HIGH STRAIN RATE SUPERPLASTICITY OF A POWDER METALLURGY, Zr-MODIFIED 2124 ALUMINUM

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Abstract

The high-temperature mechanical properties of powder metallurgy (PM) 2124 Al alloys containing 0.6wt% and 0.12wt%Zr and ingot metallurgy (IM) 2024 Al have been characterized. The alloys were tested in tension to failure at temperatures in the range from 425 to 500°C, and strain rates in the range from 8.3 x 10^{-4} to 6.7 x 10^{-1} s^{-1} (5 to 4000%/min) in air. At high strain rates (3.3 x 10^{-1} s^{-1}), 0.6wt%Zr-2124 Al exhibited superplasticity; a maximum tensile elongation of about 500% was recorded at 475°C. This high-strain-rate superplasticity (HSRS) phenomenon results from the fact that the alloy has an extremely fine grain size (~ 1 μm), as a result of the Zr addition. A comparison of the deformation properties was made between the 0.6wt%Zr-2124 Al alloy and a fine-grained 20%SiC_w-2124 Al composite, which also exhibits superplasticity at high strain rates. These deformation properties have been compared with those obtained from a non-superplastic, coarse-grained, PM 0.12%Zr-2124 and an IM 2024 Al.

Introduction

One of the major drawbacks of conventional superplastic forming is that the phenomenon is only found at relatively low strain rates, typically about 10^{-4}-10^{-3} s^{-1} [1]. Recent studies have demonstrated that superplasticity can exist at considerably higher strain rates than 10^{-3} s^{-1}. This high strain rate superplasticity (HSRS) phenomenon has now been observed principally in metal matrix composites [2-5] and mechanically-alloyed materials [6-11], but it has also been observed in more-conventionally produced metallic alloys [12-14].

The mechanism of grain boundary sliding (GBS) is generally believed to be the dominant deformation mode in fine-grained, superplastic alloys. In the case of GBS, the most dominant microstructural parameter is certainly grain size; an increased strain rate is predicted for optimal superplastic flow with a decrease in grain size. For this reason, prior efforts to produce superplasticity have centered on grain size refinement. It is well known that Zr additions to Al can form Al_3Zr precipitates that greatly reduce the grain size. In 1985 Nieh and Wadsworth [12] modified 2124 Al by adding 0.6% Zr and observed high strain rate superplasticity. Subsequently, Furushiro and coworkers [13, 14] extended this concept to 7475 Al and Al-10%Mg alloys, by modifying them with various amount of Zr, and observed HSRS. Very recently, Nieh and Wadsworth [15], reported details on the initial observation that a 0.6%Zr-modified 2124 Al alloy. In the present paper, we demonstrate the beneficial effects of Zr addition to 2124 Al alloys on the improvement of superplastic strain rate. In addition, a comparison of the deformation properties are made between HSRS in 0.6%Zr-modified 2124 Al and 20%SiC_w-2124Al [16]. These deformation properties are further compared with those obtained from non-superplastic, powder-metallurgy (PM) 0.12%Zr-2124, and ingot-metallurgy (IM) 2024 Al.

Materials and Experiments

The IM 2024 Al and PM 0.12%Zr- and 0.6%Zr-modified 2124 Al (denoted as 0.12Zr-2124 and 0.6Zr-2124, herein) alloys used in this study were prepared by ALCOA. The materials were received in the form of extruded bars and the chemical compositions are listed in Table 1. In the extruded form, the alloy did not exhibit unusual deformation properties. The material was then thermomechanically processed by rolling, using a process that was essentially analogous to the overage practice [17]. Typically, the material was solution treated at 500°C for 2 h, water quenched, and overaged at 399, 427, or 454°C, followed by isothermal rolling at 300°C.

Table 1 Compositions (wt.%) of IM 2024 and PM 2124-Zr Al Alloys

	Cu	Mg	Mn	Fe	Si	Zr	Al
PM 2124 Al-0.6%Zr	3.67	1.84	0.16	0.03	0.03	0.60	balance
PM 2124 Al-0.12%Zr	3.73	1.81	0.14	0.04	0.02	0.12	balance
IM 2024 Al	3.48	1.70	0.62	0.12	-	-	balance

Tensile samples of 12.7 mm gage length for mechanical property evaluation were machined from the rolled sheets in both the longitudinal and transverse direction with respect to the original extrusion direction. Samples were tested in tension to failure at temperatures in the range from 425 to 500°C, in air, using an Instron testing machine at a constant crosshead velocity. Strain rates in the range of 8.3 x 10^{-4} to 6.7 x 10^{-1} s^{-1} (i.e. 5 to 4000%/min) were used. The flow stresses for each of the strain rates were determined at a large fixed strain (~ 0.5). Tensile samples of the same configuration were also machined from the as-received extrusion stock and tested in order to determine the influence of thermomechanical processing.

