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Establishing a correlation between interfacial microstructures and corrosion initiation sites in Al/Cu joints by SEM–EDS and AFM–SKPFM

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ABSTRACT

A lap joint of AA3003 and pure copper was produced by friction stir welding and the induced interfaces were investigated. Interfacial regions were characterized by SEM–EDS, AFM, SKPFM, OM and Vickers micro-hardness. Multimodal Gaussian distribution (for characterization of surface potential patterns) showed the formation of multiple compounds. A quantitative correlation between microstructure constituents and Volta potential distribution was recognized and confirmed by corrosion attacked sites observations. It was observed that the Al-rich regions proximate the dispersed Cu particles and Cu–Al intermetallics were most susceptible to corrosion attack initiation due to a galvanic driving force between these surface constituents.

1. Introduction

Aluminum/copper bimetals are usually considered of high interest to electrical, aerospace, shipbuilding, and other industrial applications [1,2]. As the fusion welding of this dissimilar couple is difficult due to the formation of brittle intermetallic compounds (IMCs), friction stir welding (FSW) is considered to be an alternate process [3–11]. An investigation by Elrefaey et al. [12] on FS welded Al/Cu lap joints of AA1100-H24 to copper with a double-shoulder design showed different zones in the weld including stirred zone (SZ) and heat affected zone (HAZ); without any thermo-mechanically affected zone (TMAZ). Another study by Saeid et al. [8], demonstrated the formation of HAZ and TMAZ affected by the formation of IMCs and micro-cracks as a result of heat-input at various welding speeds. Similar studies revealed different IMCs of CuAl, $CuAl_2$, $CuAl_4$, and Cu_9Al_4 with a brittle nature close to the Al/Cu interface [6,7,9,12,13]. Xue et al. [9] showed that an excellent metallurgical bonding between aluminum and copper at the Al/Cu interface may be achieved because of the formation of a continuous and uniform Al–Cu intermetallic layer with a proper thickness of about 1 μm. It is also reported that, severe plastic deformation and thermal exposure facilitate the formation of IMCs [7,10,11]. A survey by Galvão et al. [10] also represented the formation of mixed regions of aluminum, copper, $CuAl_2$ and Cu_9Al_4 with heterogeneous structures and intermetallic content along the Al/Cu interface. Other studies [14,15] report that, during welding, copper segregates to grain boundaries and causes the $CuAl_2$ IMC to form through the eutectic reaction of: liquid → $CuAl_2$ (or θ phase) + α (Al-rich solid solution). As these Cu-rich IMCs act as micro-cathodes, the surrounding α-phase dendrites become susceptible to corrosion. Transmission electron microscopy (TEM) observation by Feng et al. [16] in a friction stir processed AA2219-T6 alloy attributed the presence of equilibrium θ to the followings:

(a) Dwelling at above 480 °C leads the semi-coherent θ′ precipitates and plate-shaped coherent θ″ metastable precipitates overage to the equilibrium θ phase.
(b) Having heated above the solvus temperature (513 °C), the metastable precipitates dissolve into the matrix and reprecipitate as equilibrium θ phase upon slow cooling rate behind the tool.

However, understanding the material affinity to corrosion and characterization of such an interface would be easier by employing multiple complementary techniques. Recently, scanning Kelvin probe force microscopy (SKPFM) is developed for concurrent mapping of topography and Volta potential distribution on metal surfaces in the air [17–25]. It combines the classical Kelvin probe

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technique with atomic force microscopy (AFM). Recently, this method has been advanced to investigate the corrosion processes under atmospheric weathering conditions on a micro or even nano-scale and has a high resolution of c.a. 1 mV in reading the surface potential [26]. The outlining of the surface Volta potential of a sample is made by employing a two-pass technique; i.e. each line of the image is scanned twice. In the first step of each scan, external voltage is not applied to the tip instead line topography is recorded using the tapping mode technique (that oscillates near the tip resonant frequency of 150–175 kHz by a Piezoelectric tool) and AFM mapping of the topography image from the sample surface. This data is then used during the second pass of scanning where a DC bias potential and an oscillating AC potential with a frequency equal to the resonant frequency of the cantilever is applied to the tip. The tip is then lifted to a selected distance and a feedback loop that controls the Z Piezo-element is turned off during the second pass. Hence, the mapping of surface potential and topography is done line by line at the same time [17].

To the present, although a number of complementary techniques have been used to describe the microstructural characteristics of FS welded joints [27], there is a paucity of research carried on the nature of the interface and its characteristics in dissimilar joints. Thus, the aim of this research is devoted to combine diverse techniques including scanning electron microscopy–energy dispersive spectroscopy (SEM–EDS), AFM, SKPFM, optical microscopy (OM) and Vickers microhardness to better characterize the Al/Cu bimetal interface. Particularly, an attempt was performed to quantitatively find out a correlation between the namely mentioned techniques in investigating the interface characteristics of an FS welded Al/Cu joint. Indeed, such information will direct one to a consistent idea of corrosion initiation sites at the induced interfaces.

