Grain boundary sliding and accommodation mechanisms during superplastic deformation of ZK40 alloy processed by ECAP

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Controlling mechanism during superplastic deformation of ZK40 alloy processed by ECAP was identified. Effects of twinning and dynamic strain ageing (DSA) on superplasticity were analyzed. Amplitude in stress oscillation was correlated with solute atom concentration theoretically. Twinning can be an enhancing factor in grain boundary sliding and DSA had apparent influence on stress fluctuation; they were accommodation mechanisms for superplastic deformation through grain reorientation and interaction between solute atoms and dislocations, respectively. The interaction between mobile and forest dislocations played a dominant role for the occurrence of DSA, when dislocation density was relatively low in large grains. The effect of DSA became more active with increasing temperature, although grain boundary sliding (GBS) was the controlling mechanism throughout the whole process of superplastic deformation under elevated temperatures.

1. Introduction

Applications of magnesium alloys has attracted more attentions from enterprises like computers, communications and consumer electronics (3Cs), owing to its excellent mechanical properties, high damping and shielding capacities, etc. But in most cases, due to the h.c.p. crystalline structure of magnesium, structural components made of magnesium alloys are manufactured through conventional casting methods, limiting wide uses of this new prosperous material in practice. Superplastic deformation technique was widely considered to be an alternative method that can be applied commercially, for its lower energy cost and relatively simple devices compared with die-casting and other techniques.

To achieve superplastic deformation, the average grain size lower than 10 μm is generally needed [1]. Many methods had been adopted to get small grain size, among which equal channel angular pressing (ECAP) was thought to be very effective [2–7]. In terms of controlling and accommodation mechanisms during superplastic deformation, grain boundary sliding (GBS) has been widely accepted as the main or controlling one. Because of the h.c.p. crystalline structure and a special c/a axial ratio compared to zinc, twinning occurred significantly in magnesium alloys when processed by compression methods. Furthermore, large strain can always be obtained after severe plastic deformation and dislocation density will be increased accordingly, thus interactions between solute atoms and mobile and forest dislocations will become more active and frequent than that in unprocessed alloys. Although the controlling deformation mechanism was generally considered to be grain boundary sliding (GBS) and stress instability during some mechanical tests were analyzed [8, 9], it is also very important to investigate other influences that accommodation mechanisms may have on the superplasticity of magnesium alloys, especially after being processed by ECAP.

2. Experimental procedures and results

Ingots of ZK40 (Mg-3.73 wt% Zn-0.64 wt% Zr) were used for the whole experiments. Pretreatment and later experiment procedures had been reported in the previous paper [10]. What is necessary to point out again is that the total strain intensities of the as-ECAPed rods after one and three passes were 1.07 and 3.22, respectively. Transmission electron microscopy (TEM) observations were performed to distinguish microstructure evolution after ECAP. Specimens were thinned at 233 K with a twin-jet polisher and then examined using a JEM200FXα

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transmission electron microscope operated at a voltage of 200 KV and a current of 60 μA. To prove the existence of grain boundary sliding and other associated phenomena, surfaces of specimens which were deformed to a specific strain or fracture were analyzed by a JXA-840 scanning electron microscope (SEM).

For the ZK40 alloy processed by ECAP for one pass (referred to as ZK40-1P hereinafter), the average grain size was 5.6 μm; while in the case of ZK40 alloy processed by ECAP for three passes (referred to as ZK40-3P hereinafter), all grain sizes were lower than 1 μm. Twinning was discovered to occur significantly in ZK40-1P, while dislocation density was very higher in ZK40-3P than that in ZK40-1P, after being processed through die for three passes. Under the same experimental conditions, ZK40-3P exhibited better superplasticity than that of ZK40-1P, e.g., when initial strain rate was set at 1 $\times$ 10^{-3} s^{-1} and the temperature at 523 K, ZK40-1P only exhibited a tendency towards superplasticity. On the contrary, ZK40-3P demonstrated apparently low temperature superplasticity under the same conditions as that for ZK40-1P, the largest elongation was as high as 612%. All these were ascribed to the simultaneous effects of finer grains, high angle grain boundaries and the homogeneity in microstructure after being processed by ECAP intensively [10].