Results

The as-received, i.e., the extruded, 0.12Zr- and 0.6Zr-2124 alloys were initially evaluated and exhibited only moderate ductilities; for example, over the strain rate range 8.3 x 10^{-4} to 3.3 x 10^{-1} s^{-1}, at 450°C, values of elongation-to-failure of less than 80% were recorded. Both alloys were subsequently thermomechanically processed. The processed 0.12Zr-2124, however, still exhibited only moderate ductility. In contrast, the processed 0.6Zr-2124 revealed superplastic behavior. The elongations-to-failure as a function of the initial strain rate, at temperatures of 425, 450, 475, and 500°C for the processed 0.6Zr-2124, are presented in Fig. 1. At low strain rates, the values of elongation-to-failure for the processed material are not much better than those of the as-received material. The elongation-to-failure, however, is noted to increase with an increase in the initial strain rate and reaches a maximum at a strain rate of about 10^{-1}-5 x 10^{-1} s^{-1} for all testing temperatures. An optimum elongation of 500% was recorded at 475°C at a strain rate of 3.3 x 10^{-1} s^{-1}. The strain rate is almost 10 to 100 times higher than the strain rates at which superplasticity occurs in most of the other advanced aluminum alloys, e.g., 7475 Al [18] and Al-Li alloys [19]. Also noted in Fig. 1, is the fact that the superplastic elongation begins to decrease at strain rates higher than 3.3 x 10^{-1} s^{-1}.

The logarithm of strain rate as a function of the logarithm of flow stress at a fixed deformation strain of 0.1 for the 0.6Zr-2124 alloy is given in Fig. 2. The stress exponent, n, in the equation $\dot{\varepsilon} \propto \sigma^n$, is approximately 5 in the low strain rate (or stress) regime (where σ is the flow stress, and $\dot{\varepsilon}$ is the strain rate). As strain rate increases, there is a transition in the flow properties. Specifically, in the high strain rate regime ($\geq 10^{-1}$ s^{-1}), the n values decrease to about 2 at each of the testing temperatures. This decrease is consistent with the observation that the tensile elongation of the alloy also increases with increasing strain rate. Also to be noted in Fig. 1, however, is the fact that the tensile elongation of the alloy decreases at extremely high strain rates, although the n value remains low. This is believed to be the consequence of extensive cavitation, and thus premature failure, taking place in the test samples at extremely high strain rates [20]. In other words, the optimum strain rate for superplasticity in this alloy is achieved under conditions whereby grain boundary sliding rates are properly balanced by accommodation processes, such as dislocation slip or atom diffusion. Otherwise, cavitation is expected to intervene and reduce tensile elongation.

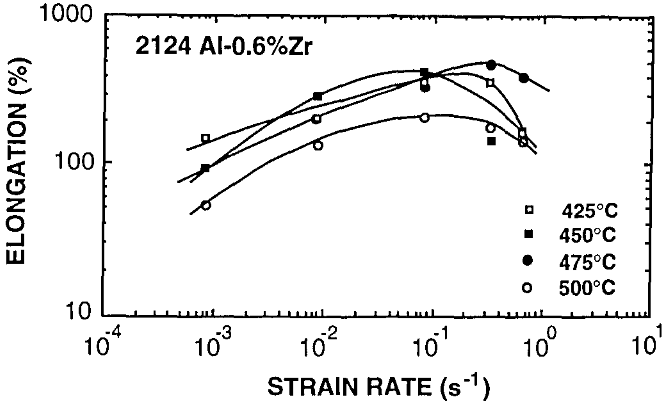

Fig. 1

Elongation-to-failure as a function of strain rate for 0.6Zr-2124 alloy at temperatures from 425 to 500°C.

