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Evaluation of diffusion and phase transformation at Ag/Al bimetal produced by cold roll welding

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Abstract: Aluminum and silver strips were cold welded by rolling and a bimetallic strip was produced. To create cold weld between Al and Ag, mating surfaces were specially prepared and various rolling thickness reductions were applied. The minimum critical thickness reduction to begin cold weld was specified as 70% which equals 0.1630 critical rolling shape factors. The bimetallic strips were treated by diffusion annealing at 400 °C and various annealing time. The Al/Ag interface of strips was observed by scanning electron microscope to investigate the formation of hard and brittle probable phases. The effect of anneal time on diffusion distance and phase transformation was also analysed by EDS analysis and line scan. A diffusion region along the interface in the Ag side was observed and its width increased with prolonging annealing time. Some δ phases were detected close to the interface after anneal treating for 3 h and δ phase was thicker and more continuous by increasing annealing time. The microhardness measurement showed that in spite of formation of δ phase due to diffusion annealing, the interface hardness was reduced.
Key words: Ag/Al bimetallic strip; cold weld; anneal; multilayer composite sheet; shape factor

1 Introduction

Layered structural composites are often considered most efficient structural materials and to have great potential in comprehensive functions they can perform. Multilayer strips consisting of two or more different material layers are widely used in various industries due to variety of properties such as high electrical conductivity, corrosion resistivity and high heat conductivity [1]. Cold roll welding is one of the techniques used to produce multilayer composite sheets and strips. Rolling pressure can create a metallurgical bond between the metal strip components in the multilayer pack [2, 3]. Metallurgical weld is creation of metallic continuity at the joint interface. The mechanical, physical and chemical properties are continuous at the interface of a metallurgical weld [4]. The application of sufficient pressure causing threshold deformation and a cold metallic bond is created between the metallic layers. The required energy for welding is provided by applying mechanical work at interface. The process parameters, such as rolling pressure and annealing temperature and time after welding must have significant effects on the weld microstructure, mechanical property and alloy element distribution at interface regions and material matrixes. In this research, one layer of aluminum was situated on a layer of silver, and then the non-bonded two layer sheets were rolled simultaneously, as shown in Fig. 1(a). This layered composite material, the aluminum–silver bimetallic strip, was used in the manufacture of electrical bimetal transition piece in bus bars line in power plant, as shown in Fig. 1(b). In the roll bonding process, there exists the difficulty of material work hardening, so the clad sheet must be annealed before rolling in order to obtain a good formability [5].

Each bimetal with 20 cm^2 connecting surface can conduct a high electrical current up to 100 A/cm^2 [6]. If copper terminal and aluminum bus are connected to each other directly without an Al/Ag bimetal transition piece, the service life of the electrical connection is low for about 1 year because of increasing temperature and intermetallic compounds formation at interface. But by using Al/Ag bimetal, the service life is increased to more

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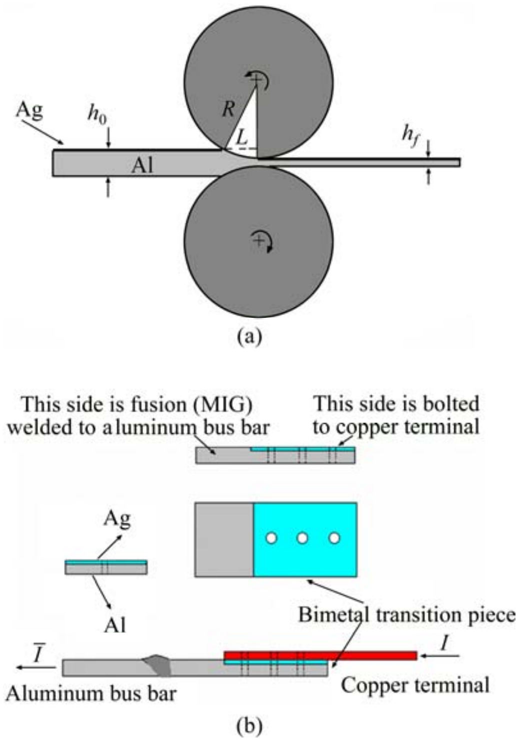

Fig. 1 Schematic diagrams of cold role welding of Al−Ag pack (a) and bimetal electrical transient joint (b)

than 20 years [6, 7]. In manufacturing process of a high quality transition piece, the electrical resistance of Al/Ag bimetal should be kept as low as possible to minimize the waste of electrical energy. Formation of intermetallic compound can increase electrical resistance of bimetal. Study of phase transformation and intermetallic formation at the interface of roll bonded Al/Ag bimetal has not been reported in previous works. Some experimental observation [8] and computer modeling [9, 10] present structure and diffusion behavior at different temperatures for Ag/Cu or Ag/Ni interface.

