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Severe friction stir processing of an Al-Zn-Mg-Cu alloy: Misorientation and its influence on superplasticity

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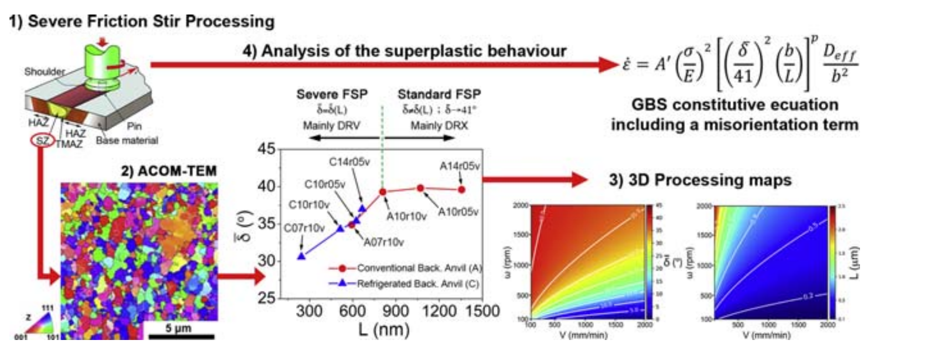
HIGHLIGHTS

- Increasing friction-stir-processing-severity conditions achieved various ultrafine grain sizes and misorientations.
- Standard and severe FSP regimes were identified based on the grain size–misorientation relationship obtained.
- 3D friction stir processing contour maps for grain size, misorientation and yield stress were obtained.
- A new GBS constitutive equation including misorientation for SPDed alloys is proposed.

GRAPHICAL ABSTRACT



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ABSTRACT

An overaged Al 7075 alloy was severe friction stir processed using two different backing anvils using different heat extraction rates. A range of processing parameters ω (rotation speed) and V (traverse speed) involving low heat index (*HI*) for obtaining severe processing conditions were employed. As a result, we obtained microstructures with a wide variety of very fine grain sizes and misorientations that gave rise to very different values of stresses and elongations. We found a relation between mean misorientation ($\bar{\delta}$) and grain size (L) that allows discerning two processing regimes: severe ($\bar{\delta} = \bar{\delta}(L)$) and standard ($\bar{\delta} \simeq 41°$). Additionally, we found analytical relations between the misorientation, the grain size and the FSP parameters that predict misorientation and grain size processing maps for each backing anvil. The further experimental data analysis of high temperature tensile tests provided the relationship $\sigma_y = \sigma_y(\omega,V)$ in the grain boundary sliding (GBS) region considering the influence of the misorientation in the superplastic behavior. Finally we propose a modified GBS constitutive equation including the influence of the misorientation. This equation describes the superplastic behavior in the severe FSP region (low *HI*), while it tends to the classical GBS constitutive equation in the standard FSP region (high *HI*).

1. Introduction

Superplasticity is the ability of a polycrystalline material to exhibit, in a general isotropic manner, very high tensile elongations prior to failure, generally at low flow stresses and in a determined strain rate and temperatures range, known as the "superplastic window" [1,2]. The benefits of superplastic deformation are the basis of the superplastic forming (SPF) [3–6], which can be used for the processing of several

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alloys with limited formability by conventional forging at elevated temperature such as the 7075 aluminum alloy. This age-hardenable alloy belongs to the 7xxx series aluminum alloys, based on the Al-Zn-Mg-Cu alloy system and is one of the strongest wrought aluminum alloys due to the high volume fraction of fine second phase particles and widely used in the aerospace industry [7,8].

It is well known that the mechanism responsible of superplasticity is grain boundary sliding (GBS), which constitutive equation is [9]:

$$\dot{\varepsilon} = A\left(\frac{\sigma}{E}\right)^n \left(\frac{b}{L}\right)^p D_{eff} \quad (1)$$

where $\dot{\varepsilon}$ is the strain rate, A is a constant, σ is the flow stress, E is the Young's modulus, $n = 2$ is the stress exponent, b is the Burgers vector, L is the grain size, p is the grain size exponent and D_{eff} is the effective diffusion. Following Hart [10] and Robinson and Sherby [11], D_{eff} can be expressed for GBS as $D_{eff} = f_L D_L + f_{GB} D_{GB}$ where D_L is the lattice-self diffusion, D_{GB} is the diffusion along the grain boundaries and f_L and f_{GB} are the fraction of atoms sites associated to each type of diffusion. At approximately $0.4T_m < T < 0.6T_m$, $p = 3$ and $D = D_{GB}$ (diffusion along the grain boundaries), while for $T > 0.6T_m$, $p = 2$ and $D = D_L$ (lattice-self diffusion) [9].

