Dynamic Strain Aging (DSA) in solid solubilized Mg-Gd alloys under compression at intermediate temperatures

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ABSTRACT: The Mg-Gd alloys compressed at intermediate temperatures exhibit flow serrations which present a significant dependence on strain rate and compression temperature. The apparition of this phenomenon is due to the dynamic strain ageing effect, that basically consists in the simultaneous and competitive movement of diffusing solute atoms, mobile dislocations and twin boundaries during deformation. Compression tests have been carried out at temperatures between 25–300 °C and strain rates in a range of 4×10^{-5} - 8×10^{-3} s⁻¹ to analyze the apparition of serrations and, implicitly, the DSA effect. A relation between the activation energy for serrated flow and the concentration of gadolinium was determined and indicates that there would be an inversely proportional relation between these two parameters.

KEYWORDS: Magnesium alloys; Dynamic strain aging; Serrated flow; Solute segregation

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RESUMEN: *Envejecimiento dinámico por deformación en aleaciones Mg-Gd solubilizadas ensayadas a compresión a temperaturas intermedias.* Tres aleaciones Mg-Gd ensayadas a compresión a temperaturas intermedias muestran un fenómeno de serrado dependiente de la velocidad de deformación y de la temperatura de ensayo. La aparición de este fenómeno se relaciona con la segregación de átomos de soluto en las dislocaciones móviles y/o en las fronteras de macla. Los ensayos de compresión se han realizado en un intervalo de temperaturas comprendido entre 25 y 300 °C y a velocidades de deformación comprendidas entre 4×10^{-5} y 8×10^{-3} s⁻¹ con el fin de analizar el fenómeno de serrado, e implícitamente, el proceso de envejecimiento dinámico por deformación. Experimentalmente se ha comprobado que el aumento de la concentración de gadolinio en solución sólida disminuye la energía de activación del proceso.

PALABRAS CLAVES: Aleaciones de magnesio; Envejecimiento dinámico; Flujo serrado; Segregación de los átomos de soluto

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1. INTRODUCTION

A wide variety of materials are used in applications in which resistance to mechanical loading is necessary. The need to use lighter alloys in structural components convert magnesium, the lightest structural metal, into a great candidate for those applications. However, magnesium alloys present severe ductility problems and, as a consequence, poor low temperature formability.

Rare Earth (RE) elements added in solid solution in magnesium alloys have a great impact in their mechanical properties which make them perfect candidates for structural applications in the automotive and aerospace industry. It was demonstrated in previous studies that the addition of RE elements in Mg enhances its formability and creep resistance (Ball and Prangnell, 1994; He *et al.*, 2007; Stanford *et al.*, 2008; Zhu *et al.*, 2010; Hantzsche *et al.*, 2010; Yuan *et al.*, 2013; Gravas *et al.*, 2016; Griffiths, 2015; Wang *et al.*, 2015). Due to their bigger atomic radius compared to the magnesium atomic radius, RE atoms trigger distortions in the crystalline magnesium lattice that lead them to interact with dislocations, twins and grain boundaries. The diffusion of RE atoms and their subsequently interactions to deformation mechanisms causes a pinning effect that retards the propagation of such mechanisms defined as Dynamic Strain Ageing (DSA). DSA is normally revealed by the appearance of a serrated flow in the stress-strain curve. DSA was detected in many magnesium alloys (some of them Mg-RE) deformed not only by tensile tests (Couling, 1959; Chaturvedi *et al.*, 1972; Chaturvedi and Lloyd, 1974; Zhu and Nie, 2004; Zhu *et al.*, 2004; Corby *et al.*, 2004; Wang *et al.*, 2006; Zhongjun *et al.*, 2007; Wang *et al.*, 2007; Fang *et al.*, 2009; Gao *et al.*, 2009a; Wu *et al.*, 2012; Cai *et al.*, 2014; Garcés *et al.*, 2015; Wang *et al.*, 2015; Wang *et al.*, 2017) but also by compression and torsion tests (Stanford *et al.*, 2010; Jiang *et al.*, 2011; Garcés *et al.*, 2018). The dynamic interaction between RE atoms with dislocations and twinning are the main responsible for its appearance.

