Effect of Processing Parameters on Superplastic and Corrosion Behavior of Aluminum Alloy Friction Stir Processed

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Abstract. Friction Stir Processing (FSP) is a variant of Friction Stir Welding, and can be used to modify the materials microstructure to functionalize it. Superplastic forming is a technological process used to produce components with very complex shapes. In the last two decades it has been a topic of major development. In Fine Structure Superplasticity (FSSP), the initial grain size exerts a strong influence on the superplastic strain rate and temperatures. Refining grain size (GS) the parameters (temperature and strain rate) of superplastic forming could be optimized. Thermal stability is also an important factor to obtain superplasticity. FSP is used to refine GS, but the optimum processing parameters are still under study over different materials. Corrosion resistance can be affected by FSP too, but the information about it is scarce.

In the present study, 7075-T651 aluminium alloy was friction stir processed under different conditions in order to improve superplastic behavior. Tool profile, rotation rate and traverse speed were analyzed. Microstructures with <4 μm grain size were obtained. The maximum superplastic elongations, in a range of 740 to 900%, at 400°C were obtained at $1x10^{-2}s^{-1}$ strain rate. The results were discussed in terms of constitutive equations and microstructure evolution. Localized corrosion potentials were obtained. Localized corrosion resistance was affected by friction stir processing.

Introduction

The study of the superplastic behavior of metallic materials has been a field of great interest and development in last years due to the importance of superplastic forming of components to obtain products with very complex geometries [1]. Superplasticity is one of several micromechanisms of deformation at high temperature, which is characterized by extensive plastic deformation prior to fracture [1], being in the case of fine-structure superplasticity (FSS), the border slip grain (GBS) mechanism that controls the superplastic deformation [2]. The activation of this mechanism is mainly determined by temperature, strain rate and grain size (GS) [3]. Superplasticity has been reported in materials with a fine and stable microstructure, which are deformed in the range of strain rates between 10^{-5} and 10^{-2} s^{-1} and temperatures usually above $0.5T_m$, being T_m the absolute melting temperature. The refinement of grain size has a strong influence on the optimum strain rate for FSS, increasing strain rate and decreasing temperature as GS decreases [1]. The thermal stability of the microstructure of the material is a critical aspect for the applicability of superplasticity.

Processing of materials by friction stir (FSP) has been recently developed and has great potential as a grain refinement method, having reported the activation of superplastic behavior in alloys processed by FSP [4]. Like Friction Stir Welding (FSW) a lot of investigations were developed on Al-alloys, being the 7075Al an interesting alloy due to their applications in aerospace industry and capability to superplasticity of FSP-microstructure [5-8].

In contrast with the enhancement that represents a friction stir modified microstructure for the plastic deformation of this alloy the resistance to corrosion in chloride environments decreases [9]. For this reason in this paper the localized corrosion susceptibility was considered.

Experimental

The plate thickness of the 7075-T651 aluminum alloy was 4 mm and the nominal composition 5.6%Zn, 2.4%Mg and 1.9%Cu. The conditions of FSP are listed in Table 1. The Velocity Index, Iv, is the ratio of tool rotation/travel speed, it was reported that the grain size of the stirred zone becomes smaller as Iv decreases [10]. The tool used was made of H13 tool steel and had a square side pin with concave shoulder of 12.5 mm in diameter. For all runs the tool inclination was 1.5°.

Table 1. FSProcessing parameters.

Identification	Tool rotation [rpm]	Travel speed [mm.min^{-1}]	Iv [rev.mm^{-1}]
514/98	514	98	5.2
388/51	388	51	7.6
514/51	514	51	10.1

The microstructure was characterized by optical microscopy (OM) and the grain size on the stirred zone was measured by mean of lineal intercept procedure according to ASTM E 112 [11].

