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Understanding the electrochemical, microstructural and morphological changes during hot rolling from a corrosion perspective

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Abstract

Thermo-mechanical processing of rolled aluminium alloys result in the formation of a deformed near-surface region with different filiform corrosion (FFC) behavior than the underlying bulk. This paper tries to correlate the microstructural, morphological and electrochemical changes occurring on the surface of aluminium sheet, during hot rolling, with the FFC behavior. The alloy under investigation is recycled AA5050. The electrochemical changes taking place in the roll-bite has been profiled and the result has been supported by electron microscopic and optical characterization for better understanding. Factors which may or may not be responsible for FFC susceptibility of rolled AA5050 have been discussed and importance of surface finish has been emphasized.

Keywords: Thermo-mechanical processing; Surface layer; Filiform corrosion

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1. Introduction

Rolling is an integral part of the fabrication of wrought aluminium alloy sheet. While the main purpose of hot rolling is gauge reduction, cold rolling additionally provides strain hardening. During rolling, the work rolls exert a load and a shear stress on the surface of the work piece, causing severe shear deformation of the near-surface region compared to the bulk microstructure. This results in the development of a surface layer (a few microns thick) [1] with different morphological [1–3], optical [4–6], microstructural [7–9] and electrochemical [9–12] properties compared to the bulk. The surface layer can control many important properties like corrosion resistance, adhesion and optical appearance and hence it is important to understand both its electrochemical behavior and microstructural details like the structure, precipitates and other features present.

The deformed surface layer has extremely fine grains compared to the bulk [1,4]. Furthermore these ultra-fine grains are pinned by rolled-in oxides [1], preventing recrystallization of the surface layer during subsequent heat treatment. This phenomenon is referred to as zener pinning [13]. Often, the surface layer comprises of a high density of sub-micron size precipitates in addition to surface features like cracks, rolling ridges, etc. [3]. Analysis using transmission electron microscopy (TEM) has revealed the composition, structure and thickness of the surface layer [7,8]. Optical reflectance of the surface layer has been found to be much less than the bulk alloy predominantly due to the presence of rolled-in oxides [4,5].

In the late nineties, it was reported that the surface layer is electrochemically active, i.e. the surface layer is more susceptible to corrosion attack, mainly filiform corrosion (FFC), than the underlying bulk [14]. FFC is usually an underfilm corrosion process which manifests itself in the form of thread-like filaments. Afseth et al. [8] combined the surface analytical and the electrochemical properties of the surface layer and formed a correlation between thermo-mechanical treatment and surface activation. They also found that hot rolling plays a more dominant role in activating the surface layer, as compared to cold rolling. Fishkis and Lin [1] had earlier reported that substantial morphological changes (cracks, holes, etc.) take place at high temperatures, i.e. initial hot rolling passes.

While in the literature there is a fairly good understanding of the microstructural and morphological changes taking place in the surface layer during rolling, the detailed mechanistic

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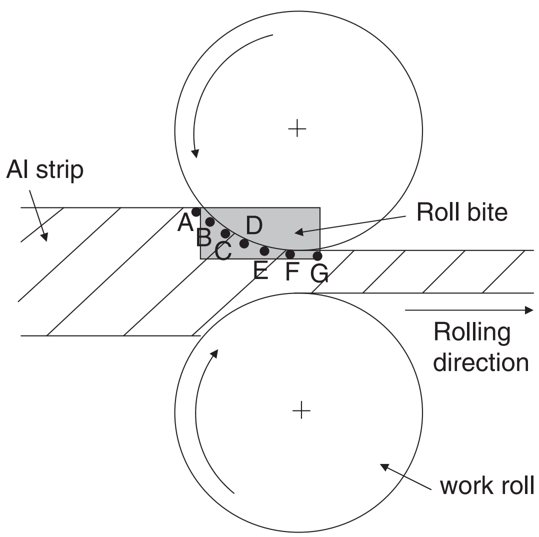

Fig. 1. Schematic representation of the roll-bite. Potentiodynamic polarization measurements using micro-cell were carried along the planes A, B, C, D, E, F and G. Planes A and G lie outside the roll-bite.

understanding of electrochemical activation is not well developed. This paper tries to develop an understanding of the electrochemical changes occurring in the near-surface after the initial hot rolling passes and also in the roll-bite. A schematic of the roll-bite can be seen in Fig. 1. The effect of mechanical deformation of the surface on the electrochemical as well as FFC behavior has been studied in detail.

