Texture evolution with superplastic deformation of a AlCu4Mg/Si₃N₄/20p composite

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Abstract

The deformation mechanisms which operate during superplastic tensile deformation at 783K in a AlCu4Mg/Si₃N₄/20p composite have been investigated by means of the crystallographic texture evolution with strain. Three stages of texture evolution have been observed: In stage I, little texture variation occurs up to a true strain of 0.4. In stage II, a progressive decrease of texture intensity up to a true strain of 0.8 is noted. This suggests that a mechanism of grain boundary sliding, GBS, typical of superplastic deformation of monolithic fine grained polycrystalline materials, prevails in this stage. At higher deformations, in stage III, the intensity of the main texture components tends to increase, indicating that the dominant deformation mechanism becomes crystallographic slip, CS. (117)

Key words. A. Metal-matrix composites (MMCs); B. Creep; B. High-temperature properties; C. Deformation; Superplasticity.

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1.- INTRODUCTION

Discontinuously reinforced metal matrix composites, MMCs, in particular aluminum alloy matrix composites, have been extensively studied in recent decades because of their good mechanical properties. They have higher Young modulus and yield strength [1] and better creep [2,3] and wear [4,5] resistance than the corresponding monolithic alloys. Because of this, and since these composites can be produced on a large scale at a competitive cost, they are becoming materials of potential interest in a number of structural applications in industrial sectors such as aerospace and ground transportation.

In particular, the potential use of these composites at high temperatures has stimulated the study of their creep behavior under several testing conditions. It has been shown that MMCs may undergo superplastic deformation when high temperature testing is involved. This is an interesting finding in view of the fact that superplastic forming might be an industrial process to fabricate complex shape components out of these composites [7]. A study of high temperature deformation mechanisms of these materials, especially when superplastic behavior is achieved, can, hence, be of importance from both the industrial and the scientific point of view.

Although much work on the high temperature mechanical response of aluminum matrix composites has been reported, the underlying mechanisms of this behavior, in particular during superplastic deformation, are still controversial.

Two types of superplastic behavior are observed in MMCs, namely internal stress superplasticity and fine grained (or structural) superplasticity [8]. The internal stress superplasticity is due to the strong interfacial stresses generated during thermo-mechanical treatments as a consequence of the different thermal expansion coefficient of both phases. These internal stresses can be partially relaxed by plastic deformation (dislocation generation) [9]. Repeated temperature changes (cycles), hence, provoke extensive plastic deformation under the action of a small applied tensile stress and a superplastic response can be obtained in the composite [10]. Usually, viscous plastic flow, with the stress exponent n equal to 1 (Newtonian flow) in the power law creep equation $\dot{\epsilon} = k \sigma^n$ (where $\dot{\epsilon}$ is the strain rate, σ the applied stress and k a parameter which is a function of the testing temperature and material microstructure) dictates the plastic response under these testing conditions. On the other hand, structural superplasticity [11] is obtained in polycrystalline alloys with small and equiaxed grains (typically, grains smaller than ~ 10

μm separated by high angle boundaries). Most of the investigations concerning the superplastic response of MMCs has concentrated on the first type of superplasticity. In this work, however, a study of superplasticity occurring in such fine grained aluminum matrix composites has been conducted.

It is well agreed that superplasticity in monolithic alloys occurs mainly by grain boundary sliding, GBS. It has been demonstrated, however, from texture evolution studies (*i.e.* the preferred orientation of the crystallites in a polycrystalline material) that crystallographic slip, CS, may also be active during this type of superplasticity [12]. Specifically, it was found in [12] a contribution of CS in competition with GBS responsible for the change in the texture during superplastic deformation of Al–5Ca–5Zn alloy. A texture evolution study has been also conducted in [13] to demonstrate that CS is never inhibited during superplastic deformation of Al-Mg alloy, dominated by GBS. In [14] the evolution of the main texture component of Al6Cu0.4Zr also indicates that both CS and GBS are competing, but that GBS dominates over CS when superplastic deformation increases.

The study of crystallographic texture evolution is, therefore, a powerful method to investigate the deformation mechanisms of polycrystalline solids. This is because the evolution of the main texture component intensities with strain is strongly dependent on the specific mechanism which dominates material deformation. To the authors' knowledge, however, this tool has not yet been used in the study of deformation mechanisms in MMCs that undergo structural superplasticity. The objective of this research is, therefore, to go deeper in the understanding of these mechanisms by a study of texture evolution in an aluminum alloy matrix composite.