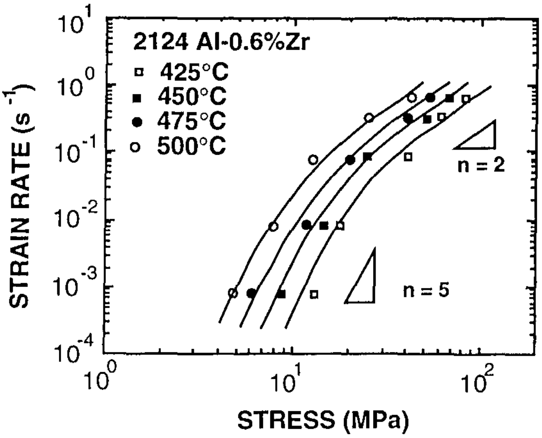

Fig. 2

Logarithm of strain rate as a function of logarithm of stress for 0.6Zr-2124 Al alloy at temperatures in the range from 425 to 500°C.

The activation energies, Q, can be estimated from the stress-strain rate data shown in Fig. 3; they are measured to be 88.6 and 267 kJ/mol in the high modulus-compensated stress ($\sigma/E = 1.8 \times 10^{-3}$) and low modulus-compensated stress ($\sigma/E = 1.8 \times 10^{-4}$) regimes, respectively. It is noted that although the activation energy for short-circuit, grain boundary, self diffusion in Al is not available, the activation energy for dislocation pipe diffusion in Al has been reported to be about 82 kJ/mol [21]. This value is similar to the Q value measured in the high stress regime (i.e., 88.6 kJ/mol). The value of 267 kJ/mol is, however, almost twice the value of the activation energy for the lattice self diffusion of aluminum [22]. The interpretation of the high Q value measured in the low stress regime is not clear, but a high Q value has been observed in many HSRS cases at low stress.

Discussion

The addition of 0.6wt% Zr to 2124 Al can lead to superplasticity. This is not the case for a more dilute addition of 0.12wt% Zr to 2124 Al. The addition of 0.12wt%Zr to 2124 Al is apparently insufficient to result in the fine grain sizes (less than ~10 μm) necessary for superplasticity. This result indicates that there is a lower bound limit of the Zr content necessary to produce superplasticity. For the commercial SUPRAL alloys (Al-6Cu based), an amount of Zr from about 0.4 to 0.5 wt.% is considered to be necessary [23]. This is in general agreement with the observations of Furushiro and Hori [13]; they carried out a systematic study on the effect of Zr additions on the superplastic behavior of 7475 Al-based alloys and found that a minimum of 0.3wt% Zr was necessary to produce significant grain refinement. Work on the influence of Zr addition on superplasticity in Al-Li alloys also led to a similar observation of the beneficial effects of increases levels of Zr [24]. It is pointed out, however, that superplasticity has been observed in some Al-Cu-Li-Mg-Zr alloys which were manufactured by ingot metallurgy and were restricted to having levels of 0.12 to 0.18wt%Zr [25]. Obviously, the exact amount of Zr necessary for leading to fine grain sizes, and thus superplasticity in an aluminum alloy, is dependent upon both the chemical composition of the alloy and processing steps. Nonetheless, the fact that superplasticity is observed in the present 0.6Zr-2124 Al alloy is generally consistent with the previous results.

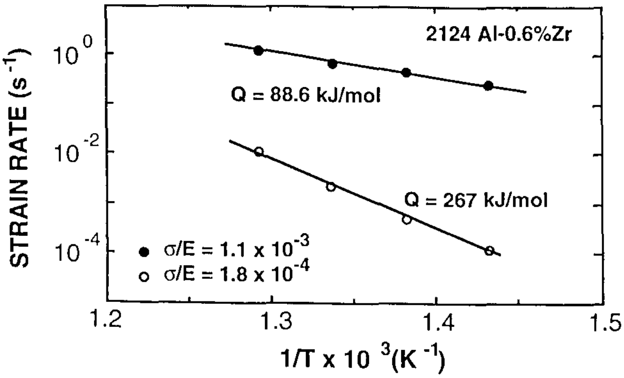

Fig. 3

Activation energies for 0.6Zr-2124 Al alloy in the low and high stress regions are calculated to be 267 and 88.6 kJ/mol, respectively.

High strain rate superplasticity has previously been observed in both SiC (SiC_w) and Si_3N_4 whiskers reinforced 2124 Al composites [2,3]. It is intriguing to compare the experimental results observed from the 0.6Zr-2124 alloy with those obtained from the studies on composites. A comparison of the flow behavior between the 0.6Zr-2124 alloy and that of the SiC_w-2124 composite is given in Fig. 4. It is obvious that in the low strain rate regime, the composite is more resistant to deformation than the alloy. In addition, in this regime, the "apparent" n value of 5 for the alloy is lower than that for the composite, which is about 8. For the composite, the high stress exponent for the composite has been associated with the existence of a threshold stress, as suggested by Pandey *et al* [26,27]. A stress exponent of 5 for the alloy appears to be consistent with a dislocation climb mechanism, as observed during the creep of metal alloys [1].

