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Electrochemical profiling of multi-clad aluminium sheets used in automotive heat exchangers

Kirill Bordo^a, Visweswara C. Gudla^{a,*}, Lionel Peguet^b, Andreas Afseth^b, Rajan Ambat^a

^a Section of Materials and Surface Technology, Department of Mechanical Engineering, Technical University of Denmark, Produktionstorvet, Kongens Lyngby, 2800, Denmark

^b Constellium Technology Center, Voreppe, 38341 France

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ABSTRACT

A combination of glow discharge optical emission spectroscopy sputtering and local electrochemical measurements was used to determine electrochemical changes upon brazing in a multi-layered Aluminium sheet (AA4343/AA3xxx/AA4343) with an additional low-Cu (AA3xxx) interlayer. E_{corr} values from potentiodynamic polarization, galvanic corrosion behaviour by ZRA, microstructure and composition by SEM and TEM were investigated and compared to those obtained for sheet without the interlayer. Inward diffusion of Si from clad, and outward diffusion of Cu from core are found to degrade the corrosion properties of conventional sheet, whereas presence of interlayer reduced outward diffusion of Cu and hence improved corrosion protection.

1. Introduction

Aluminium and its alloys are widely used in the automotive, construction, and aerospace industries due to their high strength to weight ratio, inherent corrosion resistance, ease of formability, their ability to be surface modified for different functional applications and their ease of recycling [1–6]. Aluminium is used extensively in the automotive industry for heat exchangers in radiators, air conditioner evaporators, exhaust gas recirculation (EGR) and water and air charge air cooling (W & A CAC) systems [7]. This is due to its favourable combination of mechanical, thermal, and electrochemical properties [8,9]. Over the last two decades, mechanical assembly in the production of heat exchangers has been continuously replaced by brazing due to its reduced manufacturing costs and compliance with the increased safety requirements and issues related to recyclability [10–12]. Braze heat exchangers are manufactured from clad aluminium sheets where the high melting point core alloy provides the mechanical strength (typically an Al-Mn, AA3xxx), and the clad alloy with a low melting point such as Al-Si (AA4xxx) type alloy acts as a filler for the brazing process [13,14].

In EGR and ACAC systems, the use of aluminium alloys is limited due to corrosion by the exhaust gases, which are acidic in nature and contain water [15,16]. Corrosion occurring on the air side of heat exchanger tubes is a critical issue and is currently the main issue for continued down-gauging and weight reduction [11,17,18]. The conventional AA3xxx alloy used as core material in the heat exchanger

tubes is prone to intergranular corrosion (IGC) and this results in penetration of the tube walls causing leakage and failure [19,20]. This corrosion is enhanced by diffusion of Si from the AA4xxx clad after brazing [21,22]. Afshar et al. [23–25] studied the microstructure and electrochemical behaviour of braze clad AA4xxx/AA3xxx sheets and observed that the presence of α -Al(Fe,Mn)Si intermetallic phases up to a depth of 5 μ m below the surface of the sheet support galvanic coupling with the internal layers and enhance dissolution and IGC attack. It was postulated that elimination of these corrosion susceptible sites would improve corrosion performance. Recently, significant improvement in the air-side corrosion resistance has been achieved by using cladding of multiple layers of different Al alloys where the interlayer between the AA4xxx clad and the AA3xxx core acts as an additional sacrificial layer [1,26]. This approach allows for increased diffusion distances for Si and Cu from the AA4xxx clad and the AA3xxx core respectively, and thus is expected to prevent localized precipitation of Al(Fe,Mn)Si intermetallic phases that contain Cu. In addition, the Cu concentration gradient from the core to the clad is optimized and hence the electrode potential of the metal, and thus better corrosion protection properties of the core.

The objective of the present study was to understand the specific effect of an additional low-Cu AA3xxx interlayer on the microstructural and electrochemical modifications occurring in a clad Al heat exchanger sheet after being subjected to controlled atmosphere brazing treatment (CAB, hereafter referred to as ‘brazing’). In order to characterize the local changes in the electrochemical properties as a

* Corresponding author.

E-mail address: chakri_gvc@yahoo.co.in (V.C. Gudla).

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function of depth below the surface, a combination of controlled radio frequency glow discharge optical emission spectroscopy (RF-GDOES) sputtering [27] and localized electrochemical measurements [28–33] with a lateral resolution of 1 mm was employed. Electrochemical depth profiling of the clad Al structure with a low-Cu AA3xxx interlayer was studied before and after brazing. Zero resistance ammetry measurements were also performed by coupling the individual adjacent layers before and after brazing. The microstructural and compositional modifications were analysed by scanning and transmission electron microscopy and energy dispersive X-ray spectroscopy techniques. The results were correlated with the electrochemical depth profiling data and compared to those obtained for a conventional three-layer Al clad product without a low-Cu interlayer.

2. Experimental details

2.1. Materials

Aluminium coupons of approximately 8 cm × 20 cm were cut from rolled brazing alloy sheet. The ‘core’ of the brazing sheet was a modified AA3xxx aluminium alloy, which had been clad with an AA4343 (~36 μm thick, ‘clad’) on the top of a low-Cu AA3xxx (~38 μm thick, ‘interlayer’) as shown in Fig. 1(a). For comparison, brazing sheet without the low-Cu interlayer was also analysed (Fig. 1(b)). The brazing sheet with the low-Cu interlayer is referred to as ‘**Multiclاد**’ and those without the interlayer as ‘**Reference (Ref)**’. The composition of the materials used in the as-rolled condition are shown in Table 1.

