

Article

Superplastic Behavior of Overaged 2024 Aluminum Alloy after Friction Stir Processing

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Abstract: A commercial 2024 aluminum alloy was heat treated at 280 °C for 48 h and then slow cooled in a furnace to obtain minimum hardness. This material was then friction stir processed (FSP) using three sets of processing conditions. To study the effect of the processing on the microstructure and the high temperature mechanical properties, the materials were tested in tension at an initial strain rate of 10^{-2} s^{-1} and temperature range 200 to 450 °C. Processing severity was selected as the main factor for obtaining fine grain sizes right after FSP. The grain size was enormously reduced from about 50 μm to 1 μm . This grain reduction gave rise to very high elongations to failure of about 400%. Strain-rate-change tests showed a stress exponent close to 2 at intermediate strain rates, which was related to grain boundary sliding as the controlling deformation mechanism and to superplasticity, which is strongly grain-size dependent. A possible controlling deformation mechanism by solute-drag creep, as proposed by other authors, was disregarded since tests conducted at 450 °C, where the microstructure of the FSP materials coarsens rapidly, gave a low elongation to failure and high resistance, which showed the importance of the grain size dependence of the operative deformation mechanism at 250–400 °C, which was only compatible with grain boundary sliding.

Keywords: aluminum alloy; friction stir processing; mechanical properties; creep behavior; superplasticity



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1. Introduction

The heat-treatable 2024 aluminum alloy possesses attractive features for aerospace applications due to its high strength, fatigue resistance and reasonable formability. This alloy is generally used in wing and fuselage structures subjected to tensile stresses [1,2].

The mechanical properties of these, and other, alloys can be improved using severe plastic deformation (SPD) as an effective tool for the production of ultrafine-grained material. The most important SPD techniques are friction stir processing [3,4], high-pressure torsion (HPT) [5,6], accumulative roll bonding (ARB) [7,8] and equal channel angular pressing (ECAP) [9,10].

In this work, friction stir processing (FSP) has been used. This is a solid-state process employed to modify the mechanical and metallurgical properties of many materials including aluminum, copper and magnesium. FSP consists of plunging a non-consumable rotating tool into a workpiece, traversing it along the processing direction, and finally retracting it. During the process, the FSP tool induces intense plastic deformation of the workpiece, producing a strong reduction in grain size when processing under the proper conditions [11,12].

The beneficial grain refinement produced by FSP for optimum superplastic behavior has been studied in different aluminum alloys. Charit and Mishra [13] reported superplasticity of a friction stir processed Al 2024-T4 alloy, obtaining elongations higher than

500% at 10^{-2} s^{-1} at 430 °C; Pradeep and Pancholi [14] produced a superplastic bulk area by multipass FSP in Al-Mg-Mn alloy with elongations close to 400%; Smolej et al. [15] obtained superplasticity in friction stir processed Al-4.5 Mg-0.35 Sc-0.15 Zr alloy showing elongations even higher than 1800% at 10^{-2} s^{-1} and $5 \times 10^{-2} \text{ s}^{-1}$ at 500 °C.

It is generally accepted that large elongations are obtained at high temperature by means of either solute-drag creep (SDC) or grain boundary sliding (GBS) due to their low stress exponents, $n = 3$ (SDC) or 2 (GBS). These are two independent deformation mechanisms competing to control deformation, and therefore, the slowest will be rate controlling. Both mechanisms have low stress exponents but different activation energies. The activation energy for the solute-drag mechanism corresponds to the activation energy of the diffusivity, D_{sol} , of the solute species that interact with dislocations. In the case of the 2024 aluminum alloy, the presence of Cu and Mg solutes interacting with moving dislocations may hinder dislocation glide, thereby influencing the dominant flow process. On the other hand, grain boundary sliding activation energy is the one for lattice diffusivity, D_L , at high temperature. In the case of the 2024 aluminum alloy, the two activation energies are very close and cannot be accurately discerned from the results of creep tests. Additionally, microstructural coarsening may substantially modify both apparent n and Q values. As n and Q are usually the main parameters employed by researchers to determine the controlling deformation mechanism, a controversy exists in the literature [13,16–23]. However, additional mechanical and microstructural evidences such as resistance, ductility and grain features (misorientation, size and its evolution) are taken into account in this work to determine the active deformation mechanism.