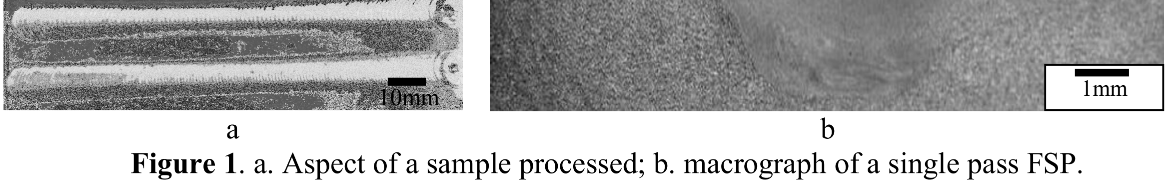
In order to evalute the superplastic behavior tensile tests at 400°C were performed. This temperature was adopted considering the thermal stability of FSP aluminum alloys studied by the authors in a previous work [12]. The tensile test specimen were obtained from the stirred zone with a gage length of 2.90 mm, 2.70 mm width and 1.70 mm in thickness. The tensile tests were carried out with three different initial strain rates: $5.0x10^{-3}$, $1.0x10^{-2}$ and $2.5x10^{-2}$ s^{-1}. Elongation to fracture was measured to compare the superplastic behavior.

Anodic potentiodynamic measurements were carried out in de-aerated 3.5wt% NaCl (pH = 6.2) solution at 24ºC for evaluating the effect of FSP on the material. Electrochemical measurements were performed using a three electrode cell. A SCE and a Pt sheet were used as reference and counter electrode respectively. The work electrodes were made with an epoxi resin and meticulously prepared. They were wet ground with 600 and 1000-grit SiC paper, then polished with alumina of 1 and 0.3 µm, ultrasonically cleaned in ethanol and dried. Considering the work of Frankel and Zhao [13] after polishing each electrode was etched by submerging it in a 1M NaOH solution at 60°C for 90 sec followed by an immersion in 70% HNO_3 for 30 sec in order to eliminate the severe plastic deformed zone induced by polishing. Results were recorded with an EG&G Princeton Applied Research model 273A potentiostat, using flat disc samples with a surface area of 0.8 cm^2 approximately. The sweep rate was 0.166 $mV.s^{-1}$. For these experiments a specimen processed with a tool rotation of 388 rpm and a travel speed of 406 $mm.s^{-1}$ was compared to the base metal.

Results and discussion

Microstructural characterization

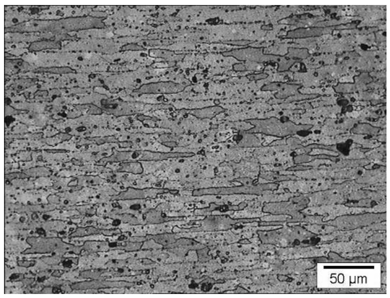
In Figure 1a can be observed a sample as processed. A macrostructure of a FSP sample is shown in Figure 1b. There is neither defects nor discontinuities and can be seen a strong refinement at the stirred zone. For the operative conditions used in this work no defects were observed.



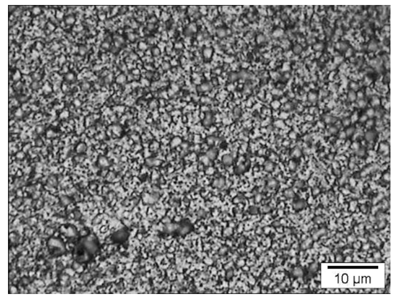
a b

Figure 1. a. Aspect of a sample processed; b. macrograph of a single pass FSP.

The grain size (mean volumetric grain diameter = 1.571 x mean lineal intercept length) on the stirred zone of sample 514/98 was 3.53 μm. GS was a little larger for sample 388/51, 3.62 μm, while the sample 514/51 has a GS of 4.65 μm. These results are in the range of 1 to 7.5 μm reported previously for similar materials and processing conditions [14]. The influence of the tool geometry could be the reason for the fine GS. It has been reported that square tools are associated to finer grain size of FSW-aluminum alloys [15]. In Figure 2 can be seen optical micrographs that have been taken from base metal (a), and from the stirred zone of different processing conditions: 514/98 (b), 388/51 (c) and 514/51 (d).



a.



