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Shielding gas effect to diffusion activities of magnesium and copper on aluminum clad

Charles SP Manurung*, Richard AM Napitupulu
Department of Mechanical Engineering, Nommensen HKBP University, Medan 20234, Indonesia
* charles.manurung1972@gmail.com

Abstract. Aluminum is the second most metal used in many application, because of its corrosion resistance. The Aluminum will be damaged in over time if it’s not maintained in good condition. That is important to give protection to the Aluminums surface. Cladding process is one of surface protection methods, especially for metals. Aluminum clad copper (Al/Cu) or copper clad aluminum (Cu/Al) composite metals have been widely used for many years. These mature protection method and well tested clad metal systems are used industrially in a variety application. The inherent properties and behavior of both copper and aluminum combine to provide unique performance advantages. In this paper Aluminum 2024 series will be covered with Aluminum 1100 series by hot rolling process. Observations will focus on diffusion activities of Mg and Cu that not present on Aluminum 1100 series. The differences of clad material samples is the use of shielding gas during heating before hot rolling process. The metallurgical characteristics will be examined by using optical microscopy. Transition zone from the interface cannot be observed but from Energy Dispersive Spectrometry it’s found that Mg and Cu are diffused from base metal (Al 2024) to the clad metal (Al 1100). Hardness test proved that base metals hardness to interface was decrease.

1. Introduction
Aluminum is a material that is widely used in various fields. Starting from household appliances, construction, electricity, aviation to reflector equipment on military vehicles. Aluminum has a Face Center Cubic (FCC) crystal structure. Aluminum is known as a light metal with 2.7 g/cm³ of density compared to steel with 7.9 g/cm³ of density[2]. Aluminum has good electrical and heat conductivity as well as corrosion resistance in the general environment with melting point is 660°C (1220 °F). Most of the Aluminum alloys can easily be formed because they have high ductility. Aluminum remains ductile even at low temperature. Therefore Aluminum is also used as a air condition elements on vehicles. Although Aluminum has good corrosion resistance, but basically Aluminum can’t be detached from the oxidation process, especially when Aluminum is in an environment containing a lot of oxygen, water or other oxides. At room temperature, the thickness of oxide layer can reach 2.5 ~ 3 nm (25 ~ 30Å). But the oxide layer that is formed is passive and does not result in great damage to the surface so it can be a protector of Aluminum from environment.

Aluminum alloy is very widely used because of its superior mechanical properties is Al-Cu alloy. The strength of this alloy is obtained through a solution heat treatment process at eutectic temperature. With that solution heat treatment, the α phase formed and distributed
throughout the Al-Cu. The $\theta$ phase is a CuAl$_2$ compound that formed as precipitate which can serve as a barrier to the dislocation movement.

![Figure 1](image1.png)

**Figure 1** Solution heat treatment process for Al-Cu alloys

![Figure 2](image2.png)

**Figure 2** Part of Al-Cu phase diagram

Al-Cu alloys have good strength but low corrosion resistance due to galvanic phenomenon on Aluminum and on the Al-Cu due to potential difference of two types of metal. Overcome that problem Aluminum alloy need to be coating with a better metal such as pure Aluminum. Metal coating is performed to provide corrosion resistance to metals, obtaining heat and electric conductivity properties and to improve the appearance of a material.

The problem are often encountered in the coating process is the adhesion between coating materials with base metal. In metallic materials, the quality of the coating can be assessed by the bonding quality between two metals, the base metal and the coating material. The type of bond that is formed also depends on the coating technique. In this research, the coating is done by cladding method using hot rolling process. This process is most widely used given the high production amount especially when using continuous rolling.

At high temperatures, aluminum is highly vulnerable to oxidation, so to produce a good coating quality the surface should be avoided from the influence of oxygen around it. This is
to prevent the formation of oxide layer on the surface which will inhibit the diffusion process between atoms from metal coatings with base metal.

2. Materials and Methode
In this research, we use Aluminum alloy from 2024 series (Al-Cu) as base metal, and Aluminum 1100 series as clad metal. Cladding material will be form by hot rolling process. Emission Spectrometry is used to get the information of alloying element on both materials as shown on table 1.

| Comp. (%wt) | Al  | Cu  | Fe | Si | Mn | Mg | Cr | Zn | Ti | others |
|-------------|-----|-----|----|----|----|----|----|----|----|--------|
| Al-Cu 2024  | 93.59 | 4.2 | 0.08 | 0.05 | 0.53 | 1.4 | 0.05 | 0.03 | - | 0.07 |
| Al 1100     | 99.17 | -   | 0.4 | -  | 0.00019 | -  | 0.0002 | 0.0166 | 0.0403 | -     |

**Figure 3** Dimension and rolling scheme

Surface of both materials used should be prepared to obtain good bonding qualities in the interface. This research was conduct from sample preparation to element examined distributing.

1. Sample Preparation.
The sample preparation begins from the material cutting and then the surface preparation to produce a good contact area between the two materials to be cladded. We use abrasive paper with. We use abrasive paper from the level of roughness 800, 1000, 1500 up to 2000, followed by a polishing process using an abrasive material with a roughness of 0.3μm. Sample surface cleaned by pickling process by using HNO₃ to remove oxide layer on the
both of samples contact area. After the pikling process, the samples are stored in a special packaging to prevent the formation of oxide layer before the rolling process is performed.

2. Hot Rolling Process
Hot rolling was done by a reduction of 5% by 9 pass. The heating process is divided into 2 variables by using protective gas and without protective gas. The protective gas used is a pure Argon gas with a flow rate of 2 liters / minute. The sample heating temperature is 450°C and 500°C and after each rolling each sample fitting is reheated for 10 minutes at predetermined temperature.