Although the composite appears to be stronger than the alloy in the low strain rate regime, the strength of the alloy is noted to be similar to that of the composite in the high strain rate regime. In fact, in the high strain rate regime, the stress exponents for both materials are noted to decrease and approach a value of 2. This indicates that a similar deformation mechanism probably operates in both materials. Specifically, a stress exponent of 2 is frequently observed in superplastic materials, in which grain boundary sliding is generally accepted to be the dominant deformation mode. The fact that the presently-measured activation energy (88.6 kJ/mol) is close to the activation energy for short-circuit diffusion (82 kJ/mol) provides indirect support for such a mechanism. A similar n value for the alloy and composite is attributed to the result that the matrix grain sizes for these two materials are similar (~1 µm). It is further pointed out that the diameter of the SiC_w (~0.5 µm) in the composite is also similar to the grain size of the matrix alloys. For boundary sliding processes, interfacial boundaries are expected to behave in a similar manner to grain boundaries. To a first order approximation, and from an interface sliding point of view, one could therefore treat the whiskers as individual grains. If this were the case, then the grain size effect applied to alloys would be mechanistically also applicable to composites.

It is of importance to note, however, that Nieh and Wadsworth [28, 29] have suggested that the presence of a liquid phase resulting from the test conditions, or the presence of a low melting point region resulting from solute segregation (e.g., at the whisker-matrix interface in metal-matrix composites), may be responsible for the observed HSRS phenomenon in the SiC_w-2124 composite. In the case of the 0.6Zr-2124, the lowest testing temperature is 425°C which is about 75°C lower than the solidus of 2124 Al (502°C [16]). The presence of a low melting point region appears to be improbable at this temperature. Furthermore, as shown in Fig. 2, there is no indication of any change in plastic flow behavior from 425 to 500°C. Therefore, despite the fact that boundary sliding processes prevail in both 0.6Zr-2124 and SiC_w-2124, the exact microscopic sliding mechanisms may be different in these two materials.

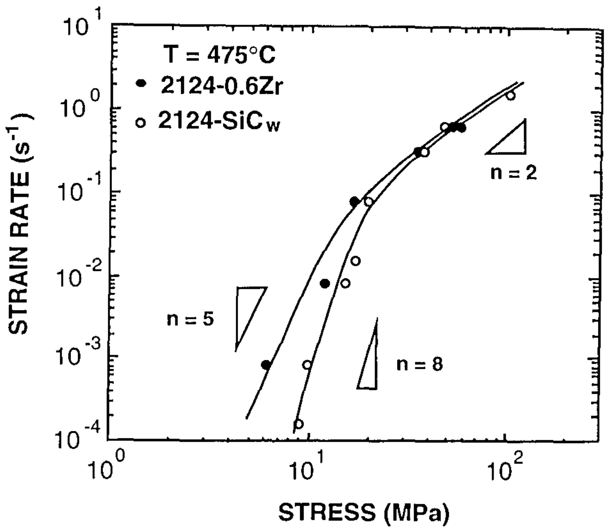

Fig. 4

A direct comparison of strain rate-stress between a 20%SiC_W-2124 Al composite and the 0.6Zr-2124 Al alloy. A transition in flow behavior occurs at a strain rate of about 10^{-1} s^{-1} in each material.

To investigate the effect of grain size, relatively coarse-grained (50 μm) IM 2024 Al and relatively coarse-grained (20 μm) PM 0.12Zr-2124 samples were prepared and tested. The deformation properties of these coarse-grained alloys are directly compared with those of 0.6Zr-2124 in Fig. 5. Neither the coarse-grained 2024 nor 2124 Al alloys are superplastic. It is pointed out in Fig. 5 that in the low strain rate regime, at a given stress, the strain rate for the fine-grained 0.6Zr-2124 is about 100 times faster than that for the coarse-grained materials. However, the n values for all these alloys are approximately 5 in this regime; this value is similar to that reported by Lilholt and Taya [30], in a study of creep of nominal 2124 Al. In the high strain rate regime (>10^{-1} s^{-1}), on the other hand, the strain rate-stress behavior for fine-grained 0.6Zr-2124 is noted to deviate greatly from those for the coarse-grained 0.12Zr-2124 and 2024 Al. Specifically, both the 2024 and 0.12Zr-2124 alloys exhibit a power-law breakdown behavior at a strain rate above about 10^{-1} s^{-1}, behavior that is similar to that in pure aluminum [31]. In contrast, above this strain rate, a grain boundary sliding mechanism appears to intervene in the case of fine-grained 0.6Zr-2124 alloy.