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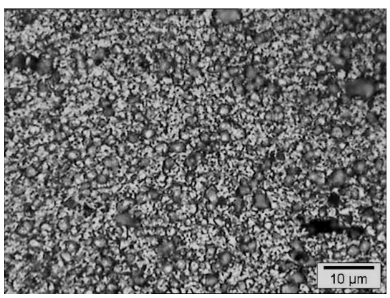

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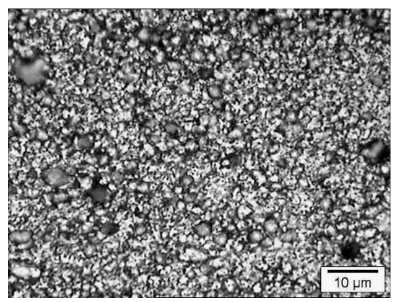

d.

Figure 2. Micrographs showing the initial microstructure of: a. base metal and stirred zones of: b. sample 514/98; c. sample 388/51 and d. sample 514/51.

In the base metal can be seen the structrure of cold worked grains of the T651 temper. In the other micrographs it can be seen that grain size increased from Figure 2b to Figure 2d. This increase is related with the Iv [10]. Figure 3 shows the variation of grain size with Iv.

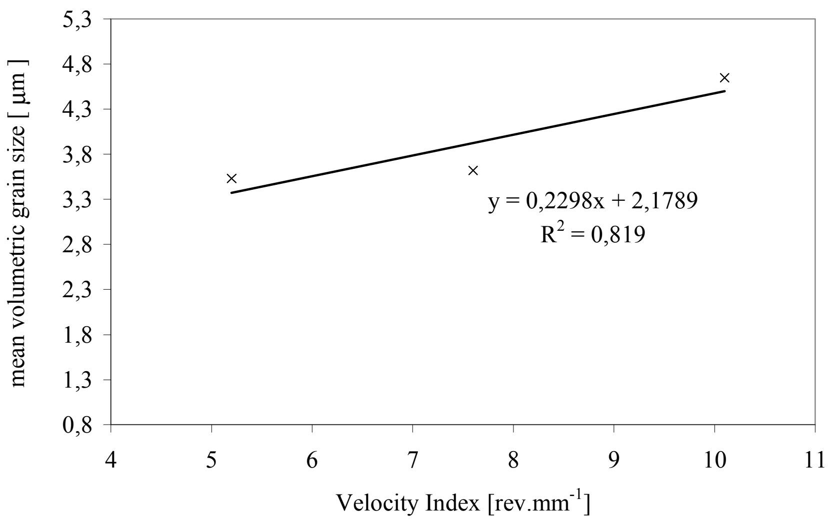

Figure 3. Velocity Index (Iv) vs. Grain size (GS) on the stirred zone.

It has been reported lower GS in the stirred zone as Iv is reduced [10]. This behavior is related with lower peak temperatures reached with lower tool rotation.

Superplastic behavior

Results from tensile tests at 400°C are shown in Figure 4. In this figure can be observed that the optimum strain rate was $1x10^{-2}$ s^{-1}, being the largest superplastic elongation 900% corresponding to condition 514/98. Also can be observed that for initial strain rates $5x10^{-3}$ and $1x10^{-2}$ s^{-1} the largest elongation correspond to the samples with lowest ratio Iv, 5.2. These results are promissory considering the elongations reported in previous works at 400°C [16] or even at higher temperatures [14]. Considering superplastic forming, temperature and strain rate are parameters of technological and economical importance, due to its effects on time of processing and energy consumption. In this sense, processing conditions that allow lower temperatures and higher strain rates superplastic forming are desired.