At 473 K, ZK40-1P demonstrated apparent strain hardening, but peak stress could not reach high value before rupture (Fig. 1a), because stress concentration could not be released efficiently at this relatively low temperature, this was also accelerated by the heterogeneity in microstructure of this alloy. When strained at 523 K, ZK40-3P exhibited low temperature superplasticity and peak stress was very lower than that of ZK40-1P (Fig. 1b), grain boundary sliding was accomplished smoothly and dislocation movement within grains was not very important in ZK40-3P, although dislocation density in it was very high. Since the interaction between dislocations and solute atoms became more active with increasing temperature as results of lattice and pipe diffusion, flow stress of ZK40-3P was higher than that ZK40-1P exceptionally (Fig. 1b); this was surely not in accordance with usual cases of superplastic deformation, in which long strain is usually corresponding to relatively lower flow stress.

3. Discussion

3.1. Grain boundary sliding during superplastic deformation

Since average grain sizes of ZK40-1P and ZK40-3P were all lower than 10 μm, grain boundary sliding occurred

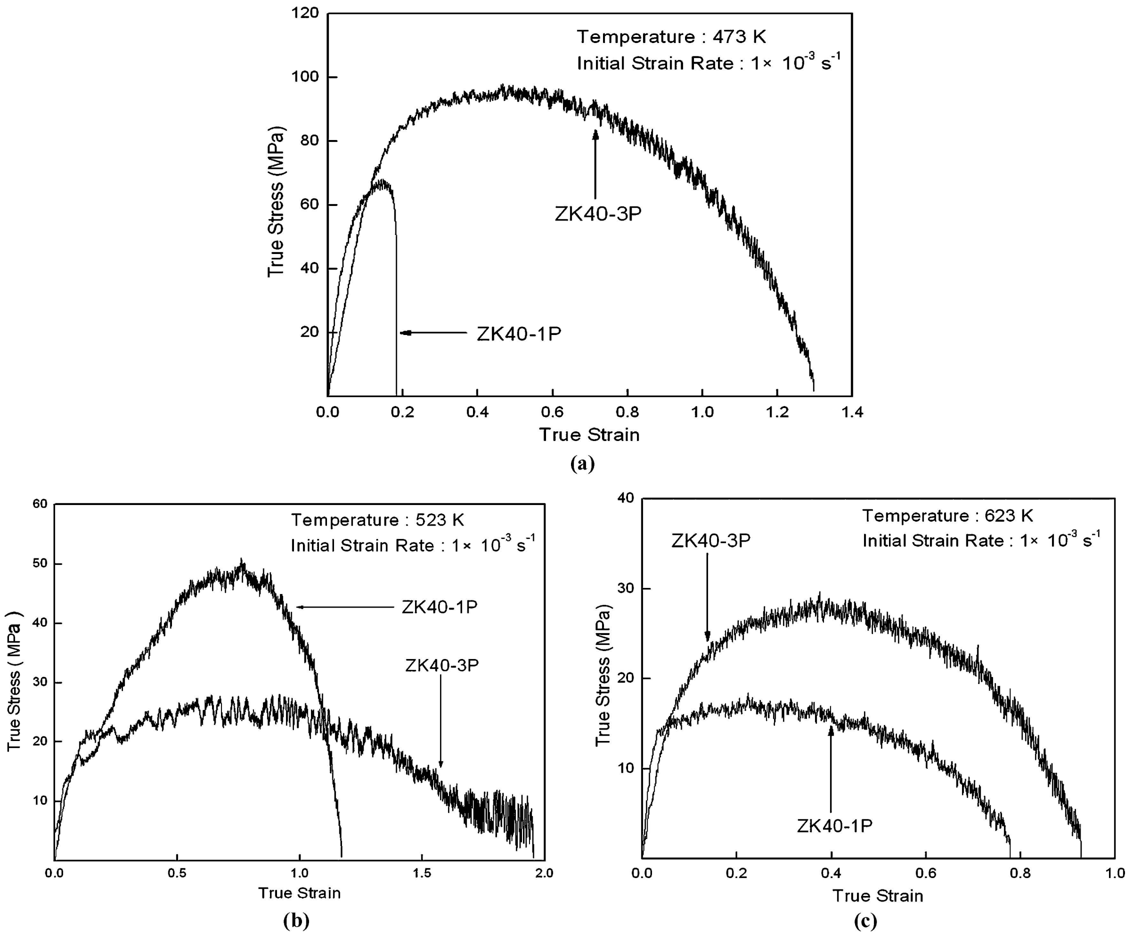

Figure 1 True stress-true strain curves of ZK40-1P and ZK40-3P at different temperatures.