The present investigation focuses on determining the critical deformation to create cold weld, evaluating the effects of diffusion annealing time on phase transformation and hardness of Al/Ag interface.

2 Experimental

The cold roll welding experiments were carried out under a laboratory hydraulic pressure rolling machine with a loading capacity of 5 t, as shown in Fig. 2. The diameter of the roll is 67 mm, the rolling speed is 9 cm/s and the rolling mill is made of 86CrMoV7 steel. During the cold welding process, the rolling pressure was recorded. In the experiments, annealed aluminum Al 1050 and pure Ag (99.99%) were used. The Al and Ag strips with initial dimensions of 100 mm×15 mm×1 mm and 100 mm×15 mm×0.5 mm were used, respectively.

The strip materials were annealed in a furnace

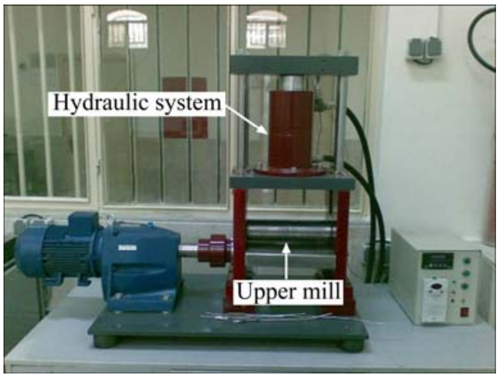

Fig. 2 Hydraulic pressure rolling machine

before roll welding to decline the hardness and increase the work ability of Al and Ag. Before roll bonding, the mating surfaces of strips were specially prepared to remove all the surface contaminates. This surface contaminates are composed of oxides, sulfides, phosphates, absorbed ions, humidity, grease and contamination particles. The surface preparation was done by detergent washing, water rinsing and then degreasing of surfaces by acetone $CH_3C(O)CH_3$ and finally scratch brushing to remove the oxide film on both surfaces of the aluminum and silver strips. The bristles of the brush were made of stainless steel. The outside diameter and rotational speed of brush were 18 cm and 650 r/min, respectively. It was reported that the oxide layer was formed very rapidly on the surface of the aluminum and acts as a barrier to the formation of a metallurgical bond [11, 12]. Therefore, the interval time between the surface preparation and rolling was kept less than 3 min to avoid the formation of a thick and continuous oxide layer on the surfaces of the strips.

The rolls were closed so the desired amount of roll pressure was applied on the strips bundle. Then by moving the rolls, the friction between the rolls and the strip pulled the bundle through the rolls.

The reduction in the strips pack can be expressed as:

$$r=(h_0-h_f)/h_0\times 100\% \quad (1)$$

where r is the reduction; h_0 is the initial thickness of strips and h_f is the final thickness.

The influences of roll diameter R and roll contact length along surface L are not considered. The geometry of the deformation zone has a strong influence on the redundant work, the frictional work, and the forming forces and critical condition of cold welding. The effect of R and L, the primary thickness and the reduction ratio on cold weld are all summarized in an complementary factor stated as the shape factor Δ. For flat rolling, shape factor Δ is simply the mean thickness $(h_0+h_f)/2=h_0(2-r)/2$ divided by the roll contact length $L=\sqrt{R(h_0-h_f)}=\sqrt{rRh_0}$ as [13]:

$$\Delta = \left(\frac{2-r}{2}\right) \times \sqrt{h_0 / rR} \quad (2)$$

Therefore, Δ is a better factor than r to evaluate the cold welding critical condition. Shape factor illustrates the effect of deformation zone geometry on the cold roll process.

For this metal combination, a series of rolling experiments were carried out using the rolling reductions from 30% to 93%. Specimen with 80% reduction with very good cold welding was selected and diffusion annealed at 400 °C for time ranging from 1 to 4 h. If the rolls were over-cambered, the residual stress pattern was the opposite. Centerline compression and edge tension may cause edge cracking, lengthwise splitting and a wavy center. The annealing was conducted in a vacuum furnace filled with an inert gas. A furnace with temperature deviation of ±3 °C was used in these experiments.

