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Resistivity and Formation of Intermetallic Layer in Aluminum-Steel Clad Strip

B Křivská¹, M Šlapáková¹, R Králík¹, L Bajtošová¹, M Cieslar¹, O Grydin², M Stolbchenko², M Schaper²

¹Charles University, Faculty of Mathematics and Physics, Ke Karlovu 5, 121 16 Prague, Czech Republic

²Paderborn University, Chair of Materials Science, Warburger Str. 100, 33098 Paderborn, Germany

E-mail: krivska.barbora@seznam.cz

Abstract. Cladding of two dissimilar materials such as aluminum and austenitic steel can bring outstanding material characteristics beyond the ones of each conventional monomaterials. The study focused on development of an Al-Fe-rich intermetallic phase formed on the aluminum/steel interface during heat treatment. Electrical resistivity measurements were performed to monitor diffusion processes and phase transformations during isochronal and isothermal annealing. Resistivity measurement results were supplemented by microscopic methods revealing the interfacial microstructure.

1 Introduction

Al-steel clad sheets manufacturing enables to get a material combining steel's high strength with a low weight of aluminum, making this material very attractive for structural applications in shipbuilding, aircraft constructions or car bodies [1,2].

Clad materials represent joints of two or more layers of different metals. They can be produced in various forms e.g. strips, foils, tubes, rods or wires. Nowadays, more than 90 % of the clad strips production is comprised by a cold or hot roll bonding process [3]. However, these methods suffer from severe drawbacks resulting in a high cost of the final product. The issue is that the substrate's preparation consists of several steps, including multi-pass hot rolling and complicated surface pretreatments.

Twin-roll casting used as a cladding process [4,5] is a novel method, which poses a solution overcoming such drawbacks since clad strips are prepared directly from a melt of one metal and a solid sheet of the other one that are fed simultaneously between the rolls of a twin-roll caster. Demands on the surface preparation are then partially relinquished.

The clad material could be subjected to a thermal exposure during manufacturing of a final product, which could activate diffusion between the metals leading to undesirable formation of brittle intermetallic compounds (IMC), influencing the final mechanical properties of the clad material [6,7]. Akramifard et al. [8] investigated microstructure and mechanical properties of a three-layered composite AA1050/304L/AA1050 prepared by hot rolling. During a subsequent heat treatment, growth of an IMC layer was observed deteriorating the joint's quality. Cracks propagated along the Al-IMC interface and

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caused an even-by-hand delamination of the bond. Al_5Fe_2 and $Al_{13}Fe_4$ phases were found as the main constituents of the IMC layer formed on the interface [8–11].

The aim of this paper is to investigate the evolution of the interface between Al and steel in twin-roll cast Al-steel clad sheet at elevated temperatures using resistivity measurements and observation of the resulting structure by light optical microscopy (LOM) and scanning electron microscopy (SEM).

2 Experimental

2.1 Material

A melt of technically pure aluminum EN AW-1070 was clad on a solid strip of austenitic steel type 1.4301. The casting process was realized by a laboratory twin-roll caster equipped with two watercooled rolls with a diameter of 370 mm and a length of 200 mm [12]. A vertical operation plane was chosen. No mold release coating was applied on the rolls' surface either before or during the casting process. The final thickness of the composite strip was 2.5 mm with the aluminum-to-steel thickness layer ratio of 4:1.

Since a subsequent heat treatment could influence the bond strength between the steel and aluminum layer, isochronal and isothermal annealing were performed using a laboratory air furnace. The isochronal step-by-step annealing scheme with step 50 °C / 25 min was applied with a water-quenching between every two steps. The isothermal annealing was done at 500 °C with a logarithmically changing time-step length.

2.2 Methods

Electrical resistivity measurements (four-point probe at the temperature of liquid nitrogen) were carried out after each annealing step and solid solution decomposition and phase transformations were investigated. Light optical microscopy (LOM, Zeiss Axio Observer metallographic microscope) and scanning electron microscopy (SEM, Quanta 200F) observations in backscattered electrons (BSE) were used for the characterization of microstructural changes.

3 Results and discussion

3.1 Initial state

Both LOM and SEM show colonies of primary phases arranged in interdendritic chains obliquely orientated in the casting direction of both outer sides of the aluminum layer (figure 1, 2a).



Figure 1. Light optical micrograph of a) Al layer surface without steel coating and b) Al/steel interface.

A slight difference in the phase distribution on the opposite sides of the aluminum layer – with and without the adjacent steel substrate – could be noticed due to different cooling rates caused by the added steel sheet [13,14]. The steel substrate acts as an additional heat barrier so the aluminum solidifies from

that side at a considerably slower rate and a position of the kissing point [13] is shifted towards the steel layer.