The activation of tensile twinning supposes a significant contribution to the plasticity in extruded magnesium alloys. Recent studies have demonstrated the segregation of gadolinium atoms in twin boundaries after static annealing in compressed samples (Nie *et al.*, 2013; Zhu *et al.*, 2017; Zhu *et al.*, 2018). This segregation induces a pinning effect of twins that delays their propagation (Nie *et al.*, 2013). Gadolinium segregation would also occur during compression tests at intermediate temperatures when twinning is produced. The magnesium-gadolinium system exhibits a wide gadolinium solubility range (up to 4.56% at. at 550 °C). Therefore, the higher the amount of gadolinium the higher the segregation effect and its influence in the

high temperature deformation. The present article studies DSA phenomenon in extruded Mg-1%Gd, Mg-3%Gd and Mg6%Gd alloys under compressive testing at temperatures between 25 °C and 300 °C in a wide range of strain rates. The critical strain for DSA will be analyzed to calculate the activation energy. This is part of a deep study of the influence of the segregation of gadolinium atoms in dislocations and twins and its influence in the mechanical strength.

2. MATERIALS AND EXPERIMENTAL PROCEDURE

Three alloys with nominal composition Mg-1wt.%Gd, Mg-3wt.%Gd y Mg-6wt.%Gd (defined as G1, G3 and G6) were fabricated by casting using pure magnesium and a Mg-20wt.%Gd master alloy at 750 °C in an electric resistance furnace. After melting, the liquid was poured in a cylindrical steel mold with a 41 mm diameter. The obtained cylinders were solubilized at 550 °C for 24 h in a container covered in magnesia powder to minimize the oxidation and quenched in water. Cylinders were extruded at 450 °C with a 25:1 extrusion ratio. The use of this extrusion temperature maintains gadolinium in solid solution. During extrusion, the extruded bars were cooled with nitrogen to quench the microstructure, maintaining gadolinium atoms in solid solution.

The crystallographic texture of the alloy was evaluated using Electron Backscattered Diffraction (EBSD) technique and the Rietveld analysis of the Synchrotron diffraction patterns. Specimens for EBSD were finally etched using a solution of 7 ml. acetic acid, 3 ml of nitric acid, 30 ml of ethanol and 10 ml of water. EBSD measurements were carried out in a direction perpendicular to the extrusion direction and data were recorded and analyzed using the Channel 5 software. The grain size was measured by the linear intercept method from at least 4 different images.

Synchrotron radiation diffraction (SRD) patterns after extrusion on the P07 beamline of PETRA III, at the Deutsches-Elektronen-Synchrotron (DESY). The gauge volume is defined by the volume generated by the beam section and the cylinder diameter ($0.8 \times 0.8 \times 5 \text{ mm}^3$). The diffraction patterns were recorded using an exposure time of a range between 0.5 s by a Perkin-Elmer XRD 1621 flatpanel detector with an array of 2048^2 pixel, with an effective pixel size of $200 \times 200 \mu\text{m}^2$. The beam energy was 100 keV that corresponds to a wavelength of 0.0124 nm. LaB6 was used as a reference to calibrate the experimental set-up. The macroscopic texture was evaluated using a Rietveld texture analysis in MAUD (Lutterotti *et al.*, 2007). The 2D detector image is converted to a set of diffraction

patterns using the Image J plugin in the software (Lutterotti *et al.*, 1997; Schindelin *et al.*, 2015), fitting the α -Mg (P63/mmc). The best fit to the experimental data was achieved using the E-WIMV algorithm with 5° resolution.

Cylindrical samples for compression tests were mechanized along the extrusion direction with dimensions of 5 mm diameter and 10 mm length. Compression tests were carried out in a universal tensile rig with a furnace for high temperature testing. Such tests were performed at different temperatures between 25°C and 300°C and at strain rates in a range of $4 \cdot 10^{-5}$ to $8.3 \cdot 10^{-3} \text{ s}^{-1}$. The temperature was measured with a thermocouple in contact with the compression sample. Data was transformed to turn the initial graphs into stress vs engineering strain curves, subtracting the machine's complying and removing the elastic zone to focusing in the plastic zone to evaluate the DSA phenomenon.