In order to evaluate the effect of annealing time on the interface microstructure, scanning electron microscope (SEM), EDS analysis and line scan were used. The metallographic samples were cut longitudinally, then ground and polished. As shown in Fig. 3, microhardness testing with 0.98 N loading was performed on the polished outside surfaces of the bimetallic specimens.

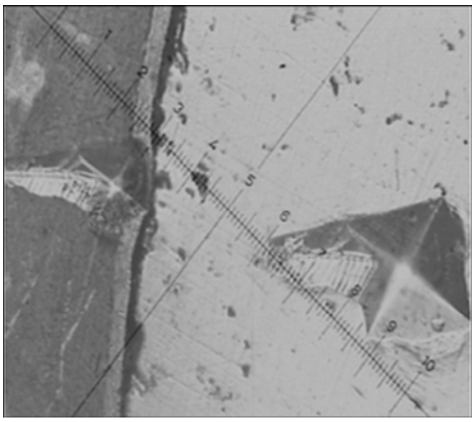

Fig. 3 OM image showing microhardness tract on bimetal specimen

3 Results and discussion

3.1 Critical reduction for cold welding

The applied pressure, reductions and the results of shape factors are presented in Table 1. After applying different amounts of pressures to create various reductions and shape factors, the cold metallic weld between Al/Ag begins at critical shape factor of Δ=0.1630 which is correspond to 70% reduction. By exceeding thickness reduction more than 90%, the composite damage is observed. The suitable range of shape factor to perform cold weld between Al and Ag is assessed between Δ=0.1630 and Δ=0.1217.

Table 1 Dimensional changes during roll bonding

Specimen number	Bimetal initial thickness/ mm	Bimetal final thickness/ mm	Applied pressure/ 10^5Pa	Reduction/ %	Shape factor
1	1.5	1.050	50	30	0.3259
2	1.5	0.900	55	40	0.2656
3	1.5	0.750	60	50	0.2227
4	1.5	0.600	65	60	0.1898
5	1.5	0.450	70	70	0.1631
6	1.5	0.375	80	75	0.1515
7	1.5	0.236	90	80	0.1409
8	1.5	0.225	100	85	0.1309
9	1.5	0.150	120	90	0.1217
10	1.5	0.101	140	93	0.1165

Figure 4 illustrates that the reduction (r) increases with increasing the applied pressure. It is seemed that the slope of curve decreases at point of 70% reduction corresponding to the critical point at the beginning of cold welding.

Figure 5 demonstrates that Δ decreases with increasing the reduction. With the increase of reduction, the variation of shape factor decreases and the shape factor goes to a constant limit of about 0.12. It is seemed that when the variation of shape factor decreases

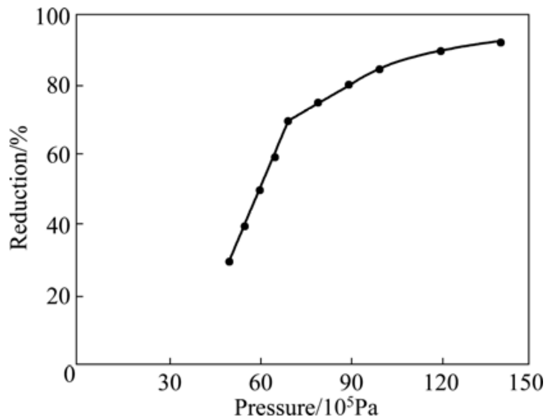

Fig. 4 Effect of pressure on thickness reduction

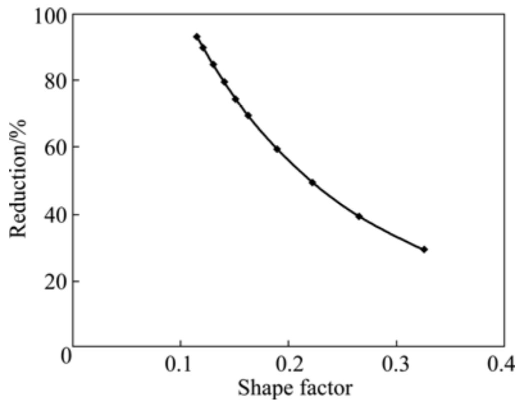

Fig. 5 Relationship between shape factor and thickness reduction

and goes to a constant limit, the cold weld is started and carried out.