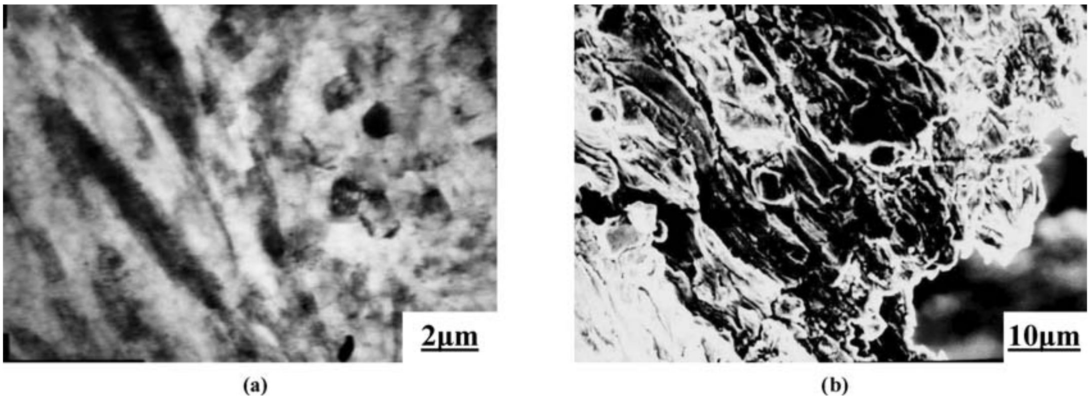

Figure 2 Heterogeneity in microstructure of ZK40-1P: (a) transition area, (b) fractograph (473 K).

steadily when deformed under elevated temperatures and relatively lower strain rates [1]. For all materials processed by ECAP for either only one pass or three passes further during the whole experiment, any concerns over the dominant effect of GBS could be summarized into two aspects including dislocation movement within grains (small or large) and transition of dislocations which might existed in non-equilibrium state and were mainly distributed in larger grains of ZK40-1P. On the one hand, dislocation densities were so high in both of the materials that dislocation movement could only be related to the interactions among them. This kind of interactions was valued to be more important for dynamic strain aging (DSA) taking effect, when dislocation density was relatively lower in ZK40-1P in comparison with ZK40-3P, as will be discussed in detail hereinafter. Provided with very high dislocation density after more passes through ECAP die, as in the case of ZK40-3P, dislocation movement would be very difficult to proceed, and should be reasonably replaced by the interactions between dislocations and solute atoms, increasing stress should be definitely attributed to the effect of DSA under such circumstance. On the other hand, no re-crystallization (RDX) was found for the two alloys during deformation. Taking ZK40-1P as example, in which dislocation density was already higher compared with that in unprocessed state, there were no sufficient reasons for the occurrence of RDX, which usually started with the movement of dislocations in non-equilibrium state towards grain boundaries and followed with the transition from non-intrinsic (or lattice) dislocations into extrinsic grain boundary dislocations (EGBDs), and the later formation of dislocation cells, owing to the pre-existing long-range stress associated generally with dislocations in non-equilibrium state and heterogeneity in coarser microstructure [10–12]. The heterogeneity (or inconsistency) in microstructure was emphasized only to explain the premature fracture of ZK40-1P, but the dominant effect of GBS could not be excluded accordingly, when considering most grains of the sizes lower than 10 μm.

