Effect of Pass Strain on Grain Refinement in 7475 Al Alloy during Hot Multidirectional Forging

Oleg Sitdikov , Taku Sakai , Alexandre Goloborodko , Hiromi Miura and Rustam Kaibyshev

Effect of pass strain ($\Delta\varepsilon$) on grain refinement was studied in multidirectional forging (MDF) of a coarse-grained 7475 Al alloy at 490°C under a strain rate of $3 \times 10^{-4}\,s^{-1}$. Samples of rectangular shape were deformed up to accumulated strains of around 6 with subsequent changes in loading direction 90° from pass to pass. The pass strains in each compression ($\Delta\varepsilon$) were 0.4 and 0.7. The cumulative flow curves integrated by each compression exhibit significant work softening just after yielding, followed by apparent steady state plastic flow at high strains. Structural changes were characterized by grain fragmentation due to frequent development of deformation and/or microshear bands followed by full evolution of new fine grains in the original grains. Increasing $\Delta\varepsilon$ accelerates significantly the kinetics of grain refinement, leading to more clear reduction of flow stresses at moderate to high strains. MDF of $\Delta\varepsilon = 0.7$ results finally in formation of a finer grained structure with an average size of around 7.5 µm at strains of above 3.5, while, the processing with $\Delta\varepsilon = 0.4$ develops a slightly coarser grain structure at higher strain of about 6. The effect of MDF on new grain evolution and the mechanisms of grain refinement are discussed in details.

Keywords: *grain refinement, multidirectional forging, aluminum alloy, shear band, grain boundary sliding*

1. Introduction

Recent investigations have shown that there is a great potential for extraordinary improvements in the chemical, physical and mechanical properties of many metals and alloys by refining their grain structure through severe plastic deformation (SPD) to (sub)micron to nanocrystalline level.[1)] Several techniques, *e.g.* equal channel angular extrusion,[1–8)] torsion under high pressure,[1,4,7)] multidirectional forging (MDF)[1,4,9–14)] *etc.*, are available for subjecting materials to ultra-high strains required to develop such fine grain structures. MDF seems to be especially attractive in these various SPD methods, because it is the easiest method without any special device and has great potentiality for scaling up of relatively large samples that can be suitable for industrial applications. This SPD technique can also be a very valuable scientific tool for studying the microstructure development in large strain deformation.[4,10–14)]

The principle of MDF, *i.e.* multidirectional forging operation, is repeating compression process with changing the axis of the applied strain $x \rightarrow y \rightarrow z \rightarrow x \rightarrow \ldots$ at each step. Redundant plastic strains can be accumulated into the material as it is repeatedly deformed at ambient to elevated temperatures. Since a work piece changes hardly its shape under MDF conditions, many repetitive passes can be undertaken to achieve very high total strains. It has been shown recently[13)] that such strain accumulation applied from various directions becomes very important for evolution of equiaxed fine grains and so the grain refinement. At the same time, grain refinement during interrupted deformation may be controlled not only by total strain accumulated, but also by a strain per each pass.[3)] There are, however, almost no experimental data on this effect in MDF, as far as the authors know.

The main aim of the present work was to study any influence of the value of pass strain ($\Delta\varepsilon$) on grain refinement during hot MDF. The 7475 Al alloy was used as one of the commercial-base materials showing continuous dynamic recrystallization (cDRX) during hot deformation.[13–15)] The microstructural evolution taking place during MDF with a step strain of 0.4 (hereafter ***MDF*** of $\boldsymbol{\Delta\varepsilon = 0.4}$) is described elsewhere.[13)] The current work analyzed the data obtained by ***MDF*** of $\boldsymbol{\Delta\varepsilon = 0.7}$ comparing with the results of $\boldsymbol{\Delta\varepsilon = 0.4}$. The effect of step strain on microstructural development and the grain refining mechanisms operating during hot MDF are discussed in details.