3. RESULTS

The microstructure the orientation image map (IOM) both materials along the extrusion direction is shown in Fig. 1(a–c). The microstructure is fully recrystallized after extrusion at 450°C . Grain size was 42 ± 1 , 29 ± 1 and $23 \pm 1 \mu\text{m}$ for G1, G3 and G6 alloys, respectively. No second phases were observed in the microstructure. Diffraction patterns have been measured to assure that gadolinium is in solid solution. As an example, the Rietveld analysis for the Mg-6Gd alloy is observed in Fig. 2. The increase in the gadolinium content in solid solution increases the lattice parameters, a and c , in agreement with previous results (Xu *et al.*, 2017). $\{10\bar{1}0\}$ and (0002) pole figures shows that the alloy shows a fiber texture with the basal plane parallel to the extrusion direction.

Figure 3 shows the plastic compression curves (subtracted the elastic zone) for the three alloys in the temperature range between room temperature and 300°C . Compression curves exhibit a sigmoidal or “S-type” shape which is indicative of the activation of extension $\{10\bar{1}2\}\langle\bar{1}011\rangle$ twinning. For G1 alloy, the yield stress at room temperature is 78 MPa, which decreases as test temperature increases up to a minimum value of 48 MPa at 300°C (Fig. 3a). After yielding, a strong work-hardening at room temperature is observed. The true stress raises rapidly from yield stress to 250 MPa at a strain of 0.1. After this strain, the stress is constant until the fracture of the compressive samples. Nevertheless, work hardening after yielding decreases as temperature increases. Such effect is more noticeable at temperatures between room temperature and 100°C or above 200°C . Curves between 100°C and 200°C have a similar behavior without any noticeable yield stress or hardening variation. In this temperature range, the compression tests are characterized by the apparition of serrated flow, which disappear above

FIGURE 1. Orientation Image Mappings (OIMs) of the extruded: a) G1, b) G3 and c) G6 alloys along the extrusion direction. The black arrow indicates the extrusion direction.

FIGURE 2. a) Rietveld analysis of the synchrotron radiation diffraction pattern of the G6 alloy, b) Evolution of the a and c lattice parameters as a function of the gadolinium content, and c) $\{10\bar{1}0\}$ and (0002) pole figure obtained from the Rietveld analysis in the G6 alloy.

200 °C. Compression curves for G3 alloy are similar to those for G1 alloy (Fig. 3b), although they are shifted to higher stress values. The yield stress at room temperature is 97 MPa. This value also gradually decreases while increasing temperature until it reaches 53 MPa at 300 °C. Serrated flow occurs before than in G1 alloy (125 °C) and disappears above 200 °C. Finally, Fig.

3c shows compression curves for the G6 alloy. The yield strength at room temperature is 124 MPa and it decreases up to 78 MPa at 300 °C. In this alloy, compression curves between room temperature and 200 °C exhibit a similar behavior, noticing a big difference in strengthening at 250 °C and 300 °C. Serrated flow is observed at temperatures between 100 °C and 200 °C.

The work-hardening increases as a function of the strain but more slowly than in G1 and G3 alloys.

The appearance and the magnitude of serrated flow depend strongly on temperature and strain rate. Figure 4 shows the range of the appearance of the serrated flow as a function of the temperature

and strain rate for the three alloys. The increase in the gadolinium content in solid solution enlarge the DSA regions with the compression temperature. Figure 5 shows the compression curves at 150 °C and 200 °C for the three alloys at different strain rates between $\dot{\epsilon} = 8 \times 10^{-3}$ and $4 \times 10^{-5} \text{ s}^{-1}$.

FIGURE 3. True stress-true strain compression curves from room temperature up to 300 °C at a strain rate of 10^{-4} s^{-1} for the a) G1, b) G3 and c) G6 alloys.

FIGURE 4. Scheme of the existence of serrated flow as a function of the compression temperature and the initial strain rate, for a) G1, b) G3 and c) G6 alloys. Green dots represent test conditions in which serrated flow appears.

