Effects of intermediate Ni layer on mechanical properties of Al–Cu layered composites fabricated through cold roll bonding

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Abstract: Layered composites have attracted considerable interest in the recent literature on metal composites. Their mechanical properties depend on the quality of the bonding provided by the intermediate layers. In this study, we analyzed the mechanical properties and bond strengths provided by the nickel layer with respect to its thickness and nature (either powder or coating). The results suggest that bond strength decreases with an increase in the content of nickel powder. At 0.3vol% of nickel coating, we found the nature of nickel to be less efficient in terms of bond strength. A different picture arose when the content of nickel was increased and the bond strength increased in nickel coated samples. In addition, the results demonstrate that mechanical properties such as bend strength are strongly dependent on bond strength.

Keywords: cold roll bonding; Al/Ni/Cu composite; mechanical testing; metallography.

1. Introduction

Metallic multilayer composites have attracted much attention due to the considerable advantages presented by their mechanical, magnetic, and electrical properties [1–2]. Multilayer composites are fabricated using various techniques, including diffusion bonding [3], cold metal transfer welding [4], explosive welding [5], electroplating [6], and roll bonding [7–8]. Of these, the roll bonding technique is most commonly used due to its low cost and overall efficiency. This process is used to manufacture multilayer composites with improved mechanical properties.

Cold roll bonding (CRB) is a pressure-welding or solid-state welding process used to join similar and/or dissimilar metals. CRB is frequently used in the production of layered composites [9–11]. The bonding between two layers is the result of the plastic deformation of both layers. Four theories have been proposed to explain the bonding mechanisms in the CRB process, including the film [12–13], energy barrier [12], diffusion bonding [14], and joint recrystallization [15] theories. Film theory is invoked more often due to the low CRB rolling temperature [7,12–13]. In the CRB process, high interfacial pressure is generated to produce high-quality bonding between layers during rolling. The formation of strong bonding depends on the deformation of the sheets. Fracture of the hard surface layers (oxide, hardened metal or coating layer) on the metal surface during deformation results in the generation of many cracks. Fresh metal extrudes through these cracks due to the roll pressure and surface expansion [16–17].

To better understand the complex nature of the bonding mechanisms, many studies have been conducted to determine the parameters that affect bond strength. Some of these parameters include a reduction in thickness [12–13,18–20], rolling speed [21–22], heat treatment of sheets before or after rolling [23–24], thickness of strips [21], surface condition [25–26], and the addition of particle reinforcement powder between layers [23,27].

Most researchers [23,27–29] have found that the addition of particle reinforcement powder between metallic layers reduces the bond strength. The main reasons for this are reported to be the prevention of the extrusion of virgin metal, reduction in the contact area between the layers, and the increased distance between layers. Although most researchers



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have shown that introducing reinforcement powders between metallic layers reduces bond strength, Lu et al. [30] reported the enhancement of bond strength between aluminum strips with the addition of SiO₂ nanoparticles. Hindrance of the dislocation movement in pile-ups around these particles, breakage of the oxide layers on the strips, and increased atomic diffusion due to the shorter length scales of smaller particles have been reported as the main parameters affecting the bond strength of nano-size SiO₂ reinforced composites [30]. In addition, Yousefi Mehr et al. [31] recently reported that increasing the thickness of the Al₂O₃ oxide layer up to 20 µm between Al-Cu strips enhances the bond strength of the composite layers. The authors noted that wider cracks appeared on the surface as the deformation of the thicker oxide layer became harder. Consequently, a larger volume of virgin metal can be extruded along these wide cracks, thereby creating stronger bonds with the opposing layer [31]. Accordingly, a comparison of the effects of an interlayer with respect to its nature as a powder or hard coating on the mechanical properties of multilayered composites is of interest.

The aim of this work was to evaluate the effects of nickel, in powder or coating form, on the mechanical properties of Al–Cu layers. To this end, we compared the bonding and bend strengths of both specimens (powder and coating).