2. Experimental

The material tested was an as-cast 7475 Al alloy with the following chemical composition: Al-6%Zn-2.5%Mg-1.8%Cu-0.23%Cr-0.16%Zr-0.04%Fe-0.03%Si-0.03%Mn (in mass pct). The ingot was homogenized at 490°C for 20 h. The initial structure was composed of coarse dendritic lamellar grains. Parts of the grain boundaries were rather straight and the others were corrugated, as shown in Fig. 1. The average spacing of the lamellas was in a range from 1 to 10 mm in longitudinal direction and from 50 to 200 µm in transverse one. Two types of dispersions distributed in the alloy were identified by TEM analysis as Al_3Zr and Al_3Cr, which were equiaxed having an average size of 20 nm and 100 nm, respectively.[15)]

Rectangular samples of 10 mm × 9 mm × 5.5 (1.8 : 1.65 : 1) mm were machined from the ingot supplied. Multipass compression was carried out with changing of the loading direction of 90° from pass to pass at 490°C under a strain rate of $3 \times 10^{-4}\,s^{-1}$. The dimension ratio of the sample did not change during repeated deformation in a pass strain of 0.7. A powder of boron nitride was used as a lubricant. The tester enabled true strain rate to be constant and was equipped with a water quenching apparatus. The metallographic analysis was carried out on a section parallel to the last compression axis by using optical microscopy after etching by a standard Dicks-Keller etchant. Scanning electron microscope (SEM)

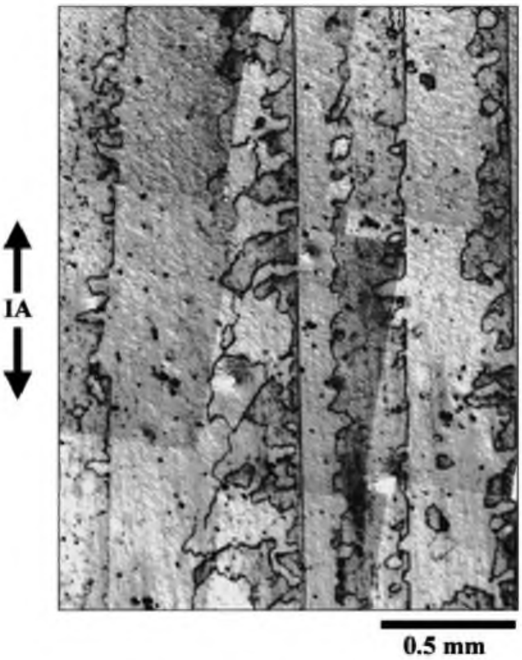

Fig. 1 Initial structure. IA is the ingot axis.

backscattering images were obtained using a Hitachi-3500A SEM with orientation imaging microscopy (OIM) OIM™ software provided by TexSem Laboratories, Inc.

3. Results

3.1 Mechanical properties

Figure 2 represents a series of true stress-true strain (σ-ε) curves plotted for 9 consecutive compression passes. The envelope flow curve summarized for MDF of $\Delta\varepsilon = 0.4$ is also represented by a dashed line for comparison. It is seen that the integrated flow curve demonstrates a sharp stress peak just after yielding followed by significant work softening. The latter takes place continuously up to strain of over 3.5 in $\Delta\varepsilon = 0.7$ and then the cumulative flow curve shows a steady-state-like flow behavior. It is remarkable to see in Fig. 2 that work softening appears more clearly in $\Delta\varepsilon = 0.7$ and deformation stress reaches rapidly a steady state flow one comparing with those during MDF of $\Delta\varepsilon = 0.4$. The ratio of the flow softening ($\Delta\sigma = \sigma_p - \sigma$) and the stress peak ($\sigma_p$), $\Delta\sigma/\sigma_p$, is around 0.6 in MDF of $\Delta\varepsilon = 0.7$, while it is around 0.5 in MDF of $\Delta\varepsilon = 0.4$. It can be seen in Fig. 2 that there is negligible small difference between the flow stresses immediately before unloading and at reloading.[16] This suggests that any structural changes during interrupted test can be mainly affected by strain accumulation applied in each compression pass. In contrast, this difference was observed to be rather large in MDF of $\Delta\varepsilon = 0.4$.[14]

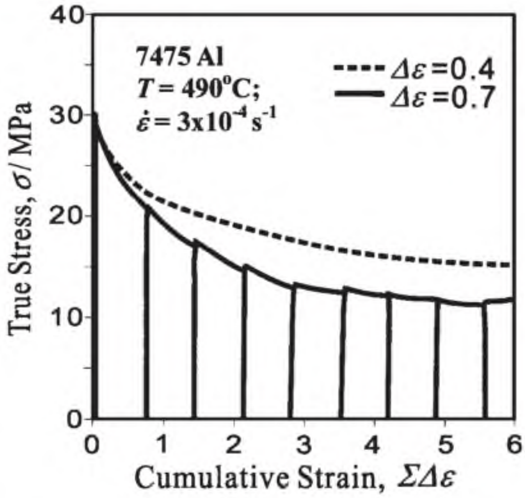

Fig. 2 Typical true stress-true strain (σ-$\Sigma\Delta\varepsilon$) curve obtained during MDF of $\Delta\varepsilon = 0.7$ at 490°C and at $\dot{\varepsilon} = 3 \times 10^{-4}\,s^{-1}$. The $\sigma - \Sigma\Delta\varepsilon$ curve in MDF of $\Delta\varepsilon = 0.4$ is represented by dashed line for reference.

