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Creep behavior of ingot and powder metallurgy 6061Al

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Abstract

The creep behavior of 6061Al alloy obtained by ingot metallurgy and powder metallurgy, IM and PM, respectively, has been investigated in the context of published studies on this alloy. The behavior of the IM alloy in a given range of temperatures where the β , Mg_2Si , precipitates are formed, is dominated by dislocation climb-controlled creep and aluminum self-diffusion as rate controlling process. A dependence of the β inter-particle distance, λ , with the applied stress, σ , of the form $\lambda \approx 1/\sqrt{\sigma}$ is found when the creep data are analyzed in the context of the sub-structure invariant model. The superior creep resistance of the PM material can be explained if a threshold stress, σ_0 , is brought into the creep equation. This term is the difference between the applied stresses needed to reach a given strain rate in the PM and the IM alloys, and correlates well with a particle-dislocation interaction mechanism according to the Artz-Wilkinson model.

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Introduction

The present investigation is motivated by the need of understanding the creep of discontinuously reinforced metal matrix composites, MMCs. In particular, of its relative improvement with respect that of the corresponding unreinforced alloys. These materials, specifically aluminum matrix composites, were developed with the aim of increasing the room temperature stiffness (Young's modulus) of the conventional aluminum alloys to be used mostly in the transportation sector and using low cost procedures [1-3]. It was soon encountered that some other properties, such as their creep behavior, also improved with respect the unreinforced alloy [4]. Several applications that take advantage of the good creep response of MMCs have been found very useful also in the above industrial sector [2,3,5]. This improvement and a significant industrial demand have led to a deep scientific interest to understand their creep deformation mechanisms. Despite the big effort devoted by many researchers, however, the basic concepts underlying this better creep behavior remains, still, as a subject of debate.

Probably, due to the fact that the initial goal was to develop composites with superior room temperature properties (stiffness), and also because of availability and easiness in their thermo-mechanical processing (rolling, extrusion), most of the aluminum alloys used in their preparation are conventional products developed for room temperature applications. This is the case of 6061Al, a typical extrudable wrought 6xxx alloy [6]. This alloy has a very complex microstructure: it is age-hardenable undergoing a well known precipitation sequence after annealing at moderate temperature from a solid solution condition. A sequence of: GP zones, incoherent, β'' , and semi-coherent, β' precipitates formation occurs before attaining a dispersion of incoherent precipitates of the stable phase Mg_2Si (β precipitates) [7-11]. With further annealing, β precipitates coarsening (Ostwald ripening) occurs. The kinetics of this precipitation sequence is strongly dependent on temperature. Its analysis, as for other typical non-continuous reactions, is usually carried out in the frame of the Jonshon-Mehl-Avrami-Kolmogorov, JMAK, and the Kissinger and/or the Ozawa models provided controlled constant heating or cooling rates from a well known initial precipitation state [11-16]. Usually, these factors are not considered in creep tests, not even in a qualitative manner. Furthermore, creep tests are commonly carried out at temperatures at which precipitation in age-hardenable alloys is fast. Thus, an unavoidable, non-controlled, evolution of the precipitation at the beginning of the creep test is a clear difficulty for a rigorous analysis of the data obtained at different temperatures since the dislocation

interaction with the precipitates will evolve. These phenomena, although barely considered in the literature [17], are highly vital to understand in depth the creep of age-hardenable aluminum alloys. In fact, and as it will be shown later, little agreement is found in the literature on the mechanisms that governs the creep of 6061Al alloy [4,17-25], and similar difficulties are found for other age-hardenable aluminum alloys used as matrices of MMCs. Since understanding the creep of MMCs should be supported on a deep knowledge of the creep of the unreinforced alloys, it is not surprising the present weak capacity to predict the creep of aluminum alloy MMCs.

The above picture is further complicated when the processing route is also considered. In particular, depending on whether ingot or powder metallurgy (IM or PM, respectively) is used in materials preparation, a fine dispersion of Al_2O_3 particles can be formed. The role of a dispersion of oxide particle on the high temperature behavior of metals has been studied by Sherby and coworkers [26]. However, whereas investigations comparing the creep of 6061Al matrix composites obtained by IM and PM routes have been conducted [27], it is surprising that a similar study carried out on unreinforced 6061Al has not yet been done.

The purpose of this investigation is, hence, to study the high temperature behavior of 6061Al alloy obtained by two different processing routes, IM and PM, and to investigate: a) the mechanisms that govern the creep of this alloy and b) the effect of the alumina particles on this behavior. As mentioned above, 6061Al alloy has been selected because it is extensively used in the preparation of discontinuously reinforced MMCs and, hence, in the studies of the creep behavior of MMCs. Thus, a significant number of investigations on the creep of this alloy is available in the literature [4,17-25]. For this reason, a review of the published studies will be conducted as a first step in this research. Understanding in depth the creep of 6061Al, and the specific role played by the alumina particles in PM alloys, will allow understanding better the creep of MMCs.

Data on the creep of 6061Al alloy

The investigations on the creep of 6061Al are mostly centered on studies of the creep of 6061Al matrix composites in which, for comparison, the unreinforced alloy has been also investigated [4,17-25]. Table I summarizes these studies and their most relevant results and figure 1 shows their creep data in a double logarithmic plot of steady strain rate or minimum creep rate, $\dot{\epsilon}$, vs. applied stress, σ . In this plot, the stress exponent, n , in the power law creep equation

$$\dot{\epsilon} = k' \sigma^n \quad (1)$$

(with k' a temperature and microstructure dependent constant) at each temperature is indicated. As seen from this plot, data in the range of eleven orders of magnitude of $\dot{\epsilon}$ are shown.