This work provides two important contributions to the study of the structural applications of alloys. The first one is the importance of the processing severity for achieving ultra-fine grain materials. For the case of the present overaged 2024 aluminum alloy, three different processing conditions have been used, resulting in a critical influence on the final microstructure. The second contribution regards the microstructural characteristics of the alloy influencing the deformation mechanisms at high temperature. In this case, grain boundary sliding has been proved to control deformation at intermediate–high strain rates for which high elongations are obtained.

2. Materials and Experimental Procedure

The Al 2024 alloy was provided from Alu-Stock in the form of rolled plates 3 mm in thickness in the T351 temper. Table 1 shows the nominal chemical composition in wt.%.

Table 1. Nominal chemical composition of the Al 2024 alloy.

Element	Cu	Mg	Mn	Si	Fe	Zn	Ti	Cr	Al
wt. %	4.3	1.5	0.6	<0.5	<0.5	0.15	0.03	0.007	Balance

The 2024-T351 aluminum alloy was heat treated at various temperatures, heating times and cooling conditions. The condition of 280 °C for 48 h and slow cooling in furnace (JH Hornos, Madrid, Spain) gave rise to minimum hardness. This temper was named TT or Al 2024-TT.

Then, the material in the TT condition was processed by FSP along the initial rolling direction. We used a tool made of a MP159 nickel superalloy. The tool had a scrolled shoulder 9.5 mm in diameter and a concentric threaded conical pin with flutes 4.7–4.1 mm in diameter and 1.8 mm in length. The aluminum alloy was processed under different severity conditions, aiming for ultra-fine grain sizes. In order to reach this goal, the material was processed using a steel backing plate at room temperature. Low heat input (HI) values through low tool rotation speed (r or ω) and high traverse speed (v), that is, $HI \propto r^2/v$ [24,25], were chosen. The lower the HI, the more severe the processing conditions. The processing conditions, combining different traverse speeds and rotational speeds, are given in Table 2.

Table 2. Designations and values of the FSP processing conditions.

r (rpm)	v (mm/min)	HI (rpm ² /(mm/min))	Designation
1000	1000	1000	10r10v
700	1000	490	07r10v
700	1400	350	07r14v

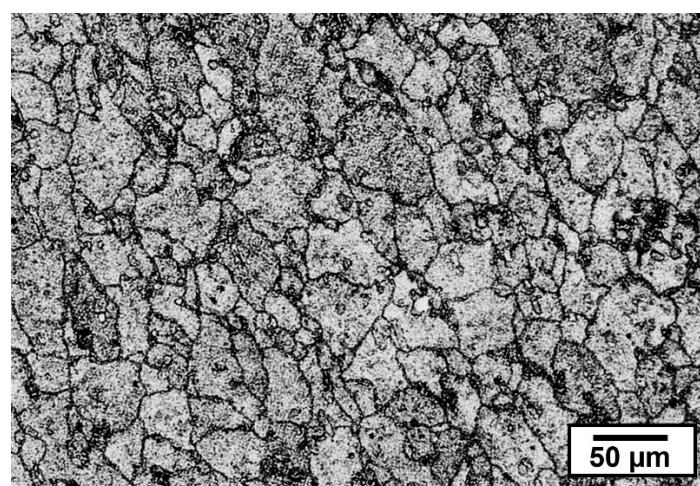
The microstructural characterization was carried out by means of light microscopy (OM) using an Olympus BH-2 microscope (Olympus Optical, Tokyo, Japan) and scanning electron microscopy (SEM) and electron backscattered scanning diffraction (EBSD) using a Hitachi ColdFEG S-4800 (HITACHI, Tokyo, Japan) and a JEOL JSM 6500 F (JEOL, Tokyo, Japan), respectively. OM samples were prepared by polishing using conventional methods and etching at room temperature with the Keller reagent (1.5% HCl, 1% HF, 2.5% of HNO₃ and 95% distilled water). SEM and EBSD samples were prepared by electropolishing at 15 V and 25 °C using a solution of 30% HNO₃ and 70% CH₃OH.

The mechanical characterization was carried out by means of constant crosshead speed tensile tests (CCST) at an initial strain rate of 10^{-2} s^{-1} . In addition, strain rate change tensile tests (SRCT) with strain rates ranging from 10^{-1} to 10^{-5} s^{-1} were performed to characterize the room- to high-temperature (25–450 °C) behavior. The tests were performed using a universal Instron 1362 testing machine equipped with a four-lamp ellipsoidal furnace. Planar dog-bone tensile samples with $6.5 \times 2 \times 1.7 \text{ mm}^3$ gage volume dimensions were electro-discharge machined so that their longitudinal axis was parallel to the initial rolling or FSP direction. The tensile samples were mirror-polished on their upper surfaces to a final thickness of 1.6 mm to monitor microstructural changes after testing. The true strain, ϵ , was calculated as $\epsilon = \ln(1 + e)$ where $e = (l - l_0)/l_0$, l_0 is the initial gage length and l is the instantaneous length. The true stress is defined as $\sigma = F/A_0(1 + e)$, where A_0 is the initial section of the sample and F is the supported load during tensile testing.