3.2 Microstructural development

Typical microstructures evolved at various strains during MDF of $\Delta\varepsilon = 0.7$ are represented in Fig. 3. The regions that appear dark in color are composed of new fine grains with an average size of around 7.5 µm, as can be seen in Figs. 3(a') and (b'), the enlarged portions outlined in Figs. 3(a) and (b). It is clearly seen in Fig. 3(a) that at a cumulative strain of 2.1, *i.e.* after a first full cycle of MDF of $\Delta\varepsilon = 0.7$, grain refinement takes place frequently and the fine-grained regions are evolved inhomogeneously accompanied with some remained original grains. Original grains are gradually replaced to fine grains with strain increasing. At a strain of 3.5 (Fig. 3(b)), such new grains are almost fully developed and the volume fraction of fine grains is about 0.85. It is also remarkable to note in Figs. 3(a') and (b') that more equiaxed grains are developed after $\varepsilon = 3.5$ compared with those at $\varepsilon = 2.1$. In contrast, the fraction of fine grains evolved at a similar strain is significantly smaller during MDF of $\Delta\varepsilon = 0.4$, as shown in Fig. 3(c). It approaches, however, a saturation value of 0.85 at a higher strain of about 6, which is similar to that in $\Delta\varepsilon = 0.7$ (see Fig. 7(a)).

3.3 OIM microstructures

Figure 4 shows typical OIM pictures for the samples processed with $\Delta\varepsilon = 0.7$ to strains of (a) 0.7 and (b) 2.1. Here the different grayscale levels indicate the different crystallographic orientations and the orientation differences (Θ) between neighboring grid points, $\Theta > 2°$, $\Theta > 4°$ and $\Theta > 15°$ are marked by thin white, narrow and bold black lines, respectively. It is seen in Fig. 4(a) that new boundaries with low to moderate and even high angle misorientations are developed within original grain interiors after a first compression. The crystal orientation is frequently changed in the regions fragmented by such boundaries (see Fig. 5). The latters may correspond to those of microshear or deformation bands, as discussed in detail elsewhere.[13,15] It is also remarkable to see that several fine grains are evolved in the side of corrugated grain boundaries. After one cycle of MDF to $\varepsilon = 2.1$, several sets of deformation bands are evolved in various directions, resulting in subdivision of original grains into separate misoriented domains, accompanied with frequent evolution of new fine grains (Fig. 4(b)). With further straining, new grains with high-angle boundaries are rapidly and homogeneously developed not only in grain boundary regions, but also in grain interiors, although some rather coarser grains still remain in the regions of fine

