Grain Structure, Texture Evolution and Deformation Mechanism during Low Temperature Superplasticity in 5083 Al-Mg Alloy

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Low temperature superplasticity (LTSP) was observed in the 5083 Al-Mg base alloy after thermomechanical treatments (TMT). The maximum LTSP elongation was 400%, occurring at 250°C and $1 \times 10^{-3} \text{ s}^{-1}$. The subgrain structures formed during TMT transformed to better defined subgrains to $\sim 0.5 \mu\text{m}$ upon heating to 250°C. Further static annealing or superplastic straining at 250°C would produce well defined fine grains to 1.5–2.5 μm , dependent on annealing time or strain level. Superplastic loading would accelerate grain evolution rate. The near brass {110}<112> and S {123}<634> texture components in the as-TMT specimens evolved into a random orientation distribution after LTSP loading to 100% at 250°C. Static annealing at 250°C itself could not alter the existing texture. Under the optimum LTSP condition, the m -value was ~ 0.5 , compared with 0.2 for the as-received coarse-grained 5083 alloy. The activation energy Q -value was around 50–90 kJ/mol for LTSP over 200–300°C and around 145 kJ/mol for HTSP over 400–550°C. It is postulated that the rate controlling deformation mechanism in the TMT processed specimens was grain boundary sliding during the optimum LTSP condition and solute drag creep during HTSP deformation.

(Received February 8, 1999; In Final Form April 20, 1999)

Keywords: low temperature superplasticity, thermomechanical treatment, 5083 aluminum alloy, grain structure, texture, deformation mechanism

I. Introduction

Superplastic forming of commercial low-priced aluminum sheets could be developed into one of the important fabrication means for future automobile, train, architecture or electric appliance industry. There are at least three main factors that need to be taken into account: (1) the alloy itself is commercially widely available and cheap; (2) the forming rate is sufficiently high; and (3) the forming temperature is as low as possible. Higher forming rate to a strain rate greater than 10^{-2} s^{-1} would satisfy the current fabrication speed⁽¹⁾; while lower forming temperature would save fabrication energy, prevent from severe grain growth, cavitation and solute-loss from surface layers, as well as maintain superior post-form properties⁽²⁾. Following these guidelines, the development of superplastic Al-Mg or Al-Mg-Si base alloys has attracted attention lately, including experimental alloys such as Al-3Mg or Al-10Mg and commercial alloys such as AA5052, 5083, 6061, 6011, etc. There have been numerous efforts in processing aluminum materials to exhibit high rate superplasticity (HRSP) and/or low temperature superplasticity (LTSP) by using thermomechanical treatments (TMT), equal channel angular (ECA) extrusion, multiple forging, cyclic extrusion, torsion under compression, or accumulative roll-bonding^{(3)–(14)} on commercial or experimental alloys. In some cases, modified alloy compositions with an addition of a small amount of Fe, Mn, Zr, Cr, or Sc^{(9)(13)–(21)} have resulted in either superior or detrimental effects.

In our pervious study⁽²²⁾, a simple rolling-type TMT

was applied to process the low-priced commercial 5083 alloy, resulting in low temperature superplasticity at around 250°C and $1 \times 10^{-3} \text{ s}^{-1}$, with an optimum tensile elongation to 400%. The TMT processed thin sheet contained subgrains measuring around 0.5 μm . At temperatures lower than 300°C, the grains grew limitedly and maintained LTSP, with failure by cavitation coalescence plus partial necking. The flow stress of the LTSP specimens dropped to nearly one half as compared with the as-received (AR) non-LTSP samples, and the strain rate sensitivity increased from 0.15–0.2 of the AR specimens to above 0.4 of the LTSP ones. In this report, the grain-structure and grain-orientation evolution, as well as the possible controlling deformation mechanisms during superplastic loading are characterized and discussed.

II. Experimental Methods

The 5083 alloy was obtained from China-Steel Aluminum Corp., Taiwan, in the form of hot-rolled thick plates to 30 mm, with a composition of Al-4.7%Mg-0.7%Mn (all in weight percent). The AR thick plates possessed elongated grains measuring $500 \times 80 \times 8 \mu\text{m}^3$ and did not exhibit any LTSP. Simple TMT processes were applied, including annealing at 500°C for 1 h, followed by air cooling and a series of low temperature rolling (25–250°C) to a final thickness of 0.5–3 mm. The rolling reduction ratio or the true strain received during TMTs varied from 90% to 98% or from 2.3 to 4.1, respectively. The trial TMT routes covered a wide range of processing conditions. Among them, an optimum TMT condition was determined. In this paper, the descriptions

on the grain structure evolution, texture transformation and deformation mechanism analysis are all centered on the results obtained from the specimens processed by the optimum route. No static recrystallization heat treatment was carried out before tensile loading. Note that the strain level experienced during TMTs in this study was around 2–4, which was smaller but comparable to the case of ECA. Both processes could produce fine grained materials. The material factory can utilize common rolling facility to produce large-scaled superplastic sheets for further forming. It does not need to redesign extrusion or pressing machine. Also, the TMT prevents from the troublesome water quenching after annealing, and it excludes the prolonged overaging heat treatment during TMTs⁽²⁾.