2. Experimental procedure

2.1. Welding procedure

An FSW adapted milling machine was employed for the welding procedure providing 1100 rpm and 50 mm/min rotation and welding feed (traverse) speeds, respectively. The process was carried out on a lap joint of an 8 mm thick plate of 3003 aluminum alloy on top and a 5 mm thick commercially pure copper at the bottom. The chemical composition of the aluminum plate was (in wt%): 1.292% Mn, 0.652% Fe, 0.157% Si, 0.124% Cu, 0.038% Mg, 0.024% Sb, 0.017% Cr, 0.011% Ti, 0.011% Zn, 0.005% Sn and Al as balance. The copper plate also consisted of (in wt%): 0.053% Al, 0.018% Fe, 0.011% Zn, 0.007% Pb, 0.006% P, 0.005% Sn, 0.003% Mn, 0.003% Mg, 0.002% Si, and Cu as balance. Both plates were cut into 20 cm × 20 cm pieces and subjected to FSW using H13 hot work double-shoulder steel tool as introduced by Khaled [7]. The tool was designed to be located at the interface between the two pieces being welded. The welding tool itself consisted of a lower 10 mm diameter and an upper 40 mm diameter concave shoulders with coaxial circular treads (scrolled-like configuration) and a primary lower tapered, parallel-treaded pin of 3 mm length beneath a secondary conic pin of 8 mm length with parallel treads (Fig. 1a). In this figure, a real optical image of the weld cross section is attached to the schematic illustration for better clarification. In the current study, the words "pin" and "shoulder" refer to the primary lower pin and the lower shoulder, respectively. Not having provided a pre-drilling start hole, the pin end initially penetrated through the top aluminum plate, and extended up to 3 mm into the bottom copper plate, whilst the tilting angle was set to 1.5°. Due to high thermal conductivity of copper, the work piece was preheated up to 200 °C prior to welding. To obtain an entirely welded interface (in order to completely surf the welding area and form a fully welded Al/Cu interface), the weld was implemented by performing a multi-pass technique with an offset distance of 8 mm between parallel weld passes that caused a 2 mm overlap between adjacent weld lines (Fig. 1a).

2.2. Sample preparation

In order to provide a suitable section for AFM, SKPFM, SEM–EDS, OM and microhardness studies, a cutoff cross-section from the weld was selected, degreased by acetone, mechanically wet polished down to 0.05 μm alumina slurry, washed with ethanol and finally dried by hot air blow. Fig. 1b reveals an SEM illustration of the three different regions investigated in this study. The P1 region corresponds to the region around the pin while the S1 region refers to the area beneath the shoulder. The P2 region, adjacent to P1, also depicts the area around the pin on which some complementary analyses were performed. Note that all the mentioned experiments have been performed on just one mirror-like surface.

2.3. Macro and microstructural investigation

Macro and microstructural changes from copper to the aluminum matrix (at both the as-polished and etched conditions) were examined using a digital camera and SEM (model: LEO 1450 VP, resolution: 2.5 nm, Max voltage: 35 kV) equipped with secondary electron (SE) and EDS analysis system (resolution: 133 eV). EDS line-scans in the P1 and S1 regions were performed to study the chemical composition transition from Cu to Al matrixes. In all backscattered SEM images, light and dark phases are related to copper and aluminum containing components, respectively. The gray colored components are those intermediate compositions consisting of both Cu and Al elements (together with other accompanying minor alloying elements).

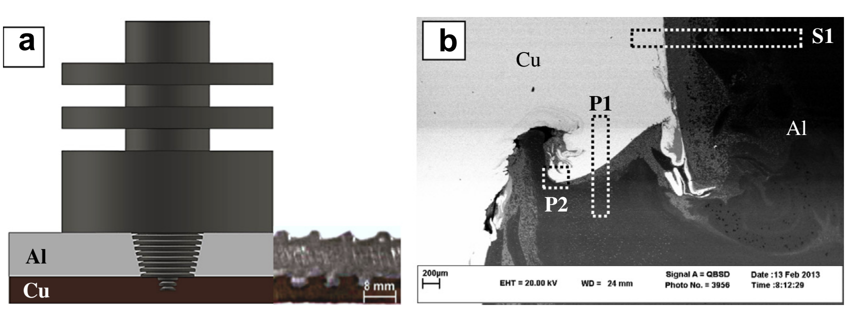

Fig. 1. (a) Schematic illustration of the FSW process. The right part shows an inserted cross-section image of the welded sample, and (b) SEM–BSE image of the interface studied in this survey. The P1 and P2 regions point to the interface around the pin and the S1 region refers to the interface beneath the shoulder.