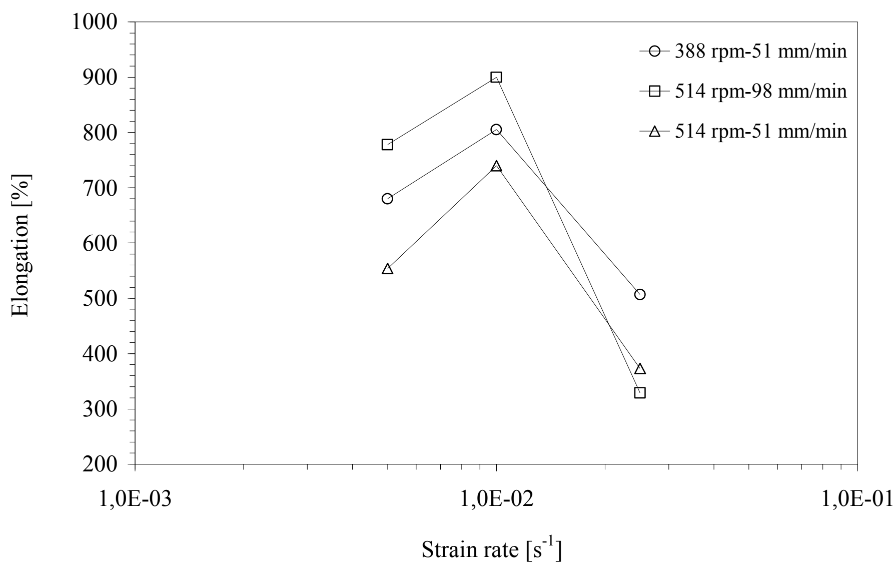

Figure 4. Elongation to fracture vs. initial strain rate of tensile tests at 400°C, for different processing conditions.

The results obtained for 514/98 are possible due to the relatively small, uniform and thermally stable grain size reached through the sample thickness [4].

In Figure 5 is shown a comparison of superplastic elongations obtained from sample 514/98 for each intial strain rate.

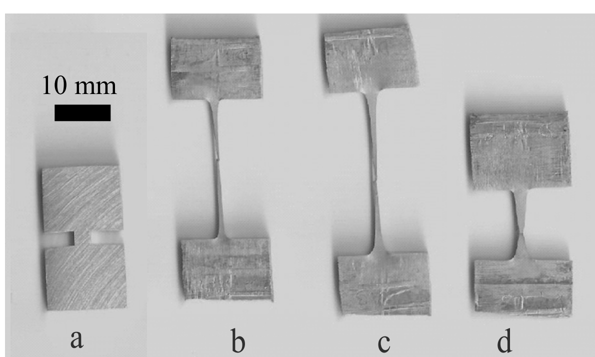

Figure 5. 514/98 tensile samples tested at 400°C under the following strain rate: a. untested sample; b. $5x10^{-3}s^{-1}$; c. $1x10^{-2}s^{-1}$ and d. $2.5x10^{-2}s^{-1}$.

The variation of flow stress (true stress, σ, at a true strain, ε, of 0.1) with the initial strain rate can be observed in Figure 6.

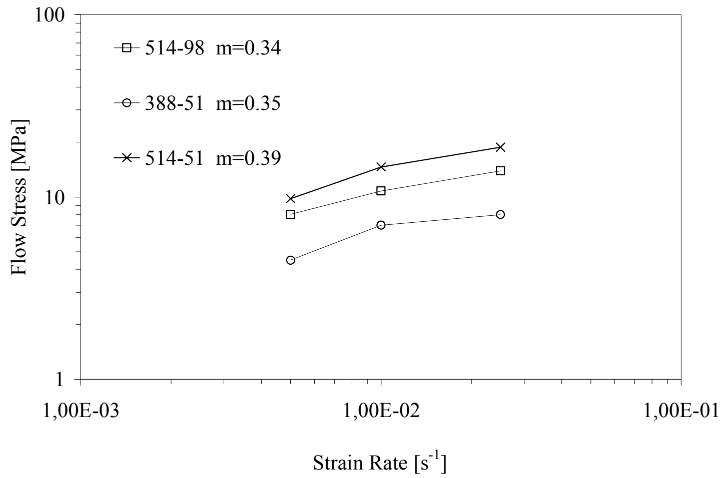

Figure 6. Variation of flow stress with initial strain rate, *m* ranged from 0.34 to 0.39.