Some AR or TMT processed specimens were prepared for exploring the thermal stability of precipitate dispersion and grain structures. For precipitation characterization, the 5083 specimens were thermally annealed at 200, 300, 400, 500, 550 and 570°C for 1 h, and then water quenched for further TEM foil specimen preparation. For the examination of grain structures, the as-TMT processed sheet specimens were heated up under the controlled heating rate, *i.e.*, 50 min from room temperature to the set temperatures such as 250, 280, and 300°C. Once the desired temperature was reached, the specimens were water quenched and ready for TEM foil specimen preparation. Another group of specimens were all heated to 250°C during 50 min, followed by further 10–60 min holding time, in order to study the effect of static annealing on the grain-structure thermal stability at this very temperature.

Constant crosshead speed tensile tests were conducted using an Instron 1125 universal testing machine equipped with a three-zone furnace, with the loading direction parallel to the rolling direction. It usually took 50 min for the tested specimens to reach in the desired loading temperature without thermal fluctuations. Different heating rates were tried, and 50 min seemed to be optimum in terms of LTSP elongation performance and instrument stability. The specimen gauge length was 8 mm.

The grain structure and grain orientation distribution were examined using a Jeol 6400 SEM, equipped with an Oxford Link Opal electron backscatter diffraction (EBSD) system. The surface of the examined specimens were first mechanically polished, followed by electrochemical polishing in 6% perchloric acid-80% ethanol-14% distilled water at room temperature and a DC voltage of 30 V. The 3 mm foil specimens sampled from the TMT processed sheets or superplastically loaded tensile specimens to different levels of plastic strain were prepared using a Struer's twin-jet polisher in a solution of 25% HNO₃-75% methanol cooled to -30°C at a DC voltage of 10 V, and examined using a Jeol 200CX or 3010 transmission electron microscope (TEM).

III. Results

1. Tensile test results

Figure 1(a) shows the typical stress-strain curves for the AR and TMT processed specimens loaded at 250°C. The TMT processed one revealed LTSP to 400% in the optimum loading condition of 250°C and $1 \times 10^{-3} \text{ s}^{-1}$, and exhibited initial strain hardening followed by strain softening behavior. The ultimate tensile stress (UTS) typically occurred at a true tensile strain of $\epsilon = 0.4$. It seemed that once the subgrains formed during TMTs transformed successfully into high angle grain boundaries during the critical initial straining over $0 < \epsilon < 0.5$, the material could further deform smoothly to large strains. At high temperatures, the flow stress exhibited continuous strain hardening effect. Figure 1(b) compares the stress strain curves of the TMT processed 5083 alloy at 250 and 550°C.

The mechanical data of tensile elongation and ultimate tensile stress obtained from the AR and TMT specimens at $8 \times 10^{-4} \text{ s}^{-1}$ are compared in Fig. 2(a), covering the LTSP ($< 400^\circ\text{C}$) and HTSP ($> 400^\circ\text{C}$) regimes. At 250°C, the tensile elongation of the AR specimens was only 84%, compared with the 350% of the TMT specimens. In Fig. 2(a), there are two maximum peaks, one of LTSP at $\sim 250^\circ\text{C}$ and the other of HTSP at $\sim 550^\circ\text{C}$. Although the values of tensile elongation e were similar to each other (both with $e \sim 300\text{--}400\%$), the controlling deformation mechanisms at these two temperatures might not be the same, as discussed in Sec. IV. With in-

Fig. 1 Typical true stress and strain curves of (a) the AR and TMT specimens loaded at 250°C and $1 \times 10^{-3} \text{ s}^{-1}$, and (b) the TMT specimens loaded at 250°C and 550°C and $8 \times 10^{-4} \text{ s}^{-1}$. The strain levels chosen for TEM examination are marked on the curve of the TMT specimen in (a).

Fig. 2 Comparison of the (a) tensile elongation, and (b) ultimate tensile stress of the AR and TMT specimens as a function of loading temperature at $8 \times 10^{-4} \text{ s}^{-1}$.

creasing loading temperature, the mechanical results of the AR and TMT groups gradually merged to similar regions. At temperatures greater than 400°C, the 5083 alloy, independent of the AR and TMT specimens, exhibited typical solute drag creep behavior with a tensile elongation over 200%.

At low temperatures over 200–300°C, the maximum tensile stresses of the TMT specimens in Fig. 2(b) were lower than those of the AR ones by 50–80 MPa. For example, the maximum stress dropped from 133 MPa of the AR materials at 250°C and $8 \times 10^{-4} \text{ s}^{-1}$ to 64 MPa of the TMT processed ones, suggesting the stress reduction effect in the fine-grained thin sheets originated from the activation of grain boundary sliding (GBS) at low temperatures. This also implies that the controlling deformation mechanism in the AR and TMT specimens should be different.

