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Grain size versus microstructural stability in the high strain rate superplastic response of a severely friction stir processed Al-Zn-Mg-Cu alloy

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ABSTRACT

An Al-Zn-Mg-Cu, Al 7075, alloy in an overaged state was subjected to friction stir processing (FSP) using a wide range of processing conditions. The goal was to diminish the heat index, increasing therefore the processing severity. Additionally, two backing anvils were selected that strongly influence the heat extraction rate. Grain sizes obtained by transmission electron microscopy were situated in the range 210–820 nm. High temperature tensile tests and the scanning electron micrographs of the topography of the tensile samples after testing revealed that grain boundary sliding (GBS) operated at an initial strain rate of $\dot{\varepsilon}=10^{-2}\ \mathrm{s}^{-1}$ in a wide range of temperatures for the processed alloy. That temperature window was wider and situated at lower temperatures for the finer grain sizes. The high processing severity imposed to create such fine microstructures introduces high values of stored energy in the material, which is the driving force for the increasing grain coarsening at high temperatures. This fact limited the operation of GBS at the highest tested temperatures in some conditions. An optimum balance between grain refinement and microstructural stability was obtained using a low heat index, corresponding to the processing conditions of ω=1000 rpm, V =500 mm/min, and the use of refrigerated backing anvil.

1. Introduction

Mechanical properties of metallic materials are highly sensitive to the grain size. At room temperature, according to the Hall-Petch relationship, a reduction in the grain size causes and increase of the yield strength [1,2]. At high temperatures, fine grain sizes are required to obtain a good superplastic response at high strain rates and relatively low temperatures [3]. In this regard, friction stir processing (FSP) is a severe plastic deformation technique based on the concepts of friction stir welding (FSW) [4] that has been demonstrated to be capable to induce ultra-fine grain sizes (<1 μm) when using severe processing conditions [5–8]. During FSP a cylindrical rotating tool with a concentric pin and shoulder is plunged in the material and traversed along the line of interest. The friction between the rotating tool and the work-piece produce a localized heating becoming the material easily plastically deformable. Therefore, the material undergoes intense plastic deformation at elevated temperature resulting in significant grain refinement when selecting appropriate conditions.

The material selected in this study is a commercial 7075 alloy, belonging to the 7xxx series aluminum alloys, based on the Al-Zn-Mg-Cu alloy system. The age-hardenable 7075 alloy is one of the strongest wrought aluminum alloys due to the high volume fraction of fine second phase particles [9]. However, it possesses a limited formability by conventional forging at elevated temperature [10]. Therefore, an acute grain refinement to enhance a superplastic response at forming temperatures is highly desirable.

In this regard, a convenient way of giving shape such materials is by superplastic forming (SPF) [11]. SPF is usually performed to obtain complex shapes with a constant thickness using much lower stresses than conventional forming processes [12]. This processing requires the activation of grain boundary sliding (GBS) as the main deformation mechanism in the processed material. The GBS mechanism is responsible for superplasticity, which is the ability of a polycrystalline material to exhibit, in a general isotropic manner, very high tensile elongations prior to failure in a determined strain rate and temperatures range, known as the "superplastic window". Additionally, high strain rate superplasticity (HSRS) is conventionally referred to the achievement of elongations over 200% at strain rates $\geq 10^{-2}\ \mathrm{s}^{-1}$ [13],

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being highly desirable for an efficient superplastic forming.

Some previous studies have shown the possibilities of FSP to obtain low temperature HSRS by reaching submicron grain sizes, ~0.7–0.8 [14,15]. However, the superplastic deformation can be hindered if the microstructure presents thermal instability. The study of the thermal stability of the processed alloys is usually carried out using static annealing processes [16–18]. It is not true that the microstructural evolution during a mechanical test could not be the same as in a static process and affect the superplastic response, as was demonstrated first in the study by Perevezentsev et. al [19] and later considered by Miles et. al [20] for superplastic deformation in general and in the particular case of SPD by Spigarelli et. al and Figueiredo et. al [21,22]. Therefore, the thermal microstructural stability under superplastic deformation for an optimum superplastic behavior must be considered as a main issue.

In this study a commercial 7075 alloy in an overaged state (showing minimum hardness) was subjected to FSP under four processing conditions. The goal was to diminish the heat index and thus, increasing the processing severity [8,23,24]. Additionally, two backing anvils, with different heat extraction rates were selected, leading to a total of eight studied conditions. The aim of choosing such a wide variety of processing conditions in this research is to find an optimum for improved superplastic performance. In this work we show that in terms of applicability of SPF, it is not only convenient to decrease as much as possible the grain size but also to get a proper microstructural stability by balancing the superplastic response due to the ultrafine grain size and its microstructural stability in dynamic conditions at high temperatures.