In the case of ZK40-1P, strain hardening took place radically when tested at relatively lower temperature (473 K) as shown in Fig. 1a, mainly due to the long-range stress associated with non-equilibrium grain boundaries within relatively large grains compared with ZK40-3P [11, 12]. The heterogeneity in microstructure of ZK40-1P, which existing mainly in transition areas of shear bands between fine and coarse grains, was also responsible for lower elongation to fracture [13, 14], as can be seen in a fractograph obtained at 473 K showing gains of various shapes (round and lenticular) and sizes (Fig. 2). As for ZK40-3P, when deformed under the same conditions as that for ZK40-1P, it arrived at steady flow stage when true strain was only 0.1, peak flow stress during whole strain was also very lower than that exhibited by ZK40-1P (Fig. 1b). Its lower temperature superplasticity was mainly attributed to small grain size and microstructure homogeneity, because stress concentration within grains can hardly take effect when grain boundary sliding have become the dominant deformation mechanism [15, 16], although dislocation density in ZK40-3P was very larger than that in ZK40-1P.

As a whole, grain boundary sliding was still thought to be the dominant deformation mechanism during superplastic deformation of the as-ECAPed ZK40 alloys, even though twinning and grain reorientation may have few important effects, which mainly existed in ZK40-1P, and the interaction between high-density dislocations and solute atoms can never be neglected during the whole process of GBS especially when dislocation density was increased further to a very higher level in ZK40-3P. DSA and twinning, with reorientation of grains being taken into consideration in some cases, can only play a subordinate role as accommodation mechanisms, when all materials are of the grain sizes lower than 10 μm, homogeneity in microstructure consisting of large fraction of high angle grain boundaries as shown in Fig. 4a was attained and relatively lower steady flow stresses were observed during whole processes of superplastic deformations at elevated temperatures [10, 15, 16].

3.2. Effect of twinning on grain boundary sliding

Because magnesium has the h.c.p. crystalline structure and a special c/a axial ratio of 1.6235, which is lower than

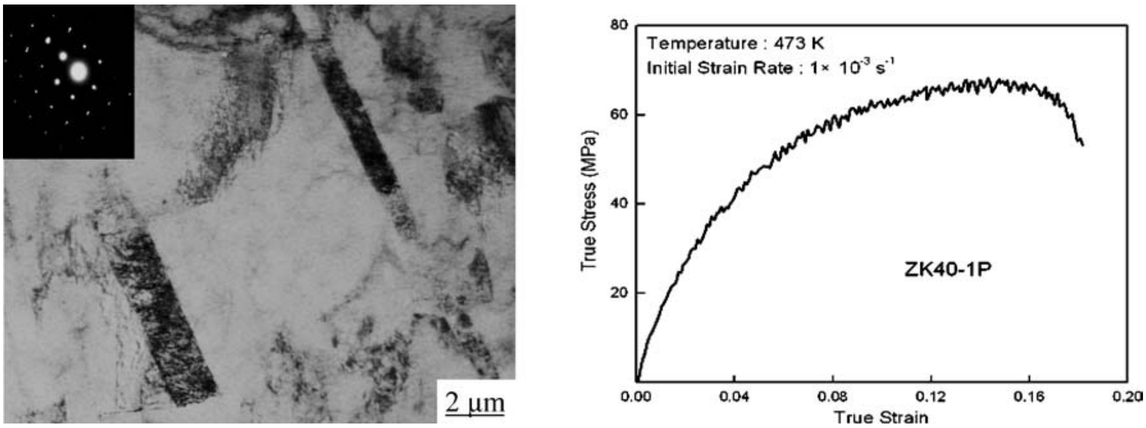

Figure 3 Twinning and serration on a true stress-true strain curve of ZK40-1P.