It is worth mentioning the investigation conducted by Park *et al* [21], probably the most complete one on the creep of 6061Al alloy up to date. Tests over six orders of magnitude of strain rate at two temperatures were performed. To compare with other investigations, Figure 1, the double shear creep data have been transformed into the equivalent uniaxial data by appropriate transformation [28]. The creep data and in particular the high n values, were analyzed in terms of the presence of a threshold stress, σ_0 , which accounts for an interaction between dislocations and a fine dispersion of coherent Al_2O_3 particles raised by the PM route. The threshold stress was analyzed using three different methods; the Orowan stress model, the local climb model, and the detachment stress model proposed by Artz and co-workers [29,30]. The one that seemed to fit best with the experimental data was the detachment stress model. It should be noted, however, that despite the detailed study, no comparison with the creep behavior of 6061Al obtained by IM (*i.e.*, without the Al_2O_3 particles) was done to confirm this.

It is also worth mentioning the study of Langdon's group [22]. The discussion of their results is centered on the occurrence of the power law breakdown in terms of the specific atomic specie that governs the diffusion coefficient. It is argued that the Mg content is sufficiently high such that creep may be controlled by either diffusion of Mg atoms in the aluminum matrix or by lattice self diffusion of Al atoms. Thus, it is concluded that the strain rate value that defines the limit between power law creep and power law breakdown depends on the rate controlling specie. This limit is 10^{-2}s^{-1} if aluminum self diffusion is rate controlling or $9 \times 10^{-2}\text{s}^{-1}$ for the case of Mg atoms diffusing in the Al lattice. The diffusing atom specie that is finally rate controlling, however, is not clearly specified.

Materials and experimental procedure

The 6061Al alloys tested for the present work were obtained by these two metallurgical procedures, IM and PM. The chemical analysis, conducted by ICP-AES, led to the data of Table II. The IM alloy was kindly supplied by Dra. C. Cordovilla, from INESPAL, Spain, as a piece extracted from a cylindrical ingot of some 200 mm in diameter. The PM alloy was processed from 6061Al powder [31,32]. Particles were spherical with a particle diameter distribution described in Table III. As a consequence of the final extrusion step (ratio of 37:1 and extrusion temperature of $T_{\text{ext}} = 763 \text{ K}$) of the PM process, a $\langle 111 \rangle + \langle 100 \rangle$ fiber texture

(with the fiber axis parallel to the extrusion direction), typical of aluminum extruded alloys [33], was developed. From the IM alloy piece, a cylinder of 40 mm in diameter and 200 mm in length was machined and extruded under similar temperature and severe extrusion ratio as the PM alloy. In this manner, a similar final fiber texture was developed in both alloys. After extrusion, the alloy bars were air quenched. Thus, all alloy elements, in particular Mg and Si, can be assumed to be in solid solution in both alloys.

Optical and scanning electron microscopy were employed to study materials microstructure, which was revealed using conventional metallographical procedures. The grain size was very similar in both alloys: of about 3 μm in the IM alloy and some 1.5 μm in the PM alloy. The grains were, as expected, slightly elongated along the extrusion axis. Thus, the solely relevant difference between the microstructure of these alloys was the dispersion of Al_2O_3 particles in the PM alloy, absent in the IM one.

The creep behavior was studied by means of stress-controlled tensile creep tests [34] at different temperatures (573-723K at increments of 50K) and applied stresses (4-50 MPa). The tests were conducted at constant stress provided by an Andrade's cam which multiplied by five the applied load. Cylindrical samples, with threaded heads and a gauge region of 3 mm diameter and 10 mm length were machined. The tensile axis was parallel to the extrusion axis. The elongation and the applied load as a function of time were recorded provided two linear variable differential transducers, LVDTs and a load cell, respectively. All data was stored through data acquisition boards. The tests were allowed to run until failure or until a minimum or a steady state creep rate was reached.

Results

The results of the creep tests of both IM and PM alloys are summarized in the plots of figure 2a) and figure 2b), respectively. These plots represent the minimum or steady state creep rate as a function of the applied stress at the different temperatures of testing in a log-log scale. The steady state creep rate, $\dot{\epsilon}_s$, of both 6061Al alloys at each temperature obeys the power law relation of equation (1). For the case of the IM alloy, n varies with the testing temperature in the range $n = 5-9$, and for the PM alloy it is of about $n=18$.

The PM alloy is much more creep resistant than the IM one. This difference is observed at all testing temperatures and is attributed to the reinforcing Al_2O_3 particles. The slightly different concentration of some alloying elements (Table I), particularly Mn and Fe, should barely account for the different creep behavior. In fact, the higher amount of these two

elements in the IM, presumably in solid solution, would favor its creep resistance contradicting the observed lower creep rate of this alloy than that of the PM one.