The variation in LTSP elongation data at 250°C obtained from the TMT processed specimens as a function of strain rate is plotted in Fig. 3. The optimum strain rate was $1 \times 10^{-3} \text{ s}^{-1}$. Above $1 \times 10^{-3} \text{ s}^{-1}$, the tensile elongation dropped appreciably.

Table 1 compares the room temperature tensile properties of the AR and TMT 5083 alloys at $5 \times 10^{-4} \text{ s}^{-1}$. The room temperature tensile strength of the as-TMT specimens was considerably higher than that of the AR samples. This was because that the as-TMT specimens were all under the strain hardening condition. Upon annealing at elevated temperatures for 0.5 h, the room temperature UTS decreased and tensile elongation increased.

Fig. 3 The dependence of tensile elongation of the TMT specimens as a function of loading strain rate at 250°C.

Table 1 Comparison of the room temperature tensile properties of the AR and TMT processed 5083 alloys.

Specimen condition	UTS/MPa	Elongation
As received (AR)	370	12%
AR+annealing at 500°C for 1 h	316	20%
As-TMT	507	10%
TMT+annealing at 100°C for 0.5 h	493	10%
TMT+annealing at 150°C for 0.5 h	481	11%
TMT+annealing at 200°C for 0.5 h	470	12%
TMT+annealing at 250°C for 0.5 h	423	18%

Nevertheless, the mechanical properties were still superior to the behavior of the AR samples.

2. Strain rate sensitivity

Figure 4(a) shows the plot of the flow stress versus strain rate for the LTSP specimens at 250°C to true strain levels of 0.3, 0.4 and 0.5. A higher local slope corresponding $m \sim 0.55$ was observed at $8 \times 10^{-4} - 1 \times 10^{-3} \text{ s}^{-1}$ and the average m was ~ 0.43 over the range of $\dot{\epsilon} = 5 \times 10^{-4} - 1 \times 10^{-3} \text{ s}^{-1}$, as shown in Fig. 4(b). An m -value greater than 0.3 was observed over the strain rate range of 2×10^{-4} to $5 \times 10^{-3} \text{ s}^{-1}$ at 250°C, compared with $m \sim 0.2$ for the AR specimens. However, all the m -values for the AR and TMT specimens at $T > 400^\circ\text{C}$ over the strain rate range of 2×10^{-4} to $8 \times 10^{-3} \text{ s}^{-1}$ merged to a similar value of 0.3.

A number of fine grained Al alloys exhibiting LTSP have been reported to possess an m -value greater than 0.3. For example, m was equal to 0.48 for the $0.3 \mu\text{m}$ Al-Cu-Zr alloy loaded at 220°C and $3 \times 10^{-4} \text{ s}^{-1}$ and m was 0.41 for the $0.8 \mu\text{m}$ 1420 alloy loaded at 300°C and $1 \times 10^{-4} \text{ s}^{-1}$ (23). The current data agreed with these results, and all of them suggested the involvement of GBS even at such low temperatures.

3. Reduction ratio of cross-sectional area in tensile specimens

The uniformity of specimen straining within the gauge section is always of concern. Figure 5 shows the spatial variation of specimen thickness, width, and cross-sectional area for a LTSP specimen loaded at 250°C and $1 \times 10^{-3} \text{ s}^{-1}$ to tensile elongations of 10%, 20%, 108%, 153%, and 216%. It was seen that apparent local necking started to occur at tensile elongations greater than 150%.

The deformation uniformity in SP samples has been evaluated in terms of the ratio of sample thickness reduction to width reduction, R_T/R_W , where $R_T = (T_f - T_o)/T_o$ and $R_W = (W_f - W_o)/W_o$ and T_f , T_o , W_f and W_o are the final and initial sample thickness and width (2). If the grain structures become equiaxed and randomly oriented, this ratio will be stabilized to a fixed value close to one. Figure 6(a) gives the typical spatial variation of R_T/R_W values, and Fig. 6(b) shows the decreasing trend of R_T/R_W with increasing strain level. The high but decreasing R_T/R_W ratio at low superplastic strains were due to the structure evolution which gradually changes the as-TMT anisotropy structures during

Fig. 4 The variation of (a) the flow stress at $\epsilon=0.3-0.5$, and (b) the average strain rate sensitivity at over $\epsilon=0.3-0.5$ as a function of strain rate for the TMT or AR specimens loaded at 250°C.

Fig. 5 The spatial variation of (a) specimen thickness, (b) specimen width, and (c) specimen cross-sectional area within the gauge section of the TMT specimens loaded at 250°C and $1 \times 10^{-3} \text{ s}^{-1}$ to different strain levels as marked in Fig. 1(a).