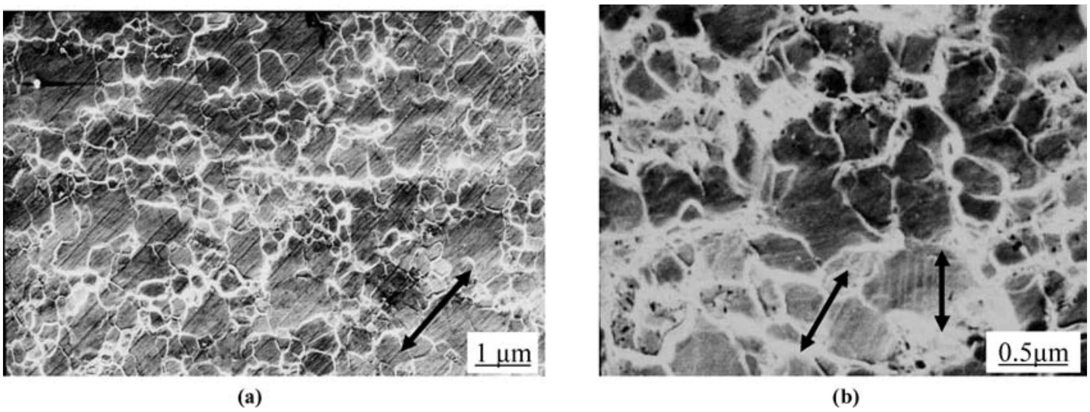

Figure 4 SEM observations of the microstructure of ZK40-3P deformed to a true strain of 0.1 at the temperature of 623 K: (a) grip section; (b) gage section.

1.732, twinning had been discovered to occur dramatically in its alloys especially when processed by the methods of compression. For the purpose of grain refinement, ECAP was adopted to achieve small grains, but when the total accumulated strains was not very large (processed for only one pass through die) and grain refinement was not very complete, twinning was likely to take place in those comparatively large grains as discovered in ZK40-1P.

Although twinning was generally unfavorable for large strain in tensile test, stress concentration was released to some extents or at least partially, as demonstrated by the serration on a true stress-true strain curve in Fig. 3. This jerky stress was found under relatively lower temperature (473 K), at which dislocation movement could only be related to strain hardening on a large scale, solute atoms could not diffuse and accumulate on dislocation lines easily. Because during the progress of tensile test those parts formerly thought to be disadvantageous to superplastic deformation can become favorable for further slip through reorientation of lattice, for which new slip systems can be activated in the areas twinned, twinning should be the only factor causing stress fluctuation under relatively lower temperature.

Nevertheless, this small serration on curve can not be regarded as the oscillation that will be discussed hereinafter. Besides the positive effects of twinning on releasing stress concentration, this serration on curve was also ascribed to the facts that some grains formerly located at hard orientation (Fig. 4a) unfavorable to twinning became to be located at soft orientation (Fig. 4b) through grain rotation both in ZK40-1P and ZK40-3P during grain boundary sliding. The only difference in this rotation should lie on that finer grains will make it to take place more easily. Thus, further twinning can be activated during superplastic deformation in those grains at favorable orientation and new slip systems will be also available for large strain accordingly. But it must be pointed out that the relatively large grain size and heterogeneity consisting of shear bands and transition areas between large and small grains were surely to be responsible for poor superplastic behaviors of ZK40-1P (Fig. 2); twinning, as the results of either ECAP before tensile test or grain rotation during deformation, can only serve as an enhancing factor in releasing stress concentration within local areas, strain hardening can still happen dramatically.

3.3. Correlation of dynamic strain aging (DSA) and stress oscillation

As mentioned above, large strains could be achieved after severe plastic deformation, e.g., equal channel angular pressing (ECAP) adopted in this experiment for the purpose of grain refinement. Definitely, the more strains accumulated on material, the higher dislocation density achieved. As results of being processed by ECAP, the interactions between dislocations and solute atoms became more active than ever before [17]. In addition, Orowan had proved that the average distance between two forest dislocations was inversely proportional to the square root of dislocation density ($\lambda = \rho_f{}^{-1/2}$, where λ is the mean distance and ρ_f is the density of forest dislocations). So the possibility of interaction between dislocations and solute atoms in ZK40-3P must be more frequent than that in ZK40-1P [18–20], because total strains accumulated in the former (3.22) were three times higher than that in the later (1.07) and dislocation density in ZK40-3P was surely higher than that in ZK40-1P [21].

As shown in Fig. 1b, stress oscillations were discovered for both of the two materials. In case of ZK40-1P, true stress became to fluctuate when true strain was about 0.6 and the maximum oscillation in stress was relatively small compared to that exhibited by ZK40-3P. This should be explained as that dislocation movements in comparatively large grains were likely to be absorbed under elevated temperatures when the true strain was small whereas the strain rate was not very large [22, 23]. But when true strain was increased to large values, effects of dynamic strain aging (DSA) became more apparent, because grain boundary sliding was hampered by microstructure inconsistency in ZK40-1P, thus dislocation density became higher than that in the initial stage of strain and more non-equilibrium grain boundaries which would cause difficulties for grain boundary sliding were formed in large grains [13, 14]. So effect of DSA could increase with true strain in ZK40-1P, but could not increase all the way because of strain hardening which could result premature rupture.