3. Results and Discussion

3.1. Microstructures

A light micrograph of the Al 2024 alloy under TT temper is presented in Figure 1. After this temper, the alloy presented coarse grains.

**Figure 1.** Light micrograph of the Al2024 alloy under TT temper.

Most of the grains were about 20 μm in size, and some of them were even larger, around 50 μm. Grain shape was slightly elongated.

The microstructure changed drastically after friction stir processing. Figure 2 shows an EBSD crystallographic orientation map of a FSP sample processed at 1000 rpm and

1000 mm/s. The material consists of a very fine-grained microstructure, about 1 μm average grain size, a high density of high-angle grain boundaries and practically random misorientations. The average grain sizes (Feret diameter, D_F) were 1.12, 1.05 and 0.93 μm for the 10r10v, 07r10v and 07r14v conditions, respectively.

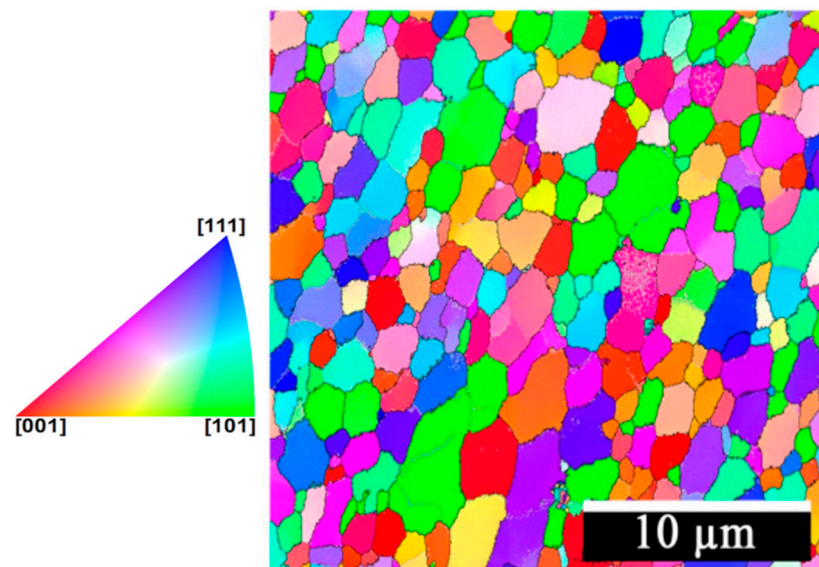


Figure 2. EBSD OIM micrograph of Al 2024-TT FSP sample 10r10v (processed at 1000 rpm and 1000 mm/s).

3.2. Tensile Tests at Medium and High Temperatures

True stress–true strain curves for the Al 2024 alloy for the TT temper at an initial strain rate of 10^{-2} s^{-1} and at temperatures ranging from 200 to 450 $^{\circ}\text{C}$ are presented in Figure 3. In general terms, the stress values decrease drastically with increasing temperature. The ductility increases with increasing temperature, but at 450 $^{\circ}\text{C}$, the elongation to failure decreases substantially. The greatest tensile ductility is observed at 400 $^{\circ}\text{C}$ for all 3 severe conditions, accompanied with the lowest stress values.

Elongation to failure curves at 10^{-2} s^{-1} as a function of temperature for the TT and FSP conditions are given in Figure 4. The ductility continuously increases with temperature up to 400 $^{\circ}\text{C}$. At this temperature, the ductility is larger than 400% for all FSP-processed materials. Similar values are observed for the three FSP processing conditions, followed by a strong ductility decrease at 450 $^{\circ}\text{C}$. This phenomenon will be discussed later. On the other hand, the non-processed material shows a moderate increase in ductility up to 450 $^{\circ}\text{C}$ with values lower than 100%. These values are much lower than those for the processed materials, revealing the presence of different deformation mechanisms.