Fig. 6 (a) The spatial variation of specimen thickness reduction ratio R_T versus width reduction ratio R_W measured from the TMT specimens loaded at 250°C and $1 \times 10^{-3} \text{ s}^{-1}$ to different strain levels as marked in Fig. 1(a). (b) The variation of R_T/R_W as a function of true local strain ϵ' .

the initial stage into equiaxed and randomly oriented structures. It seems that the specimen needs at least a local plastic true strain of $\epsilon' = 1.3$ (a tensile elongation of $\sim 150\%$) for the grain structure evolution to be completed, so as to exhibit a stabilization of R_T/R_W ratios near unity.

Previous studies on the LTSP 8090 Al-Li base alloy⁽²⁾ at 350°C also revealed that a minimum tensile elongation of 200% was required for R_T/R_W to reach the plateau level. In contrast, the partially recrystallized HTSP 8090 alloy at 525°C exhibited $R_T/R_W \sim 1$ from the very early stage of superplastic straining. A similar observation on the HTSP 5083 alloy at 550°C⁽⁴⁾ was reported; for that alloy the R ratio (=the reverse of R_T/R_W) maintained a relatively fixed value of ~ 0.75 during the entire superplastic straining.

4. Microstructure evolution

(1) Precipitates

The most notable precipitates in the current alloy were the Al_6Mn phase, as evident from Fig. 7. The Al_6Mn precipitates remained relatively fixed in size, most within 0.1–0.3 μm , in specimens thermally annealed at 200, 300, 400, 500, 550 and 570°C followed by water quenching. The interparticle spacing for the Al_6Mn precipitates was in the neighborhood of 0.3–1 μm , which might have something to do with the resulting subgrain size of 0.5 μm in the TMT processed sheets.

(2) Microstructures before LTSP loading

The coarse grained structures in the AR and AR + an-

Fig. 7 TEM bright field micrographs of the 5083 alloy annealed at (a) 250°C, and (b) 550°C for 1 h, showing the similar size of the Al_6Mn particles.

nealing (at 500°C for 1 h) specimens were examined using optical microscopy, and the grain size was $500 \times 80 \times 8 \mu\text{m}^3$ and 30 μm , respectively. Systematic characterization of the grain structures using TEM was done on specimens under the (i) as-TMT condition; (ii) TMT + heating to 250°C (taking approximately 50 min) condition; and (iii) step (ii) plus further holding time at 250°C for another 10 to 60 min, *i.e.*, static annealing for different periods of time.

Figure 8(a) shows a representative TEM micrograph of the 5083 alloy under the as-TMT condition. The typical microstructures seen from the rolling plane were poorly defined subgrain-like substructures measuring 0.3–1 μm under the as-TMT condition. The major preferred sample orientations can be traced by the associated diffraction patterns. All diffraction patterns were taken using an selected area aperture covering 8 μm in diameter. The TEM foil normal orientation, or the rolled sheet normal, of the as-TMT specimens scattered from near [110] (Fig. 8(b)), to near [112] (Fig. 8(c)), or other zones such as [100], [113], or [123] (Fig. 8(d)). This suggests that the texture contained multiple components. In most cases, the foil normal was oriented along a direction within the range of [110] to [112]. The mutual angular misorientation between neighboring subgrains and grains can also be roughly estimated by the diffraction patterns. For example, the maximum misorientation in Fig. 8(b) was at

Fig. 8 (a) TEM bright field micrograph and (b)–(d) the associated diffraction patterns (using a fixed size of selected area aperture) for the as-TMT processed specimens. The zone axis was close to [110] in (b), close to [112] in (c), and close to [100] in (d).

Fig. 9 TEM bright field micrographs and their associated diffraction patterns for the TMT specimens (a) upon heating to reach the stable temperature of 250°C without temperature fluctuation (note that the heating rate was controlled to be fixed and it took 50 min for the specimens to heat from room temperature to the stable temperature), and (b) subjected to further 50 min holding time at 250°C.

most 10°, suggesting the prevailing occurrence of low angle subgrain boundaries.

After heating to 250°C, the specimens contained better defined subgrains measuring $\sim 0.5 \mu\text{m}$ under the TMT+heating to 250°C condition, as shown in Fig. 9(a). Although different zone axes were observed, a near [110] diffraction pattern is selectively presented in order to compare with the pattern in Fig. 8(b). Note that even though the grain structure in Fig. 9(a) seems to be better defined, the relative misorientation did not alter much. It can be seen that the current LTSP sheet, after heating to the optimum temperature of 250°C, contained roughly equiaxed grains or subgrains with small angled boundaries at the onset of superplastic loading.