The analysis of the curves presented in Figs. 3 and 5 determine the conditions for which serrated flow is observed and the critical strain, ε_C , defined as the offset strain at which serration starts. An increase in the strain rate, the macroscopic strain and the compression temperature tend to eliminate the serrated flow in compression curves. At 150 °C, the serration amplitude increases when the gadolinium content is increased from 1 to 3%. This is especially significant for the lowest strain rate, $4 \times 10^{-5} \text{ s}^{-1}$. However, when

the amount of gadolinium content is doubled up to 6%, the stress amplitude decreases although the strengthening is the highest, as found in G1 and G3 alloys. The appearance of serrated flow during the plastic behavior is not the only aspect that reveals the DSA process. The negative strain rate sensitivity (SRS) is characteristic of the DSA. SRS causes an increase in the flow stress with the decrease in the strain rate. Figure 6a, c and e show compression jump curves for G1, G3 and G6 alloys at 100,

FIGURE 5. True stress-true strain compression curves at a, c, e) 150 °C and b, d, f) 200 °C in the strain rate range from $4 \times 10^{-5} \text{ s}^{-1}$ to $8.3 \times 10^{-3} \text{ s}^{-1}$ for the a, b) G1, c, d) G3 and e, f) G6 alloys.

200 y 300 °C changing the strain rate from 10^{-3} s^{-1} to 10^{-4} s^{-1} , starting from 10^{-3} s^{-1} . At 100 and 200 °C, a decrease in the strain rate from 10^{-3} s^{-1} to 10^{-4} s^{-1} results in an increase of the stress. Moreover, it seems that the higher is the gadolinium content the higher is the stress difference between both strain rates. Therefore, even when the serrated flow is not observed in G6 alloy, DSA occurs. At 300 °C, the strain rate sensibility shows the typical behavior; a decrease in the flow stress the strain rate is decreased.

The critical strain for serrated flow, (ε_c), depends on microstructural parameters such as dislocation density and concentration of solute atoms or test parameters such as test temperature and strain rate. The critical strain in the DSA phenomenon is generally described:

$$\varepsilon_c^{(m+\beta)} = K \cdot \dot{\varepsilon} \cdot \exp\left(\frac{Q}{RT}\right) \quad (1)$$

FIGURE 6. True stress-true strain compression curves at 100 °C, 200 °C and 300 °C changing the strain rate from $\dot{\varepsilon}_1 = 10^{-3} \text{ s}^{-1}$ to $\dot{\varepsilon}_2 = 10^{-4} \text{ s}^{-1}$ for the a) G1, c) G3 and e) G6 alloys. A detail of curves a), c) and e) for 100 °C and 200 °C for the b) G1, d) G3 and f) G6 alloys.

where K , m and β are a constant, $\dot{\epsilon}$ is the strain rate, R is ideal gas constant, T is the temperature and Q is the activation energy. Initial studies proposed that vacancies produced during plastic deformation will allow solute atoms to diffuse faster onto dislocations, moving more slowly as their density

FIGURE 7. Variation of the critical strain for the onset of flow serrations in logarithmic scale (ϵ_c) with a) the strain rate at 150 °C and b) 200 °C in logarithmic scale. c) Variation of the critical strain for the onset of flow serrations in logarithmic scale (ϵ_c) with the inverse of the test temperature at strain rate of $10^{-3} s^{-1}$.

increases (Cottrell, 1953a; Cottrell, 1953b; Kubin and Estrin, 1990). Therefore, the activation energy represents solute mobility. More recent studies propose that solute diffusion along forest dislocations, pipes which intersect the mobile dislocation segment, is the mechanism controlling strain ageing (Kubin and Estrin, 1990). The activation energy, in this case, represents the pipe diffusion.

Figures 7a and b show the dependence of the critical strain with the strain rate in double logarithmic scale for the three alloys at 150 °C and 200 °C. As the gadolinium content increases, serrations start at lower strains. From equation 1, the constant $(m + \beta)$ can be calculated from the slope of the linear fitting. In all cases, $(m + \beta)$ values are near 1. These values are used to calculate the activation energy for the process. Figure 7c represent the temperature dependence of logarithm of the critical strain. The slope of the linear fitting defines:

$$\frac{Q}{(m + \beta) \times R} \quad (2)$$

Although the activation energy is so sensible to $(m + \beta)$ values, activation energies are close at 150 and 200 °C. Therefore, the average value for both temperatures has been calculated. The activation energies are 55, 48 and 40 $kJmol^{-1}$ for G1, G3 and G6, respectively. It seems that the activation energy decreases with an increase in the gadolinium content.