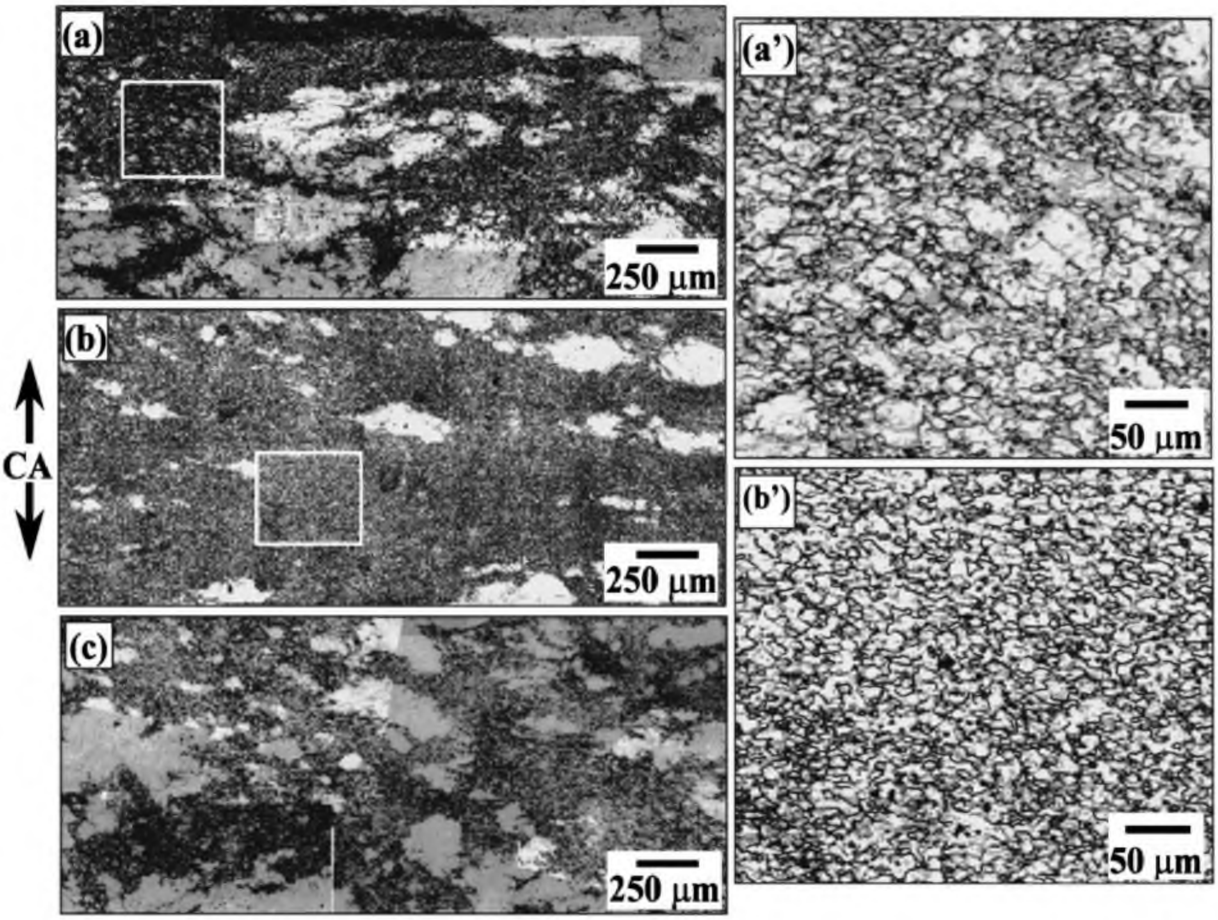

Fig. 3 Microstructures developed in 7475 Al during MDF at 490°C and at $\dot{\varepsilon} = 3 \times 10^{-4}\,\mathrm{s}^{-1}$: (a), (a') $\Sigma\Delta\varepsilon = 2.1$, $\Delta\varepsilon = 0.7$; (b), (b') $\Sigma\Delta\varepsilon = 3.5$, $\Delta\varepsilon = 0.7$; (c) $\Sigma\Delta\varepsilon = 3.6$, $\Delta\varepsilon = 0.4$. Regions with dark color are composed of new fine grains, as shown in enlarged portion outlined in (a) and (b). CA is the last compression axis.

grains (Fig. 3(b)). New deformation-induced boundaries were scarcely developed in such remained original grains, which existed stably even at a strain of 6.3.

Figure 5 represents the distribution of typical point-to-point ($\Delta\Theta$) and cumulative (point-to-origin) ($\Sigma\Theta$) misorientations developed in grain interiors along the lines T_1, T_2 and T_3 indicated in Fig. 4. The value of $\Delta\Theta$ defines the relative difference in crystal orientation between two adjacent scan points with step of 1 μm. In Fig. 5(a), $\Delta\Theta$ does not exceed generally 2°-3° except some spots with $\Delta\Theta > 10°$ to 15°, which corresponds to deformation-induced new boundaries. It is also remarkable to see that $\Sigma\Theta$ changes discontinuously and crystal orientation is alternated at the same places. This suggests that highly heterogeneous deformation takes place in coarse grain interiors during hot deformation, leading to rigid local lattice rotation and then formation of dislocation subboundaries. These can be similar to transition bands of microshear or kink bands.[18–20] The density and misorientation angle of such boundaries increase with further repeated MDF to $\varepsilon = 2.1$ (Figs. 5(b) and (c)). New high angle boundaries with misorientations ranging from 15° to beyond 50° are often developed in some grain interiors, leading to almost full development of fine grains at high strains.