2. Materials and experimental method

The material used in the present study was Al 7075 (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, bal. Al) aluminum alloy in an overaged state in the form of 3 mm thickness sheets. The overaging treatment was conducted starting from the commercial 7075-T6 state, aging treated at 265 °C for 13 h and furnace cooling. The grain size was ~60–100 μm in the rolling direction (RD) and ~10 μm in the transverse direction (TD). The sheets were then subjected to FSP using four different combinations of rotation rate (ω) and traverse speed (V). These combinations were selected by reducing by half the heat index [25] from the hottest to the coldest feasible combination of processing conditions, and therefore an increase in the processing severity. Additionally, two backing anvils with different cooling rate were used. The nomenclature for the eight processing combinations is presented in Table 1. The tool was made of MP159 superalloy and possessed a scrolled shoulder 9.5 mm in diameter and a concentric threaded pin with flutes 3 mm in diameter and 2 mm in length.

The high temperature mechanical behavior was characterized by means of constant crosshead speed tensile tests. Planar dog-bone tensile samples with 6 mm×2 mm×1.8 mm gage dimensions were electro-discharge machined along the traverse direction. The sample geometry and size were selected for including the area of interest: *nugget* or stir zone (SZ). Therefore, sample dimensions should be smaller than the SZ in order to test just this area. Additionally, when obtaining superplasticity, samples can reach huge elongations that could exceed the available displacement of the testing machine. Thus, superplastic small samples can be tested within the displacement limits unlike longer gauge samples and their use is a common practice in this field [14,26–28]. The samples were tested at elevated temperatures (200–450 °C) and at constant crosshead speed of 0.065 mm s^{-1}, equivalent to an initial strain rate ($\dot{\varepsilon}$) of 10^{-2} s^{-1}, in a universal Instron 1362 testing machine equipped with a four-lamp ellipsoidal furnace. The samples were grounded and polished to a 1 μm finish prior to the mechanical testing.

Table 1
Combinations of backing anvil, rotation rate and traverse speed selected in this study and the designated nomenclature.

Backing anvil	ω (rpm)	V (mm/min)	Heat index (rpm^2/(mm/min))	Nomenclature
Conventional martensitic stainless steel (A)	1400	500	3920	A14r05v
	1000	500	2000	A10r05v
	1000	1000	1000	A10r10v
	700	1000	490	A07r10v
Copper refrigerated with liquid nitrogen (C)	1400	500	3920	C14r05v
	1000	500	2000	C10r05v
	1000	1000	1000	C10r10v
	700	1000	490	C07r10v

Transmission electron microscopy (TEM) was selected for the characterization of the fine microstructure just after FSP. The used TEM model was a JEOL JEM 2000 FX II operating at 200 kV. TEM samples consisted in ϕ=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, analysing more than 400 grains for each processing condition. 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.

The characterization of the samples topography after high temperature testing was carried out in a scanning electron microscope (SEM), model Hitachi Cold FEG S-4800.

3. Results

3.1. Microstructure

Fig. 1 presents grain size values as Feret diameter for the processed samples including the confidence interval under different processing conditions. It can be observed that processing over the refrigerated backing anvil leads to significantly higher grain refinement than processing over the conventional anvil. Additionally, increasing the processing severity towards lower heat indexes for each backing anvil produce finer microstructures. Additionally, TEM micrographs from the conditions A14r05v and C07r10v are presented in Fig. 1 to show the huge difference in grain size between these two extreme conditions, 820 vs. 210 nm respectively. The condition A14r05v with the highest energy input (3920 rpm^2/(mm/min), highest heat index in Table 1) and the use of the non-refrigerated backing anvil (A) presents the coarsest microstructure, with nearly equiaxed grains. The grain boundaries are well defined, indicating high misorientation values. On the other hand, the condition C07r10v with the lowest energy input (490 rpm^2/(mm/min), lowest heat index in Table 1) and the use of the refrigerated backing anvil (C) presents the finest microstructure. In addition, this condition presents less misorientation than the condition A14r05v, indicated by boundaries slightly less defined. The rest of conditions show progressive microstructural evolution among these two limit conditions [8].

3.2. High-temperature mechanical properties

Fig. 2 presents stress-strain curves at temperatures ranging 200–450 °C and at a constant crosshead speed equivalent to an initial strain rate of 10^{-2} s^{-1} for the overaged 7075 alloy. The σ-axis and the ε scales ranging 0–350 MPa and 0–2, respectively, were selected in order to make more evident the comparison with the figures of the processed material (Fig. 3). A decrease in yield stress ($\sigma_{0.2}$) and in maximum flow stress (σ_{max}) with increasing testing temperature can be observed. It is worth to mention that the overaging treatment leads to a significant