3.2 Interdiffusion and phase formation

The understanding of the Ag−Al system depends on the individual metal properties of Al and Ag. Both materials have a FCC crystallographic structure with lattice parameters of 0.2889 and 0.2863 nm for Al and Ag, respectively, and the lattice mismatch is about 0.9%. The segregation properties of the two metals depend on the surface-free energy of each material, which is smaller in Al (1.16 J/m^2) than in Ag (1.25 J/m^2), implying that Al tends to segregate at the surface of Al/Ag alloy (≥3% of Ag). The Ag−Al phase diagram indicates that the solubility of Al in Ag is high even at room temperature, while at the same temperature, the solubility of Ag in Al is negligible. The Ag/Al equilibrium ground state structures are FCC for Ag, HCP for Ag_2Al and FCC for Al. The Al segregates into the FCC solid solution and the HCP intermediate δ phase. In general, the transfer rate of Al atoms through interface is greater than Ag atoms and the experiment result also confirms it.

Aluminum and silver line analysis across the interface is shown in Fig. 6. The diffusion of Al along the

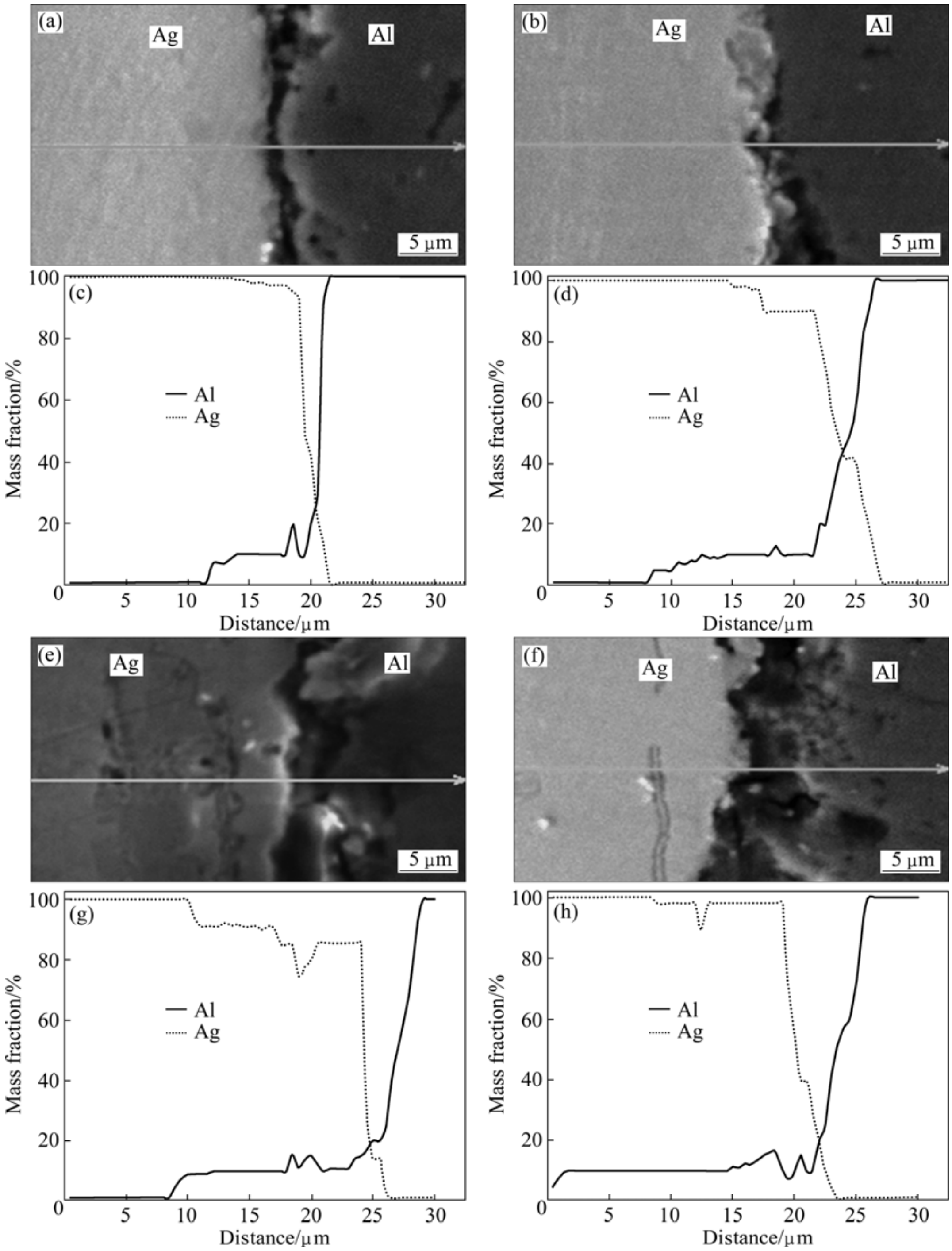

Fig. 6 Line analysis of interface region for Al−Ag bimetallic specimens annealed at 400 °C for 1 h (a, c), 2 h (b, d), 3 h (e, g), 4 h (f, h)