4. DISCUSSION

The addition of gadolinium improves the mechanical strength over the complete RT-300 °C temperature range. Gadolinium atoms are in solid solution in the three alloys. Solid solution strengthening is considered to be the most reinforcing mechanism in Mg-Gd alloys (Gao *et al.*, 2009b; Xu *et al.*, 2018; Xu *et al.*, 2020). Solid solution strengthening induced by gadolinium additions is higher than that reported for aluminium or zinc but lower than that of other rare-elements such as yttrium. Gadolinium atoms enhance the bond energy between gadolinium and magnesium atoms and between magnesium atoms themselves as well.

At intermediate temperatures, between 100 and 200 °C, a DSA process is clearly identified by, the appearance of serrated flow during plastic regime and the negative evolution of the SRS. An increase in gadolinium concentration promotes DSA phenomenon with the temperature. It is well established that DSA phenomenon involves a competition between diffusion of solute atoms along forest dislocations and movement of mobile dislocations. The increase in the temperature enhances the diffusivity of gadolinium atoms,

which favors local locking of mobile dislocations. Dislocations require an additional stress to cause local unlocking. Above 200 °C, climb of dislocations precludes the locking effect of gadolinium atoms. Similarly, an increase of gadolinium atoms in solid solution also favors dislocation pinning. However, when the amount of gadolinium atoms is high enough to pin mobile dislocations independently of the local unpinning, serrations in the flow stress disappear showing a higher work hardening as observed in G6 alloy, especially at low strain rates.

An increase in dislocation density and strain rate makes harder dislocation pinning by gadolinium atoms. It is interesting to point out that a threshold dislocation density is needed for the appearance of serrated flow. However, serrations tend to disappear at high strain rates because of the high density of dislocations accumulated within grains. Although gadolinium atoms pin dislocations, there are always free mobile dislocations slipping within magnesium grains.

During compression tests, deformation is not only controlled by dislocation slip but also by twinning. Figure 8 shows IOMs for the three alloys after 8% of plastic deformation. Twins are clearly observed within magnesium grains. Gadolinium atoms segregate also at twin boundaries (Nie *et al.*, 2013; Zhu *et al.*, 2017; Zhu *et al.*, 2018) avoiding efficiently their movement and increasing their mechanical strength (Nie *et al.*, 2013). Gadolinium is demonstrated as an effective element to segregate into twin boundaries to improve the mechanical strength of magnesium alloys (Pei *et al.*, 2019). The segregation of gadolinium atoms also promotes DSA phenomenon (Garcés *et al.*, 2018). The pinning-unpinning process of twin boundaries from gadolinium atoms induces serrations with lower amplitude (in both stress and strain) than when the process is only controlled by pinning-unpinning of mobile dislocations.

The critical strain for serrated flow increases its value as the strain rate increases, but it decreases when temperature is increased, such dependence is generally expressed by equation 1. The $(m + \beta)$ parameters are similar in the three alloy around 1. These values are lower than other values observed in the bibliography (Table 1) for magnesium alloys. Activation energies are also lower than other alloys observed. As it was commented above, the activation energy is related to pipe diffusion ($Q \sim 0.6-0.7$ $Q = 80-94$ kJmol^{-1}). The closer values are reported in Mg-Zn-Nd and Mg-Ag alloys. The activation of twinning and, therefore, gadolinium segregation at twin boundaries could contribute to reduce activation energy for pipe diffusion in comparison with tensile tests in which only the pinning of mobile dislocations takes place. Atomic radius of Gd and Nd atoms are similar (0.18 and 0.182 nm, respectively) and higher compared to magnesium atomic

FIGURE 8. Orientation Image Mappings (OIMs) of the deformed a) G1, b) G3 and c) G6 alloys along the extrusion direction at a strain of 8%. The colour representation of the three main orientations for the hexagonal lattice is the same showed in Fig. 1. The extrusion direction is also the same of Fig. 1.

radius (0.160 nm). In contrast, atomic radius of Ag (0.144 nm) is lower than the magnesium. However, recently (Zhao *et al.*, 2019; Liu *et al.*, 2019) the segregation of Ag atoms at twin boundaries has been

TABLE 1. Magnesium alloys in which DSA has been reported. $(m + \beta)$ parameters and activation energies and the cause of DSA phenomenon. Rare-earth elements are printed in bold.