2.2. Brazing

The Al samples were degreased in acetone, dried and placed into a Controlled Atmosphere Brazing (CAB) furnace in a dry, inert nitrogen gas atmosphere for simulating the temperature cycle during the brazing treatment. The materials were heated up to 600 °C (at 30 °C/min), and were held at that temperature for 2 min and then cooled down at a specified rate of 60 °C/min. The process temperature was selected to be close to the liquidus temperature of the clad material (~612 °C) and much lower than the solidus temperature of the core material (~640 °C) [11,34].

2.3. RF-GDOES

Both the as-rolled, and brazed sheet materials were then etched to certain depths by Argon ion sputtering using radio frequency glow discharge optical emission spectroscopy (RF-GDOES, GD-Profilier 2, Horiba Jobin Yvon). The applied sputter power was 40 W and the Ar pressure in the sputtering chamber was 650 Pa. The diameter of the sputter crater was 4 mm. The compositional depth distributions of the main alloying elements (Si, Cu, Fe and Mn) in the sheet materials were used to identify the interfaces between the individual cladding layers and to define the required sputtering depth for further electrochemical depth profiling analysis.

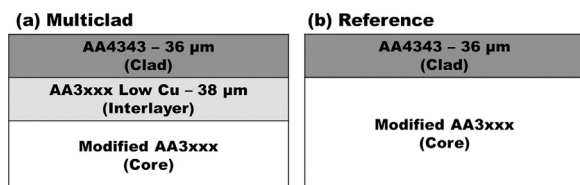


Fig. 1. Schematic cross-section of multiclاد Al brazing sheet showing the different alloy types and their relative thicknesses used in this study. The relative thicknesses are not to scale.

2.4. Electrochemical behaviour

The electrochemical behaviour of both the as-rolled, and brazed materials was determined at different depths using a Duoscope EC localized electrochemical cell (Semilab Germany GmbH, formerly DME Nanotechnologie GmbH, Germany) having a small opening (1 mm diameter plastic pipette tip) which ensured the contact between the electrolyte and the working electrode (see Fig. 2). The system employed an electrochemical head consisting of the reference and counter electrodes, which were open to a reservoir cavity containing the electrolyte. A plastic pipette tip with 1 mm diameter opening was attached to this reservoir cavity to create a hanging droplet of the electrolyte onto the test surface. The hanging droplet was achieved by the precise airtight solution pumping mechanism. The size and shape of the droplet were continuously monitored and controlled using a microscope and a digital video camera attached to the cell [23,35,36]. Prior to each experiment, a small amount of solution was always dispersed from the tip followed by gentle cleaning by scrubbing against a flat surface. The tip was then brought into contact with the surface of the working electrode and then slowly retracted to a height of 100 μm from the surface of the working electrode. The hanging droplet of the electrolyte (of 1 mm diameter) is achieved due to surface tension, and guarantees wetting of the working electrode surface while maintaining the size and shape of the droplet.

For all the electrochemical measurements a saturated Ag/AgCl reference electrode and a Pt wire counter electrode were used. Potentiodynamic polarization scans were performed with a scan rate of 1 mV/s. Prior to each measurement, the open circuit potential (OCP) was allowed to stabilize for 30 min. A solution of 0.1 M HCl (pH ~ 1, analytical grade) was used as the electrolyte based on a parametric study by Peguet et al. [37,38] which compared the use of ASTM G69 solution (1 M NaCl + H₂O₂ 9 mL/L) [39] with 0.1 M HCl. Results showed that the 1 M NaCl based electrolyte was very aggressive and penetrating pits of more than 100 μm depth were formed which was not suitable for thin gauge sheets. Lower Cl concentration with use of 0.1 M NaCl resulted in longer time for stabilization of potential. The 0.1 M HCl solution allowed faster de-passivation of the surface and stabilising of the surface potential. All experiments were conducted at room temperature and no aeration was applied to the electrolyte.

Zero Resistance Ammetry (ZRA) measurements were performed in a conventional 3-electrode cell using the same potentiostat. The measurements were conducted in a 0.1 M NaCl solution of pH 2.8 (adjusted with HCl; 400 mL volume electrolyte) at room temperature for 3 h to determine the evolution of the galvanic current density between the individual layers in the brazing sheet and the galvanic potential. These measurements were conducted before and after the brazing treatment. In each ZRA experiment, two pieces of the similar sheet material which had been sputtered to different depths by RF-GDOES were used. This allows for coupling between the clad (WE1) and the interlayer (WE2); and the interlayer (WE1) and the core (WE2). Only the sputtered craters (of 4 mm diameter, 12.56 mm² area; surface area ratio 1:1) were exposed to the electrolyte in the ZRA setup, and the remaining surface of each sample was covered with a protective lacquer. The samples (electrodes) were placed facing each other and the distance between the anode and the cathode was 5 ± 0.5 cm. The potentiodynamic polarization tests and the ZRA testes were repeated at least three times.

2.5. Microstructural characterization

The microstructures of the as-rolled, and brazed materials were examined using a scanning electron microscope (SEM, Quanta 200 ESEM FEG, FEI) equipped with an energy dispersive X-ray spectrometer (EDS, 80 mm² X-Max silicon drift detector, Oxford Instruments). Additional characterization of the microstructure before and after brazing was performed using a transmission electron microscope (TEM, Tecnai T20 G2, FEI) coupled to an energy dispersive X-ray spectrometer (EDS, 80 mm² X-Max silicon drift detector, Oxford Instruments).

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