After further holding to different periods of time from 10 to 60 min, the microstructures were evolved into better defined subgrains, as an example shown in Fig. 9(b)

Fig. 10 The variation of (sub)grain size observed in the TMT specimens after static annealing at 250°C for 0–60 min holding time. In all cases the heating time was 50 min.

for the condition of TMT+heating to 250°C+holding for 50 min. From the associated diffraction pattern in Fig. 9(b), the relative misorientation and texture also did not evolve much. This was true for all the statically annealed specimens at 250°C for 10–60 min, though the grain boundaries became much clearer. This indicated that the recovery process during static annealing could not change grain orientation. It was observed there was gradual grain structure evolution during the static annealing with increasing holding time. The subgrain boundaries seemed to gradually evolved into more clear and distinct boundaries, reflecting the static recovery process at all boundaries. The variation of the average subgrain size or grain size as a function of static annealing time is depicted in Fig. 10. Here we use the term of '(sub)grain' size in Fig. 10, since in the measurements were made without the differentiation of subgrains or grains. The highest average (sub)grain size was less than $2.0 \mu\text{m}$, suggesting that the microstructure without mechanical loading appeared to be fairly thermally stable at 250°C.

The grain structures in the 5083 alloy were highly sensitive to the static annealing temperature. For example, when the TMT processed specimens were heated to 280 and 300°C, also taking ~ 50 min, the grains grew to around 9 and $13 \mu\text{m}$, respectively, compared with the $0.5 \mu\text{m}$ when heated to 250°C. The much larger grain size at 280°C and 300°C would effectively destroy the LTSP characteristics.

(3) Microstructures after LTSP loading

TEM characterizations were also conducted on specimens that were subjected to superplastic loading at 250°C and $1 \times 10^{-3} \text{ s}^{-1}$ to tensile elongations of 10% (true strain $\epsilon \sim 0.1$), 20% ($\epsilon \sim 0.2$), 108% ($\epsilon \sim 0.7$), 153% ($\epsilon \sim 0.9$), and 216% ($\epsilon \sim 1.2$), as marked on the representative stress-strain curve in Fig. 1(a). The specimens were first strained to the set level of tensile elongation, and then stopped and unloaded. The exact local true plastic strain ϵ' , measured from the location in the tested tensile specimen at which the 3-mm TEM disc

Table 2 Summary of the corresponding values of specimen gauge tensile engineering elongation e , specimen gauge true strain ε , and local plastic true strain ε' measured from the positions for TEM and EBSD examinations.

Engineering elongation of the gauge length, e	True strain of the gauge length, ε	Local plastic true strain, ε'
10%	0.1	0.10
20%	0.2	0.25
108%	0.7	0.97
153%	0.9	1.31
216%	1.2	2.21

samples were punched, was determined. This value was calculated in accordance with $\varepsilon' = \ln A_0/A_f$, where A_0 and A_f are the initial and final cross-sectional areas at that position. For the above five tensile specimens to different tensile elongations, ε' were calculated to be 0.1, 0.25, 0.97, 1.31, and 2.21, respectively. **Table 2** summarizes the corresponding values of specimen tensile elongation e , true strain ε , and local true plastic strain ε' . These local true strain ε' values at later stages were higher than the overall tensile strain ε due to the local necking effect.

Both the grain structure and the grain orientation evolved with increasing strain, starting from the subgrain structures of $\sim 0.5 \mu\text{m}$ in Fig. 9(a). The structures gradually transformed into better defined fine grains to (i) $\sim 0.9 \mu\text{m}$ at a tensile elongation of $e=10\%$ or true local strain of $\varepsilon'=0.1$, as shown in Fig. 11(a); (ii) $1.4 \mu\text{m}$ at $e=20\%$ or $\varepsilon'=0.25$, as shown in Fig. 11(b); (iii) $\sim 1.8 \mu\text{m}$ at $e=108\%$ or $\varepsilon'=0.97$, as shown in Fig. 11(c); (iv) $\sim 2 \mu\text{m}$ at $e=153\%$ or $\varepsilon'=1.31$, as shown in Fig. 11(d); and (v) $\sim 2.5 \mu\text{m}$ at $e=216\%$ or $\varepsilon'=2.21$, as shown in Fig. 11(e). Note that severe dislocation activity can be traced from the dense dislocation tangles inside the grain interior in Figs. 11(a) and (b), reflecting the predominant deformation mechanism related to dislocation glide during the initial stage of straining over $\varepsilon'=0-0.3$. In contrast, less dislocations were observed inside the grains in specimens subjected to $\varepsilon'=0.97$ or above. Grain boundary sliding should have been active in such later stages. A well defined grain structures to $2.5 \mu\text{m}$ in diameter with few dislocations in the grain interior seemed to be a stable microstructure when loaded at 250°C .

The variation of the average (sub)grain size as a function of true local strain ε' and superplastic loading time during LTSP loading is depicted in Figs. 12(a) and (b); the latter can be compared with the case of static annealing shown in Fig. 10. The time to attain a tensile elongation of 216% (corresponding to the true local strain of $\varepsilon'=2.21$) was around 20 min. Tensile loading at 250°C for 20 min will result in an average grain size of $2.5 \mu\text{m}$, much larger than the $0.75 \mu\text{m}$ when static annealing at 250°C for 20 min. It is apparent that the grain growth was accelerated under loading.