As mentioned previously, in the low strain rate regime, an apparent stress exponent of 5 in the 0.6Zr-2124 alloy indicates that a dislocation climb mechanism prevails. In the case of climb-controlled creep, the constitutive equation can be expressed as [1]:

$$\dot{\varepsilon} = A\cdot\left(\frac{Gb}{kT}\right)\cdot\left(\frac{\sigma}{E}\right)^5\cdot exp\left(-\frac{Q}{RT}\right) \quad (2)$$

where G is the shear modulus, b is Burgers vector, k is Boltzmann's constant, R is the gas constant, T is the absolute temperature, E is Young's Modulus, Q is the activation energy, and A is a material constant, which is noted to be independent of grain size. Examining the above equation, the difference in creep rates between 0.6Zr-2124 and the other two alloys does not appear to be obvious and cannot be accounted for by differences in modulus. Such differences have sometimes been accounted for by invoking variables contained in the value of A [32]. In the present case, the three alloys have virtually the same chemical compositions. It is difficult to conceive that microstructural parameters, such as grain, subgrain, and stacking fault energy, can cause a major difference in the A value in these alloys. Therefore, despite the fact that the apparent stress exponent is about 5, the deformation mechanism in 0.6Zr-2124 may not be dislocation climb.

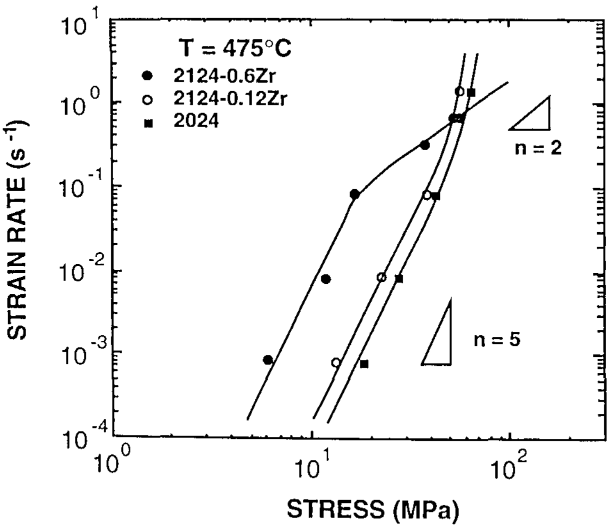

Fig. 5

Comparison of strain rate-stress data from fine-grained, PM 0.6Zr-2124 Al alloy (1 μm) and coarse-grained IM 2024 (50 μm) and PM 0.12Zr-2124 Al (20 μm) alloys.

Because of a high Zr concentration, and thus a high density of Al_3Zr particles, it is anticipated that there exists an appreciable threshold stress, σ_{th}, during the creep of 0.6Zr-2124 alloy. This threshold stress, in combination with a grain boundary sliding mechanism (n = 2), is expected to result in an apparent n value higher than 2 at stresses near σ_{th}; this is schematically illustrated in Fig. 6. Within the strain rate range used in the present study of the 0.6Zr-2124 alloy, the apparent n value is about 5. It is worth noting that a similar concept has been proposed by Gregory *et al.* [10], in the study of high strain rate superplasticity in mechanically-alloyed, nickel-based materials, MA754 and MA6000. In these two materials, Gregory *et al.* argued that a combination of slip with Coble creep (in which the Coble creep exhibits a threshold stress) can explain the phenomenon. It is also of interest to point out in Fig. 2 that the threshold stress is temperature-dependent; σ_{th} decreases with increasing temperature.