2.- EXPERIMENTAL PROCEDURE

The AlCu4Mg/Si₃N₄/20p composite material studied in previous work [15,16] has been used for the present research. Material preparation involved a powder metallurgical route: Powder of 2124Al (Al-Cu-Mg), of average diameter smaller than 20 μm , was mixed with 20vol% of α -Si₃N₄ equiaxial particles of average diameter, $d < 1 \mu\text{m}$. The powder mixture was extruded to a bar at 773 K and an extrusion ratio of 100:1. The chemical composition (in wt.%) of the Al alloy powder was: Cu 4.20, Mg 1.48, Zn 0.12, Mn 0.64, Ti 0.06, Cr 0.06, Si 0.12, Fe 0.09, Al bal.

Tensile tests along the extrusion direction were conducted on cylindrical samples with threaded heads at 783 K under stress controlled condition (8 MPa) at different strains, as

described elsewhere [15]. These are very appropriate testing conditions to obtain superplasticity in this material. The resulting strain rate under these conditions was $2 \times 10^{-1} \text{ s}^{-1}$. Different samples were strained at several true strains levels, namely: $\varepsilon = 0, 0.15; 0.45; 0.82$, and 1.19 .

The texture of the aluminum matrix phase in the un-deformed material and deformed samples was subsequently determined by x-ray diffraction in a Siemens diffractometer equipped with a closed Eulerian cradle. Monochromatic $\text{Cu } K_{\alpha}$ x-ray radiation, of wavelength $\lambda = 1.5406 \text{ \AA}$, was used. Samples consisted of small discs sectioned perpendicularly to the tensile direction, *i.e.*, the normal direction to the disc surface was parallel to the extrusion (and tensile) axis. (111), (200), (220) and (311) pole figures were measured. From them, recalculated pole figures were obtained assuming cubic symmetry of the crystal lattice and the cylindrical symmetry of the extruded bar. The orientation distribution function $f(\varphi_1, \Phi, \varphi_2)$, ODF, a function of the three Euler angles $(\varphi_1, \Phi, \varphi_2)$, was obtained from the recalculated pole figures by the series expansion method [17] using commercially available software. From the ODF data, inverse pole figures could be obtained and the volume fraction of crystallites belonging to the main texture components was calculated numerically, applying the Simpson method [18]. To ensure the reliability of these texture measurements, longitudinal samples (with the tensile axis parallel to the samples surface) were also prepared on which further measurements were conducted following the same procedure described above.

3.- RESULTS AND DISCUSSION

The texture of as received material was typical of extruded FCC metals, *i.e.*, a fiber texture (with the fiber axis parallel to the extrusion axis) with two main components, namely, $\langle 111 \rangle$ and $\langle 100 \rangle$. Figure 1 shows the inverse pole figure of the un-deformed material in which these two components are expressed. Contrary to previous work [19], the $\langle 100 \rangle$ component is more intense than $\langle 111 \rangle$. The deformed samples maintained, qualitatively, the same texture at the different strain levels. Only the intensity varied with strain, as shown in the plot of Figure 2. This figure reveals the evolution with tensile strain of the volume fraction of crystallites associated with the $\langle 111 \rangle$ and $\langle 100 \rangle$ components, V_{111} and V_{100} , respectively. As can be seen, both V_{111} and V_{100} remain roughly constant up to approximately 0.4 strain. These values subsequently decrease progressively up to a true

strain of 0.8, and from there increase again to failure. It is also observed that the evolution of V_{hkl} with strain for both texture components ($dV_{111}/d\varepsilon$ and $dV_{100}/d\varepsilon$) is very similar.

The evolution of the volume fraction of crystallites associated with the texture components with deformation supplies valuable information regarding the deformation mechanisms responsible for superplasticity in this composite. For simplicity, the effect of cavity formation, in principle, will not be taken into consideration as little evolution of cavities during tensile testing, in comparison to other superplastic composites, occurs [15,16].

