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Influence of grain coarsening on the creep parameters during the superplastic deformation of a severely friction stir processed Al-Zn-Mg-Cu alloy

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Abstract

During grain boundary sliding in ultrafine-grain materials at intermediate temperatures and high strain rates ($\sim 10^{-2} \text{ s}^{-1}$) apparent creep parameters usually deviate from the theoretical values, due to microstructural coarsening. An analysis has been carried out in a severely friction stir processed (FSP) 7075 alloy with three different ultra-fine grain sizes (L), obtaining explicit grain size dependence of the creep parameters $n_{ap}=n_{ap}(L)$ and $Q_{ap}=Q_{ap}(L)$, confirming the validity of the theoretical values of these parameters in the constitutive equation.

Keywords: aluminum alloys; friction stir processing; grain boundary sliding; grain growth; creep

Main Body:

Bulk nanostructured materials usually present good superplastic response at high strain rates and relatively low temperatures due to their exceptional microstructures in the form of highly misorientated ultrafine grains [1–6]. In this regard, friction stir processing (FSP) is a severe plastic deformation (SPD) technique based on the concepts of friction stir welding (FSW) [7] capable of producing ultra-fine grain sizes ($< 1 \mu\text{m}$) using appropriate parameters [8–12]. The effects of FSP on the microstructure modification and further mechanical properties and formability have been extensively studied in the Al 7075 alloy [11–20].

Two deformation mechanisms usually operate at intermediate to high temperatures in fine grain materials containing precipitates and solutes, like the Al 7075 alloy: constant substructure slip-creep [21] and grain boundary sliding (GBS) [1]. In this study we focus on GBS, which requires fine ($\lesssim 15 \mu\text{m}$), equiaxed and highly misoriented grains [22,23]. Its constitutive equation is [1]:

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

where $\dot{\epsilon}$ is the strain rate, K is a constant, σ is the flow stress, E is Young's Modulus, b is Burgers vector modulus, L is the grain size, p is the grain size exponent, Q is the activation energy, R is the ideal gas constant and T is the temperature. The stress exponent is $n=2$ for GBS. At approximately $0.4T_m < T < 0.6T_m$, $p=3$ and $Q=Q_{GB}$, while for $T > 0.6T_m$, $p=2$ and $Q=Q_L$ being T_m the absolute melting temperature.

However, the theoretical n and Q values are not observed under certain testing conditions [24,25]. On one hand, fine L can lead to the occurrence of GBS at temperatures and $\dot{\epsilon}$ over the power law breakdown ($\text{PLB} \equiv \dot{\epsilon}/D_L > 10^{13} \text{m}^{-2}$) [26], showing $n_{ap} > 2$ and $Q_{ap} \neq Q_{GB}, Q_L$ [27–31]. On the other hand, grain coarsening during the superplastic deformation alters the resulting values of the apparent creep parameters [33]. In this study, superplastic ultra-fine grained FSPed Al7075 alloy data showing non-model n_{ap} and Q_{ap} values are rationalized taking into account the L dependence of the GBS constitutive equation.

We used 3 mm thickness sheets of a commercial 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 the T6 temper, with average L~60-100 μm in RD and ~10 μm in TD, that were subjected to FSP as described elsewhere [11]. Three different L were obtained using different processing conditions, i.e. L(1) = 1065 nm (1400 rpm, 500 mm/min, conventional backing anvil), L(2) = 530 nm (1000 rpm, 500 mm/min, liquid nitrogen refrigerated backing anvil) and L(3) = 385 nm (1000 rpm, 1000 mm/min, liquid nitrogen refrigerated backing anvil). For discussion purposes, the L(2) condition has been selected as the reference material.