2. Experimental

2.1. Materials

The materials used in this study were commercially pure Al and Cu strips, whose chemical compositions are presented in Table 1. The strips with the dimensions of 100 mm $\times 25$ mm $\times 1$ mm were cut from primary cold-rolled sheets. To ensure consistent specimen hardness, Al strips were annealed at 663 K for 2 h and Cu strips were annealed at 773 K for 2 h. Prior to the CRB process, Nickel powder with the average particle size of less than 40 µm was manually dispersed between the two strips. The powder specifications

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were determined using an energy dispersive spectroscopy (EDS)-equipped scanning electron microscope (SEM, Seron AIS 2300) operated at 20 kV, and the results are shown in Fig. 1(a). Using the electroplating method, we coated some Cu strips with nickel. This process has been explained in detail in previous studies [1,24]. Electroplating was performed under an applied voltage of 8 V for 15, 30, 60 and 120 min. The thickness of the nickel layer obtained through the electroplating process was determined by SEM cross-sectional analysis. Fig. 1(b) shows the EDS results for the nickel coating. Table 2 shows the volume percentages of nickel used as a powder or coating.

2.2. Sample preparation

To produce acceptable bonding quality via warm roll bonding and CRB, it is essential that the contamination layers on the surface of the two metals be eliminated prior to rolling [1,12,32]. The surface preparation processes we used in this study consisted of degreasing the surfaces in an acetone bath and thoroughly scratch-brushing the strip surfaces using a circular steel brush.

Prior to rolling, the sheets were fixed with a steel wire. To study the effect of the intermediate nickel layer, we coated some copper strips with nickel and added nickel powder between the layers in a number of samples. Then, we rolled the strip samples to achieve thickness reductions ranging from 20% to 80%. In this study, we performed the rolling process in a laboratory rolling mill at room temperature.

2.3. Investigation of mechanical properties

In accordance with the ASTM: D1876-01, the bonding quality of the joined Al–Cu strips was measured using the peel test. In this test, the breaking-off forces were measured as previously described in Ref. [24] and the following equation (Eq. (1)) was used to calculate the average peel strengths (*S*) of the samples [27]:

$$S = \frac{P}{BW}$$
(1)

Material	Chemical composition										
A 1 1	Al / %	Si / ppm	Fe / ppm	Mn / ppm	Ni / ppm	Zn / ppm	Ti / ppm	Sb / ppm	V / ppm	Cu / ppm	Cr / ppm
Allayer	99.713	730	1640	70	17	160	150	14	90	0	0
Material layer					Che	mical compo	osition				
Material layer	Cu / %	Si / ppm	Fe / ppm	Mn / ppm	Cher Ni / ppm	mical compo Zn / ppm	osition Sn / ppm	Pb / ppm	Cr / ppm	Al / ppm	S / ppm

Table 1. Chemical composition of the materials used

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Fig. 1. Specifications of interlayer nickel as powder (a) and coating (b).

Table 2.	Amounts of	nickel	used in	cold r	oll bondin	g
Table 2.	Amounts of	IIICKCI	uscu m	colu l	on bonum	ĸ

Mass of nickel powder / g	Thickness of nickel coating / μm	Volume percentage of nickel between layers / %
0.11	5	0.3
0.22	10	0.7
0.45	20	1.3
0.67	30	2.0

where *P* is the average load in N and BW is the bond width in mm. Peel tests were performed by a Hounsfield H50KS tensile testing machine using a crosshead speed of 20 mm/min. The fracture surfaces of the samples were examined through optical microscopy (OM) and scanning electron microscopy (SEM).

The mechanical properties of different samples were investigated using the three-point bending test. Eq. (2) was used to calculate bend strengths of samples [33];

$$\sigma = \frac{6FL}{4WT^2} \tag{2}$$

where σ is the bend strength of samples, *F* is the bending load, *L* is the distance between the two supports, and *W* and *T* are the sample's width and thickness, respectively. All mechanical

tests were performed three times at room temperature.

3. Results and discussion

3.1. Presence of nickel between layers and reduction in thickness

Fig. 2 shows the effect of thickness reduction on the average peel strength of Al–Cu strips with 0.7vol% of Ni powder and coating, respectively, as compared with nickel-free samples. We can see two different phenomena in this figure. First, there is enhancement of the bond strength via further reduction in thickness and, second, there is a decrease in the bond strength due to the presence of the nickel interlayer.



Fig. 2. Variation of average peel strength of the samples for different thickness-reduction percentages.

Fig. 2 shows that the average peel strength of samples both with and without nickel increased more than 10 times after 70% thickness reduction. Generally, in CRB, bonding quality improves with a greater reduction in thickness [10,18–21,23–24]. According to film theory [12–13], the coherent fracture of the brittle surface layer of metals during rolling leads to the formation of numerous cracks perpendicular to the rolling

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direction. The formation of metallic bonds during rolling occurs due to the extrusion of virgin metal through these cracks.