Figure 6 represents changes in the misorientation distributions of deformation-induced dislocation boundaries with hot MDF. It is seen in Fig. 6(a) that most boundaries developed at $\varepsilon = 0.7$ exhibit low-to-medium angle misorientations of below 20°, although a small number of high angle boundaries with over 40° is detected. With further deformation, the fraction of low and moderate angle boundaries rapidly decreases and that of high-angle ones conversely increases in fine-grained regions. The misorientation distribution at high strains approaches the Mackenzie distribution of a fully annealed grain structure, as shown by dashed line in Fig. 6(d).[21]

Figure 7 represents changes in several characteristic parameters of the microstructure developed with MDF of $\Delta\varepsilon = 0.7$. The data of $\Delta\varepsilon = 0.4$ are included in Fig. 7 for comparison. The volume fraction of new grains, V_{rex}, the average misorientation, Θ_{ave}, in the fine-grained regions and the average size of new grains, d_{rex}, are plotted against accumulated strain in Figs. 7(a), 7(b) and 7(c), respectively. V_{rex} increases with deformation and approaches a saturation of about 0.85 at $\varepsilon \approx 3$. Θ_{ave} increases rapidly at low strains, gradually at moderate strains and then approaches a saturation value of around 34° at $\varepsilon \approx 2$. It is remarkable in Figs. 7(a) and (b) that new grains are more rapidly formed in a low to moderate strain range with increase in pass strain from 0.4 to 0.7, that is, larger pass strain under MDF conditions can promote the occurrence of grain refinement. On the other hand, d_{rex} rapidly drops at earlier stages of

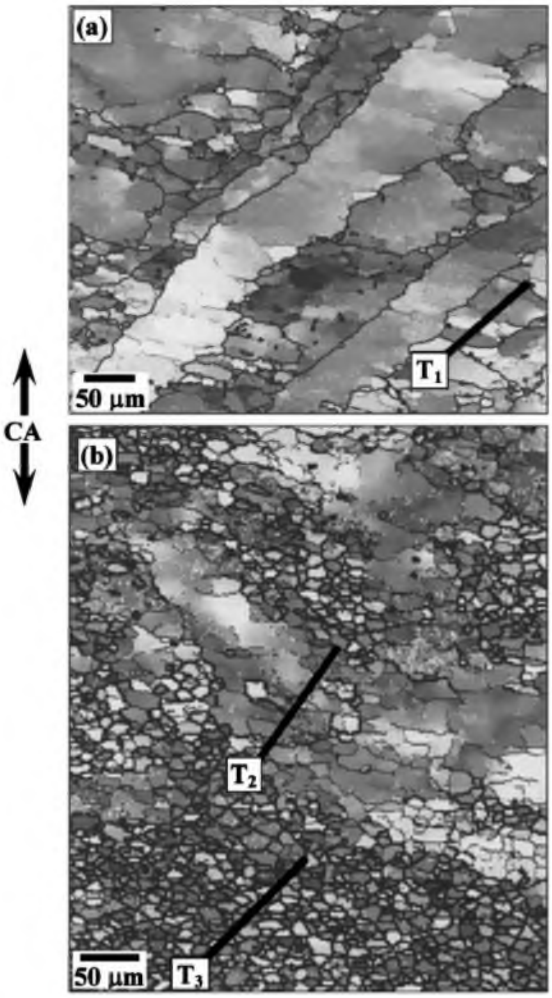

Fig. 4 Typical OIM pictures showing the microstructures developed in 7475 Al alloy during MDF at cumulative strains of (a) 0.7, and (b) 2.1. CA is the last compression axis.