The superplastic strain rate sensitivity, *m*, ranged from 0.34 to 0.39. These values of *m* were previously reported in the work of Liu and Ma [16] for low temperature superplasticity of a FSP 7075 alloy.

Corrosion behavior

Typical potentiodynamic polarization curves are shown in Figure 7. In the figure can be observed that the breakdown potential for the base metal sample was -750 mV SCE with a mean value of ten runs of -748 mV SCE, while for the friction stir processed sample an abrupt increase in current density can be identified at -850 mV SCE being the mean value -850 mV SCE. In all runs hydrogen bubbles were observed in isolated sites on the work electrode. This can be presumed due to cathodic reaction near the active sites [17, 18].

Observation of the electrodes by OM indicated that an intragranular attack was predominant in base metal samples, while for FSP samples pitting attack was prevalent, especially in the HAZ and lesser in the TMAZ. This is in agreement to previous results reported in the literature for similar conditions [9, 18, 19].

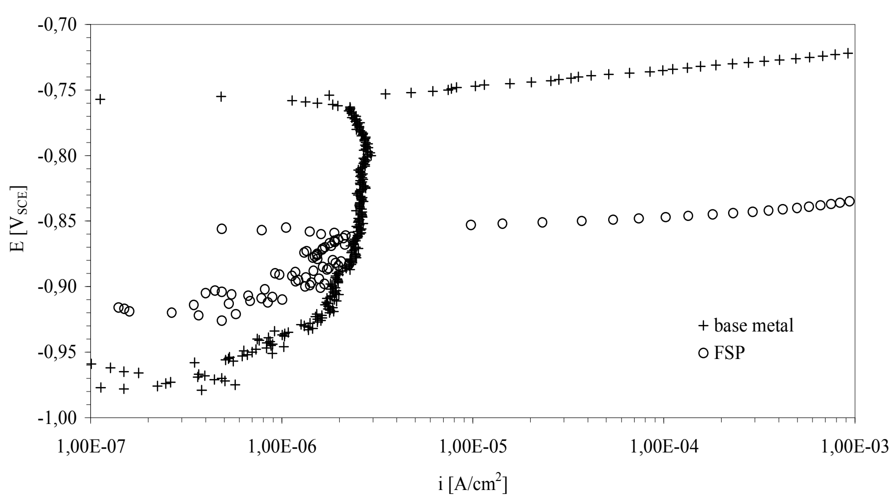

Figure 7. Characteristic potentiodynamic curves for base metal and FSP 7075-T651.

Conclusions

Under conditions analyzed in this work the following can be concluded:

1. successfully grain size refinement was achieved on 7075-T651 alloy by mean of FSP, reaching GS of stirred zone ranged from 3.53 to 4.65 µm;
2. there was observed a correlation between the velocity index (Iv) and the GS obtained in the stirred zone;
3. superplastic elongation up to 900% were obtained at relative low temperature 400°C and high strain rate $1x10^{-2}s^{-1}$, related to the fine and thermally stable grain structure obtained using 514 rpm for tool rotation, 98 mm/min for welding speed and a tool with a square pin;
4. the superplastic strain rate sensitivity was between 3.34 to 3.39;
5. the breakdown potential of the FSP was 100 mV below that the breakdown potential of base metal;
6. the HAZ and the TMAZ were more susceptible to localized corrosion that the stirred zone and base metal

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