The flow stress as a function of temperature at an initial strain rate of 10^{-2} s^{-1} is represented in Figure 5 for the TT temper and FSP conditions. The figure shows a strong decrease in the stress with temperature from very high values at 200 $^{\circ}\text{C}$ down to very low values at 350 $^{\circ}\text{C}$ for all FSP-processed materials. On the other hand, the non-processed Al2024-TT material presents a moderate continuous decrease in stress values, with the stress being the same at 450 $^{\circ}\text{C}$ as for the FSP materials.

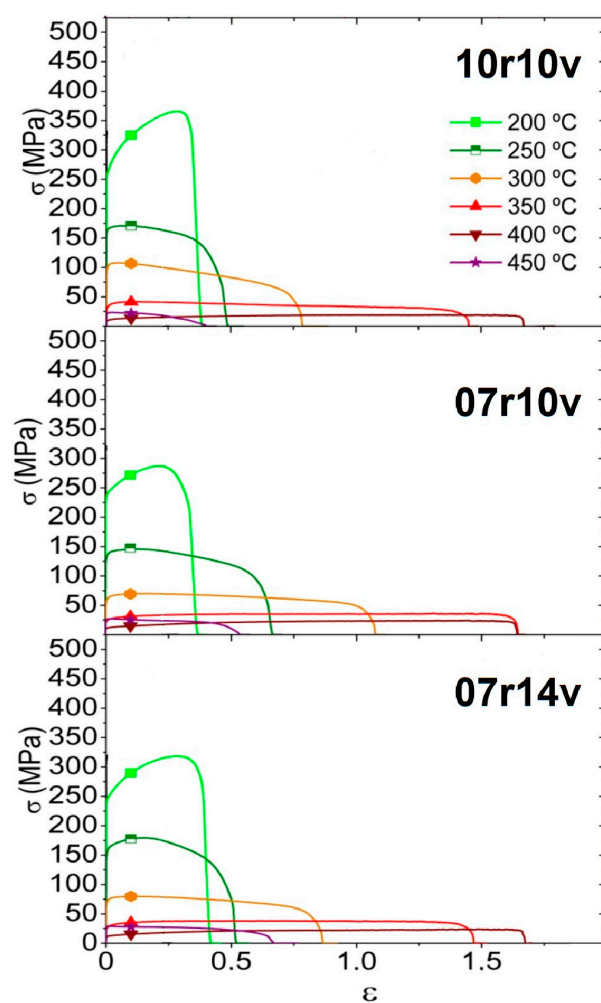


Figure 3. Stress as a function of strain at 10^{-2} s^{-1} for the Al 2024-TT in three FSP conditions and temperatures from 200 to 450 °C.

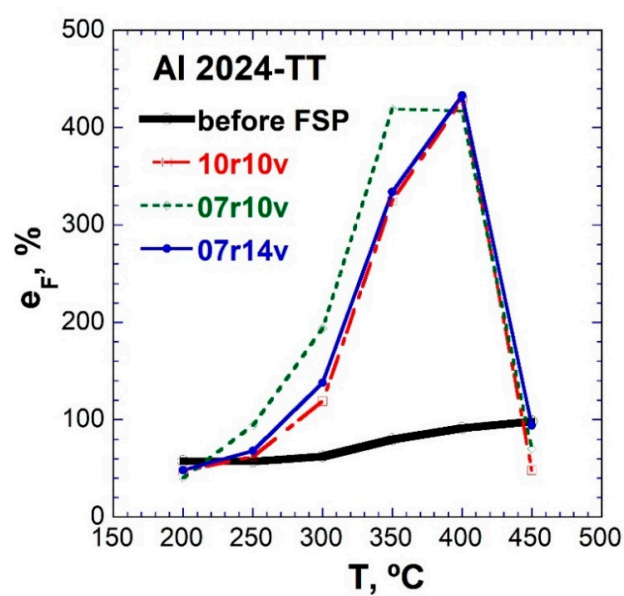


Figure 4. Elongation to failure as a function of temperature at 10^{-2} s^{-1} for the three FSP conditions and the non-processed Al 2024-TT.