However, when the interaction between solute atoms and dislocations were valued more importantly for the occurrence of DSA, the interaction between mobile and forest dislocations must be taken into consideration [24], it was especially the case for ZK40-1P which consisted of relatively large grains. According to the models proposed by Ball, Hutchisont [25] and Mukherjee, [26], dislocations are likely to form at ledges and triple junctions of grain boundaries during GBS, then they glide and or climb within grains in order to release stress concentration. It was also deemed by Gifkins [27] that dislocations formed at grain boundaries can also glide and or climb within grains, i.e., grain boundary dislocations can become lattice dislocations, accommodating GBS efficiently. So DSA was not discovered in ZK40-1P initially at 523 K (Fig. 1b), it was partially because that new slip systems were activated in the areas twinned, but it was mainly attributed to the fact that dislocations formed initially could be absorbed or annihilated to some extents when gliding and climbing within grains. When the strain was increased, stress concentration could not be released further due to the jostle of dislocations generated from the primary slip systems. As the results of dislocation jostle which was usually associated with long-range stress [11, 12], secondary slip systems were activated accordingly, likely bringing about the emergence of forest dislocations. Fig. 5 showed the formation of dislocations at the triple junction of grain boundaries (Fig. 5a) and the interaction among dislocations of different origins within a grain (Fig. 5b). Therefore, it can be proposed that the interaction between mobile and forest dislocations also played an important role for the occurrence of DSA, when dislocation density was not very high within large grains [28].

According to Kubin, Estrin and Louat [29, 30], the hardening caused by solute segregation is proportional to the excess solute concentration on dislocations. The maximum increment in stress f_0 is of the form as Equation 1,

$$f_0 = B\frac{C\exp(W/KT)}{1 + C\exp(W/KT)} \quad (1)$$

where B is a constant, C is the solute atom concentration, W is the binding energy between a solute atom and a dislocation, and k and T are the usual parameters. So the largest oscillation in stress during tensile test could

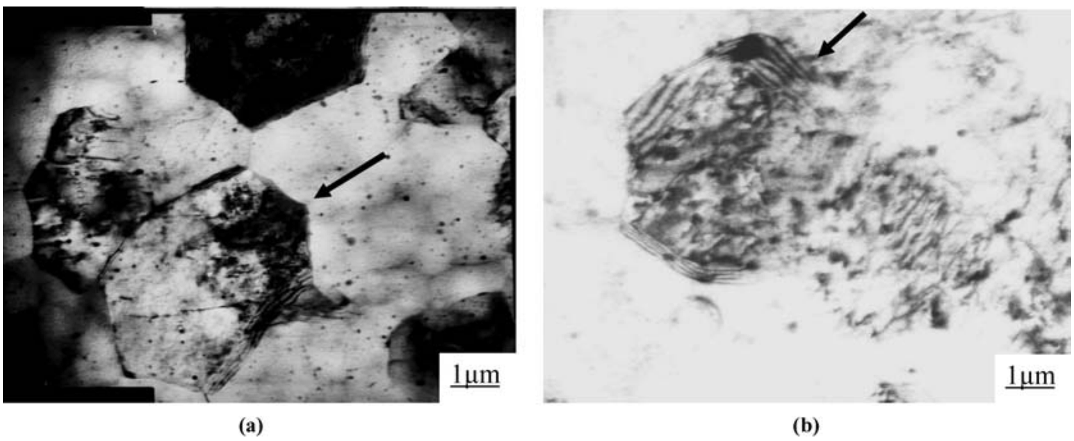

Figure 5 Microstructures of ZK40-1P during deformation at 523 K: (a) dislocations generated gliding and climbing, (b) interaction among dislocations of various origins.

only be affected by solute atom concentration of metallic materials, provided that other parameters set down and alloys of same chemical composition tested under same experimental conditions. For the whole experiment, only one material of same chemical composition but different working procedures, under which they were processed by ECAP, was tested. Solute atom concentration in ZK40-3P could be assumed to be higher than that in ZK40-1P, although these two alloys are of the same chemical composition as each other. As a result, DSA was found when true strain was only 0.2 for ZK40-3P in which dislocation density was very higher than that in ZK40-1P (Fig. 1b).