interface to Ag side is considerable. For a binary system of Al−Ag in diffusion annealing process, the composition has to change from high to low Al-content. By line scan analysis, a pick of Al is observed near the interface at Ag side in Figs. 6(a) and (b). This concentration of Al near the interface in Ag side causes the formation of a new phase gradually. As shown in Figs. 6(c) and (d), a layer of δ phase is formed and becomes thicker with increasing the annealing time.

Figures 7(a) and (b) show the interface after 1 and 2 h annealing at 400 °C, respectively. In this case, the diffusion zone could be seen but no phase near the interface is observed. As shown in Fig. 7(c), after annealing at 400 °C for 3 h, in the Ag side a δ phase appears. δ phase becomes thicker and more continuous near the interface after 4 h. Figure 7(d) shows a δ phase near the interface formed proximate in the Ag side.

Several samples were analyzed by EDS point analyzer near the interface, and the results are presented in Table 2. The point composition shows that the content of Al in Ag side raises with increasing the annealing time. The analysis results are in the range of δ phase compositions in the Al and Ag diagram phase.

Table 2 EDS analysis of point located at 7 μm from interface in Ag side after annealing at 400 °C

Annealing time/h	x(Al)/%	x(Ag)/%	w(Al)/%	x(Ag)/%
1	36.08	63.92	12.37	87.63
2	39.25	60.75	13.91	86.09
3	41.79	58.21	15.22	84.78
4	44.44	55.56	16.67	83.33

The effect of annealing time on the hardness of aluminum and silver strip near the interface is shown in Fig. 8. The hardness of the aluminum and silver side drops significantly as the annealing time increases to 4 h. It is observed that the hardness of the strip does not increase with δ phase formation and interdiffusion. It may be due to the work hardening recovery.