The high temperature, 473.15-723.15 K (200-450 °C), mechanical behavior was characterized by means of constant crosshead speed tensile tests (CCST) equivalent to an initial $\dot{\epsilon}=10^{-2} \text{ s}^{-1}$ which have been compensated to an equivalent constant $\dot{\epsilon}$ at 10^{-2} s^{-1} throughout the test (constant strain rate tests, CSRT), and by strain-rate-change tensile tests (SRCT) ranging $\dot{\epsilon}=10^{-1}-10^{-5} \text{ s}^{-1}$. Since $\dot{\epsilon}$ decreases with increasing ϵ in CCST, the observed σ values correspond to different $\dot{\epsilon}$ when deforming the material, which is noticeable at the high ϵ values usually considered in superplastic materials. In our calculations, it is crucial to use σ - ϵ data keeping $\dot{\epsilon}$ constant. Therefore, we first calculate the true $\dot{\epsilon}$ ($\dot{\epsilon}_t$) for each deformation as $\dot{\epsilon}_t=v/(l_0+d)$, where $v=0.065 \text{ mm/s}$ is the crosshead speed, $l_0=6.5 \text{ mm}$ is the initial gauge length and d is the instant displacement. Once $\dot{\epsilon}_{\text{true}}$ is known, the CCST stress (σ_{CCST}) can be compensated by comparing Eq. [1] at two different $\dot{\epsilon}$, assuming no change in the deformation mechanism during the test. Therefore, the compensated stress (σ_{comp}) can be obtained using $\sigma_{\text{comp}} = \sigma_{\text{CCST}}(\dot{\epsilon}_i/\dot{\epsilon}_t)^{1/n_{\text{ap}}}$, where $\dot{\epsilon}_i=10^{-2} \text{ s}^{-1}$ is the initial $\dot{\epsilon}$ and n_{ap} is the apparent stress exponent, obtained from the CSRT (see Fig. 2 description). The tests were performed using a universal Instron 1362 testing machine equipped with a four-lamp ellipsoidal furnace. Dog-bone tensile samples with $6 \times 2 \times 1.8 \text{ mm}^3$ gage dimensions were electro-discharge machined along the traverse processing direction in such a way that the gage section is fully inside the stir zone. The topography of the samples after testing was evaluated in a Hitachi Cold FEG S-4800 microscope. Grain sizes were obtained as area values and then converted into Feret diameter using Sigma Scan Pro software in more than 400 grains.

True stress – true strain curves at temperatures ranging 573.15 to 673.15 K (300 to 400 °C) and an initial $\dot{\epsilon}=10^{-2} \text{ s}^{-1}$ for the microstructure $L(2) = 530 \text{ nm}$ are shown in Fig. 1. The material presents low flow stresses (σ), especially at 623.15 K (350 °C), and extraordinary elongations to failure (e_F) of 450 and 507 % at 573.15 and 623.15 K (300 and 350 °C) respectively, pointing to GBS as the deformation controlling mechanism. Nevertheless, while at 573.15 K (300 °C) σ remains quite constant from $\epsilon = 0.4$ to the beginning of necking, at 623.15 K (350 °C) σ increases during the whole plastic regime, from the yield point to the ultimate tensile strength (UTS). At 673.15 K (400 °C), a return to low e_F and higher σ is observed, pointing to slip creep as the dominant mechanism as discussed in [19] [32]. The topography after testing is also presented in Fig. 1. At 573.15 – 623.15 K (300 – 350 °C) the micrographs show the typical surface aspect of a material deforming by GBS, with equiaxed grains emerging from the sample interior. The observed L is ~ 1.2 and $\sim 1.9 \mu\text{m}$ at 573.15 and 623.15 K (300 and 350 °C), respectively. The microstructure at 673.15 K (400 °C) exhibits very coarse grains. This extreme coarsening leads to the change of mechanism provoking both large stress increment and ductility drop at 673.15 K (400 °C) [32].

Fig. 1. True stress – true strain (σ – ϵ) at a constant strain rate of 10^{-2} s^{-1} for the FSPed alloy with a grain size of $L(2) = 530 \text{ nm}$ at three different testing temperatures. On the top, micrographs corresponding to the topography of the samples after testing.