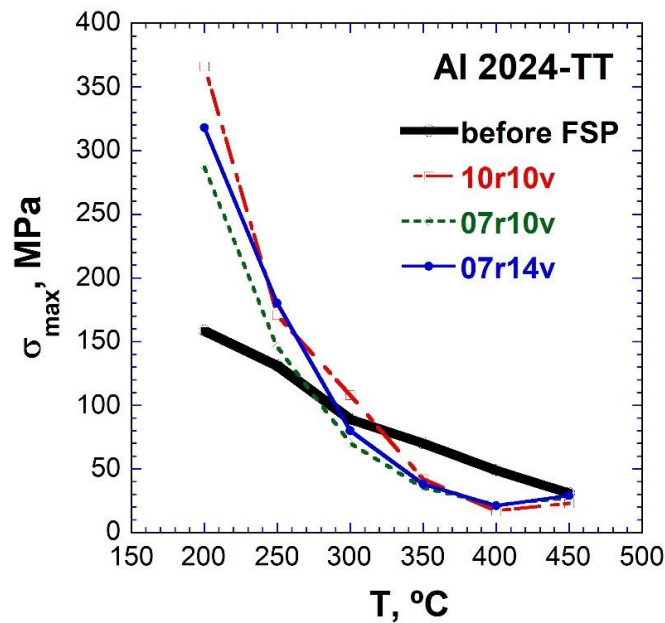


Figure 5. The flow stress, $\sigma_{\max}$, as a function of temperature at 10^{-2} s^{-1} for the Al 2024-TT before and after FSP conditions.

3.3. Strain-Rate Change Tensile Tests

With the goal of assessing the deformation mechanism at various temperatures in the FSP Al 2024-TT materials, a series of strain-rate change tests at various temperatures was conducted for three FSP conditions to determine the stress exponents and the activation energies. Strain rate–true stress curves at 300, 350, 400 and 450 °C are represented in Figure 6.

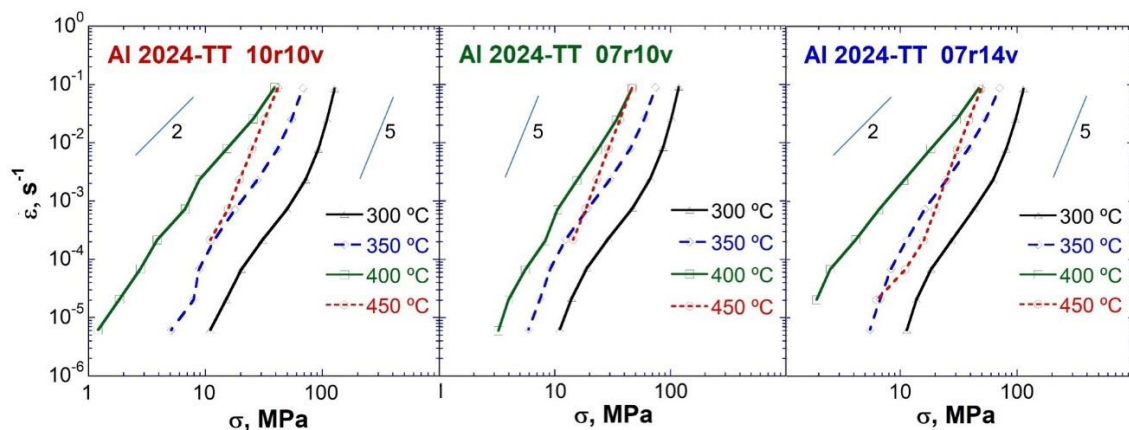


Figure 6. Double logarithmic scale representation of the strain rate–stress pairs at different test temperatures of the three FSP Al 2024–TT alloys.

The materials' curves show, in general, a sigmoidal shape with high apparent (experimental) stress exponents, n_{ap} , at high and low strain rates. At intermediate strain rates, the materials reveal smaller n_{ap} of about two to three. This decrease in the n_{ap} is consistent with the increasing elongation at this strain rate zone. All three materials have similar resistance at a given temperature and strain rate. The tests at 450 °C, as in Figures 3 and 4, clearly present an anomaly, pointing to a change in the deformation mechanism. The curves for the three FSP conditions can be related to the microstructures present in these materials.

3.4. Microstructures after Testing

The grain size rapidly coarsens after testing at high temperatures, as shown in the microstructures of the FSP materials in Figure 7 after testing at 350 and 400 °C. The grain size at 350 °C is about 3 µm and increases to about 4 µm at 400 °C. However, the grain size of the three FSP conditions is very similar at each temperature.

