

# Development of a recovered/recrystallized multilayered microstructure in Al alloys by accumulative roll bonding

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## Abstract

A high-purity Al alloy and a supersaturated Al–0.3 wt.% Sc alloy (Al(Sc)) were accumulative roll bonded at 200 °C to generate sheet material consisting of alternating layers of Al and Al(Sc). The deformation structure within these layers consisted of lamellar bands aligned parallel to the rolling direction. Compared with those bands in Al(Sc), the bands in the Al layers were less refined but contained a larger fraction of high angle grain boundaries (HAGBs). Subsequent annealing at 350 °C generated alternating layers of coarse grains (Al layers) and a recovered substructure (Al(Sc) layers); the latter were stabilized by the precipitation of Al<sub>3</sub>Sc particles. Within the Al layers, annealing did not significantly alter the rolling texture (β-fibre), although the strong Brass component was largely eliminated; this behaviour has been explained using the “ReNuc” model of recrystallization whereby nucleation is deemed to occur on HAGBs between the β-fibre components of the lamellar bands in conjunction with orientation dependent recovery.

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## 1. Introduction

Severe plastic deformation (SPD) is a method of producing ultrafine grained (UFG) materials. The usual objective is to produce materials that possess novel combinations of properties beyond the property space of conventional materials. Accumulative roll bonding (ARB), originally developed by Saito et al. [1], is a relatively simple method of generating UFG structures in which two metal sheets are bonded during rolling at a relatively high reduction (~50%). The bonded sheets are repeatedly cut, stacked together and roll bonded for a number of cycles depending on the strain hardening behaviour of the material. ARB has a number of advantages over similar processes because it can introduce ultrahigh plastic strain without geometrical change. The result is a method

for bulk production of UFG alloys using a relatively simple technique that can be carried out in a conventional rolling plant with easy-to-control parameters (e.g. percentage reduction, rolling speed, roll diameter, bonding temperature, etc.). The early work on ARB was largely applied to Al alloys [1–3], with recent extension to other materials like Cu, Mg and Fe [1,4,5]. To date, up to eight-cycle ARB sheet has been produced, with an equivalent true strain of ~5 [2]. A review of ARB is given by Tsuji [6].

The ARB process is amenable for producing sheet products containing alternating layers of different metal types (Cu–Zn, etc. [7]), as well as heat-treatable and non-heat-treatable alloys of the same base metal. In the latter, roll bonding sheets of particle-free and particle-containing Al alloys may generate a composite structure containing alternating recrystallized and unrecrystallized layers by subsequent annealing. Unfortunately, particle-containing alloys exhibit a high work hardening rate which may be problematic during ARB. However, it is possible to exploit the

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interesting properties of Al–Sc alloys whereby deformation may be carried out in the supersaturated condition followed by low temperature annealing to rapidly generate a relatively uniform dispersion of nanosized  $\text{Al}_3\text{Sc}$  particles that strongly pin both dislocations and grain boundaries [8]. This type of stabilized microstructure resists recrystallization (discontinuous coarsening) at temperatures up to  $\sim 500^\circ\text{C}$  [9]. By controlling the type of alloys, thickness ratio of the sheets and processing conditions (total strain during ARB and the annealing parameters), it is possible to generate a hybrid microstructure with a particular combination of properties. For example, recrystallized commercial purity Al has a hardness of  $\sim 30$  VHN whereas a simple Al–0.2 wt.% Sc alloy remains unrecrystallized for the same annealing conditions and has a hardness of  $\sim 65$  VHN [9]. Hence, a unique combination of strength and ductility may be achieved in such a composite material if the recrystallized/unrecrystallized ratio is controlled. A similar type of property control has been demonstrated recently in various alloys produced by plastic deformation followed by careful annealing to generate a bimodal grain size distribution [10–12]. The ARB process is highly suited to controlling the fraction of the total material recrystallized via the thickness of the individual alloy layers.

A number of detailed studies of SPD have been carried out individually for both Al and Al(Sc) alloys [9,12], but there is no information on the deformation and annealing behaviour of these materials in the form of a multilayered structure. This paper describes an ARB/annealing process for generating an aluminium composite containing coarse-grained (ductile) Al layers separated by UFG (high strength) Al(Sc) layers with particular emphasis on the effect of deformation and annealing on microstructural development.