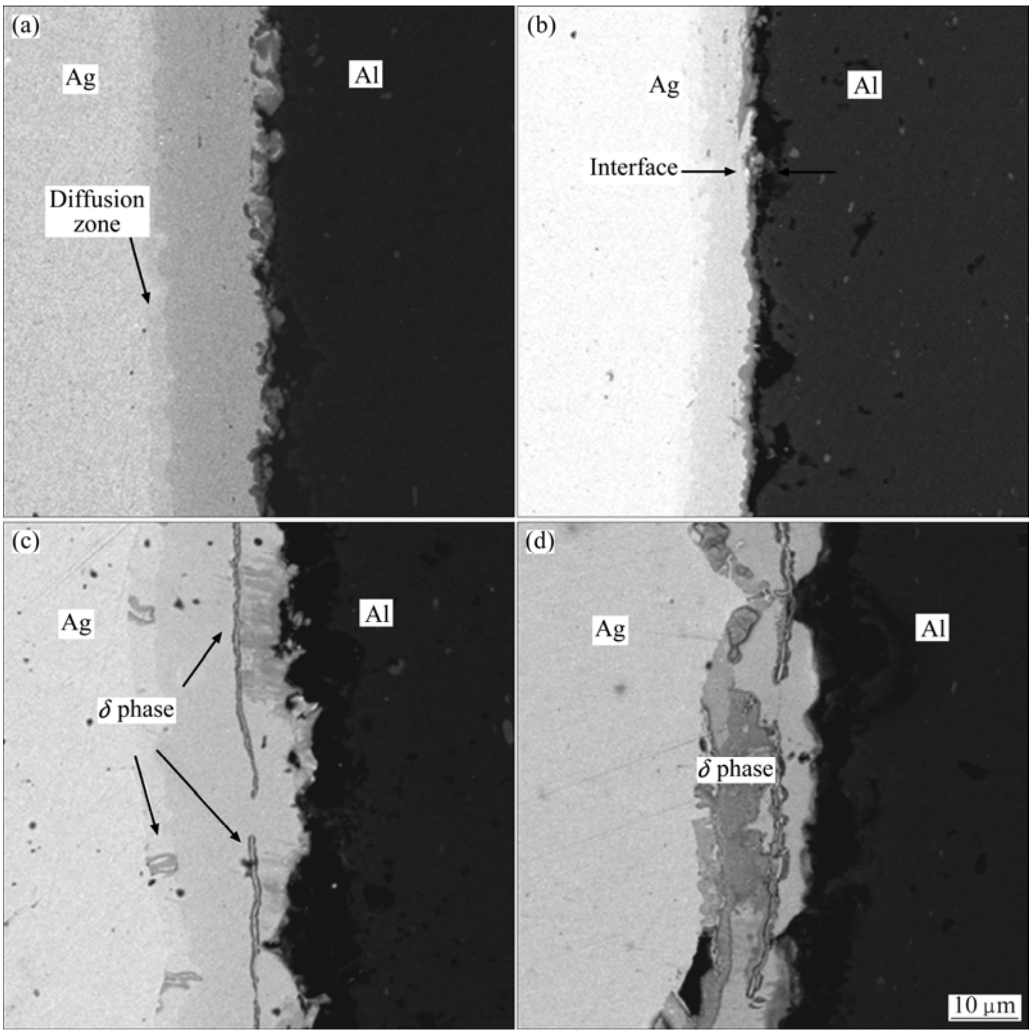

Fig. 7 SEM images of interface region for Al−Ag bimetallic specimens annealed at 400 °C for 1 h (a), 2 h (b), 3 h (c), and 4 h (d)

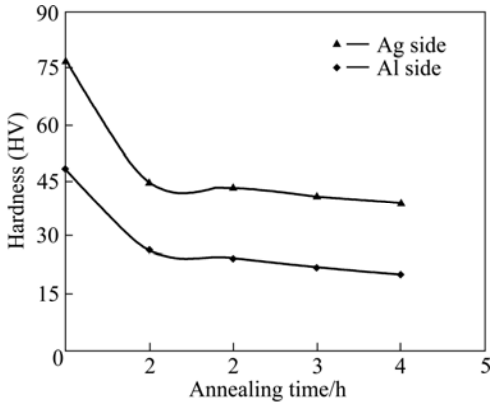

Fig. 8 Hardness of Al and Ag near interface of bimetallic specimens vs annealing time

4 Conclusions

1) The minimum critical reduction to begin the cold weld between Al and Ag is specified as 70% equal to 0.1630 critical rolling shape factor. It is seemed that when the variation of shape factor decreases and goes to a constant limit, the cold weld would be started and carried out.

2) By increasing the pressure and thickness reduction more than 90%, composite damage is observed. The suitable range of shape factor to perform cold weld between Al and Ag is assessed between 0.1630 and 0.1217.

3) Diffusion annealing at 400 °C of 80% reduction samples is enhanced, the driving force for phase transformation and the intermediate *δ* phase is formed after 3 h. By increasing annealing time to 4 h, *δ* phase is thickened in the form of a layer near the interface. To prevent the formation of continues *δ* layer and prevent the weakening of the cold weld, it is proposed to limit the annealing time to 3 h.

4) The hardness of the Al and Ag components near the interface decreases with increasing interdiffusion and *δ* phase formation.

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冷轧焊接制备 Ag/Al 双金属材料的扩散和相转变

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摘　要：通过轧制 Al 条和 Ag 条制备 Ag/Al 双金属条。为使 Al 和 Ag 条之间发生冷焊，对表面进行处理并设置不同的轧制压下量。实验表明：发生冷焊的最小临界轧制厚度压下量为 70%，相当于轧制形状因子 0.1630。对双金属条进行均匀化退火，于 400 °C 保温不同时间。利使用扫描电子显微镜观察 Al/Ag 界面，研究可能存在的硬脆相。通过 EDS 分析和线扫描分析退火时间对扩散距离和相转变的影响。在界面上 Ag 侧观察到一个扩散区，其宽度随退火时间的延长而增加。退火处理 3 h 后在靠近界面处观察到一些 *δ* 相，而且随着退火时间的延长，*δ* 相变得更粗、更连续。显微硬度测试表明：尽管通过均匀化退火生成 *δ* 相，界面硬度却有所降低。

关键词：Ag/Al 双金属条；冷焊；退火；多层复合板；形状因子

(Edited by FANG Jing-hua)