the thickness of the diffusion layer generally increases exponentially with respect to the annealing time, satisfying the Arrhenius equation:

\[
\ln k_2 = \frac{Q}{R} \frac{1}{T} + \ln k_0
\]

The logarithm of the two sides is available as follows:

\[
\ln k_2 \propto \frac{1}{T}
\]

where \( k_0 \) is the pre-exponential factor (cm\(^2\) s\(^{-1}\)), \( Q \) is the growth activation energy of the intermetallic compound (kJ mol\(^{-1}\)), and \( R \) is the gas constant (8.314 J mol\(^{-1}\) \cdot K). Growth activation energy can be obtained by linearly fitting the curves of \( \ln k_2 \) and \( 10^4/T \), as shown in figure 9.
The intercept and slope were obtained according to the fitting results in figure 9. The factor $k_0^2$ was calculated to be 11 mm$^2$ s$^{-1}$ and the activation energy $Q$ of the IMC was 108 kJ mol$^{-1}$, which are very close to the values measured by other researchers. In Braunovic’s research, there are two diffusion mechanisms in the temperature range from 250 °C to 520 °C [33]. As the activation energies are different: for 72 kJ mol$^{-1}$ in the range of 250 °C to 325 °C and 137 kJ mol$^{-1}$ in the range of 325 °C to 520 °C. Between 250 °C and 325 °C, the grain boundaries and dislocations are the main reasons for diffusion process. Above 325 °C, the volume diffusion is activated by the increasing thermal energy.

3.3. Properties
3.3.1. Tensile test
Figure 10 shows the tensile stress-strain curve of cold-rolled sheets and different temperatures annealed sheets (annealing time is 2 h). The tensile strength and yield strength of the cold-rolled sheet are 363 MPa and 287 MPa, respectively, and the fracture elongation is 4.4%. As the annealing temperature increases, the tensile strength gradually decreases and the elongation increase. When the composite sheet is annealed at 300 °C for 2 h, the tensile strength, yield strength and elongation are 305 MPa, 225 MPa and 16%, respectively. When the annealing
temperature higher than 300 °C, the yield strength decreased rapidly, and the yield strength of 350 °C annealed composite sheet is only 77 MPa, the elongation increased to 37.5%.

Annealing heat treatment improves the elongation but reduce the tensile strength of Cu/Al composite sheets as figure 10 shows. The strengths decrease is less at annealing temperature at 300 °C than those at higher temperature. It is due to the severe deformation in Al and Cu matrix at cold rolled composite sheets. After annealing at 300 °C, complete recrystallization occurs in aluminum matrix and the equiaxed grains can be attained, but due to the high recrystallization temperature and incomplete recrystallization occurs in copper matrix. The equiaxed Al grains are beneficial for dislocation slip and climb, reduce the strength, but the incomplete recrystallized Cu grains working as the strengthen phase still has strong tensile strengthen. After annealing temperature higher than 350 °C, due to the softening of copper matrix the yield strength decreases dramatically. Meanwhile, with the growth of Al grains at high temperature, dislocation pile-up and stress concentration can be easily formed at the grain boundaries and a small stress drive dislocation slip across the grain boundaries lead to the yield strength of Cu/Al composite sheet decrease.

The elongations are governed by the interaction between the matrix and the interface layer. During the tensile deformation process, the Cu and Al matrix remain cooperatively deformed before fracture; the two matrixes have the same strains, while the respective stresses of the Cu and Al matrix will be different due to the differences in mechanical properties of Cu and Al. Generally, uniform elongation is closely related to the strain-strengthening ability of the material, which is jointly controlled by the generation of dislocations and the dynamic recovery of dislocations, both of which compete with each other. By promoting the generation of dislocations or inhibited the dynamic recovery of dislocations, the plastic deformation ability of the material can be improved [34]. During the tensile tests of the composite sheet, the large amount of formation and accumulation of dislocations at the Al matrix and the interface caused by stress differences significantly improve its strain-strengthening ability. Secondly, the copper layer can effectively inhibit the neck fracture behavior of the aluminum layer during the stretching and necking stage. Thirdly, due to the dramatically grow of interfacial intermetallic compounds at high temperature and thicker interlayer inhibits the dislocation slip across the interface, the cooperate deformation of Cu/Al become difficulty. When the copper and aluminum matrix cannot work as a whole, the interlayer will fracture. So with the increase of annealing temperature the elongations increase slowly.