## 2. Experimental procedure

### 2.1. Initial materials processing and ARB

Sheets of commercial purity aluminium (99.8 wt.% purity) (Al) and aluminium containing 0.3 wt.% Sc (Al(Sc)) were processed prior to ARB. An as-cast slab of Al was cold rolled from a thickness of 13 mm to 1 mm, followed by recrystallization for 45 min at  $375^\circ\text{C}$  and air cooling, to generate an equiaxed microstructure of grain size  $50 \pm 30\ \mu\text{m}$ . Another 20 mm thick as-cast slab, of Al(Sc), was homogenized for 48 h at  $640^\circ\text{C}$ , cold rolled to 1 mm thickness, solution treated for 48 h at  $640^\circ\text{C}$  and cold water quenched. This generated a coarse-grained microstructure spanning the full sheet thickness. After thorough brushing and cleaning,  $1 \times 50 \times 100$  mm sheets were mechanically stacked and held for 5 min at  $200^\circ\text{C}$  in a preheated furnace then rolled at this temperature without lubrication to 50% reduction in a single pass using a two high rolling mill. The rolled material was cut into two pieces and roll bonded in an identical manner while maintaining the alternating sequence of Al and Al(Sc) layers.

After five cycles plus final rolling to 50% reduction, the method produced 0.5 mm sheet containing 32 alternating layers. The as-rolled material was heat treated for up to 6 h at  $350^\circ\text{C}$  followed by air cooling. This heat treatment temperature was selected due to the detailed understanding of the restoration processes that occur in both Al and Al(Sc) [9].

### 2.2. Analysis of microstructure

Normal direction (ND)–rolling direction (RD) sections of the as-rolled and annealed materials were examined in an FEI DualBeam™ platform by both ion channelling contrast (ICC) imaging using focused ion beam (FIB) microscopy and electron channelling contrast imaging using field-emission gun scanning electron microscopy (FEGSEM). Microstructural investigations were carried out on mid-thickness regions of the sheet. Using the DualBeam™ platform in FEGSEM mode, EBSD was carried out using a TSL OIM™ system operating with an accelerating voltage and working distance of 10 keV and 10 mm, respectively. Using ICC imaging, suitable areas of microstructure were selected for analysis by EBSD. A low angle grain boundary (LAGB) was defined by a misorientation between adjacent grains ( $\theta_m$ ) of  $5^\circ < \theta_m < 15^\circ$ , and a high angle grain boundary (HAGB) was defined by  $\theta_m > 15^\circ$ . For transmission electron microscopy (TEM), site-specific foils were prepared using FIB for analysis of appropriate regions of interest. The individual layers (Al and Al(Sc)) and the interface between these layers were investigated by TEM using a Philips CM200 FEGTEM. The analysis of dislocation structure and interactions between  $\text{Al}_3\text{Sc}$  particles and the deformation substructure during annealing were carried out by bright field imaging as well as convergent beam electron diffraction (CBED) for investigating orientations in the microstructure at high resolution.

## 3. Results

### 3.1. Deformation microstructures and textures

Fig. 1 is a typical ICC micrograph of the material after ARB showing an Al layer between two Al(Sc) layers. The average Al(Sc):Al thickness ratio was  $\sim 4:3$  which corresponds to average true strains in the Al and Al(Sc) layers of 4.2 and 3.9, respectively. The small bright islands located at the interfaces of the layers in Fig. 1 (arrowed) were identified to be oxide debris formed during the intermediate heating stage before roll bonding. The oxides are discontinuous and irregular in shape and, therefore, expected not to have an adverse effect on bonding quality. The Al layer in Fig. 1 is composed of strip-like substructures aligned along RD that resemble the typical high-strain substructures known as lamellar bands (LBs) [2,13–15]. In the Al(Sc) layers, this type of substructure is so fine that it appears diffuse and beyond the resolution of FEGSEM, but there are easily recognizable coarse substructures within the layers,

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