To better understand the role of thickness reduction in the presence of 0.7vol% nickel powder, Fig. 3 shows the results of an SEM analysis of the fractured surface of copper. We can see that the bonded area stretched with a greater reduction in thickness and, in fact, the greater the degree of thickness reduction, the larger are the sizes of the cracks on the surface. Therefore, by further reducing the thickness, the average peel strength is enhanced due to the formation and growth of cracks. This means that more areas suitable for bonding are made available due to the extrusion of more virgin metal, as shown in Fig. 3. Moreover, further reducing the total thickness leads to the formation of stronger bonding, as more atoms obtain the activation energy necessary for bond formation [7,9,13,23,4,34]. In addition, reducing the distance between layers and increasing the width of cracks (see Fig. 3) lead to improved bonding quality due to the reduction in thickness. These findings are consistent with those of other researchers [2,7–12,19,23–24].



Fig. 3. SEM micrographs of copper peeled surfaces in the presence of 0.7vol% nickel powder: (a) 40% thickness reduction; (b) 50% thickness reduction; (c) 60% thickness reduction; (d) bonding area after 60% thickness reduction with higher magnification.

Furthermore, as indicated in Fig. 2, the presence of 0.7vol% of an intermediate nickel layer as powder or coating for various reductions in thickness reduces the bonding quality of the Al–Cu strips. This may be due to either an increase in the distance between the layers due to the presence of Ni, a reduction in the bond surface due to the presence of Ni, or a decrease in the friction coefficient between the layers [24].

Fig. 4 shows the peeled surfaces of samples after a 60% reduction in thickness in the presence of intermediate nickel layers. We can see the formation of cracks and the extrusion of virgin metal through the cracks as dark traces perpendi-



We propose that the quality of bonding between layers can be evaluated using the bonding area ratio, which is the ratio of the bonded surface to the peeled surface. We measured these quantities using ImageJ software and Fig. 5 shows this ratio for samples in the presence of 0.7vol% nickel and free-nickel samples after a 60% reduction in thickness. This ratio decreased 21% and 32%, respectively, with the addition of nickel powder and coating interlayers, compared to the Al–Cu multilayer. Therefore, we can conclude that the number of suitable regions for bonding had decreased due to the presence of the nickel interlayer, which led to a decrease in the bond strength, as shown in Fig. 4.

In addition, the samples with Ni powder showed greater enhancement in bond strength as a result of the reduction in total thickness (Fig. 2). This could be attributed to the following factors: cular to the rolling direction in the fracture surface of the free-nickel sample (Fig. 4(a)). The extrusion of virgin copper metal through these cracks led to the formation of a strong bond between the layers. As we can see in this figure, the bonding area between the Al and Cu strips decreased due to the presence of the intermediate nickel layer, as either powder or coating, between the Al–Cu layers. We can also see that cracks have formed on the surfaces, but the number of cracks has decreased considerably. Therefore, because of the extrusion of a lesser amount of virgin metal through the cracks in the presence of nickel, weaker bonding has resulted between the layers.



Fig. 4. Peeled surfaces of copper strips after the peeling test for samples with 60% thickness reduction: (a) without nickel; (b) with 0.7vol% nickel powder; (c) with 0.7vol% nickel coating.



Fig. 5. Bonding area ratio for samples with and without nickel after 60% thickness reduction.

(a) Ni coating covers the Cu surface more uniformly, which leads to a reduction in areas that are bond-

ing-appropriate on the coating. Therefore, the bonding area ratio decreases in the Ni-coated samples (see Fig. 5), which leads to lower bond strength in these samples.

(b) The friction coefficient between layers is an effective parameter in CRB [24–25]. The bonding quality of the Ni-coated samples decreases due to the smooth surface of the Ni coating and the lower friction coefficient of the Al layer.

3.2. Effect of increase in interlayered nickel content

Fig. 6 shows that increasing the content of nickel between the Al-Cu strips has two different effects on the bond strength of the layers, depending on whether the nickel layer is in the form of powder or coating. The average peel strength of the strips increases from 4 to 10 N/mm when the content of coated nickel increases from 0.3vol% to 2.0vol%. In contrast, the average peel strength of the strips decreases from 8.4 to 3.6 N/mm when the content of nickel powder increases from 0.3vol% to 2.0vol%. To gain insight into this unexpected behavior, we used OM to examine the fractured surfaces of different samples. Fig. 7 shows the fractured surfaces of samples with different nickel contents and a 60% thickness reduction. An excessive number of cracks have formed on the surface layer for specimens rolled with 0.3vol% of nickel powder, which contrasts with the results observed in the 0.3vol% nickel-coated samples (see Figs.