Analysis of the creep data of 6061Al alloy

As mentioned above, a thorough and rigorous analysis of the creep behavior of 6061Al in terms of deformation micro-mechanisms is very complex. The specific interaction of dislocations with the precipitate particle, either at room or high temperature, dictates the mechanical behavior of the alloy. At room temperature, such a study, even for different stages of the precipitation sequence, is relatively easy since the precipitation process is virtually frozen. This has been done by several researchers, mostly by hardness tests [17,25, 31,35-37]. At moderate or high temperatures (*e.g.*, above 400 K), however, this task becomes further more complex because the precipitation velocity is strongly dependent on temperature. This is crucial for the case of creep tests which may extend for long time periods during which a significant precipitation can occur. Thus, a comparison of data obtained at different temperatures (for example, with the aim of determining Q_c) may be criticized since different degrees of precipitation may results also in a different alloy “microstructure”. This picture points out the great complexity in establishing a solid frame to describes the creep deformation mechanisms of 6061Al alloy. Nevertheless, some remarkable consequences can be deduced from the analysis of the published work on the creep of 6061Al. This analysis has allowed to understand in more depth the creep data of the 6061Al alloys of this research.

a) Analysis of literature data

To go deep into the creep behavior of 6061Al, the data will be analyze assuming that self diffusion of aluminum atoms is rate controlling during creep of the alloy. The following creep equation should, then, be obeyed,

$$\dot{\epsilon} = K' \left(\frac{D_L}{b^2} \right) \left(\frac{\sigma}{E} \right)^n \quad (2)$$

where K' is a material constant, D_L is the lattice diffusion coefficient, $D_L = D_o \exp(-Q_L/RT)$ (where D_o and Q_L are: the pre-exponential term, equal to: $D_o = 1.7 \times 10^{-4}$ m²/s, and the activation energy for lattice self diffusion, equal to: $Q_L = 142$ kJ/mol, for the case of aluminum [38], respectively, R is the universal gas constant, equal to $R=8.314$ kJ/mol K, and T is the absolute temperature), b is de Burgers vector, equal to: $b= 2.86 \times 10^{-10}$ m in aluminum, and E is the Young’s modulus. For this analysis, the strain rate and stress data of

figure 1 have been normalized with D_L and with the temperature dependent Young's modulus, $E(T)$ [39], respectively. The resulting log-log plot is shown in Figure 3. For comparison, also the creep data of pure aluminum [40] at different temperatures are also included in this plot. Whereas the creep data of pure aluminum leads to an excellent correlation (as it has been established earlier [41]), a more scattered correlation results in the case of the 6061Al alloy. Nonetheless, all the data tend to group under a power law dependence over several orders of magnitude reasonably well considering the different data sources and the very likely different precipitations stages of the alloys studied. It should be also considered the possible effect of other parameters of the microstructure (initial dislocation density, texture) coming from alloys prepared by different suppliers as well as the inherent scatter in data coming from different laboratories [42]. Only the data from Luster et al [20] at 423 K and from Matsuda et al. [23] at 773 K separate quite clearly from the common general trend.

The idea that aluminum self diffusion is the rate controlling process during creep of several aluminum alloys and discontinuously reinforced aluminum alloy MMCs has been assumed in previous investigations [26,41,43-53]. Whereas this assumption may be criticized for some alloys, it can be well justified for the case of 6061Al. Specifically, a solid justification relies in that at many of the temperatures under study in figure 3, the most important alloying elements in this alloy, Mg and Si in a stoichiometric proportion, Table II, precipitate to form Mg_2Si . Therefore, it is reasonable to assume in a simplified model, that self-diffusion of aluminum atoms dictates the rate mobility of dislocations in 6061Al at those temperatures (little effect of dislocation pipe diffusion is also considered). Furthermore, Q_c values close to that for lattice self diffusion of aluminum have been found in some studies [18,24].

An explanation for the deviation of Luster [20] and Matsuda's [23] data is found in the temperature of testing and the corresponding microstructure developed in the alloy during testing. Luster's data were taken at 423 K, a typical annealing temperature for a T6 condition. In fact, the alloy was T6 treated after annealing at 433 K for 24h, at only 10 K above the creep tests temperature. Hence, the precipitation state of the alloy during creep should be formed by a very fine homogeneous distribution of coherent β'' and semi-coherent β' precipitates. The interaction of dislocations with these precipitates differs dramatically from the interaction when the coherent and large β precipitates are developed during creep at higher temperatures, *e.g.*, above 473 K. All this explains the strain rate

dependence and the high stress exponent obtained by Luster ($n=22$) at 423 K, very different from the general trend shown in figure 3.

Contrarily to Luster's alloy, Matsuda's alloy tested at 773 K [23] shows a substantially weaker behavior than that defined by the general trend of figure 3. At this temperature of testing it is likely that all alloying elements, in particular the predominant Mg and Si atoms, are in solid solution. Thus, solute drag by dislocations should be the dominant deformation mechanism, consistently with the stress exponent of nearly $n=3$ obtained. *I.e.*, the material behaves like a class I solid solution alloy [41]. A reinforcing effect attributed to the coherent Mg_2Si particles is, hence, absent and the alloy is much weaker. It is difficult to obtain a meaningful value of Q_c from these data and those obtained by Matsuda at 573 K since at this temperature precipitation of the incoherent Mg_2Si particles should occur, leading to a dramatically different interaction with dislocations, as in fact is revealed by the different stress exponents, Table I.

It is worth mentioning that, surprisingly, the data from Park *et al* [21] and Pickens *et al* [18], which correspond to the PM alloys, exhibit slightly weaker behavior than the IM alloys, figure 3. It would be expected that the fine dispersion of Al_2O_3 particles of those alloys would act as effective barriers for dislocation motion, as in oxide dispersion strengthened, ODS, alloys, leading to a higher strength than the IM alloys. In fact, a higher stress exponent from that of the IM alloys results. There is no at present an explanation that can account for this result apart from the above mentioned idea of the different precipitation state, and justifies the present investigation to compare the creep of IM and PM 6061Al alloys using the same experimental set up.

b) Analysis of the present results

As suggested from the previous analysis, the present creep rate and stress data have been also normalized with D_L and $E(T)$, respectively. The result is shown in the log-log plot of figure 4. For comparison, the region which groups the data of the general trend of figure 3 (dotted region), the data from Luster *et al* [20] and from Matsuda *et al.* at 773 K [23], and the data from pure aluminum [40,41], are also indicated. The data from Park *et al.* [21] have been also specified within the dotted region. The values of the present study fall in this region and expand in almost three orders of magnitude the diffusion compensated strain rate data reported in the literature on the creep of 6061Al.