The competition of GBS and CS mechanisms can be analyzed on the basis of the variation of V_{hkl} with strain. On the one hand, GBS involving sufficient grain rotation (approximately above some 15°), in particular of $\langle 111 \rangle$ and $\langle 100 \rangle$ grains, would provoke a decrease of V_{111} and V_{100} . This is because grains oriented initially with a crystallographic direction $\langle 111 \rangle$ or $\langle 100 \rangle$ parallel (or nearly parallel) to these fiber texture components would rotate or deviate from these orientations in Euler space in a random manner; leading, as mentioned, to a decrease of V_{111} and V_{100} . Thus, the contribution of GBS correlates well with the negative value of $\Delta V_{hkl}/\Delta\varepsilon$ observed, in particular in stage II, figure 2. On the other hand, the activation of CS alone would lead to well defined grain rotation towards particular stable orientations in Euler space if specific slip systems are operative. If the initial texture components are already stable orientations, then CS would provoke an increase in the intensity of these components. It might also be possible that the initial texture components were not stable ones for given crystallographic slip systems. In such a case, a “displacement” in Euler space, from the initial texture component orientation to the final stable one, would be observed. The path traveled by the ODF maximum (main texture component) in Euler space gives also information on the specific CS systems operating during deformation in this case.

In the case of the present composite, both texture components are known to be stable for uniaxial tensile testing along the fiber axis and assuming that CS occurs under the usual crystallographic slip systems in FCC metals: *i.e.*, $\{111\}\langle 110 \rangle$ [20]. In other words, grains initially oriented at random would rotate towards either $\langle 111 \rangle$ and $\langle 100 \rangle$ as deformation progresses. As a consequence, an increase of both V_{111} and V_{100} would, therefore, be expected. This is contrary to the observations reported here in stages I and II. The fact that the fraction of crystallites associated with these two components remains constant or decreases, as in Figure 2, is an indication that other deformation mechanisms should be active during straining. Because of the small grain size of the matrix alloy associated with

the small inter-particle reinforcement distance and the high strain achieved (superplasticity) it is our contention that, to some extent, a GBS mechanism involving grain rotation should also contribute to deformation. Whereas no previous studies report in depth that GBS is a dominant deformation mechanisms in MMCs under isothermal testing conditions, the small grain size of this material, the high strain rate sensitivity ($1/n$), and accompanying high strain, supports the proposal that GBS also contributes to deformation in this case [7].

On the other hand, a previous study [15] of this AlCu4Mg/Si₃N₄/20p composite has shown that a transition in mechanism, associated with a change in n , occurs at 8 MPa. This is seen from the plot of figure 3, taken from ref. [15], in which true strain rate data as a function of the applied stress are represented in double logarithmic scale. As can be seen, the slope is higher in the lower stress regime. Insufficient data is reported in the plot of figure 3, but the increase in trend in n from $n \approx 2$ towards $n=3$ or even higher as the applied stress decreases suggests a change from GBS to CS. This indicates that the present tests were very probably conducted under conditions where GBS and CS are competitive deformation mechanisms [12,13]. The calculation of the precise magnitude of each contribution from the change in the slope of $\Delta V_{hkl}/\Delta \epsilon$ with strain, figure 2, is very complex and beyond the scope of this research. However, the fact that the slope, $\Delta V_{hkl}/\Delta \epsilon$, is essentially similar for $\langle 100 \rangle$ and $\langle 111 \rangle$ texture components is an indication that uniform deformation occurs throughout all grains of the aluminum alloy matrix of this composite.

Furthermore, two possible interactions exist when two deformation mechanisms occurring in the plastically deforming phase are contributing to material's deformation under given testing conditions: they can be independent or interdependent mechanisms [21,22]. In the first case, the dominating mechanism would be the faster one for the given testing conditions, and in the latter case, the slower one would dominate the creep rate. The dependence or interdependence of deformation mechanisms can be known from the variation of n with applied stress (or strain rate) in the double logarithmic representation, as schematically shown in figure 4. In this figure, the competition of two mechanisms is depicted, with stress exponents n_1 and n_2 , with here $n_2 < n_1$. The evolution of n with the applied stress (or strain rate) of the experimental $\epsilon - \sigma$ data (as represented by the two thick-solid lines), would dictate whether they are dependent or interdependent mechanisms, as indicated. The evolution of n in the plot of figure 3 and the comparison with figure 4 allows assessment of the interdependence of the deformation mechanisms

involved during superplasticity of this composite. This, together with previous findings on the superplastic behavior of this composite [15,16] and the study of the main texture components evolution summarized in the plot of figure 2, give further hints on the interaction between GBS and CS, at least in a qualitative manner.