7(a) and 7(c)). This indicates that more virgin-metal surfaces were in contact, thereby increasing the number of sites available for interlayer bonding. In agreement with this microstructural observation, we found the quality of the bonding between layers to be enhanced. In addition, we evaluated the bonding area ratio for these samples and the results are presented in Fig. 8. As we can see in the figure, in the presence of 0.3vol% of nickel, the powdered sample has a higher bonding area ratio. This means that more virgin-metal surfaces were in contact and more metal bonding occurred in the presence of nickel powder compared to nickel coating.



Fig. 6. Variation of average peel strength of the samples with different contents of nickel.



Fig. 7. Copper peeled surfaces for samples with 60% thickness reduction: (a) 0.3vol% nickel powder; (b) 2.0vol% nickel powder; (c) 0.3vol% nickel coating; (d) 2.0vol% nickel coating.



Fig. 8. Variation of bonding area ratio with nickel content.

Fig. 6 also shows that an increase in the content of interlayer nickel has different outcomes depending on the nature of the nickel layer (coated or powdered). Increasing the amount of interlayer nickel powder decreases the bond strength and, as explained above, increasing the amount of nickel coating enhances the bond strength of the layers. As previously reported [23,27-29], in CRB, the quality of bonding decreases when the intermediate powder layer is enhanced. Using a greater amount of powder at the interfaces increases the thickness of the powder layer and, consequently, the bonding distance between layers. Therefore, a greater degree of deformation is required to bond the layers. As a result, increasing the amount of powder reduces the bond strength. Since the powder layer covers the surface, the number of surface cracks in contact with the Al layer decreases significantly (see Fig. 7(b)). Consequently, less virgin metal can extrude and reach the surface of the layers, thereby decreasing the bond strength. Fig. 9 shows SEM micrographs of the samples' fracture surfaces after 60%

thickness reduction for different powder contents, in which traces on the surfaces reveal the bonding area. We can see that in the sample without nickel, the bonding line covers almost the entire surface, which is conducive to the formation of strong bonding. We can see a reduction in bonding quality when the amount of powder was increased to 0.7vol% and 2.0vol% (see Figs. 7 and 9). However, Lu et al. [30] reported different results for the addition of interlayered nanoparticles. The authors used SiO₂ nanoparticles to enhance bond strength and explained their finding based on the following hypotheses. First, shear deformation occurs along the contact interfaces and nano-sized particles hinder the movement of the dislocations, which leads to a pile-up of dislocations near the particles. Therefore, the interfaces harden locally and the bond strength increases. Second, since the embedded nanoparticles have greater hardness than the metal matrix, the hardened surface layer breaks due to the presence of these particles. The particles cause fresh surfaces to be exposed, and when there is insufficient time to reform an oxide layer, fresh new sites are generated for more efficient bonding. Third, due to atomic diffusion, the bonds between the reinforcement and the matrix are related to the maximum static friction. Smaller atomic particles more effectively diffuse from the particle to the strips due to the shorter diffusion paths. This improves the bonding quality, and in turn, increases the bond strength [30]. Nevertheless, as shown in Fig. 8, by increasing the content of nickel powder to 2.0vol%, the bonding area ratio decreased more than 40%. Therefore, as explained above, the reduction in the bonding area on the contact surfaces is due to the presence of nickel powder (see Figs. 7 and 9).



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In Fig. 6, unlike the behavior of nickel powder, a thicker nickel coating yields a greater increase in the bonding quality. We found bond strength to significantly improve with high contents of nickel (more than 2.0vol% or more than 20 μ m coating). We observed stronger bonds in the samples with more than 20 μ m of nickel coating compared to the samples without any nickel coating. Fig. 8 shows the variation in the bonding area ratio with increased nickel-coating thickness and it is evident that the enhancement of nickel coating from 0.3vol% to 2.0vol% increases this ratio, which leads to higher bond strength.

In Fig.7(c), the coating with a thickness of less than 20 μ m was easily deformed at a constant pressure, which led to the formation of many surface cracks and the extrusion of fresh virgin metal from these cracks. The bonding quality is not high due to the small crack size. In the samples with coatings thicker than 20 μ m (Fig. 7(d)), deformation of the intermediate coating layer requires higher stresses and yields very few nucleated cracks on the surface. Nevertheless, the roller pressure leads to crack propagation, as shown in the notable enhancement of the crack widths in Fig. 7(d).