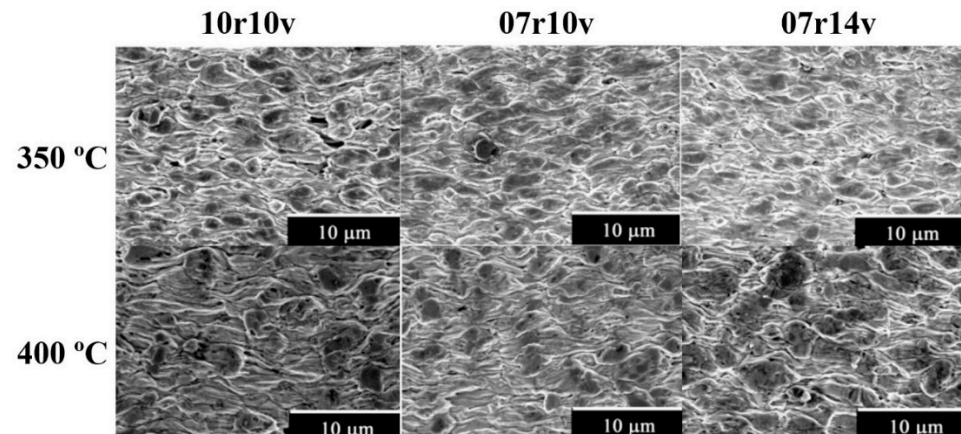


Figure 7. Microstructures of FSP Al 2024-TT samples after tensile testing at 10^{-2} s^{-1} at 350 and 400 °C.

Figure 8 presents the microstructure of the 07r10v material at 450 °C, as an example, as similar microstructures were obtained for the other FSP conditions. The material presents quite large grains, coarser than the considered limit of 20 µm for the activation of GBS. This explains the low elongation to failure observed in Figure 3 and the anomalous behavior in the strain-rate change tests in Figure 6 at this testing temperature.

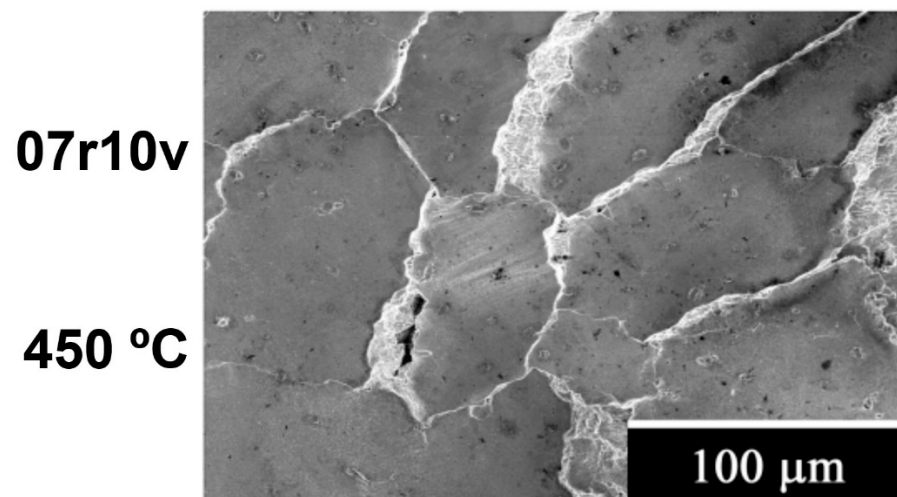


Figure 8. Microstructure after tensile testing at 10^{-2} s^{-1} and 450 °C the Al 2024-TT FSP 07r10v material showing very coarse grains.

In relation to the activation energy for deformation, all FSP conditions have values of about 140 kJ/mol in the superplastic range. This value is close to that corresponding to the self-diffusion of aluminum, 142 kJ/mol [26].

3.5. Deformation Mechanisms

The stress exponent values were calculated from the slopes of the curves in Figure 6 for each temperature and are mostly between $n = 2$ and 3. A stress exponent of 3 is usually

associated with solute drag or viscous glide creep [27]. During high-temperature plastic deformation, the dislocations may interact with the solute atoms, limiting their movement. It is convenient to remind that dislocation movement rate is governed by the slowest of the two possible serial movement stages, glide along the slip plane and climb to overpass obstacles such as second-phase particles. While climb is usually the slowest stage in most of the cases, in materials with a relatively high solute concentration, glide may be slower and thus rate controlling. Under such conditions, a three-power law may follow naturally:

$$\dot{\epsilon} = A_1 D_{\text{sol}}/b^2 (\sigma/E)^3 \quad (1)$$

where A_1 is a constant, b is the Burgers vector and E is the average unrelaxed polycrystalline Young's modulus. In this case, the associated stress exponent is $n = 3$.