System		Values	Reference
Md-Y-Nd	Diffusion of Y-Nd solute atoms	$m + \beta = 2.2$ $Q = 75 \text{ kJmol}^{-1}$	Zhu and Nie (2004)
Mg-Gd	Diffusion of Gd solute atoms	$m + \beta = 2.85$ $Q = 80 \text{ kJmol}^{-1}$	Gao <i>et al.</i> (2009a)
Mg-Gd-Zn	Fracture of coarse precipitates during deformation	$m + \beta = 2.17$ $Q = 83 \text{ kJmol}^{-1}$	Wu <i>et al.</i> (2012)
Mg-Gd-Zn	Diffusion of Gd and Zn solute atoms		Garcés <i>et al.</i> (2018)
Mg-Dy-Zn	LPSO 18R – solute diffusion atoms LPSO 14H – particle fracture		Cai <i>et al.</i> (2014)
Mg-Er	Diffusion of Er solute atoms	$m + \beta = 2.62$ $Q = 78 \text{ kJmol}^{-1}$	Zhongjun <i>et al.</i> (2007)
Mg-Gd-(Mn,Sc)	Diffusion of Gd, Mn and Sc solutes atoms		Fang <i>et al.</i> (2009)
Mg-Y-Gd-Zn	Diffusion of Y and Gd solute atoms	$m + \beta = 3$ $Q = 77 \text{ kJmol}^{-1}$	Garcés <i>et al.</i> (2015)
	Dislocations slip in channel generated between lamellar precipitates	$m + \beta = 4.3$ $Q = 156 \text{ kJmol}^{-1}$	
Mg-Y	Diffusion of Y solute atoms		Gao <i>et al.</i> (2009c)
Mg-Zn-Nd	T4 treatment. Diffusion of solute atoms	$m + \beta = 1.8$ $Q = 45 \text{ kJmol}^{-1}$	Wang <i>et al.</i> (2007)
	T6 treatment. Diffusion of solute atoms. Secondary DSA due to dissolution of precipitates	$m + \beta = 2.3$ $Q = 73 \text{ kJmol}^{-1}$	
Mg-Nd-Zn	Diffusion of Nd and Zn solute atoms		Wang <i>et al.</i> (2017)
Mg-Gd Mg-Ce	Diffusion of RE solute atoms		Stanford <i>et al.</i> (2010)
Mg-Ag	Diffusion of Ag solute atoms	$m + \beta = 1.44$ $Q = 50 \text{ kJmol}^{-1}$	Chaturvedi <i>et al.</i> (1972, 1974)
Mg-Ca-Zn	Diffusion of Ca solute atoms		Zhu <i>et al.</i> (2004)
Mg-Al-Zn	Al determine the dynamics of pile-up formation Zn catalyser enabling the formation of a forest dislocations		Corby <i>et al.</i> (2004)
Mg-Li-Al	Low strain rate: shearing effect of precipitates by dislocations High strain rate: Diffusion of Li solute	$m + \beta = 2.73$ $Q = 83 \text{ kJmol}^{-1}$	Wang <i>et al.</i> (2006)
Mg-Th			Couling (1959)

reported inducing a strong effect on the strengthening of magnesium alloys.

5. CONCLUSIONS

The effect of gadolinium concentration in solid solution on the DSA phenomenon were studied using compression tests in three Mg-Gd alloys in a temperature range between room temperature and 300 °C and in a wide range of strain rates. The following conclusions can be drawn:

- The dynamic strain ageing in Mg-Gd alloys is caused by the interaction of gadolinium atoms and mobile dislocations as well as twin boundaries. Gadolinium atom pin both defects, inhibiting their movements.
- The increase in the gadolinium concentration in solid solution widens the range of parameters,

temperature and strain rate, in which the presence of serrated flow is presented. Furthermore, for higher gadolinium contents, solute atoms are so effective to avoid the unpinning effect.

- The $(m + \beta)$ parameter is independent on gadolinium concentration. However, the activation energy for DSA tends to decrease with an increase in solute concentration.

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