In this way, it can be accepted, in principle, that *only* a balance between CS and GBS contributions can maintain the strain evolution of V_{111} and V_{100} in stage I. However, during deformation, the storage of geometrically necessary dislocations, GNDs, around the undeformable ceramic particles should also occur in this stage. When the dislocation density achieves a certain level, grains locked by the Si_3N_4 particles are allowed to undergo slip and grain rotation (*i.e.*, GBS) accommodated by CS. This phenomenon plays a crucial role in the evolution of texture from stage I to stage II. [23].

The increase of V_{111} and V_{100} in stage III is, finally, an indication that the CS mechanism prevails. At present, however, the microstructural features responsible for this change are not fully understood. Probably, the formation of a liquid phase at cavities developed in this superplastic composite plays a role in this behavior. As suggested in [15], cavity growth during superplasticity in this composite is restricted by the presence of a liquid phase, probably associated with the relaxation of stress concentration due to GBS accommodated by CS. It can be assumed, then, that a liquid phase is an additional accommodation mechanism for GBS in this composite. During stage I and predominantly in stage II, the liquid phase at cavities allows GBS to occur as cavities grow with superplastic deformation. Despite the fact that cavity growth is not as rapid as in other superplastic composites or as in superplastic 7475 Al alloy [15], it is possible that coalescence of cavities in stage III has impeded the formation of sufficient liquid at cavities. Consequently, GBS accommodated by this phase is suppressed, leading to the predominance of CS. This would provoke the observed increase of V_{111} and V_{100} in stage III and to final composite failure.

Nevertheless, more thorough understanding of the competition between GBS and CS, in particular in stage III, would be needed. It may be that the liquid phase formed at cavities acts as a new accommodation mechanism by stress relaxation for GBS, additionally to CS. As has been reported previously by other authors [24], at low stress levels fracture in these particle reinforced materials occurs at dimples formed by cavity coalescence controlled by diffusion. Coalescence of these cavities in stage III prevents the liquid phase in filling them up. Hence, the accommodation mechanism is inhibited. The absence of this

accommodation leads, then, to the predominance of CS over GBS in stage III, to the increase in texture and subsequent composite failure.

5.- CONCLUSIONS

Crystallographic texture evolution with superplastic deformation of AlCu4Mg/Si₃N₄/20p composite tested at 8 MPa and 783 K has been determined by x-ray diffraction. This study has allowed an analysis of the deformation mechanism involved during composite deformation. The main conclusions are:

- 1.- Two interdependent deformation mechanisms, namely: grain boundary sliding, GBS, and crystallographic slip, CS, are responsible for the superplastic behavior of this composite under the conditions investigated.
- 2.- Three different stages of texture evolution have been observed. In Stage I little variation of the volume fraction of crystallites associated with the main texture components is seen. This reveals a competition between GBS and CS for plastic deformation. In stage II, a decrease of the volume fraction is appreciated. This indicates that GBS prevails as opposed to a CS mechanism. Finally, in stage III, CS takes over from GBS and the volume fractions of crystallites associated with the main texture components increase with increasing strain.

A more detailed understanding of the deformation mechanisms involved during superplastic deformation of this AlCu4Mg/Si₃N₄/20p composite and the specific competition between GBS and CS, particularly that occurring in stage III, is needed.

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Figure 1.- Inverse pole figure for the extrusion axis direction of the un-deformed composite material showing the two main texture components: $\langle 100 \rangle$ and $\langle 111 \rangle$. Maximum intensity is 4.7 at the $\langle 100 \rangle$ pole.

Figure 2.- Strain evolution of the volume fraction of crystallites, V_{111} and V_{100} , associated with the two main texture components, namely, <100> and <111>, respectively. The three stages of volume fraction evolution with strain are indicated.

Figure 3.- Double logarithmic plot of the strain rate as a function of stress showing the transition of n from $n=3$ to about $n=2$ with increasing stress (from ref. [15]).

Figure 4.- Schematic representation of the competition between independent and interdependent (or sequential) deformation mechanisms which influences the evolution of n with the applied stress (or strain rate) in a double logarithmic representation of strain rate vs. stress, in arbitrary units (a.u.).

FIGURES CAPTIONS

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