On the other hand, grain boundary sliding, GBS, has an associated stress exponent of $n = 2$. In the case of the present FSP Al2024 materials, the grain size, L , is much finer than $20 \mu\text{m}$, which is usually considered the maximum grain size for the activation of GBS. The constitutive equation describing this mechanism is [28]

$$\dot{\epsilon} = A_2 D_L/L^2 (\sigma/E)^2 \quad (2)$$

where $A_2 = 2 \times 10^9$ for high stacking-fault energy materials, such as aluminum and its alloys. In this case, at high temperature, the associated grain size exponent, p , is usually $p = 2$.

On the other hand, slip creep is the controlling mechanism for coarse grains and at high strain rates. This mechanism is usually described by the following equation [29]:

$$\dot{\epsilon} = A_3 D_L/b^2 (\sigma/E)^5 \quad (3)$$

where A_3 is a constant, $n = 5$ and $p = 0$ (grain size independent, as for the solute drag mechanism in Equation (1)).

As can be observed in Figure 6, the three FSP Al 2024-TT present at high strain rates a stress exponent of about 5 or higher. This is because the materials start to deform in the power law breakdown region, where slip creep controls deformation under these conditions of temperature and high strain rates. On the other hand, at lower strain rates, the stress exponents are between 2 and 3, and it is not possible to confidently ascribe dislocation glide or GBS as the deformation mechanism. We should therefore seek other features that characterize these two deformation mechanisms.

At 400°C , the processed materials present very large ductility, about 400% (Figure 4), and very low stress values (Figure 5), which contrast with the non-FSP Al 2024-TT values. Additionally, the n values are very close to 2 for the 10r10v and 07r14v materials at 400°C . Lee et al. [16] assumed that the rate-controlling mechanism for an Al-2024 alloy is dislocation glide. This is attributed to the large size differences between the solute and the matrix atoms in Al-Mg and also Al-Cu alloys. They also assumed dislocation glide even for very fine grains after ECAP processing although elongations to failure were about 500% or even larger. Such elongations, at high strain rates, are usually higher than those typical of a dislocation glide or solute-drag mechanism.

A close inspection of our results gives a different perspective on the deformation behavior of the Al 2024 alloy. All the data presented in this work are congruent with the activation of a grain boundary sliding mechanism in certain temperature and strain rate windows. According to Figure 4, maximum elongations are observed at 400°C for the FSP materials, which are much higher than those for the coarse-grained Al 2024-TT alloy. Above this temperature, the microstructure of the FSP materials coarsens rapidly, impeding the activation of GBS and thus dramatically diminishing the ductility. This grain-size dependency is not compatible with dislocation glide. Additionally, Figure 5 shows minimum stress values for the three FSP materials at 400°C , followed by higher stress values at 450°C . This is confirmed by the strain-rate change test results given in

Figure 6, showing higher stress values at 450 °C than at 400 °C, and even at 350 °C at some strain rates (see dotted red lines). This behavior, associated with large grain coarsening (Figure 8) is again not compatible with the dislocation glide mechanism, which is grain-size independent.

On the other hand, in the superplastic regime, Equation (2) well describes the behavior of this Al 2024 alloy. This can be visualized by plotting the diffusion and grain size normalized strain rate as a function of modulus-compensated stress for the FSP materials assuming a final grain coarsening up to 4 µm; see Figure 9. The selected grain size is a reasonable value considering the micrographs after tensile testing presented in Figure 7. The 07r10v condition at 400 °C shows less ideal superplastic behavior compared with the other two FSP conditions. This can be attributed to the slightly bimodal microstructure after FSP present in this condition, showing some lower angle boundaries, meaning that the microstructure is less stable. It is worth mentioning that any coarsening would give a higher stress for a given strain rate, moving the curve in Figures 6 and 9 rightwards and showing higher apparent stress. In fact, as shown in Figure 4, the ductility is unexpectedly lower at 400 °C than at 350 °C for this 07r10v condition.