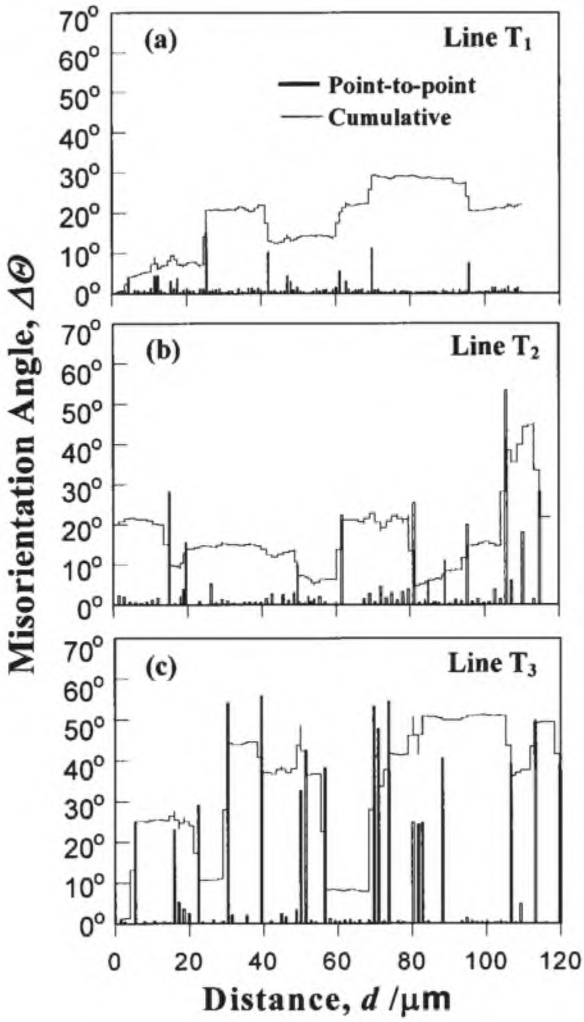

Fig. 5 Typical point-to-point and point-to-origin misorientations developed in original coarse grain interiors in 7475 Al alloy during MDF at $T = 490^\circ C$ and $\dot{\varepsilon} = 3 \times 10^{-4}\,s^{-1}$. (a) $\Sigma\Delta\varepsilon = 0.7$, (b) and (c) $\Sigma\Delta\varepsilon = 2.1$. Misorientations in (a), (b) and (c) were measured along the lines T_1, T_2 and T_3 indicated in Fig. 4, respectively.

deformation and approaches a roughly constant value after a cycle of MDF, *i.e.* at $\varepsilon = 1.2$ in MDF of $\Delta\varepsilon = 0.4$ and $\varepsilon = 2.1$ in MDF of $\Delta\varepsilon = 0.7$ (Fig. 7(c)). It should be noted in Fig. 7(c) that d_{rex} in $\Delta\varepsilon = 0.7$ is smaller than that in $\Delta\varepsilon = 0.4$. The smaller grain size evolved in $\Delta\varepsilon = 0.7$ may result in a lower flow stress (Fig. 2), as discussed in the *next Sections* in more details.

4. Discussion

4.1 Microstructural development during MDF

The present results mentioned above show that new grains are mainly developed during hot MDF due to grain fragmentation process accompanied by frequent evolution of deformation-induced subboundaries with low to moderate angle misorientation, which are recognized as the boundaries of deformation and/or microshear bands. The authors[13,15] showed that during hot compression of the current alloy grain boundary sliding (GBS) takes place frequently and inhomogeneously in the unsymmetric coarse-grained structure. GBS can occur with different rates along straight and corrugated segments of the original grain boundaries (see Fig. 1), resulting in formation of high strain gradients and stress concentrations in grain interiors. For relaxation of the latters, microscopic shear bands can be developed in the columnar grains even at early stages of hot deformation.[13]

The same discussion can be applied to the structural mechanism operating during MDF of both $\Delta\varepsilon = 0.4$ and 0.7. It is possible to consider that deformation-induced boundaries such as microshear bands are developed parallel to each other during first compression (Fig. 4(a)). When uniaxial compression is continuously applied to the material, *i.e.* under a constant strain path condition, the misorientation of such boundaries is increased and their spacing is gradually reduced with compression, eventually leading to formation of high-density two-dimensional planar microstructure, *i.e.* layered lamellar structure.[13] In contrast, changes in strain path during repeated MDF can promote development of such boundaries in various directions. They are continuously formed by strain accumulation and microstructural heterogeneities evolved in each compression pass, accompanying with their intersection in grain interiors.[13] Further MDF to large strains results in progressive transformation of the crystallites fragmented by microshear bands into new grains.