The stress exponent of the normalized creep data of the IM alloy in the range 573-723 K is about $n=6.5$, figure 4. As can be seen, a reasonably good correlation is obtained for the four

temperatures of testing. This correlation is better than that for the literature data, figure 4. This should be attributed to the initial state of the alloy and to the heating step prior to creep tests: both affect the state of the Mg_2Si precipitation. This precipitation is more similar among the samples of this study than among the different alloys of the literature studies. Still, the scatter of data in the IM 6061Al alloy is attributed to the different precipitation kinetics at the different temperatures: *i.e.*, to different alloy “microstructures”.

The reasonably good correlation of the IM alloy supports the importance of lattice self diffusion of aluminum atoms as rate controlling mechanisms for dislocation motion and creep behavior in this alloy, as suggested in the previous section. This result, and the fact that a stress exponent of $n=6.5$ is obtained, differs from Nieh’s idea that dislocation drag, (with $n=3$), as in class I solid solution alloys, is the dominant deformation mechanism [4]. It is also seen that the IM alloy is, as expected, more creep resistant than pure aluminum. This is due to the reinforcing effect of the Mg_2Si precipitates.

On the other hand, the normalized creep data of the PM 6061Al alloy fits well with a stress exponent value significantly higher; $n=12.3$. As expected, the PM alloy is more creep resistant than the IM alloy, and this is attributed to the presence of the alumina particles.

It is worth mentioning the common behavior manifested by the PM 6061Al alloy studied by Park *et al* [21] and the present PM 6061Al alloy. The correlation between both sets of data is excellent, with virtually the same n value: $n=12.3$ for the present data and about $n=11.8$ for Park’s *et al* data [21]. This similar behavior, which extends over nine orders of magnitude of normalized strain rate, reveals the importance of the dispersion of the alumina particles on the creep of aluminum alloys. The small difference in creep strength, being stronger the present alloy than Park’s *et al* one [21], may be attributed, as it will be seen later, to a slightly different dispersion of the alumina particles (larger particles and higher inter-particle distance in the Park *et al* alloy) or to the small differences in alloying elements (the alloying elements described in the Park alloy are, in wt%: 1%Mg, 0.6%Si, 0.28Cu, 0.2%Cr).

Discussion

As mentioned in the previous chapter, it is very reasonable to assume that lattice self-diffusion of aluminum is the dominant rate controlling mechanism when creep occurs at temperatures within an interval in which the precipitation of the coherent Mg_2Si happens. Then, if dislocation-climb is the dominant deformation mechanism during creep of 6061Al, a value of $n=5$ or $n=8$ should be obtained if the substructure, defined by a barrier spacing for dislocation motion, evolves with σ or is constant with σ (in accordance with Sherby’s sub-

structure invariant model), respectively [48]. However, it is $n=6.5$ for the IM alloy and about $n=18$ for the PM one. On the following, the creep data of the IM and PM alloys will be analyzed to understand the difference in n obtained from the above expected values.

a) Creep of the IM 6061Al alloy

Considering the temperatures at which creep tests have been conducted (573-723K, within the temperature interval defined by the data of dotted region of figure 3), it can be assumed that the microstructure during creep of 6061Al alloy is formed by some elements in solid solution in aluminum plus a dispersion of incoherent β particles. This dispersion is defined by an inter-particle distance, λ , which must be proportional to a barrier spacing for dislocation motion. Then, this alloy should creep under constant structure conditions, with $n=8$ if λ remains constant during testing according to the creep equation for the sub-structure invariant model,

$$\dot{\epsilon} = K \left(\frac{D_L}{b^2} \right) \left(\frac{\lambda}{b} \right)^3 \left(\frac{\sigma}{E} \right)^8 \quad (3)$$

where K is a material unitless constant (equal to about 10^9 for high staking fault energy materials) [26]. A dependence of λ with the applied stress, however, should be taken into account due to the Ostwald ripening phenomenon. This can be very different at the different applied stresses because the time of testing can be also dramatically different. At low applied stress, long testing times are needed to measure creep rates, whereas much shorter times are needed at high applied stresses. Consequently, at a low applied stress, long time of annealing and, hence, long time for Oswald ripening process allows for the formation of large particles, determining a long inter-particle distance, λ_1 in the scheme of Figure 5. On the contrary, high applied stress implies short time for Oswald ripening and smaller inter-particle distance; λ_2 in Figure 5, *i.e.*, $\lambda_1 > \lambda_2$. Therefore, for appropriate experimental data fit in figure 5, with a stress exponent value of $n=6.5$, it is proposed that creep in this alloy occurs creep under the basis of the sub-structure invariant model, equation (3), in which the interparticle distance, λ , increases with a decrease in σ , according to:

$$\lambda = A b \left(\frac{\sigma}{E} \right)^{-m} \quad (4)$$

where A is about four and it should be $m=1/2$. A detailed analysis to account for this dependence without conducting extensive TEM work to study in depth the shape and growth

rate of these particles would be rather speculative. This TEM work is, at present beyond the present investigation. However, a dependence of the type $\lambda \approx 1/\sqrt{\sigma}$ should be obeyed.