### 3.3.2. Tensile-shear experiment

To verify the bonding strength of the interface, the tensile shear of cold rolling and annealed sheets at different temperature for 2 h were test, the result as table 3 shows. The cold rolled sheet shows relatively low tensile-shear strength, which is 98 MPa. After annealing for 2 h, the shear strength of the composite sheet increased with the increase in the annealing temperature, reached a maximum value of 117 MPa at 400 °C, and then decreased as the annealing temperature was increased to 450 °C.

Usually the tensile shear strength of the sheet is determined by the interface strength. In the cold rolled composite sheet, the element diffusion isn’t significant, and the interface intermetallic compound layer is thinner lead to the low shear strength [35]. During the annealing process, the two metal atoms diffused into the adjacent metal matrix through the bonding interface at high temperature, meanwhile, the grain boundary migrates, creating a larger bonding probability for the two elements at the interface. Due to the rearrangement movement of atoms, more coincident lattice positions and common grain boundaries were formed, and the degree of interface metallurgical bonding enhanced. When the annealing temperature was increased further, the grains began to coarsen and the thickness of the intermetallic compound increased. The hard and brittle phase gradually reduced the bonding strength of the bonding surface, which could seriously damage the shear strength of composite sheet.

### 3.3.3. Conductivity

Figure 11 shows the conductivity of composite sheet annealing at different temperatures and time. It can be seen, during annealing at temperatures of 300 °C, 350 °C, 400 °C and 450 °C, the conductivity increased at the beginning and then decreased. The peak conductivities occurred after annealing for 4 h at all temperatures and reached a maximum of 96.4% IACS (International Annealed Copper Standard) for the sheet annealed at 350 °C. After annealing at 450 °C for more than 4 h, the conductivity decreased significantly.

| Table 3. Tensile-shear strength at different annealing temperatures. |
|----------------------|-------|-------|-------|-------|-------|
| Annealing Temperature (°C) | cold rolling | 250 | 300 | 350 | 400 | 450 |
| Tensile-shear Strength (MPa) | 98 | 103 | 106 | 110 | 117 | 107 |

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**Notes:**

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The conductivity of Cu/Al composite sheet is related to Cu and Al matrix and intermetallic compound layer. In general, the resistances of Cu/Al intermetallic compounds are 3~6 times higher than the pure Al. Obviously, the intermetallic compound layer is disadvantageous to improve the conductivity of the Cu/Al composite sheet. However, the conductivity is also related to the dislocations and crystal defects such as vacancies and interstitial atoms [36]. During the annealing, the dislocations and crystal defects could recover therefore the defect concentration reduce and the atomic lattice vibration strengthen, which enhance the conductivity. After cold rolling, the intermetallic compound layer is discontinuous, and then the continuous layer formed with the increase of annealing temperature and time. When the annealing time exceed 4 h, the thickness of intermetallic compound layer increases. Due to the growth of compound layer and the nuclear vibration near the equilibrium position intensifier, the electron blocking and scattering effects deteriorate, the conductivity decreased gradually.

4. Conclusions

(1) During the annealing of cold-rolled explosive welding Cu/Al composite sheets, the Al\textsubscript{2}Cu, Al\textsubscript{Cu}, and Al\textsubscript{4}Cu\textsubscript{9} compounds formed in interface, and the total activation energy of the compounds is 108 kJ mol\textsuperscript{-1}.

(2) After annealed at 300 °C for 2 h, the tensile strength, yield strength and elongation of Cu/Al composite sheets are 305 MPa, 225 MPa and 16%, respectively. while the tensile shear strength is 117 MPa, after annealed at 400 °C for 2 h.

(3) The conductivity of the composite sheets is greatly affected by the annealing time, which reached to 96.4% IACS after annealing at 350 °C for 4 h.

(4) In the cold-rolled explosive welding Cu/Al composite sheets, the temperature had greater effect on the growth of diffusion layer than the annealing time.

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