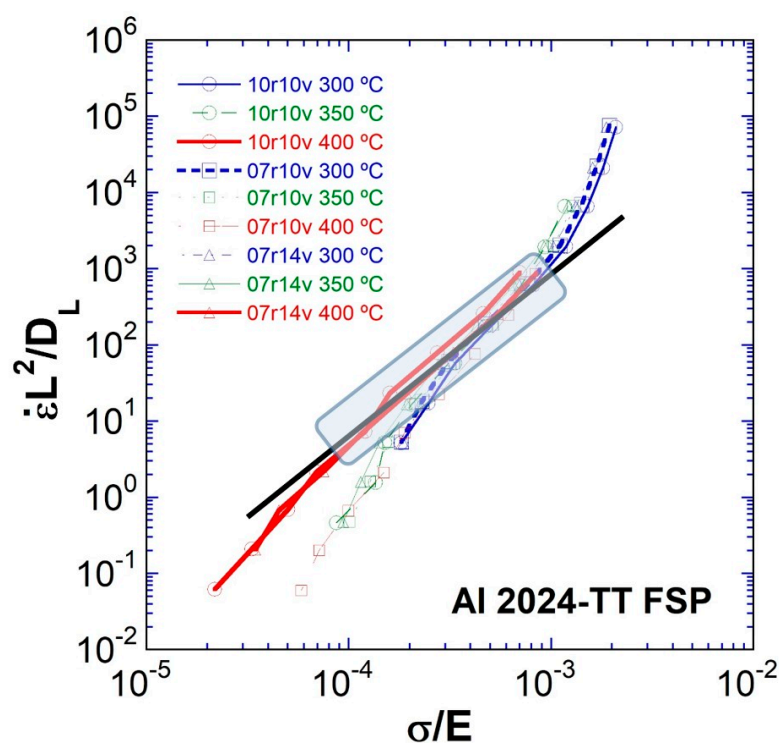


Figure 9. Diffusion— and grain—size—normalized strain rate as a function of modulus—compensated stress for the FSP conditions ($L = 4 \mu\text{m}$) at three temperatures.

A thick black line is displayed in Figure 9 that corresponds to a pre-exponential constant $A = 6 \times 10^8$. This value is 3 times lower than the generally considered in superplastic materials (2×10^9). Additionally, data for other superplastic alloys have been included in the colored band of Figure 9. These data correspond to alloys with compositions close to that of Al 2024 [13,30–37]. All these alloys have similar resistance in the superplastic regime and therefore similar A values. Other aluminum superplastic alloys have stress exponents close to 2, and large elongations to failure are also inside the colored area of Figure 9 [38–41]. All these alloys deform by grain boundary sliding when their microstructures consist of fine, equiaxed and highly misoriented grains, and therefore, they follow similar behavior as the processed materials. Therefore, comparison with other aluminum alloys reveals similar high temperature resistance despite the very different precipitation characteristics, pointing out the importance of the grain size of the alloys for controlling the superplastic behavior.

In this regard, FSP is a suitable processing method able to ultra-refine the microstructure, and thus, it allows easier formability at high strain rates and intermediate temperatures even with highly resistant aeronautical aluminum alloys.

4. Conclusions

A tempered 2024 aluminum alloy was subjected to three FSP conditions. The tempering, named TT, consisted of heat treatment at 280 °C for 48 h and slow cooling in a furnace to obtain minimum hardness.

- (1) After the TT temper, the microstructure was formed by coarse, barely elongated grains. Grain size was considerably reduced by severe FSP processing, reaching values about 1 μm .
- (2) A rapid decrease in the stress with temperature from 200 °C to 350 °C for all FSP processed materials was observed. On the other hand, the non-processed Al 2024-TT material showed a slower decrease, with the same stress at 450 °C as for the FSP materials.
- (3) The ductility increases substantially with temperature up 400 °C. At this temperature, the ductility is higher than 400% for all FSP processed materials. However, a strong decrease in ductility is obtained at 450 °C, due to extensive grain coarsening, and thus, grain boundary sliding is no longer the controlling deformation mechanism. On the other hand, the coarse-grained non-processed Al 2024-TT alloy presents a moderate increase in ductility up to 450 °C with values lower than 100%.
- (4) Similar strain rates at a given stress are observed in the superplastic regime, which are attributed to similar grain sizes in the three FSP materials during deformation.
- (5) The controlling mechanism in the low-stress regime is grain boundary sliding, as expected from fine, equiaxed and highly misoriented grains after friction stir processing.

Author Contributions: Conceptualization, F.C., A.O.-C. and O.A.R.; Methodology, A.O.-C. and F.C.; Validation, O.A.R. and F.C.; Investigation, M.Á.-L.; Resources, F.C. and A.O.-C.; Data curation, M.Á.-L.; Writing—Original draft preparation, O.A.R. and A.O.-C.; Writing—Review and editing, F.C., A.O.-C. and O.A.R.; Visualization, M.Á.-L. and F.C.; Supervision, F.C., A.O.-C. and O.A.R.; Project administration, F.C. All authors have read and agreed to the published version of the manuscript.

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