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Strategy for severe friction stir processing to obtain acute grain refinement of an Al–Zn–Mg–Cu alloy in three initial precipitation states

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ABSTRACT

An Al–Zn–Mg–Cu, Al 7075, alloy was subjected to friction stir processing (FSP) using several processing conditions, two different backing anvils and three initial precipitation states in order to reach the maximum feasible processing severity to produce ultrafine grain sizes. Microstructures formed by fine, equiaxed and highly misoriented grains were obtained. Grain sizes were situated in the range of 200–1000 nm, making FSP competitive with other severe plastic deformation techniques. No influence of the initial precipitation state in the processed grain size was perceived. In fact, the processing conditions and the cooling rate determine the observed grain size. It was found that the selection of the appropriate processing conditions delivered an ultrafine grain size, thus allowing suitable microstructural control.

1. Introduction

The Al 7075 alloy is an age-hardenable aluminium alloy belonging to the Al–Zn–Mg–Cu family. The excellent combination of high strength-density ratio with excellent mechanical properties makes this alloy a reference in the aeronautical industry [1,2]. Nevertheless, it possesses a limited formability by conventional forging at elevated temperature. Thus, a convenient way of forming this alloy is by means of superplastic forming (SPF) techniques, which take advantage of the benefits of superplastic deformation. Superplasticity is known as the ability of a polycrystalline material to exhibit, in a general isotropic manner, very high tensile elongations thanks to the activation of the grain boundary sliding (GBS) mechanism. This mechanism operates in a determined strain rate ($\dot{\varepsilon}$) and temperatures range, known as the "superplastic window". The operation of this mechanism requires fine (usually <15 μm), equiaxed and highly misoriented grains and is characterized by low flow stresses (σ) and low stress exponents ($n \sim 2$) [3,4]. Eq. (1) is the constitutive equation for this mechanism, where $\dot{\varepsilon}$ is the strain rate, K is a constant, σ is the flow stress, E is the elastic modulus, b is the Burgers vector, L is the grain size, p is the grain size exponent, Q is the activation energy, R is the universal gas constant and T is the temperature. In general, the values of p and Q are $p = 3$ and $Q = Q_{GB}$ when $0.4T_m < T < 0.6T_m$ and $p = 2$ and $Q = Q_L$ when $T > 0.6T_m$, [5]. It can be inferred from Eq. (1) that the finer the grain size (L), the higher the strain rate ($\dot{\varepsilon}$) and the lower the temperature where GBS can operate, highlighting the importance of the microstructure refinement for an efficient superplastic response.

$$\dot{\varepsilon} = K\left(\frac{\sigma}{E}\right)^2\left(\frac{b}{L}\right)^p \exp\left(-\frac{Q}{RT}\right). \tag{1}$$

Consequently, SPF allows obtaining pieces with complex shapes and homogeneous thickness, using lower flow stresses than in conventional forming processes, with the corresponding energy saving. Nevertheless, to perform SPF, it is necessary a previous thermomechanical treatment to obtain an appropriated microstructure able to sustain grain boundary sliding. In this regard, a recommendable thermomechanical processing to obtain such fine microstructures is through severe plastic deformation (SPD) techniques, which are an attractive and cost-effective way to achieve fine microstructures.

SPD is a generic term describing a group of metalworking techniques involving very large strains which are imposed without introducing any significant changes in the overall dimensions of the specimen or workpiece to obtain ultrafine or nanostructured microstructures [6,7]. In this way, during recent years, friction stir processing (FSP) has been developed as one of the most promising techniques to obtain fine microstructures in large sheets [8,9].

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FSP was developed on the basic principles of friction stir welding (FSW) which is a solid state joining process developed in 1991 by Wayne Thomas at The Welding Institute (TWI) in the UK [10]. FSP consists in traversing a rotation tool, formed by a pin and shoulder, inserted in the material across a worksheet, producing intense plastic deformation through the friction between the tool and the worksheet. This processing generally results in a deformed zone with a fully recrystallized, equiaxed and very fine grain size [11–13]. Nevertheless, grain sizes obtained by FSP are in general larger than 2 μm [14–16], well over the size range commonly considered in ultrafine-grained materials, 0.1–1 μm [17,18], which will provide a much better superplastic response as predicted by Eq. (1).

Although the overall process seems to be simple, the optimization of FSP is a difficult issue due to the combination of processing parameters that affect the final microstructure. In general, the processing conditions selected for FSP are not usually appropriate for grain refinement due to the high energy input that they impose. High rotation speeds lead to a high frictional heat generation and low traverse speeds hinder heat dissipation leading to grain growth. However, those processing conditions are commonly selected because they assure no processing flaws in the processed material. Therefore, a systematic experimental procedure for the optimization of the process parameters is the best option to reach the finest suitable microstructures. Some recent publications have dealt with the optimization of FSP conditions in order to reach superior mechanical properties [19–20]. However, these publications do not come into aspects related to the attainment of the finest microstructures.