In Fig. 2, the $\dot{\epsilon} - \sigma$ data obtained from the SRCT at 473.15–673.15 K (200–400 °C) in the L(2) material have been normalized by the lattice self-diffusivity (D_L) and the Young's modulus (E) for each temperature [33], respectively. D_L was calculated by using the Arrhenius-type equation $D_L = D_0 \cdot \exp(-Q_L/RT)$, using pre-exponential factor (D_0) and lattice self-diffusion activation energy (Q_L) as $D_0 = 1.71 \cdot 10^{-4} \text{ m}^2$ and $Q_L = 142 \text{ kJ/mol}$ [34]. The data are compared with data from pure aluminum (open circles) [35]. A horizontal dashed line represents the PLB at $\dot{\epsilon}/D_L \approx 10^{13} \text{ m}^{-2}$ indicating roughly the upper limit for the applicability of power-law-like constitutive equations [26,36]. Pure aluminum $\dot{\epsilon}/D_L - \sigma/E$ data pairs below PLB are distributed along a line with a slope (n_{ap}) about 5, while at values of $\dot{\epsilon}/D_L > 10^{13} \text{ m}^{-2}$ the values of n_{ap} increase rapidly, being consistent with the operation of the mechanism of slip-creep with stress-dependent subgrain formation ($n \approx 5$) [26]. It is observed in the Fig. 2 that the L(2) material (color filled symbols) is stronger than pure aluminum in the entire temperature range. Regarding the n_{ap} values, at 473.15 K (200 °C) data are distributed along a high slope. The data group ranging 523.15–623.15 K (250–350 °C) lay on a sigmoidal curve. At the highest $\dot{\epsilon}$ at 523.15 K (250 °C) the data fall in a line of a slope around 8 and at lower $\dot{\epsilon}$, the slope tends to decrease to values close to 2. Finally, at extremely low $\dot{\epsilon}$ at 573.15 and 623.15 K (300 and 350 °C), the data is again distributed along a line with a higher slope. Regarding the data set for 673.15 K (400 °C), the material turns back to high σ and lay on a line with slope close to 8, aligned with the previous data for 473.15 K (200 °C). This behavior is consistent with the operation of two deformation mechanisms: i) constant substructure slip creep mechanism ($n \approx 8$) at 473.15 K (200 °C) and 673.15 K (400 °C) [21,37], and ii) GBS at 523.15–623.15 K (250–350 °C), centered at 573.15 K (300 °C) [38]. This mechanism change has been previously reported in similar severely deformed alloys [39–42], but this change is now around and above the PLB which is interesting from an industrial standpoint.

Fig. 2. $\dot{\epsilon}/D_L - \sigma/E$ data pairs obtained from the strain rate changes tensile tests at different temperatures for pure aluminum (open circles, obtained from [36]) and the processed Al 7075 alloy with an grain size in the stir zone after FSP of $L(2) = 530$ nm (color filled symbols).

In order to better understand the results shown in Fig. 2, we have plotted the variations of n_{ap} and Q_{ap} with temperature for the three FSP materials and compared with the as-received Al 7075-T6 alloy in Fig. 3. n_{ap} and Q_{ap} were calculated using the $\dot{\epsilon}-\sigma$ data pairs from the SRCT by applying the corresponding equations: $n_{ap} = \Delta \ln \dot{\epsilon} / \Delta \ln (\sigma/E)$ for each temperature and $Q_{ap} = nR(\Delta \log(\sigma/E) / \Delta(1/T))$ at $\dot{\epsilon} = cte$. The as-received alloy and L(1) present similar trends, with decreasing n_{ap} and Q_{ap} values when increasing the temperature. Both materials reach the minimum value of n_{ap} at 723.15 K (450 °C) but, while in the case of the unprocessed alloy n_{ap} is ~ 6 , this value is 2 for the processed L(1). Regarding the Q_{ap} values, a similar trend is observed when increasing the temperature, when at 623.15 K (350 °C) Q_{ap} approaches to Q_L . The apparent values of both creep parameters are consistent with the operation at low temperatures of the constant substructure slip creep mechanism ($n \approx 8$) affected by microstructural changes in the unprocessed material [21,37], changing the L(1) material to a GBS mechanism with increasing temperature.

Fig. 3. Values of (a) n_{ap} and (b) Q_{ap} vs. testing temperature at $10^{-2} s^{-1}$ obtained from the SRCT for the starting Al 7075-T6 alloy and the FSPed alloy with three different grain sizes after processing.