Equation (4) with $m=1/2$ accounts, as expected, for a weaker dependence of λ with σ than that due to subgrain formation, where it is $m=1$, leading to the classical creep equation for pure metals, equation (2), with $n=5$ [41].

b) Creep of the PM 6061Al alloy

The creep of the PM alloy, such as ODS alloys [26] and Park's *et al.* 6061Al PM alloy [21], can be explained bringing in a threshold stress term, σ_0 , into the creep equation in its general form, equation (2) [26],

$$\dot{\epsilon} = K \left(\frac{D_L}{b^2} \right) \left(\frac{\sigma - \sigma_0}{E} \right)^n \quad (5)$$

The usual way to determine σ_0 is well described elsewhere [21,43,44]. It consists on “imposing” an n value on equation (5) such that, at a given temperature and barrier spacing, a straight line should be obtained when the $1/n$ power of creep strain rate data is plotted as a function of σ/E . Then, the extrapolated value of σ at zero strain rate is σ_0 . Typically, the fits to obtain σ_0 are conducted using stress exponent values of $n=3$, $n=5$, and $n=8$, which are identified with specific deformation mechanisms [49]. As done by Park *et al* [21], the n value which best fits a straight line and the corresponding σ_0 are the values to be included in equation (5). Upon σ_0 normalization of the creep data (i.e., plotting $\dot{\epsilon}/D_L$ vs. $(\sigma - \sigma_0)/E$ in log-log scale), two important consequences derive: first, a “rational” value of Q_c should be obtained, and second, a good correlation of data with those of a similar alloy but without the dispersion of particles must result. Whereas the first consequence can be easily assessed by re-calculating Q_c from the new log-log plot, the second one request a comparison of creep data from alloys. This is more difficult to verify due to the lack of comparable data in the literature.

Because the precipitation evolution of 6061Al during testing and the different kinetics at the different temperatures the above procedure does not seem to be fully appropriate to calculate σ_0 . In fact, a stress exponent of 6.5 is obtained in the present case. To solve this, an alternative and rigorous procedure to calculate σ_0 from creep data of both alloys (with and without the alumina particles) is proposed. The procedure is based on the above second requirement that the creep data of both materials should correlate in a log-log plot of $\dot{\epsilon}/D_L$

vs. $(\sigma - \sigma_0)/E$. The creep behavior of the PM alloy should, then, obey the following equation [26],

$$\dot{\epsilon} = K \left(\frac{D_L}{b^2} \right) \left(\frac{\lambda}{b} \right)^3 \left(\frac{\sigma - \sigma_0}{E} \right)^8 \quad (6)$$

Where the dependence of λ with, now, $\sigma - \sigma_0$ is supposed to be similar as in the ingot alloy, equation (4). Under this condition, σ_0 at each temperature and any given strain rate can be readily taken as the difference between the normalized creep data of the PM and the IM alloys. This can be done with the present data since, as mentioned, the unique significant difference between the microstructure of the IM and the PM alloy is the dispersion of alumina particles. It is also assumed that the precipitation of the Mg_2Si particles and its kinetics is not affected by the presence of the alumina dispersion (the interparticle distance is very different).

Figure 6 represents the resulting σ_0/E values as a function of σ/E for the different testing temperatures calculated by this procedure. As can be seen, σ_0 barely changes with σ at all temperatures except at the lowest one, 573 K, at which a rapid drop of σ_0/E with σ/E is observed. There is no presently explanation for this results, but indicates that the origin of σ_0 at this temperature differs from that at higher temperatures.

The plot of figure 5 points out an essential difference between this new procedure to determine σ_0 and the classical one. The classical method imposes that σ_0 is independent of σ and that only variations with temperature are expected. In fact, this restriction justifies the expression “threshold” to this stress; the one that should be overcome for creep to progress. This term is microstructure dependent “only”. However, it might be possible that equation (5) “requests” a stress term σ_0 value that may vary with applied stress, as it occurs at 573 K (then, it would not be formally a threshold stress any more in the above sense). In other words, σ_0 would not be only a microstructure and temperature dependent term, but would depend also on test conditions (applied stress). This is not feasible with the classical method but, as deduced by the present one for the lowest temperature, is not physically impossible.

The resulting log-log plot of $\dot{\epsilon}/D_L$ vs. $(\sigma - \sigma_0)/E$ is shown in figure 7. As expected, the correlation is very good. Of course, on calculating σ_0 by this new procedure, the expected data correlation between the IM and the PM alloys is redundant since it is imposed by the method, but can give further insight into the significance of σ_0 .

c) Threshold stress: analysis and significance

As can be seen from figure 6, σ_0 decreases with increasing T for the highest three temperatures. This is better seen from the plot of figure 8 in which the average value of σ_0 is represented as a function of T . This dependence can be understood if the data are analyzed on the basis of the detachment model proposed by Artz *et al* [29,30]. This model considers an attractive force between particle and dislocation. In this manner, an extra stress is needed to be externally applied to “detach” the dislocation from the particle. This detachment stress is described by the equation,

$$\sigma_0 = 0.84 \frac{G b}{\lambda' - d} \sqrt{1 - k^2} \quad (7)$$

where G is the temperature dependent matrix shear modulus, λ' the alumina inter-particle distance, d the average particle diameter, and k is a temperature dependent parameter that accounts for the degree of interaction between the dislocation and the dispersoid. Artz and Wilkinson [29] defined this parameter as the ratio of the dislocation line energy in the matrix-reinforcement interphase, T_p , and in the matrix $T_M = G \frac{b^2}{2}$; therefore:

$$T_p = k G \frac{b^2}{2} \quad (8)$$

It varies between $k=1$, when there is no interaction ($\sigma_0=0$), and $k=0$ when the interaction is maximum. It is proposed here that the attractive interaction between dislocations and particles is null ($k=1$) at a sufficient high temperature, then;

$$T_p = G_0 \frac{b^2}{2} \quad (9)$$

where G_0 is the matrix shear modulus at the temperature where $k=1$. In the most simple case, *i.e.*, considering that the temperature dependence of k is only that of the temperature dependence of G , it is readily found an interpretation of the parameter k through equations (8) and (9), according to,

$$k = \frac{G_0}{G(T)} \quad (10)$$

where G_0 is the matrix shear modulus at the temperature T_0 , at which the extrapolated data of σ_0 in the plot of figure 8 results in $\sigma_0 = 0$. It is found by linear extrapolation that this temperature is $T_0 \approx 810$ K, at which the shear modulus is, $G_0 = 17970$ MPa [39].

It is possible now to predict σ_0 from the model, equation (7), and compare with experimental data, giving realistic values of λ' and d . To this purpose, it is necessary to consider the precise size distribution of the 6061Al powder, Table III, and the severe extrusion ratio employed in the preparation of the PM alloy. The particle size distribution of the aluminum powders of Table III leads to a volume fraction of alumina particles of $f_v=0.087\%$. The Al_2O_3 particles were considered as platelets of about 4 nm in thickness (which is the thickness of the Al_2O_3 surface layer [21]). It is assumed that the large extrusion ratio leads to a severe breakage of the alumina particles. However, it is not expected alumina particles with a diameter smaller than the thickness of 4 nm. In figure 8, the model prediction for particles of 4 nm in thickness and three particle diameters: 4, 7 and 10 nm, is indicated. A good correlation with experimental data for the case of particles with a diameter in the range of 10-7 nm is evident. This good correlation supports the validity of the Artz-Wilkinson model to understand the interaction between dislocations and the ceramic particles. The dependence of the experimental data with temperature is slightly more pronounced than that predicted by the model, which suggests that possibly other effects should be also taken into account.

In this figure, also the threshold stress data from Park et al [21] is shown. The dependence of σ_0 with T is very similar to that of the present investigation. The trend predicted by the model for the case of particles of 50 nm in diameter and 4 nm in thickness (which is the size of the alumina particles in this investigation), is indicated (in this case, $T_0 \approx 780$ K, at which $G_0 = 18480$ MPa). It is also seen a very good correlation between experimental data and model prediction giving further support to the validity of the Artz-Wilkinson model, the origin of the threshold stress for PM alloys in equation (6), and its dependence with temperature; in essence that derived from the temperature dependence of the matrix shear modulus, as deduced from equations (7) and (10).

Conclusions

The creep behavior of 6061Al alloy obtained by ingot and powder metallurgical routes (IM and PM, respectively) has been studied and analyzed in the context of the creep data from published investigations on this alloy. The following are the most relevant findings of this investigation:

- 1.- The creep of the ingot 6061Al alloy within a given interval of tests temperatures in which the Mg_2Si precipitates are formed, is dominated by a climb-controlled dislocation mechanism in which the rate controlling process is self-diffusion of aluminum atoms.

2.- The substructure invariant model (equation 3) has been successfully used to analyze the creep of this alloy, where a dependence of the interparticle distance with the applied stress of the form $\lambda \propto 1/\sqrt{\sigma}$ is found. This dependence leads to a stress exponent value of $n=6.5$.

3.- The creep of the powder metallurgy 6061Al alloy can be explained in terms of the behavior of the ingot alloy with an additional stress, σ_0 , or threshold stress due to the interaction of dislocations with the dispersion of Al_2O_3 particles present in powder metallurgical Al alloys.

4.- The threshold stress can be readily calculated as the stress difference between the applied stresses needed to reach a given strain rate in the powder metallurgy and the ingot metallurgy alloys. This stress term can be well interpreted as a particle-dislocation interaction mechanism according to the Artz-Wilkinson model. The temperature dependence of σ_0 appears naturally in this model through the temperature dependence of the shear modulus.

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References

- [1] McDanel DL. Metall. Trans. 16A (1985) 1105-1115.
- [2] Clyne TW, Withers PJ. An introduction to Metal Matrix Composites (Cambridge, MA): Cambridge Univ Press; 1993.
- [3] Geiger AL, Walker JA. The processing and properties of discontinuously reinforced aluminum composites, JOM 1991; 45(8):8.
- [4] Nieh TG. Metall Trans. 15A (1984) 139-145.
- [5] Ashby MF. In *Materials Selection in Mechanical Design*. Pergamon Press. Oxford, 1992.
- [6] Aluminum and Aluminum alloys. ASM Specialty Handbook. J.R Davis, Davis & Associated Eds. ASM International. Materials Park, OH. 1993.
- [7] Borrego A, González-Doncel G. Mater Sci. Eng. A245 (1998) 10-18.
- [8] García-Cordovilla C, Louis E, Narciso J, Pamies A. Mater Sci. Eng. A189 (1994) 219-227.
- [9] Papazian, JM. Metall. Trans. 19A (1988) 2945-2953.