2. Experimental

The wrought architectural aluminium alloy, AA5050, was manufactured at the test mill facility of Hunter Douglas Europe BV, Rotterdam. The alloy was cast from recycled aluminium and hence had a high concentration of impurities like Fe and Si. The chemical composition of this alloy is shown in Table 1. Samples were provided from different stages of the fabrication (as-cast, hot rolled 1st and 2nd pass, final hot rolled, cold rolled) and also from the roll-bite of the first 3 passes of a 6-stand tandem hot rolling mill. The width of the cast bar was 23 cm and the gauge was 25 mm. The entry temperature in the hot rolling mill was around 525 °C and the gauge reductions in the first two hot rolling passes were 50% and 48% respectively.

Glow discharge optical emission spectroscopy (GDOES) was used for depth profiling the concentration of various alloying elements. The instrument used was a Leco SDP-750 dc GDOES with 4 mm diameter copper anode. This instrument has a high sensitivity and can theoretically measure concentration as low as 10 ppm. A discharge voltage of 700 V and a discharge current of 20 mA were applied. The sputtered depth was calculated from sputtering time using the multi-matrix calibration approach [15].

Microstructural characterization was carried out using backscattered electron imaging using a LEO 1455VP Scanning Electron Microscope (SEM) fitted with an Energy Dispersive X-ray (EDX) analyzer. Transmission Electron Microscopy (TEM) was carried out using CM30T from Philips. Cross-sectional TEM specimens were prepared using a Gatan PIPS-691 Ion Miller. The surface was protected using glue.

Acidified salt spray (ASS) test was performed on the samples from different stages of fabrication in order to have a better estimation of the FFC susceptibility of the alloy under investigation. These tests were performed in accordance with European coil coating association (ECCA) T-8 test (EN 13523-8), at room temperature for 500 h. Test specimens were degreased in Z19 (phosphoric acid bath) at 50 °C for 20 s and rinsed. Thereafter the samples were pre-treated using Unicon 87 (Cr-free). Finally a transparent polyester topcoat was applied followed by curing. The coated specimens were then scribed and placed in the salt spray test chamber. Test solution, containing 1% CH_3COOH and 5% NaCl, is continuously sprayed on the walls of the test chamber so as to have them suspended in the test environment. The samples were examined after 168 h and after 500 h. Corrosion number, a standard used in the industry, is defined in Eq. (1). A lower corrosion number implies a more corrosion resistant surface.

$$\text{Corrosion number} = (\text{fraction of scribe corroded}) * (\text{average length of filament}). \tag{1}$$

An electrolyte containing 5% NaCl+HCl (pH 2) was used for the electrochemical characterization. The pH of this electrolyte is similar to that at the head of a filiform filament [11]. Macro-electrochemical characterization was performed over an exposed area of 0.79 cm^2 ($\phi = 1$ cm). After a steady-state potential was developed, the test samples were polarized anodically with a scan rate of 10 mV/s. A standard 3-electrode set-up was used with Ag/AgCl (3 M KCl) as the reference electrode and platinum as the counter electrode.

Due to their curved geometry, the roll-bite samples could not be characterized electrochemically on a macroscopic scale. This problem was overcome by characterizing the roll-bite specimen using a capillary-based micro-cell [16,17]. The micro-cell is used for small area measurement, in which a glass capillary is used to restrict the area of measurement. The electrolyte is filled up in the glass capillary and the size of the capillary tip determines the size of the working area because the capillary tip is in contact with the working electrode, i.e. the sample. The counter electrode and the reference electrode are connected to the other end of the glass capillary. This technique has been used in understanding the breakdown in association with

Table 1
Composition of alloying elements in the commercial AA5050

Element	Si	Fe	Cu	Mn	Mg	Cr	Zn	Ti	Pb	Be	Na
AA5050 (wt.%)	0.28	0.8	0.12	0.33	1.20	0.05	0.07	0.03	0.01	0.0006	0.003