The presence of the IMC layer at the Al-steel interface in the as-cast state was not confirmed by LOM or SEM (figure 1b, 2b). The observation is in contradiction with results given in [2,15], where a formation of the IMC during the casting was reported. It should be noted that clad strip casting conditions were different for the compositions used in our study and in the referenced works. In work of Grydin[2], a thicker Al-layer, a thinner steel layer and a higher Al pouring temperature were used. In work of Stolbchenko [15], the same clad parameters were used (Al layer 2.0 mm, steel layer 0.5 mm) but with higher casting temperature and casting rate. Thus, a higher strip outlet temperature could result in the IMC growth without any additional annealing.



Figure 2. BSE images a) of aluminum and b) interface region.

3.2 Annealing

Figure 3a shows electrical resistivity annealing curves of the composite and separate Al and steel layers during isochronal annealing. A pronounced resistivity drop was measured in the composite and Al sample between 200 °C and 450 °C. Since very similar shapes of the resistivity curves were observed for Al and the composite sample, phase transformation ongoing in Al layer has a controlling role in the resistivity curves evolution. The decrease of resistivity is connected with a higher purity of the matrix and a depletion of the solid solution. Contrarily, a backward increase above 450 °C could be a sign of particle dissolution.



Figure 3. a) Relative changes of electrical resistivity during isochronal annealing, b) resistivity annealing spectra calculated from resistivity data.

The calculated negative differential curves $-(d\rho/dT)/\rho_0$, referred to as resistivity annealing spectra, exhibit a pronounced peak at 400 °C for both aluminum and composite samples (figure 3b).

Since the derivative directly corresponds with an intensity of resistivity changes, peaks in positive values are related to precipitation, coarsening of the precipitates and solid solution depletion. In contrast, negative values measured above 450 °C are connected with solid solution enrichment and dissolution of the particles. A small peak present below 250 °C in Al sample is often observed in Al alloys and it is related to a redistribution of Si atoms [16]. The effect is very weak and it can be overlapped by other transformations ongoing at higher temperatures. Partial suppression of the peak in the composite could be the result of a different heat loss due to the added steel layer.

Noticeable coarsening of existing particles corresponding with the peak in resistivity annealing spectra is confirmed between 350 °C and 450 °C by LOM micrographs (figures 4a, b, c). Above 450 °C, predicted particle dissolution was also observed (figure 4d). The steel layer remained unchanged during all annealing experiments and the resistivity of the steel sample is rather constant during the whole annealing process.



Figure 4. LOM images of the particles distribution in Al during isochronal annealing up to: a) 200 °C, b) 350 °C, c) 400 °C, d) 500 °C.

The development of the interface region was studied by SEM (figure 5). Growth of an IMC layer at the Al-steel interface was observed above 500 °C. Its influence on the resistivity curves remains rather disputable. However, the slope of the resistivity annealing spectra above 450 °C was slightly steeper in the case of the composite sample (figure 3b).

Considering the convincing development of the interface region above 500 °C, the isothermal annealing was performed at 500 °C. The resistivity curves are shown in the figure 6a. A similar evolution of the Al and composite resistivity was obtained, indicating the main influence of the phase transformations in the Al layer. In contrast to the isochronal annealing, the resistivity curves of the Al and composite samples during isothermal annealing do not contain the backward increase suggesting that 500 °C annealing leads only to a depletion of the matrix and coarsening of existing particles. However, a larger drop of resistivity observed in the clad material indicates an influence of formation

of an IMC layer at the Al-steel interface. Similarly, as observed by several researches [9–11], the interfacial phase layer grows according to the parabolic law (figure 6b and figure 7).



Figure 5. BSE images of the interfacial phase evolution during isochronal annealing up to: a) 400 °C, b) 500 °C, c) 550 °C.



Figure 6. a) Relative changes of electrical resistivity, b) time evolution of the IMC layer thickness d during isothermal annealing.

Formation of the thick IMC layer could result in formation of cracks between Al and IMC and deterioration of the bond [8,15]. Isochronal annealing up to 600 °C leads to full delamination of the aluminum layer from the steel sheet. Isothermal annealing at 500 °C for 32 h results only in crack formation in Al along the IMC layer (figure 7c). EDX analysis confirms formation of Al-Fe rich phases [17] observed in Al-steel clad sheets [8–11].



Figure 7. BSE images of the interfacial region after annealing at 500 °C for: a) 8 h, b) 16 h, c) 32 h.

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4 Summary

Evolution of microstructure, resistivity and IMC layer in twin-roll cast Al-steel clad material were studied during annealing. Significant coarsening of precipitates was observed in the Al strip below 450 °C during isochronal annealing. Corresponding maximum in the resistivity spectrum was detected at 400 °C. Partial dissolution of coarser particles occurs above 450 °C, resulting in presence of negative values in the resistivity spectrum. A similar evolution of resistivity was also observed in the composite material. The influence of the IMC layer growth on the resistivity of the clad is rather small, however, evinced by larger decrease of the composite resistivity at 500 °C. The interfacial IMC phases grow parabolically with time.

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