Journal of Materials Science & Technology 32 (2016) 774–782

Contents lists available at ScienceDirect

Journal of Materials Science & Technology

journal homepage: www.jmst.org

Influence of the Accumulative Roll Bonding Process Severity on the Microstructure and Superplastic Behaviour of 7075 Al Alloy

P. Hidalgo-Manrique *,†, A. Orozco-Caballero †, C.M. Cepeda-Jiménez ‡, O.A. Ruano, F. Carreño

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

ARTICLE INFO

Article history:
Received 8 July 2015
Received in revised form
23 October 2015
Accepted 13 November 2015
Available online 7 June 2016

Key words:
Accumulative roll bonding (ARB)
Al—Zn—Mg—Cu alloys
Grain refining
Precipitate coarsening
Recrystallisation
Superplastic deformation

The 7075 Al alloy was processed by accumulative roll bonding (ARB) at 350 °C using 2:1, 3:1 and 4:1 thickness reductions per pass (R_p) up to 8, 6 and 3 passes, respectively. Microstructural examinations of the processed samples revealed that ARB leads to a microstructure composed of equiaxed crystallites with a mean size generally lower than 500 nm. It was found that, due to both the stored energy throughout the processing and the particle pinning effect, the alloy is affected by discontinuous recrystallisation during the inter-pass heating stages, the precise microstructural evolution being dependent on R_p. Mechanical testing of the ARBed samples revealed that the main active deformation mechanism in the ARBed samples in the temperature range from 250 to 350 °C at intermediate and high strain rates is grain boundary sliding, the superplastic properties being determined by both the microstructure after ARB and its thermal stability.

1. Introduction

Mechanical properties of metallic materials are very sensitive to grain size. On the one hand, a reduction of grain size increases the yield stress at room temperature according to the Hall–Petch relationship[1,2]. On the other hand, fine grains promote good superplastic properties[3–5]. Superplasticity, characterised by enhanced ductility and reduced flow stress[6–11], is an important field of scientific research because it is the underlying basis for superplastic forming (SPF). SPF is a well-established industrial process in the transport industry for the fabrication of components with a complex shape and uniform thickness in a single forming operation[12]. This procedure eliminates the need for assembly of separately made parts and leads to a reduction of tooling costs. In addition, according to the constitutive equations for grain boundary sliding (GBS)[13,14], fine grain sizes favour decreasing temperatures (typically at ~$0.5 \times T_m$, where T_m is the absolute melting temperature of the material) and increasing strain rates (typically of the order of $10^{-4}\,s^{-1}$) for optimum superplasticity[15–21], leading to lower production costs and quicker production rates. Thus, grain refinement gives the opportunity to increase the viability of SPF, extending its commercial application.

Methods that subject metals to severe plastic deformation (SPD) are proven to successfully produce ultrafine-grained microstructures, whose mean grain sizes are smaller than 1 μm, without changing the specimen dimensions and without additions of alloying elements[22]. Accumulative roll bonding (ARB)[23], using the conventional rolling system, is the only SPD technique suitable for fabrication of large bulky materials. The ARB process consists of multiple cycles of cutting, stacking and roll bonding. By applying this procedure on the same sample, very high strains can be introduced into the material. As a result, significant structural refinement can be achieved. In fact, the ARB process has succeeded to manufacture UFGed sheets or plates, the most widely used material shape in the industrial field, of several pure metals and alloys[22]. Microstructural investigations showed that grain refinement resulting from the ARB process could be achieved by continuous recrystallisation, which can be explained in terms of grain subdivision and extended recovery, characterised by a strain-induced progressive rotation of subgrains with little boundary migration[24–27].

In the ARB process, rolling is usually performed at elevated temperature to enhance bonding and material workability[28]. However, due to the high energy stored in the deformed state and the

* Corresponding author.
E-mail address: paloma.hidalgo-manrique@manchester.ac.uk (P. Hidalgo-Manrique).
† Present address: School of Materials, The University of Manchester, Manchester M1 7SH, UK.
‡ Present address: IMDEA Materials Institute, C/Eric Kandel 2, 28906 Getafe, Madrid, Spain.

http://dx.doi.org/10.1016/j.jmst.2016.06.004
1005-0302/

presence of structural heterogeneities, high temperature may cause discontinuous recrystallisation, thus relieving all the accumulated strain[11,29]. Regarding the imposed strain per cycle, the ARB process has been mostly performed by 2-layer stacking and rolling by 50% reduction. However, there is no restriction in the number of layers or in the strain applied per pass, as long as the rolling mill capacity is large enough and edge cracking does not occur. In fact, since the redundant shear strain play a key role in grain misorientation[24], an increase in the thickness reduction per pass (R_p) may accelerate the evolution toward an ultrafine microstructure subdivided by high angle boundaries (HABs). In other words, if a certain amount of strain is required for the formation of ultrafine grains surrounded by HABs, an increase in R_p might result in a decrease in the number of passes and hence in a higher productivity[30,31]. Moreover, since the applied stress usually increases with increasing the thickness reduction, the attained grain size is expected to be smaller, and therefore, the refining effectiveness of the ARB process would be higher with increasing R_p, i.e., with the processing severity[32,33]. Finally, increasing R_p leads to a superior bonding strength between the stacked layers[30,31].