- [10] Jacobs MH. *Phil Mag.* 26 (1972) 1-13.
- [11] Song Y, Baker TN. *Mater Sci. Eng. A201* (1995) 251-260.
- [12] Kovács I, Lendvai J, Nagy E. *Acta Metall.* 20 (1972) 975-983.
- [13] Mittemeijer EJ. *J. Mater. Sci.* 27 (1992) 3977-3987.
- [14] Borrego A, González-Doncel G. *Mater Sci. Eng. A245* (1998) 149-152
- [15] Starink MJ. *Int Mater Rev.* 49 (2004) 191-226.
- [16] Doan LC, Ohmori Y, Nakai K. *Mater Trans JIM.* 41 (2000) 300-3005.
- [17] Requena GC, Deguischer HP. *Z. Metallkd.* 96 (2005) 805-811.
- [18] Pickens JR, Langan TJ, England RO, Liebson M. *Metall. Trans.* 18A (1987) 303-312.
- [19] Morimoto T, Yamaoka T, Lilholt H, Taya M. *J. Eng. Mater. & Technol.* 110 (1988) 70-76.
- [20] Luster JW, Thumann M, Baumann R. *Mater. Sci. & Technol.* 9 (1993) 853-862.
- [21] Park KT, Lavernia EJ, Mohamed FA, *Acta Metall. Mater.* 42 (1994) 667-678.
- [22] Furukawa M, Wang J, Horita Z, Nemoto M, Ma Y, Langdon T, *Metall. Mater. Trans.* 26A (1995) 633-639.
- [23] Matsuda N, Akaike J, Hongo K, Matsuura K. *Mater. Sci. & Eng. A234* (1997) 751-754.
- [24] Sakaris SP, McQueen HJ. In "Advances in production and fabrication of light metals", ed. by M.M. Avedesian et al. Pub. by CIM Montreal (1992) 605-617.
- [25] Bhagat RB, House MB. *Mater Sci. Eng. A144* (1991) 319-326.
- [26] Lin J, Sherby OD. *Res Mechanica*, 2 (1981) 251-293.
- [27] Li Y, Langdon TG. *Mater. Sci. Eng. A245* (1998) 1-9.
- [28] Mohamed FA, Murty KL, Morris Jr JW. *Metall Trans.* 4 (1973) 935-940.
- [29] Artz E, Wilkinson SD. *Acta Metall.* 34 (1986) 1893-1898.
- [30] Rösler J, Artz E. *Acta Metall Mater.* 38 (1990) 671-683.
- [31] Borrego A, Ibáñez J, López V, Lieblisch M, González-Doncel G. *Scripta Mater.* 34 (1996) 471-478.
- [32] Borrego A, Fernández R, Cristina MC, Ibáñez J, González-Doncel G. *Comp. Sci. Technol.* 62 (2002) 731-742.
- [33] McHargue CJ, Jetter LK, Ogle JC. *Trans Met Soc AIME.* 215 (1959) 831-837.
- [34] Bruno G, Fernández R, González-Doncel G. *Mater. Sci. Eng. A382* (2004) 188-197.
- [35] Nieh TG, Karlak RF. *Scripta Metall.* 18 (1984) 25-28.
- [36] Dutta I, Allen SM, Hafley JL. *Metall. Trans.* A22 (1991) 2553-2563.
- [37] Kovács I, Lendvai J, Nagy E. *Acta Metall.* 20 (1972) 975-983.

- [38] Frost HJ, Ashby MF. in *Deformation Mechanisms Maps*. Pergamon Press. Oxford. (1982).
- [39] Simmons GS, Wang H. in *Single Crystal Elastic Constants and Calculated Aggregate Properties: a Handbook*. MIT Press. Cambridge, Mass. (1971).
- [40] Servi IS, Grant NJ, Trans AIME. 191 (1951) 909-916.
- [41] Sherby OD, Burke P. Prog Mater Sic. 13 (1968) 325-390.
- [42] Nieh TG, Chellman DJ. Scripta Mater 18 (1984) 925-928.
- [43] González-Doncel G, Sherby OD. Acta Metall Mater. 41 (1993) 2797-2805.
- [44] Mishra RS, Pandey AB. Metall Trans. 21A (1990) 2089-2090.
- [45] Clauer AH, Hansen N. Acta Metall. 32 (1984) 269-278.
- [46] Spigarelli S, Cabibbo M, Evangelista E, Langdon TG. Mater. Sci. Eng. A328 (2002) 39-47.
- [47] Krajewski PE, Allison JE, Jones JW. Metall Trans. 24A (1993) 2731-2741.
- [48] Sherby OD, Klundt RH, Miller AK. Metall Trans 8A (1977) 843-850.
- [49] Sherby OD, Walser B. Scripta Metall. 16 (1982) 213-219.
- [50] Peng LM, Zhu SJ, M ZY, Bi J, Chen HR, Wang FG. Mater. Sci. Eng. A259 (1999) 25-33.
- [51] Carreño F, Ruano OA. Metall Mater Trans. 30A (1999) 371-376.
- [52] Mishra RA, Pandey AB, Mukherjee AK. Mater. Sci. Eng. A201 (1995) 205-210.
- [53] Kassner ME, Pérez-Prado MT. Prog Mater Sic. 13 (2000) 1-102.

Figure captions

Figure 1.- Double logarithmic plot of minimum or steady state strain rate as a function of applied stress of all the data published in the open literature on the creep behavior of 6061Al alloy (see symbols legend in Table I). Numbers denote the stress exponent.

Figure 2.- Steady state or minimum creep rate as a function of the applied stress in a double logarithmic plot of, a) the IM 6061Al alloy (E183) and b) the PM alloy (E220). Numbers denote the stress exponent at each temperature.

Figure 3.- Normalized minimum creep rate with D_1 as a function of the normalized applied stress with E for the different studies taken from the literature on creep of 6061Al alloy (see figure 2). For comparison, the creep data of pure aluminum [40] at different temperatures are also shown (see symbols legend in Table I).