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With respect to the peeling test mechanisms, we theorized that in samples with almost the same bonding area ratio, crack width will critically affect the bond strength and control the bonding quality. Fig. 10 shows a schematic illustration of the peeling test after cold rolling, in which the hard surface layer (here the coating layer) is broken during rolling, and the extrusion of virgin metal through these cracks leads to the formation of metal bonding. Fig. 10 shows that, during the peeling test, the maximum force is applied to only a small portion of the bonded surface. In fact, during peeling, certain bonded areas (cracks) tolerate the applied force. This means that the width of the bonded area is an important parameter affecting the bond strength. Therefore, the simultaneous significant enhancement in crack widths (see Fig. 7(d)) and the bonding area ratio (see Fig. 8) from the increased nickel-coating thickness is the main cause of increased bond strength. Consequently, we can say that the nickel-coating layer and SiO₂ nanoparticles induce similar effects on bond strength, but their explanations involve the effect of different microstructural mechanisms on the observed behavior.



Fig. 10. Schematic illustration of the peeling test.

In brief, we can say that the bonding sites in the nickel-coated samples increased with an increase in nickel content and the reverse occurred in the nickel-powder samples. The nature of the nickel layer (powder or coating) is the key factor determining the bond strength of these composites.

Fig. 11 shows the effect of a nickel interlayer on the bend strength of the Al–Cu composite. We can see that variation in the bend strength of the composite due to increases in nickel content is similar to the variation in bonding quality between Al–Cu strips (see Fig. 6). It has been reported that the bonding quality of layers directly affects the bend strength in multilayer composites [34].



Fig. 11. Bend strength variation versus nickel content.

In the bending mechanism of layered composites, the layers experience different amounts of tension, so local sliding of the layers occurs in the contact surface, which leads to their separation and a reduction in the bend strength. Consequently, an appropriate degree of bonding prevents lamination of the layers, so composites can withstand higher loads and have higher bend strengths. As shown in Fig. 11, with the addition of 0.3vol% nickel between the Al–Cu layers in powder and coating forms, the bend strength decreased from 498 MPa to 490 and 390 MPa, respectively. In addition, increasing the content of nickel in the powder and coating forms had different effects on bend strength. An increase in nickel powder from 0.3vol% to 2.0vol% led to a reduction in the bend strength (to approximately 460 MPa).

In contrast, the bend strength of the strips increased to 505 MPa when the amount of nickel coating was increased to 2.0vol%.

Fig. 12 shows cross sections of as-rolled and bending specimens, in which we can see that bending resulted in the separation of the cold-rolled layers. However, this separation did not occur in the same way in all samples. As shown by the arrows in Fig. 12(e), with a 0.3vol% nickel coating between the layers, separation occurred during the bending test. Figs. 12(e)–12(h) show that with the enhancement of the nickel coating, separation between the Al–Cu layers decreased during the bending test. Consequently, the bend strength of the composite improved due to the enhancement of the layer bonding quality.



Fig. 12. Cross sections of as-rolled specimens with 0.3vol% (a), 0.7vol% (b), 1.3vol% (c), and 2.0vol% (d) of nickel coating and bending specimens with 0.3vol% (e), 0.7vol% (f), 1.3vol% (g), and 2.0vol% (h) of nickel coating and 0.3vol% (i), 0.7vol% (j), 1.3vol% (k), and 2.0vol% (l) of nickel powder.

Conversely, in the nickel-powder samples, we can see that increases in the content of nickel powder causes delamination to occur between the Al–Cu layers (arrows in Figs. 12(k) and 12(l)), which led to the reduction in the mechanical properties (i.e., bond strength and bend strength).

4. Conclusions

In this study, we prepared Al/Ni/Cu layered composites and tested the strength of these composites based on the content of nickel and the nature of the nickel layer (either powder or coating of copper strips). We can summarize our study results as follows:

(1) Bond strength increased more effectively in the nickel-powder composites via thickness reduction due to the enhancement of the bonding area ratio.

(2) With 0.3vol% nickel between the Al–Cu layers, the bond strength was reduced. However, increasing the content of nickel gave rise to quite different responses, depending on the nature of the nickel layer (powder or coating).

(3) The bonding quality of the layers had a direct effect on mechanical properties such as bend strength. The enhancement of the bond strength led to an increase in the bend strength of the samples.

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