In this study, the 7075 Al alloy was deformed by ARB at 350 °C by rolling reductions per pass of 2:1, 3:1 and 4:1. A throughout microstructural characterisation was performed on the ARBed samples by transmission electron microscopy (TEM), electron backscatter diffraction (EBSD) and X-ray diffraction (XRD). In addition, the ARBed material was mechanically characterised by tensile tests in the temperature range between 250 and 350 °C and in a wide range of strain rates. The alloy under study is widely used for the construction of airplane structures, such as wings and fuselage, because of its excellent strength/weight ratio. Application of the ARB to the 7075 Al alloy would result in superplasticity, the optimum superplastic regime being displaced toward lower temperatures and/or higher strain rates than in conventional Al alloys, and thereby in important savings in the aeronautical industry. The purpose of the present study was to clarify the effect of the thickness reduction per pass on the resulting microstructure of the 7075 Al alloy and the mechanical properties at different conditions. Moreover, the microstructure evolution during the ARB process for every reduction of thickness and the deformation mechanisms of the ARBed samples were carefully investigated.

2. Experimental Procedure

The material under study was the commercial 7075 Al alloy, with the following chemical composition (wt%): 5.68 Zn, 2.51 Mg, 1.59 Cu, 0.19 Cr, 0.19 Fe, 0.052 Si, 0.025 Ti, 0.007 Mn, and Al (balance), provided as a 2-mm thick sheet in the T6 condition. Microstructural examinations revealed the presence of strain-free grains slightly elongated in the rolling direction (RD) and flattened in the normal direction with dimensions of 60 μm × 47 μm × 4 μm. Orientation characterisation showed a pronounced cube texture with some scatter along RD.

From the as-received alloy sheet, two pieces 180 mm long and 30 mm wide were cut and cleaned with methyl ethyl ketone. One was put on top of the other and they were fastened with steel wires. The resulting 4-mm thick specimen was held for 5 min at 350 °C in a preheated electric furnace and straight afterwards rolled by a single pass to a X:1 (X = 2, 3 or 4) thickness reduction, equivalent to a rolling true strain of 0.7, 1.1 and 1.4, respectively. The rolling true strain (ε_r) was calculated as $\ln(h_0/h)$, where h_0 is the initial thickness and h is the final thickness. Then, the rolled sample was divided into identical pieces that were supplied to the next ARB pass. From then on, the procedure described above was repeated several times maintaining the rolling direction. Rolling was performed in non-lubricated conditions using a two-high mill with a roll diameter of 131 mm and a peripheral speed of 346 mm/s. After each rolling pass, the ARBed samples were immediately water cooled. The maximum number of passes was 8 for R_p = 2:1, 6 for R_p = 3:1 and 3 for R_p = 4:1. The ARBed samples were designated by a prefix indicating the number of passes (N) followed by the applied thickness reduction per pass. Hence, the 3p-3:1 sample was processed by three passes of 3:1 thickness reduction.

Microstructural examinations on the ARBed samples were performed by TEM in a JEOL JEM 2100 microscope operating at 200 kV. The macrotexture was analysed by XRD. The (111), (200) and (220) pole figures were measured using Cu$K\alpha$ radiation in a Siemens D500 diffractometer equipped with an open Euler ring working with Schultz geometry. From these experimental data, the orientation distribution functions (ODFs) were derived by means of the series expansion method using the TexTools software. EBSD was also carried out using a field emission gun scanning electron microscope (JEOL 6500 F FEG-SEM) operating at 20 kV, with a sample tilt of 70° and a working distance of 15 mm. The EBSD analyses were completed using the commercially available HKL Channel 5 software. Because of the limited angular resolution of the EBSD system, misorientations below 2° were neglected. For TEM investigations, disks of 3 mm in diameter were thinned to perforation using a twin-jet electropolishing facility with a solution of 30% nitric acid and 70% methanol at 15 V and –5 °C. Samples for EBSD and XRD preparation were ground and polished using standard metallographic techniques. Afterward, the specimens for EBSD inspections were electropolished for some seconds in the same solution that was used for the TEM specimens at 15 V and –15 °C.

Tensile dogbone specimens with a gauge length of 10 mm, width of 3 mm and thickness varying from 1 mm to 2 mm, depending on the thickness reduction per pass, were electro-discharge machined from the as-received sheet and the ARBed samples with their loading axis perpendicular to the RD. Uniaxial tensile tests were performed up to failure at 250, 300 and 350 °C using a Servosis ME 405/10 and an Instron 1362 testing machine equipped with an elliptical furnace provided with four quartz lamps. Before the start of the tests, the specimens were kept for ~20 min at the testing temperature, which was measured using a thermocouple clamped close to the specimens. Upon completion of the tests, the specimens were immediately water-cooled to preserve the microstructure. A set of tests was performed at a constant crosshead speed corresponding to the initial strain rate of $10^{-2}\,s^{-1}$. In this case, the elongation to failure (e_F) was determined from the true stress (σ)–true strain (ε) curves. Another set of tests was performed using the strain rate change (SRC) method. First, the initial strain rate is consecutively reduced in eight steps from $10^{-1}\,s^{-1}$ to $10^{-5}\,s^{-1}$. Then, the strain rate is consecutively increased in three steps from $10^{-5}\,s^{-1}$ up to $10^{-2}\,s^{-1}$. From the SRC tests the true strain rate ($\dot{\varepsilon}$)–true stress (σ) pairs corresponding to the decreasing strain rate steps were extracted in the steady state. Using these data, the apparent stress exponent (n_{ap}) was determined as the slope at three points of the $\dot{\varepsilon}$–σ data at a given temperature (T) by the following expression:

$$n_{ap} = \left.\frac{\partial \ln \dot{\varepsilon}}{\partial \ln \sigma}\right|_T \quad (1)$$

The surfaces of the tested samples were examined in a HITACHI S 4800 cold FEG-SEM.

3. Results

TEM micrographs showing the evolution of the microstructure with N at each R_p are displayed in Fig. 1. It is observed that the microstructure evolves from a cell structure with a very high dislocation density to a structure composed of more misoriented boundaries