On the other hand, the two finest materials in Fig. 3 follow the same behavior of decreasing n_{ap} values with increasing temperature, towards $n_{ap}=2$, but close to 3 ($n_{ap(min)} = 2.8$ at 623.15 K (350 °C)) at a temperature range of 473.15–623.15 K (200–350 °C). Nevertheless, at 673.15 K (400 °C) the n_{ap} values increase again towards $n_{ap}=8$. Regarding the Q_{ap} values, a sustained decrease with increasing temperature is observed. However, three regimes can be identified. The first one from 473.15–523.15 K (200–250 °C) corresponds to a strong decrease in the Q_{ap} values, while in the 523.15 to 573.15 K (250 to 300 °C) range a moderate reduction is observed with Q_{ap} being close to Q_L . At $T > 573.15$ K (300 °C) the Q_{ap} values decrease drastically and even negative values are observed at 673.15 K (400 °C). This unexpected behavior is related to the sudden abnormal increment of σ from 623.15 to 673.15 K (350 to 400°C) shown at Fig. 2. It is our contention that after the initial transition from the constant substructure slip creep mechanism ($n \approx 8$) at 473.15 K

(200°C) towards GBS at 523.15–623.15 K (250–350 °C), the materials suddenly change back to the initial constant substructure slip creep mechanism at higher T. Indeed, the extremely low and even negative values of Q_{ap} at $T > 623.15$ K (350 °C) are a consequence of the change of mechanism from the “soft” GBS towards the “hard” constant substructure slip creep. Nevertheless, the n_{ap} and Q_{ap} values never reach the theoretical $n = 2$ and $Q = Q_L, Q_{GB}$ contained in the constitutive GBS equation as observed in materials with coarser L [43–45]. However, the topography in Fig. 1 is evidence of the operation of GBS even with $n_{ap} > 2$ and $Q_{ap} \neq Q_L, Q_{GB}$. Additionally, L dependence is observed, as corresponds to the operation of GBS.

The microstructural changes undergone during testing give rise to differences in experimental n_{ap} and Q_{ap} values from the theoretical ones of $n=2$ and $Q=Q_L, Q_{GB}$ and also lead to important changes in predictions by the constitutive equations, due to the L dependence of GBS (such as L^{-p} , being $p = 2 - 3$). In fact, Fig.1 shows very high strain hardening in some temperatures, especially at 623.15 K (350 °C). This huge difference in the flow stress from YS to UTS is related to grain coarsening during the test, which, in turn causes important changes in the values of n_{ap} and Q_{ap} . To show this, data of SRCT and CSRT (10^{-2} s^{-1}) for the L(2) material at 623.15 K (350 °C) is given in Fig. 4a where the maximum $\Delta\sigma$ (UTS–YS) was observed in Fig. 1. The SRCT curve can be divided in three regions: in the first one the $\dot{\epsilon}$ decrease in small jumps from 10^{-1} to 10^{-5} s^{-1} , in the second one $\dot{\epsilon}$ is increased again until 10^{-2} s^{-1} and finally in the third one $\dot{\epsilon}$ is kept at 10^{-2} s^{-1} until the end of the test. Each $\dot{\epsilon}$ is kept constant for 0.02. It can be observed that the σ in the first 10^{-2} s^{-1} step for the SRCT is lower than for the CSRT at the same equivalent strain of $\epsilon=0.45$. This is likely to be due to less grain growth since the initial $\dot{\epsilon}$ is higher and hence it takes less time to reach the same strain level in the SRCT than in the CSRT. In Fig. 4b the $\dot{\epsilon} - \sigma$ pairs from the SCRT in Fig. 4a are plotted for the decreasing $\dot{\epsilon}$ and the increasing $\dot{\epsilon}$, showing higher σ for the increasing $\dot{\epsilon}$ region than the equivalents for the decreasing $\dot{\epsilon}$ region. If the microstructure remained constant during the test, the σ for both regions should be the same. Nevertheless, the increase in σ during the test is indicative of grain coarsening (ΔL).

Fig. 4. (a) Comparison of the SRCT and CSRT (σ – ϵ) curves at 623.15 K (350 °C) and (b) $\dot{\epsilon}$ – σ data pairs from the SRCT test at 623.15 K (350 °C) showing the increase in the flow stress due to the grain coarsening during the duration of the test. Both representations belongs to the FSPed alloy with a grain size $L(2) = 530$ nm.