The first objective of this study is establishing a methodological strategy to produce the finest feasible microstructures, exploring the FSP severity limits. The second objective is to determine the influence of the initial precipitation state of the alloy in the final processed microstructure, as this issue remains still uncertain. In order to achieve both objectives, four combinations of processing conditions over two different cooling anvils, which are applied to three different initial precipitation states are proposed. This study will permit determination of the appropriate processing conditions to obtain a desired grain size, allowing FSP to be performed with microstructural control and establishing the influence of the initial precipitation state.

2. Experimental procedure

A commercial 7075 alloy (Al–5.68 wt.% Zn–2.51 wt.% Mg–1.59 wt.% Cu–0.19 wt.% Cr–0.19 wt.%Fe–0.052 wt.% Si–0.025 wt.% Ti–0.007 wt.% Mn) prepared as 3 mm thick rolled sheets was chosen for investigation. Three precipitation states were studied: T6 treatment (7075-T6), solution treatment (7075-S) and over-aged treatment (7075-O), obtained as follows:

- 7075-T6: solution treatment at 465 °C for 1 h, quenching in water at 40 °C maximum temperature and artificial ageing for 12–16 h at 135 °C.
- 7075-S: starting from 7075-T6 state, solution treatment at 465 °C for 12–15 h, quenching with N_2 (8 bar).
- 7075-O: starting from 7075-T6 state, ageing treatment at 265 °C for 13 h, furnace cooling.

Sheets of dimensions 290 × 110 × 3 mm in the three precipitation states were subjected to FSP using a *MTS PDS-4 Intelligent-Stir* equipment. The tool used was made of MP159 and the design includes a scrolled shoulder 9.5 mm in diameter and a concentric threaded pin with flutes 3 mm in diameter and 2 mm in length. Two backing anvils were selected in order to perform processing at two different cooling rates. The first one is a conventional martensitic stainless steel anvil (A) where the sheet cools at room temperature. The second one consists in a copper anvil refrigerated by liquid nitrogen (C), generating a much faster cooling rate. Table 1 shows the different combinations of rotation rate (ω) and traverse speed (V) used in the present study, including the designated code for each combination. The given order of these combinations corresponds to a progressive decrease in the heat index and linear energy [21], and therefore, an increase in the processing severity.

The nomenclature used in the present study for each processing condition include, first, the used backing anvil (A for the conventional uncooled anvil and C for the cooled anvil refrigerated with liquid nitrogen), second, the processing code (extracted from Table 1) and finally the initial precipitation state (T6, O and S); for example C07r10v-O corresponds to the material in the 7075-O precipitation state, processed over the copper anvil refrigerated with liquid nitrogen, using a rotation rate of 700 rpm and a traverse speed of 1000 mm/min.

The three initial precipitation states and the friction stir processed transversal section microstructures were analysed by means of optical microscopy (OM), model *Olympus BH-2*. The samples were grounded and polished to a 1 μm finish followed by Keller etching. The microstructure of the initial materials and the friction stir processed stir zone was analysed by means of transmission electron microscopy (TEM), model JEOL JEM 2000 FX II, operating at 200 kV. TEM samples were ø 3 mm discs, electropolished using a *Struers Tenupol 5* operating at 12 VDC, at −25 °C and using a solution of 30% vol. HNO_3 − 70 vol.% CH_3OH until light detection.

Grain sizes were measured using the Sigma Scan Pro software in TEM images. More than 400 grains for each processing condition were analysed. Size distribution histograms were obtained from these measurements. Data fell into lognormal distributions, so the geometric mean value was chosen as the measure of the grain size.

3. Results

Fig. 1 shows optical micrographs from the three starting precipitation states in the ND–TD plane. OM shows a fully recrystallized microstructure composed of elongated-shaped grains with dimensions of 60–100 μm in TD and approximately 10 μm in ND. Additionally, large irregular-shaped particles, 5–10 μm in size are observed in the three samples, corresponding to particles consisting mainly of Al–Cu compounds containing Fe and Si, which are very resistant to dissolution, often referred to as “constituent” particles [22,23].

Fig. 2a–c displays TEM micrographs from the three starting precipitation states showing small dispersoid particles, 100–200 nm in size. The dispersoid particles have the composition $Al_{18}Mg_3Cr_2$ [22]. The TEM micrographs show that the 7075-T6 has the highest dispersoid volume fraction, followed by the 7075-O and 7075-S. In addition, the dispersoid size is slightly greater in the 7075-O than in the other two precipitation states. A second type of particle, fine and homogeneously distributed is found in the 7075-T6 in Fig. 2d, known as strengthening precipitates. This phase, ranging 10-100 nm in size and composed mainly by $MgZn_2$, is responsible for the exceptional precipitation hardening behaviour of the 7xxx series alloy [24].

Fig. 3 corresponds to an OM micrograph showing the friction stir processed transversal section for the condition A10r05v-T6. As a result of the tool action, distinct regions can be distinguished: the red dashed-line surrounded area corresponds to the stir zone (SZ), where the pin tool interacts directly with the material; the orange dashed-line surrounded area highlights the thermomechanical affected area

Table 1
Combinations of rotation rate and traverse speed selected in this study and the designation code.

ω (rpm)	V (mm/min)	Code
1400	500	14r05v
1000	500	10r05v
1000	1000	10r10v
700	1000	07r10v