3. Metallographic test
Metallographic testing is performed to observe the interface area of both materials. The etching solution used was the modification of Keller solution with a composition of 10ml HNO3 + 1.5 ml HCL + 1.0 ml HF + 87.5 ml Aquadest.

4. Emission Dispersive Spectrometry Test (EDS Test)
The EDS test is performed to find out where the presence of atoms diffuses during the heating and rolling process. This test is aimed at the Mg and Cu atoms that are clearly not found in the clad material of Al 1100. The method used is line analysis method ranging from 600μm from the base metal interface (Al-Cu 2024) to 300μm from the interface to the clad metal (Al 1100). The test is continued by the spot area method to calibrate the elements obtained from the line analysis method.

5. Hardness test
Hardness testing is done from base metal area until clad metal passes through interface area. This test is performed to determine the change in the distribution of violence due to the displacement of atoms due to diffusion.

3. Result and Discussion
Based on the results of metallographic testing on the interface area can be seen differences in the thickness of each sample as shown in the following table:

| Sample | T (°C) | 450°C | 500°C |
|--------|--------|-------|-------|
| Shielding gas | Yes | No | Yes | No |
| Speciment Number | 1 | 2 | 3 | 4 |
| Thickness (μm) | 1.06 ± 0.34 | 2.06 ± 0.61 | 0.95 ± 0.21 | 1.59 ± 0.56 |
From the observations it can be concluded that the heating conditions without the use of protective gas resulted in the presence of oxide layers that block the unification process of both types of materials. This can be seen both at the heating temperature of 450°C and 500°C. In the heated sample using protective gas, it appears that the interface area has a better transition region.

**Figure 4.** Microstructure of cladding material sample

**Figure 5.** Part of EDS & EDAX examination

- a. line analysis method
- b. selected area to calibrate the elements
EDS tests shown the distribution of Cu and MG elements that diffuse as shown in the following table.

**Table 3.** The distribution of Cu and Mg elements in the observation area

| Temperature (°C) | Shielding gas | Element (%) | Distance from Interface | To base metal | To clad metal |
|------------------|---------------|-------------|-------------------------|---------------|--------------|
|                  |               |             | 600 μm | 300 μm | 150 μm | interface | 150 μm | 300 μm |
| 450              | Yes           | Cu          | 6.64  | 5.92  | 4.49  | 2.61     | 2.30   | 2.27   |
|                  |               | Mg          | 3.51  | 3.38  | 2.76  | 2.10     | 2.05   | 1.96   |
| 450              | No            | Cu          | 5.60  | 5.13  | 4.79  | 3.80     | 1.91   | 1.78   |
|                  |               | Mg          | 3.33  | 3.20  | 3.15  | 2.67     | 1.95   | 1.93   |
| 500              | Yes           | Cu          | 6.62  | 5.99  | 5.34  | 2.87     | 2.27   | 0.26   |
|                  |               | Mg          | 3.53  | 3.30  | 3.02  | 2.02     | 1.98   | 1.88   |
| 500              | No            | Cu          | 6.68  | 5.53  | 3.76  | 3.63     | 2.16   | 0.00   |
|                  |               | Mg          | 3.47  | 3.26  | 2.56  | 2.41     | 1.97   | 1.45   |

Table 3 shown that use of protective gas at the time of heating can reduce the formation of oxide layer on the surface. Thus the diffusion process is not obstructed, seen from the amount of Cu and Mg elements diffusing towards Al 1100 which previously did not contain elements Cu and Mg.

In samples that do not use protective gas during heating there is an increase in Cu and Mg elements in the interface area. It can be concluded that there is an obstruction of oxide to the diffusion process. Testing Hardness in the transverse direction from base metal to clad metal can be seen in the following table.

**Table 4.** Distribution of hardness throughout the observation area

| Temperature (°C) | Shielding gas | Vickers Hardness Number (VHN) |
|------------------|---------------|-------------------------------|
|                  |               | Distance from Interface       |
|                  |               | Base Metal | Clad Metal |
|                  |               | 2 mm | 300μm | 200 μm | 100 μm | 100 μm | 2 mm | 300μm | 200 μm | 100 μm | 100 μm |
| 450              | Yes           | 100 ± 2.9 | 97 ± 6.2 | 93 ± 4.8 | 85 ± 10.4 | 28 ± 2.3 | |
| 450              | No            | 139 ± 15.4 | 135 ± 117 | 122 ± 15 | 114 ± 6.0 | 39 ± 1.4 | |
| 500              | Yes           | 96 ± 1.7 | 93 ± 3.6 | 92 ± 3.1 | 86 ± 4.6 | 25 ± 1.9 | |
| 500              | No            | 97 ± 1.8 | 92 ± 2.2 | 84 ± 2.5 | 84 ± 2.4 | 31 ± 2.7 | |

Hardness test result show a decrease in hardness in the base metal area leading to the interface. This is due to the decrease in metal properties due to reduced alloying elements in the area because it has diffused into clad metal.

**4. Conclusion**

1. The use of protective gas when heating the material sample can suppress the formation of the oxide layer in the interface area. This is evidenced by the diffusion activity of Cu and Mg atoms. In Table 3 for heated samples 450°C and 500°C without the use of protective
gases it appears that the activation of Cu and Mg atoms is retained in the interface area. While the sample using protective gas seen diffusion of Cu and Mg atoms more to the clad metal area (Al 1100).

2. The thickness of the interface area is affected by the use of protective gases. This is due to the number of oxide layers on the surface of the interfaces being formed more frequently during heating. The heated sample without the use of a protective gas has a thicker interface area.

3. Base metal hardness in the interface direction decreases due to atom diffusion from base metal to clad metal.

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