It was also discovered that the amplitude in stress oscillation for ZK40-3P was larger than that for ZK40-1P, because solute atom concentration was supposed to be higher hypothetically in ZK40-3P than in ZK40-1P. Since solute atoms distributed at one place could be regarded as either a Cottrell, Snoek or Suzuki atmosphere outside dislocation, or a core atmosphere in the core areas of forest dislocations. Hence, the mobility of dislocations was limited by both volume diffusion in crystal lattice and pipe diffusion along dislocation cores [31]. But the amplitude in stress oscillation during the whole strain of ZK40-3P was almost the same as the beginning of deformation, because grain boundary sliding could be fulfilled steadily and dislocation density could be assumed to be constant although very high, the oscillation amplitude was only affected by the interaction between solute atoms and dislocations, as discussed above.

Finally, effect of DSA was found to increase with temperature, as shown Fig. 1c. When strained at 623 K, the average peak flow stress of ZK40-3P was unexpectedly higher than that of ZK40-1P, even though GBS was accomplished more smoothly in the former than in the later; DSA also became to take effect at the initial stage for both. Definitely, diffusion of solute atoms can become more active at higher temperature, thus the mobility of dislocations will be constrained more intensively and efficiently, so the effect of DSA was thought to account for more of peak flow stress during superplastic deformation at relatively higher temperatures. This exceptional result can also be related to the fact that mobility of solute atoms is proved to be of the same order as that of moving dislocations in most cases when temperature is increased sufficiently. As a result, solute atoms are able to diffuse over relatively long distance to pin moving dislocations. On the other hand, strain hardening is directly associated to the waiting time of solute atoms before or around moving dislocations during deformation of alloy under the effects of DSA. Thus, given the temperature and the concentration of solute atoms, influence of DSA increased with waiting times. That is why ZK40-3P showed a higher flow stress at 623 K, compared with the corresponding value tested under a relatively lower temperature of 523 K. As for the comparison with ZK40-1P which exhibited a comparatively lower flow stress under the same condition, increasing effect of DSA that contributed effectively to the flow stress as discussed above must be firstly related to the hypothetically higher solute concentration in ZK40-3P, although GBS was achieved smoothly under such high temperature. Moreover, dislocation absorption and annihilation was dramatically accelerated at 623 K in those large grains of ZK40-1P, due to more efficient diffusion rate of magnesium atoms, compared with aluminum under elevated temperatures. In summary, both hypothetically higher solute concentration and increasing effects of DSA could be enhancing factors in ZK40-3P exhibiting unexpectedly a higher flow stress than that of ZK40-1P superplastically deformed under the same conditions. This exception in flow stress should be an apparent demonstration of DSA contributing noticeably to flow stress under higher temperatures, even though this increase in flow stress could be partially offset by the enhancement of finer grains of sub-micrometer scale to grain boundary sliding (GBS).

4. Conclusions

(1) Grain boundary sliding was the controlling deformation mechanism during superplastic deformation of ZK40 alloys processed by ECAP for one and three passes through die.

(2) Twinning could be an enhancing factor for grain boundary sliding when new slip systems were activated after reorientation of lattice atoms of twined areas, thus accommodated grain boundary sliding; reorientation of grains during superplastic deformation could be favorable to grain boundary sliding.

(3) Oscillation in stress during strain was ascribed to effects of dynamic strain aging during superplastic deformation.

(4) The more total strain accumulated on alloy, the larger amplitude in stress fluctuation found, because solute atom concentration could be regarded to be increased hypothetically after being processed by ECAP for more passes through die.

(5) Dynamic strain aging was accommodation mechanism for superplastic deformation; effects of DSA was found to increase with temperature, because diffusion of solute atoms can become more active and dislocation density was very high in ZK40 alloy after being processed by ECAP.

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