A distinct change in diffraction patterns was seen for the specimens experienced superplastic straining. As shown in Figs. 11(a) to (e), the texture with [110] to [112]

Fig. 11 TEM bright field micrographs and their associated diffraction patterns for the TMT specimens loaded at 250°C and $1 \times 10^{-3} \text{ s}^{-1}$ to a true local strain of (a) $\varepsilon'=0.1$, (b) $\varepsilon'=0.25$, (c) $\varepsilon'=0.97$, (d) $\varepsilon'=1.31$, and (e) $\varepsilon'=2.21$.

Fig. 12 The variation of (sub)grain size observed in the TMT specimens under superplastic loading as a function of (a) true local strain ε' , and (b) superplastic loading time.

zones parallel to the foil normal gradually transferred to a much more random orientation distribution. Even to a true local plastic strain of $\varepsilon'=0.1$, corresponding to the

Fig. 13 The {100} pole figures, inverse pole figures for the sheet normal and inverse pole figures for the sheet rolling direction obtained from the SEM electron backscattering diffraction from the specimens under the condition of AR+annealing at 500°C for 1 h.

load-raising stage in Fig. 1(a), the grain orientation has started to evolve appreciably. After $\epsilon' = 0.97$, the original [110] or [112] zone could hardly be seen. Because of the grain growth effect, less grains were included in the fixed-size selected area aperture, and hence the diffraction patterns in Figs. 11(c)–(e) were simpler than those in Fig. 11(a) or (b).

Micro-cavitation was sometimes observed in the TEM specimens with $\epsilon' = 1.31$ or 2.21, all associated with grain boundaries or grain triple points. The length of the micro-cavities was mostly of the order of 0.5 to 1.5 μm . The coalescence of such fine cavities would result in final failure, which was caused by a mixed mode of partial necking and partial cavitation.

5. Texture evolution

Based on the EBSD results obtained from a number of Al-Mg alloys, it was found that the texture under the AR+annealing at 500°C condition was relatively weak, as shown in Fig. 13. When subjected to TMT processes, preferred orientation texture components were seen. As the TMT was performed using cold rolling at 25°C, a highly strong texture near the α -brass $\{110\}\langle 112 \rangle$ type was observed, with a maximum intensity contour of ~ 100 in the {100} pole figure. With increasing TMT temperature, the texture became weaker. Figures 14(a) and (b) are two examples for the specimens subjected to a TMT process at two different warm rolling temperatures ($T_{14(a)} < T_{14(b)}$). The highest texture intensity contour gradually decreased to ~ 40 in Fig. 14(a) to ~ 20 in Fig. 14(b). The LTSP tensile elongation became higher for a warm rolling TMT. For example, the highest LTSP elongations at 250°C for the cases of cold rolling, or warm rolling for Fig. 14(a) and Fig. 14(b) were $\sim 150\%$, 250% and 400%, respectively. Under the optimum TMT process, there exhibited multiple texture components containing the α -brass $\{110\}\langle 112 \rangle$ type and S $\{123\}\langle 634 \rangle$ type plus a minor copper $\{112\}\langle 111 \rangle$ type. Figure 14(b) shows the pole figures for the specimens processed by the optimum TMT and exhibiting the highest LTSP elongation. The following texture pole

Fig. 14 The {100} pole figures, inverse pole figures for the sheet normal and inverse pole figures for the sheet rolling direction obtained from the SEM electron backscattering diffraction from the specimens under the as-TMT processed condition. The warm rolling temperature in (a) was lower than that in (b).

figures presented were all further evolution from Fig. 14(b). It was apparent that once the texture after TMT was dispersed more widely, the resulting LTSP properties became better. The EBSD layouts can be used as an index in predicting the characteristics of LTSP. Also, the EBSD texture components are consistent with the preferred diffraction pattern zone axes observed in the TEM diffraction patterns with zero specimen tilt. Upon

heating to 250°C, the texture shown in **Fig. 15** remained similar to the one of the as-TMT specimens shown in **Fig. 14(b)**.

Once the TMT-processed tensile specimens were loaded at LTSP temperatures, the textures gradually transformed to more random orientation distribution. **Figure 16** shows the texture evolution when loaded at 250°C and $1 \times 10^{-3} \text{ s}^{-1}$ to $\epsilon' = 0.1, 0.25, 0.97, 1.31$, and 2.21 , respectively. At $\epsilon' = 0.97$ or above, a much more uniform orientation distribution was observed. The highest texture contour decreased from 20–25 under the as-TMT condition to around 10 after $\epsilon' = 2.21$.

Based on the TEM diffraction patterns and EBSD pole figures, the preferred orientation and the small misorientation between neighboring subgrains or grains were gradually evolved during the initial superplastic straining over $0 < \epsilon' < 1.0$. At $\epsilon' \sim 1.0$ (or $\epsilon \sim 0.7$, or a tensile elongation of 100%) the grain structure and grain orientation have both transformed into a state favorable for overall GBS. At $\epsilon' > 1.3$ (or $\epsilon > 0.9$, or a tensile elongation of 150%), the effective GBS operating at most

grain boundaries would result in a nearly isotropic deformation characteristic of a R_T/R_W ratio near unity.