These microstructural changes lead to variations in the measured n_{ap} and Q_{ap} and can be related analytically [46–48]. When GBS is operative, the analytical relation for $n_{ap}=n(L)$ can be derived using its definition ($n=\partial \ln \dot{\epsilon} / \partial \ln (\sigma / E)$ at $T=cte$) and the constitutive equation (Eq. [1]) as follows:

$$n_{ap}(L) = n - \left(\frac{p\sigma}{L} \right) \left(\frac{dL}{d\sigma} \right) \Big|_T \quad (2)$$

being, theoretically, $n=2$, and if $Q = Q_L$ then $p = 2$ or if $Q = Q_{GB}$ then $p = 3$. It is predicted that when the microstructure remains stable, $\Delta L=0$, and $n_{ap}=n=2$. Nevertheless, when the microstructure coarsens $n_{ap}>2$. Likewise, obtaining the analytical expression of $Q_{ap}=Q(L)$ from its definition ($(Q=nR\partial \log(\sigma/E) / \partial (1/T))$ at $\dot{\epsilon}=cte$) and the constitutive equation (Eq. [1]):

$$Q_{ap}(L) = Q - \left(\frac{pRT^2}{L} \right) \left(\frac{dL}{dT} \right) \Big|_{\dot{\epsilon} \text{ or } \frac{\sigma}{E}} \quad (3)$$

where, $p = 2$ for $Q=Q_L$, or $p = 3$ for $Q=Q_{GB}$. Similarly to $n_{ap} = n(L)$, when the microstructure remains constant, $Q_{ap}=Q_L, Q_{GB}$, but if coarsens then $Q_{ap}<Q_L(p=2), Q_{GB}(p=3)$.

The original equations defining n and Q are for an ideal situation of high temperature behavior in steady state. This is not often the experimental condition since grain growth takes place during deformation of a given test. As an

approximation, we introduced Eqs. (2) and (3) for consideration of the sensitivity to grain coarsening of n and Q . Therefore, it is assumed that successive steady states are obtained for increasing L . These equations are deduced for small ΔL as a function of stress and temperature for a given $\dot{\epsilon}$. Larger increments would require integration of those equations.

However, Eq. (1) is always valid for large ΔL and can be used to estimate the initial effective grain size (L_i) from measurements of the final L assuming a GBS mechanism [49]. It has to be remembered that $\dot{\epsilon}$ and T remains constant during the test as follows $L_i = L_f \cdot (\sigma_i / \sigma_f)^{n/p}$. It is reasonable considering L_i at $\epsilon \approx 0.1$ in Fig. 1. At 623.15 K (350 °C), $L_f = 1.9 \mu\text{m}$, σ_i (at $\epsilon = 0.1$) = 24 MPa, $\sigma_f = \text{UTS} = 64 \text{ MPa}$, $n = 2$ and $p = 2$ thus $L_i = 0.71 \mu\text{m}$, or if $p = 3$ then $L_i = 0.99 \mu\text{m}$. These values are consistent with the experimental evidence and the constitutive equation for GBS, as slip creep would not predict a strengthening by microstructural coarsening. As a consequence of this large grain coarsening during the test the n_{ap} and Q_{ap} values differ from the theoretical ones.

In summary, the high temperature creep behavior of a severely deformed FSP 7075-T6 alloy with three different ultra-fine grain sizes (0.38, 0.53 and $1.1 \mu\text{m}$) has been rationalized. The detailed analysis of the deformation mechanism confirms that GBS is controlling deformation as evidenced by low σ , large elongations, low stress exponents and tested sample topography, in the superplastic window. However, the experimental creep parameters n_{ap} and Q_{ap} do not correspond to the theoretical values. This is mainly due to the microstructural changes that occur during deformation. Explicit L dependence of the creep parameters $n_{ap} = n(L)$ and $Q_{ap} = Q(L)$ have been derived from the operating constitutive creep equation. Calculations of the dependence of n and Q with the changed L confirm the validity of the theoretical values of the constitutive equation parameters for GBS.

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