An overview of the superplastic behavior of metal alloys, and in particular of Al alloys, to demonstrate the overwhelming effect of grain size, is given in Fig. 7. In this figure, the elongation-to-failure is shown as a function of strain rate for (i) 7475 Al, (ii) a USSR alloy V96Ts that is rather similar in composition to the 7000 series alloy (but contains Zr as a grain refining element instead of Cr), (iii) Al-Li 2090 alloy, (iv) the commercial SUPRAL alloys, (v) SiC_w/Al and $Si_3N_{4(w)}$/Al composites, (vi) the Zr-modified Al 2124 alloy of the present study, (vii) a Zr-modified Al 7475, and (viii) mechanically-alloyed Al 9021. The grain size ranges of these alloy groups are 10-20 μm for the 7475 Al alloys, about 5 μm for the USSR alloy, 2-3 μm for the 2090 Al and SUPRAL alloys, 1 μm for the SiC_w/Al and $Si_3N_{4(w)}$/Al composites and Zr-modified Al 2124 and 7475 Al, and 0.5 μm for Al 9021 and Al 90211. Obviously, grain size plays a dominant role in determining the optimal superplastic strain rate. (It should be emphasized that a fine grain size is a necessary but insufficient condition for superplasticity [28].) The general trend in Fig. 6, i.e., an increased strain rate for optimal superplastic flow with a decrease in grain size, is predicted from the phenomenological equation for superplasticity [1], i.e.,

$$\dot{\varepsilon} = A' DG(\frac{b}{d})^p (\frac{\sigma}{G})^n \qquad (3)$$

where D is the appropriate diffusion coefficient, d is the grain size, and A' and p are constants. The p value usually is found to be equal to 2 or 3. With a decrease in grain size, the superplastic strain rate therefore rapidly increases. Typically, refinement of the grain size by a factor of 2 would be expected to increase the optimum strain rate for superplastic flow by a factor of from 4 to 8 depending upon the precise grain size relationship as described above.

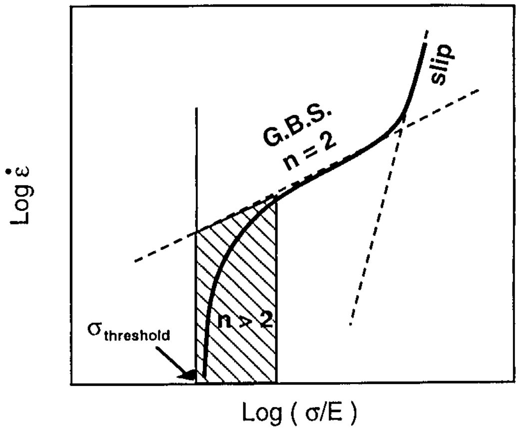

Fig. 6

Schematic representation of deformation mechanism regimes, including a grain boundary sliding regime in which a threshold stress is present. The threshold stress is associated with the presence of a large volume fraction of particles.

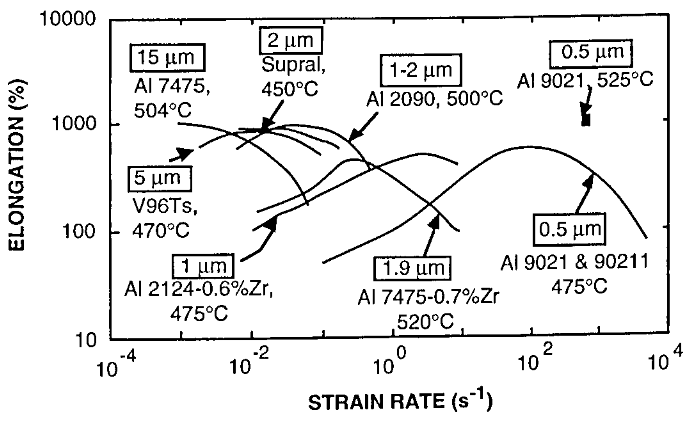

Fig. 7

An overview of the superplastic behavior of Al alloys to illustrate the grain size effect.

Conclusion

The high-temperature deformation properties of a 2124 Al alloy containing 0.6wt%Zr have been characterized. As a result of the Zr addition, the alloy has a relatively fine grain size (~ 1 μm), as compared to the grain size of 50 μm in conventional ingot-metallurgy 2024 Al and 20 μm in powder-metallurgy 2124 Al containing only 0.12wt% Zr. At relatively low strain rates ($<10^{-2}$ s^{-1}) and elevated temperatures (approximately 425-500°C), the fine-grained 0.6Zr-2124 Al alloy behaves like the coarse-grained alloys, i.e., they all deform by a dislocation climb mechanism. At high strain rates, however, the fine-grained 0.6Zr-2124 Al alloy exhibits superplasticity, similar to that observed in SiC and Si_3N_4 whisker-reinforced 2124 Al composites. The maximum tensile elongation is about 500%, recorded at 475°C, and at a strain rate of 3.3 x 10^{-1} s^{-1}. The high strain rate phenomenon is consistent with the general trend observed in aluminum-based alloys, namely, an increased strain rate for optimal superplastic flow with a decrease in grain size.

Acknowledgment

This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.

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