



# Evolution of the microstructure, texture and creep properties of the 7075 aluminium alloy during hot accumulative roll bonding

P. Hidalgo-Manrique<sup>\*,1</sup>, C.M. Cepeda-Jiménez<sup>1</sup>, A. Orozco-Caballero, O.A. Ruano, F. Carreño

Department of Physical Metallurgy, CENIM, CSIC, Av. Gregorio del Amo 8, 28040 Madrid, Spain

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## ABSTRACT

The 7075 Al alloy was severely deformed at 350 °C by a 3:1 thickness reduction per pass accumulative roll bonding (ARB) process up to six passes. It was found that discontinuous recrystallisation occurs during the inter-pass annealing stages from the third pass on, attributable to the increment of the mean particle size during processing. As a consequence, the mean crystallite size did not decrease, but remained approximately constant at 440 nm along the present ARB process and the mean boundary misorientation angle reached a maximum of 30° for the 3-passes sample. However, since nucleation of new grains takes place at the pre-existing grain boundaries, discontinuous recrystallisation results in slight changes in texture throughout the processing, being the orientations in the ARBed samples predominantly located along the typical rolling  $\beta$ -fibre. Uniaxial tests conducted at 300 °C and 350 °C revealed that the operating deformation mechanism in the processed alloy at such temperatures was grain boundary sliding; the optimum superplastic strain rate being  $3 \times 10^{-3}$ – $10^{-2}$  s<sup>-1</sup>. Boundary misorientation and thermal stability are the two main factors that contribute to high elongations to failure.

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## 1. Introduction

Accumulative roll bonding (ARB) is a severe plastic deformation process developed in 1998 by Saito et al. [1], consisting of multiple cycles of cutting, stacking and roll bonding. So, large strains can be accumulated in the material and significant structural refinement can be achieved [2]. Since 1998, extensive studies regarding the evolution of microstructure during ARB have been conducted [3–9]. According to these studies, grain refinement takes place in a gradual manner which can be characterised as continuous recrystallisation. In the particular case of aluminium, studies on the microstructural evolution during ARB have been widely performed on pure aluminium [6] and several alloys such as AA3003 [5], AA6061 [4] and AA8006 [8], while a few studies have been performed on the aeronautical 7075 Al alloy [10–12], despite the large advantages that would arise from refining its microstructure. A previous work [10] revealed that, as a high-strength alloy, the 7075 Al alloy needs to be processed at a minimum temperature of 300 °C in order to attain sufficient bonding and good workability. However, it must be taken into account that this alloy contains

thermally unstable precipitates [13,14], which for certain processing temperature may alter or hinder the expected grain refinement process.

Although extensive studies have been conducted regarding the microstructural evolution during ARB, there is only limited quantitative information available on the texture evolution during this process [15–18], despite the fact that the texture affects the ductility and the mechanical anisotropy which, in turn, determine the material formability. Moreover, texture measurements during ARB have been made after the roll-bonding steps, but never after the inter-pass heating stages, where significant texture changes may take place as a consequence of certain restoration processes, such as discontinuous recrystallisation [12]. That is, no studies on the influence of the inter-pass heating stages on the texture evolution have been performed. Several works revealed that the texture developed by ARB in the mid-thickness regions of the samples is very similar to that developed by conventional rolling and, as such, it is characterised by rolling-type components. However, textures developed by ARB are normally weaker [1,6,19] and even less symmetric [1]. In order to understand these differences it must be considered that shear strain is introduced into the sample surface during any rolling process due to the large friction between the rolls and the sample. Consequently, the surface regions of the rolled samples exhibit shear texture. In the ARB process, in the cutting and stacking steps, surface regions

<sup>\*</sup> Corresponding author. Tel.: +34 91 549 34 22.

E-mail address: [paloma.hidalgo@imdea.org](mailto:paloma.hidalgo@imdea.org) (P. Hidalgo-Manrique).

<sup>1</sup> Present address: IMDEA Materials Institute, C/Eric Kandel 2, 28906 Getafe, Madrid, Spain.

are transferred into the sample interior. The introduction of shear strain may alter the slip pattern from that which characterises conventional rolling. Additionally, although shear texture is easily destroyed, the introduction of shear orientations may retard the development of a strong rolling texture.

The superplastic forming (SPF) technology has already found numerous industrial applications, as it can be used to fabricate components with a complex shape and uniform thickness in a single forming operation. This procedure eliminates the need for the assembly of separately made parts and leads to a reduction of tooling costs [20]. The underlying basis for SPF is superplasticity, characterised by enhanced ductility and low stress, operating through the so-called grain boundary sliding (GBS) creep mechanism [21,22]. In high strength aluminium alloys, such as 7075, superplasticity has been traditionally developed by complex thermomechanical processes [23–27]. The resulting materials with a grain size around 10  $\mu\text{m}$  exhibited optimum superplasticity at temperatures close to 500 °C and at strain rates of the order of  $10^{-4} \text{ s}^{-1}$ . These conditions lead to so elevated costs which necessarily confine the applications of SPF to low-volume production. Experiments [28,29] show that a shift to lower deformation temperatures and/or higher strain rates can be attained by further grain refinement. Thus, ARB gives the opportunity to make SPF cost-effective, extending its commercial application.

In the present work, the 7075-T6 Al alloy was deformed at 350 °C by a 3:1 thickness reduction per pass ARB process up to six passes. The evolution of the microstructure, texture and creep properties was carefully examined. Moreover, the superplastic behaviour of the processed alloy at temperatures lower and strain rates higher than conventional for optimum superplasticity was evaluated.