Figure 4.- Normalized creep rate with D_1 as a function of the normalized applied stress with Young's modulus for the IM and PM alloys. The trend marked by the pure aluminum and the region defined by the creep data reported in the literature (Figure 3) is also included, for comparison. Within this region, the data from Park's *et al* [21] PM alloy is specified. A good correlation with the present data of the PM alloy (E220), covering altogether a common behavior over nine orders of magnitude of D_1 compensated strain rate, is observed.

Figure 5.- Scheme of the proposed model to account for the stress exponent of about $n=6.5$ of the IM 6061Al alloy on the basis of the sub-structure invariant model, equation (3), and the weak dependence of λ with σ (stress and strain rate logarithmic scales in arbitrary units, a.u.). Due to the Ostwald ripening phenomenon, Mg_2Si precipitates (represented as spheres for simplicity) are larger and more spaced ($\lambda_1 < \lambda_2$) at low than at high applied stress because of longer testing time.

Figure 6.- Dependence of the Young's modulus normalized threshold stress as a function of the Young's modulus normalized applied stress for the PM 6061Al alloy, at the four temperatures studied.

Figure 7.- Double logarithmic plot of the diffusion coefficient normalized creep rate as a function of the Young's modulus normalized effective stress for the IM and PM 6061Al alloys of the present investigation.

Figure 8.- Dependence of the average threshold stress, σ_0 , with testing temperature in the range where this stress is approximately constant with applied stress and prediction of this dependence according to the Artz-Wilkinson model. A good correlation is observed between experimental data and model prediction. The experimental data from Park *et al* [21] are also included in the plot as dark circles. The drop of σ_0 with T also agrees quite well with the prediction of the Artz-Wilkinson model for the particle size of $d=50$ nm.

Author [ref.]	Symbol in Figures	Processing	Applied stress (Mpa)	Test mode	Test temperature (K)	Heat Treatment	Remarks
T.G. Nieh [4]	◇	IM	25 - 70	Tensile	561	-----	Dislocations viscous glide. Absence of primary creep.
T. Morimoto <i>et al.</i> [19]	◁	IM	26 – 38	Tensile	573	T6 (not specified)	Better creep resistance than Nieh's alloy because of T6 condition.
J.W. Luster <i>et al</i> [20]	▵	IM	190-270	Tensile	423	T6*	Dislocation climb is not rate controlling. Lack of steady state. Premature onset of tertiary creep.
K.T. Park <i>et al</i> [21].	⊕ ⊗	PM	15.7 – 43.1	Double Shear	648, 678	-----	Threshold stress analysed by three methods.
M. Furukawa <i>et al</i> [22]	⊠ ⊡	IM	13.1 - 60	Tensile	623, 673	T6**	Diffusion of either Mg or Al atoms governs diffusion coefficient for the occurrence of power law breakdown.
N. Matsuda <i>et al</i> [23]	⊙ ⊛	IM	2.1 - 5.3 and 26.4 - 65.2	Tensile	573, 773	803 K / 2h	Transition between 573 K and 673 K of alloy's structure. Data analysis by the θ projection method.
J.R. Pickens <i>et al</i> [18]	△	PM	13.9 – 47.4	Torsion	700	-----	$Q_c = 142$ kJ/mol
P. Sakaris and H.J. McQueen [24]	◁	IM	25.3 – 44.4	Torsion	723	-----	Alloy undergoes dynamic recovery and particle coarsening. Absence of dynamic recrystallization. $Q_c = 188$ kJ/mol
G.C. Requena and H.P. Deguischer [17]	▽	IM	15 - 70	Tensile	573	T6***	Dislocations viscous glide at <50 MPa. Particle coarsening of Mg_2Si influencing stress exponent.
R.B. Bhagat and M.B. House [25]	▷	PM	60	Tensile	561	-----	---

* (solubilization 796 K/30 min., water quench, and annealing 433 K/24 h)

** (solubilization 803 K/90 min., water quench, pre-ageing 20 h at room temperature and annealing 448 K/8 h)

*** (solubilization 803 K/30 min., water quench, annealing 433 K/24 h, overaging 573 K/2 h)

Table I. Summary of the creep studies on 6061Al alloy conducted by different authors.

	Mg	Si	Cu	Cr	Mn	Fe	Ti	Zn	Al
6061Al IM	0.89 ±0.02	0.46 ±0.01	0.239 ±0.001	0.1262 ±0.0001	0.0736 ±0.0006	0.3614 ±0.0008	-	-	Bal.
6061Al PM	0.96 ±0.02	0.45 ±0.01	0.27 ±0.02	0.16 ±0.01	0.0023 ±0.0004	0.15 ±0.01	0.0195 ±0.0001	0.0049 ±0.0001	Bal.

Table II. Composition of the IM and PM 6061Al alloys investigated in the present work.

Class (size, μm)	6	8	10	20	30	40	50	63	74	104	150
Frecuency (%)	1.46	1.67	2.11	21.37	26.14	16.54	29.14	0.84	0.30	0.2	0.2

Table III. Particle diameter distribution of the 6061Al powder used in the preparation of the 6061Al PM alloy.

Figure 1.- Double logarithmic plot of minimum or steady state strain rate as a function of applied stress of all the data published in the open literature on the creep behavior of 6061Al alloy (see symbols legend in Table I). Numbers denote the stress exponent.

a)

b)

Figure 2.- Steady state or minimum creep rate as a function of the applied stress in a double logarithmic plot of, a) the IM 6061Al alloy (E183) and b) the PM alloy (E220). Numbers denote the stress exponent at each temperature.

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