IV. Deformation Mechanism and Discussion

The controlling deformation mechanisms for LTSP have been discussed previously⁽²⁾⁽²⁴⁾. For fine-grained Al-Mg base alloys deformed at around 250°C, a number of possible mechanisms should be considered. Firstly, the typical superplastic deformation controlled by grain boundary sliding would exhibit an m -value of ~ 0.5 ($n = 1/m \sim 2$) and a Q -value close to the one for grain boundary or lattice diffusion, with strong grain size dependency⁽²⁵⁾, i.e.,

$$\dot{\epsilon} = A \frac{Eb}{kT} D_{\text{eff}} \left(\frac{b}{d} \right)^p \left(\frac{\sigma - \sigma_0}{E} \right)^2, \quad (1)$$

where $\dot{\epsilon}$ is the strain rate, A the coefficient related to grain boundary structure, E the elastic modulus, b the Burgers' vector, T the test temperature, k the Boltzmann's constant, D_{eff} the effective diffusivity, d_{eff} the grain or grain-group size active for GBS, p the grain-size exponent close to 2–3, σ the applied stress, and σ_0 the threshold stress below which no superplastic straining or dislocation creep will occur. The second acting mechanism might be the power law dislocation creep governed by dislocation climb, with an average m -value of ~ 0.2 ($n \sim 5$, ranging from 4 to 8) and no grain size dependence⁽²⁶⁾, i.e.,

$$\dot{\epsilon} = B \frac{Eb}{kT} D_{\text{eff}} \left(\frac{\sigma - \sigma_0}{E} \right)^5, \quad (2)$$

where B is another coefficient for dislocation creep. Thirdly, the solute drag mechanism for aluminum alloys with a sufficient amount of Mg should be active. The rate

Fig. 15 The inverse pole figures for the sheet normal and rolling direction obtained from the SEM electron backscattering diffraction from the specimen under the condition of TMT+heating to 250°C.

Fig. 16 The inverse pole figures for the sheet normal obtained from the SEM electron backscattering diffraction from the TMT specimens loaded at 250°C and $1 \times 10^{-3} \text{ s}^{-1}$ to a true local strain of (a) $\epsilon' = 0.1$, (b) $\epsilon' = 0.25$, (c) $\epsilon' = 0.97$, (d) $\epsilon' = 1.31$, and (e) $\epsilon' = 2.21$.

equation is⁽²⁷⁾

$$\dot{\varepsilon} = C \frac{kT}{E} D_s \left(\frac{\sigma}{E} \right)^3, \quad (3)$$

where C is a coefficient, D_s is the diffusivity of the solute in Al matrix. Other low temperature deformation mechanisms include several dislocation glide controlled processes, e.g., the thermally activated dislocation glide mechanism⁽²⁸⁾. The latter ones are not considered here since the loading temperature of 200–400°C for current LTSP might be too high and the strain rate of 2×10^{-4} – $8 \times 10^{-3} \text{ s}^{-1}$ might be too low for them to control the deformation.

For the coarse grained AR 5083 alloy, the m -values at 200–300°C were consistently around 0.2, suggesting that power law creep might have been controlling. Similar results have been reported by Taleff *et al.*⁽¹⁴⁾ We postulated that the involvement of solute drag creep might be very limited over this low temperature range. The transition between solute drag creep at higher temperatures and lower stresses to power law creep at lower temperatures and higher stresses has been discussed by Woo *et al.*⁽¹¹⁾ The flow stresses of the AR specimens loaded at 200–300°C and 5×10^{-4} – $5 \times 10^{-3} \text{ s}^{-1}$ were all above the critical transition stress, and fell well within the power law region. As temperature increased to 400°C or above, the AR alloy lay within its typical solute drag mechanism regime.

As for the TMT processed fine grained 5083 specimens, the m -values at 250°C over the optimum narrow strain rate range of 5×10^{-4} – $2 \times 10^{-3} \text{ s}^{-1}$ were mostly scattered within 0.4–0.55, which resided close to the upper bound of 0.5 for GBS and the lower bound of 0.33 for solute drag process. Recall that the maximum stress dropped from 133 MPa of the AR materials at 250°C and $8 \times 10^{-4} \text{ s}^{-1}$ to 64 MPa of the TMT processed ones. Such a stress reduction effect in the fine-grained superplastic materials is usually originated from the activation of GBS. This implies that GBS might be the controlling mechanism under the optimum LTSP condition.