There are two main requirements for a material to deform by GBS. The first one is fine grain size, in such a way that as predicted by the constitutive equation, the finer the grain size the higher the strain rate and the lower the temperature where GBS could be operative [12]. Additionally, a second requirement is high misorientation values in order to ease the sliding between grains and accommodate the high deformation values, avoiding premature necking [1,2,9,13,14]. While the influence of grain size on superplasticity has been widely studied [15–22], the role of misorientation remains uncertain and only few studies have pointed out its importance. Watanabe et al. studied the misorientation dependence in grain boundary sliding for specific types of boundaries by testing bicrystal zinc samples and determined that specific high misorientation ranges in this material lead to higher sliding rates [23]. In a further study, Watanabe determined that the contribution of grain boundary sliding to the total amount of creep increases in general with increasing misorientations (with the exception of some specific coincident boundaries) [24]. Additionally, Somekawa and Mukai reported that the amount of sliding increases with grain boundary energy [25]. Kokawa et al. studied the evolution of grain boundaries during deformation in coarse-grained polycrystal and bicrystal aluminum and determined that different misorientation can lead to different hardening rates during superplastic deformation [26]. More recently, in the revealing studies by Tóth and coworkers, the misorientation evolution during increasing plastic strain during severe plastic deformation is studied in detail but no direct relation with the further mechanical behavior was presented [27,28]. These kinds of studies are crucial from the grain boundary-engineering point of view and relevant at the microscale, but insufficient for understanding the influence of the mean misorientation on the superplastic properties at the macroscale. In general, superplasticity studies assume that the higher the misorientation the better the superplastic performance [1,29], but there is a lack of quantitative studies in this regard. Possibly, one of the reasons behind this is that misorientation is commonly measured using electron back-scattered scanning diffraction (EBSD), which is based on the acquisition of Kikuchi patterns that are difficult to resolve in materials with microstructures prone to GBS, as they are usually formed by ultra-fine grains with high dislocation densities [30,31]. The most immediate way to overcome this issue is using an alternative technique, such as the automated crystal orientation-mapping tool attached to a transmission electron microscope, also known as ACOM–TEM [32]. This technique analyzes diffraction spot patterns instead of the Kikuchi lines used in EBSD, as they are less sensitive to the internal stresses resulting from the complex thermomechanical processing required to produce such microstructures [32–36].

Microstructures prone to GBS can be obtained by friction stir processing (FSP), a severe plastic deformation technique based on the concepts of friction stir welding (FSW) [37] and first described by Mishra et al. [38]. During FSP a cylindrical rotating tool, formed by a concentric pin and shoulder, is punched in the material and traversed along the line of interest. The friction produced between the tool and the workpiece creates localized heating that favors plastic deformation of the material, resulting in significant grain refinement and an increase in the mean misorientation [39–41], which has been demonstrated to be especially effective in aluminum alloys [14,42–47].

In this study, the 7075 alloy has been processed by FSP in a range of different processing severities, obtained by combining different processing conditions and two backing anvils with different cooling rates, in order to obtain a collection of microstructures with different grain sizes and misorientations. The proper measurement of the misorientation with ACOM-TEM and the study of further superplastic behavior were used to answer the following questions: i) What is more important for an optimum superplastic performance, grain size or misorientation? ii) Is it enough to just consider the classical approach where grain size is the only key parameter? iii) Is the commonly disregarded misorientation value playing an important influence? The results reveal the role of the misorientation in the superplastic behavior and how it is related to grain size. Additionally, misorientation, grain size and superplastic flow stress maps are constructed from the relations between the processing parameters–microstructure–mechanical performance. Finally, we propose a modified GBS constitutive equation including the influence of the misorientation.

2. Materials and experimental method

We used 3 mm thickness rolled sheets of a commercial 7075-T6 aluminum alloy, with chemical composition is shown in Table 1.

The alloy, in the initial commercial T6 temper, was subjected to an overaging treatment at 265 °C for 13 h and furnace cooling in order to obtain thermally stable precipitates. This temper is the starting material and designated as 7075-O. 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). We selected these combinations by halfing the heat index (HI), $HI \propto \omega^2/V$ [48] from the hottest to the coldest feasible combination of processing conditions, and thus, increasing the processing severity. While HI is more indicative of the processing temperature, the linear energy (LE) is more related to the processing strain during FSP. LE is defined as the tool rotation rate divided by the tool traverse speed, $LE = \omega/V$, and is the inverse of the weld pitch, i.e. the tool advance per rotation. It is reflected macroscopically as the spacing between the characteristic FSP/W "scars". When selecting the processing conditions it is critical to consider the definitions of HI and LE, as for instance, two different processing combinations could have different HI but identical LE, i.e. the combination 2000 rpm | 1000 mm/min has $HI = 4000$ rev^2/(mm·min^{-1}) and $LE = 2$ rev/min while the combination 1000 rpm | 500 mm/min has $HI = 2000$ rev^2/(mm·min^{-1}) and $LE = 2$ rev/min. This means that while both combinations impose similar deformation values (identical LE), the heat input is double in the first combination than in the second (double HI). This fact can have further implications in the evolution of the precipitates shapes and sizes or in the thermal stability of the processed material [49]. Therefore, in our study we selected our processing combinations not only to decrease the HI but also LE. The nomenclature for the four processing combinations is presented in Table 2. Additionally, we selected

Table 1
Chemical composition (wt%) of the Al 7075 alloy.

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