## 2. Material and experimental procedure

The material used for the present work was a 2 mm-thick sheet of the commercial 7075 aluminium alloy in the T6 condition. The chemical composition is shown in Table 1. Microstructural examinations showed a microstructure composed of pancake-shaped grains of dimensions  $60 \times 47 \times 4 \mu\text{m}^3$ . The orientations characterisation showed a pronounced cube texture with rolling direction-scatter [11].

From the as-received alloy sheet two pieces with dimensions 2 mm  $\times$  30 mm  $\times$  180 mm were cut, cleaned with methyl ethyl ketone, put one on top of the other and fastened by steel wires. The resulting 4 mm-thick specimen was held for 5 min at 350 °C in a preheated electric furnace and straight afterwards underwent a thickness reduction of 3:1, equivalent to a true strain of 1.1, by a single rolling pass. True strain ( $\epsilon$ ) was calculated as  $-\ln(h_0/h)$ , where  $h_0$  and  $h$  are the initial and the final thicknesses of the rolled specimen, respectively. Rolling was performed in non-lubricated conditions using a two-high mill with a roll diameter of 131 mm and a peripheral roll speed of 346 mm/s. Immediately after rolling, the resulting 1.33 mm-thick specimen was water quenched. Afterwards, it was divided in three identical pieces that were supplied to the next ARB pass. From that moment on, the procedure described above was executed 5 more times maintaining the rolling direction (RD).

**Table 1**  
Chemical composition of the 7075 aluminium alloy studied (mass%).

Si	Mg	Fe	Zn	Cu	Ti	Cr	Mn	Al
0.052	2.51	0.19	5.68	1.59	0.025	0.19	0.007	Bal.

The microstructures of the ARBed samples were characterised on the rolling plane in a section located at a depth of 40% from the surface by transmission electron microscopy (TEM) and electron backscatter diffraction (EBSD). The TEM studies were carried out in a JEOL JEM 2000 FX II microscope operating at 200 kV. The mean crystallite size ( $d$ ) was directly calculated from the TEM micrographs as the mean boundary spacing. In this work, the term crystallite is used to describe volumes separated from the neighbouring volumes by boundaries of any misorientation angle. For TEM investigations disks of 3 mm diameter extracted from the ARBed samples were thinned to perforation using a twin-jet electropolishing facility with a solution of 30% nitric acid and 70% methanol at 15 V and  $-25$  °C.

The EBSD mapping was conducted in a JEOL JSM 6500 F field emission gun scanning electron microscope operating at 20 kV with a working distance of 15 mm and a step size of 80 nm. Data acquisition and analysis were performed using the commercial Channel 5 software. Because of the limited angular resolution of the EBSD system, misorientations below or equal to 2° were neglected. Boundaries with misorientations from 2° to 15° were defined as low angle boundaries (LABs), while those with misorientations greater than 15° were defined as high angle boundaries (HABs). The mean boundary misorientation angle ( $\theta$ ) was calculated from the EBSD data. In addition, the mean spacing of HABs ( $d_{\text{HAB}}$ ) was determined from the maps as mean linear intercepts along the transversal direction (TD). Specimens for EBSD investigations were mechanically ground and polished and then electropolished in the preceding solution at 15 V and  $-15$  °C.

Textures of the ARBed samples were determined by X-ray diffraction (XRD) in a section located at a depth of 40% from the surface. The (111), (200) and (220) pole figures were measured using  $\text{CuK}_\alpha$  radiation in a Siemens D500 diffractometer equipped with an open Euler ring working with Schultz geometry. From these experimental pole figures and using the TexTools software the orientation distribution functions (ODFs) were derived by means of the series expansion method. The ODFs were represented in the form of sections through the Euler space, which due to the cubic crystal symmetry and the orthotropic sample symmetry are defined by  $0^\circ \leq \varphi_1, \Phi, \varphi_2 \leq 90^\circ$ . In particular, equal distance sections along the  $\varphi_2$  angle in 5° steps are used.

Tensile dogbone specimens with gauge length of 10 mm and width of 3 mm were electro-discharge machined from the as-received sheet and the ARBed samples with their tensile axis perpendicular to the rolling direction (RD). Uniaxial tensile test to failure was conducted at 300 °C and 350 °C using a Servosis ME 405/10 and an Instron 1362 testing machine and an elliptical furnace provided with four quartz lamps in air. The tensile specimens were held at the testing temperature for 20 min before the tests and water quenched immediately after the tests. A set of tensile tests was performed at a constant crosshead speed corresponding to the initial strain rate of  $10^{-2} \text{ s}^{-1}$ . Note that initial strain rate is given by  $v/l_0$ , where  $v$  is the crosshead speed, which can be measured with high accuracy, and  $l_0$  is the initial specimen gauge length. Yield stress ( $\sigma_{0.2}$ ) and elongation to failure ( $e_f$ ) were determined from the true stress ( $\sigma$ )–true strain ( $\epsilon$ ) curves. Another set of tensile tests was performed using the strain rate change (SRC) method. First, the initial strain rate is consecutively reduced in several steps from  $10^{-1} \text{ s}^{-1}$  to  $10^{-5} \text{ s}^{-1}$  utilising constant crosshead speeds. Then, the strain rate is consecutively increased from the lowest strain rate, in several steps up to  $10^{-2} \text{ s}^{-1}$ , also utilising constant crosshead speeds. From the SRC tests the true strain rate ( $\dot{\epsilon}$ )–true stress ( $\sigma$ ) pairs corresponding to the decreasing strain rate steps were extracted in the steady state. As the calculations take into account the increasing specimen gauge length during testing, the accuracy of the  $\dot{\epsilon}$ – $\sigma$  data pairs is maximised. Using these data, the apparent stress exponent ( $n_{\text{ap}}$ )

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