As for the activation energy Q -values, they were extracted from either $\ln \dot{\varepsilon}$ under a constant stress condition, as shown in Fig. 17, or $\ln \sigma$ under a constant strain rate condition, as presented in Fig. 18. In Fig. 17, the LTSP $\ln \dot{\varepsilon}$ data are plotted against $1000/RT$, and the slope or the average apparent activation energy Q for $\varepsilon=0.4$ and 0.5 is $\sim 97 \text{ kJ/mol}$. Considering the temperature dependence of elastic modulus $E(T)$ ⁽²⁹⁾, Q should be around 90 kJ/mol. On the other hand, if we calculate the activation energy under a constant strain rate condition, the slope in Fig. 18 represents mQ . Taking the average m -value as 0.4 over 200–300°C for the LTSP data in Fig. 18(a), the average apparent activation energy turns out to be $Q \sim 58 \text{ kJ/mol}$ for LTSP. Similarly, taking the average m -value as 0.33 over 400–550°C for the HTSP data in Fig. 18(b), the average apparent activation energy is $Q \sim 151 \text{ kJ/mol}$ for HTSP. With the consideration of temperature dependence of elastic modulus $E(T)$, Q should be around 51 kJ/mol for LTSP and 145 kJ/mol for HTSP.

Fig. 17 The plot of $\ln \dot{\varepsilon}$ versus $1000/RT$ for LTSP data on the TMT processed 5083 alloy loaded over 200–300°C to $\varepsilon=0.4$ and 0.5. From the slope, an average apparent $Q \sim 97 \text{ kJ/mol}$ is obtained.

Fig. 18 The plot of $\ln \sigma$ versus $1000/RT$ for (a) LTSP data at $T=200$ – 300°C , and (b) HTSP data at $T=400$ – 550°C obtained from the TMT processed 5083 alloy strained to $\varepsilon=0.4$ and 0.5. From the slope and then divided by corresponding m , an average apparent $Q \sim 58$ for LTSP and 151 kJ/mol for HTSP is obtained, respectively.

The Q -values of $\sim 145 \text{ kJ/mol}$ for HTSP at $T=400$ – 550°C is consistent with the activation energy of $\sim 136 \text{ kJ/mol}$ for Mg diffusion in Al⁽³⁰⁾, and has been reported in a number of similar alloys. And the Q -values for LTSP at $T=200$ – 300°C obtained from the two approaches were either 90 or 51 kJ/mol. Although they are widely scattered, they are both much lower than the 142 kJ/mol for Al lattice diffusion and 136 kJ/mol for Mg diffusion in Al, and might be closer to the activation energy for grain boundary diffusion ($\sim 84 \text{ kJ/mol}$) or dislocation pipe diffusion ($\sim 82 \text{ kJ/mol}$)⁽³¹⁾. The current results of Q -values are similar to our previously reported LTSP in the 8090 Al–Li alloy at 350–450°C⁽²⁾, but the measured m -values are not. In LTSP 8090 alloy, m was ~ 0.3 , compared with the current range of 0.4–0.55. The preliminary overall results of a low Q -value and a high m -value, couple with the significant reduction of flow stress, for the LTSP in the fine grained 5083 Al–Mg alloy might suggest the governing role of grain boundary sliding. The solute drag glide-controlled creep or power law climb-controlled creep might play minor contribution for the current LTSP at a very low temperature of 200–300°C.

V. Summary

Low temperature superplasticity (LTSP) was observed in the 5083 Al-Mg base alloy after thermomechanical treatment (TMT). The maximum LTSP elongation was 400%, occurring at 250°C and $1 \times 10^{-3} \text{ s}^{-1}$. The microstructure evolution and deformation mechanisms of the LTSP specimens were examined and discussed. The following conclusion can be drawn.

(1) The subgrain structures formed after TMT transformed to better defined subgrains to $\sim 0.5 \mu\text{m}$ upon heating to 250°C. Further static annealing or superplastic straining at 250°C would result in well defined fine grains to 1.5–2.5 μm , dependent on annealing time or strain level. The grain evolution rate seemed to be accelerated by superplastic loading.

(2) Multiple texture components of the type of brass $\{110\}\langle 112 \rangle$ or S $\{123\}\langle 634 \rangle$ were observed in the as-TMT specimens. After superplastic straining at 250°C to 100% or above, a much more random orientation distribution was formed. Static annealing at 250°C itself could not alter the existing texture.

(3) The high but decreasing R_T/R_W ratio at low superplastic strains was due to the structure evolution which gradually changed the as-TMT anisotropy structures during the initial stage into equiaxed and random structures. It seems that the specimen needs at least 150% strain for the grain structure evolution to be completed, so as to exhibit a stabilization of R_T/R_W ratio near unity.

(4) Under the optimum LTSP condition, the m -value was ~ 0.5 , compared with 0.2 for the as-received coarse-grained 5083 alloy at 250°C. The activation energy Q -value was around 50–90 kJ/mol for LTSP over 200–300°C and around 145 kJ/mol for HTSP over 400–550°C. It is postulated that the rate controlling deformation mechanism in the TMT processed 5083 specimens was grain boundary sliding during the optimum LTSP condition and solute drag creep during HTSP deformation.

Acknowledgment

The authors would like to gratefully acknowledge the sponsorship from National Science Council of ROC under the project no. NSC 87-2216-E-110-018.

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