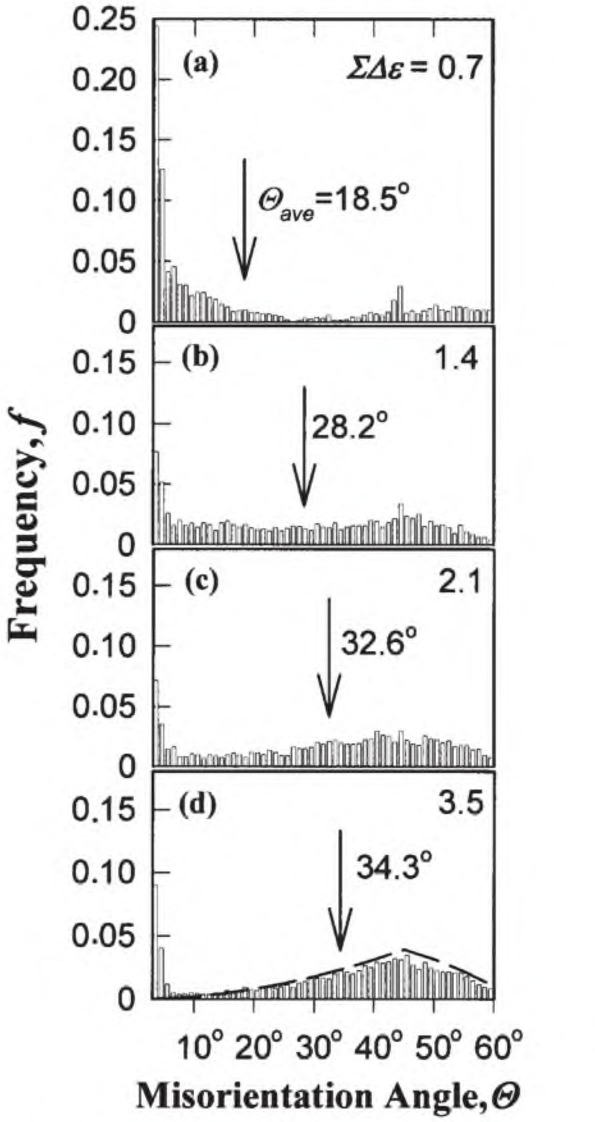

Fig. 6 Evolution of misorientation angle distribution of strain-induced boundaries in fine-grained regions developed during MDF of $\Delta\varepsilon = 0.7$. The broken line indicates the random misorientation distribution evaluated by Mackenzie.[21)]

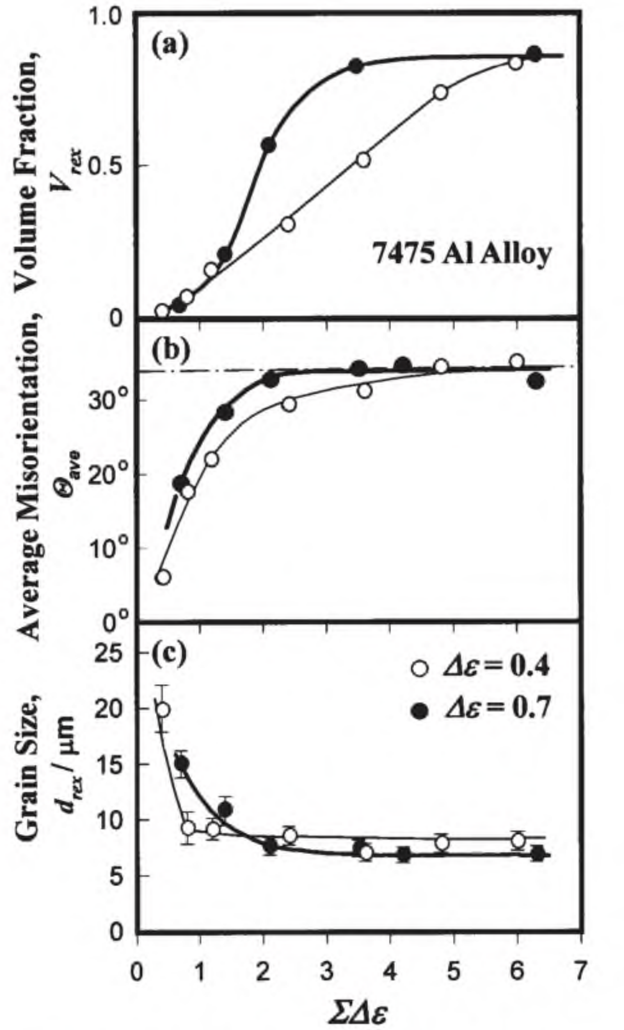

Fig. 7 Changes in the volume fraction of new grains, V_{rex}, the average misorientation angle of strain-induced boundaries, Θ_{ave}, and the average size of new grains, d_{rex}, evolved during MDF with various pass strains.

This is considered to be a kind of continuous reactions sometimes called as continuous dynamic recrystallization (cDRX).[4,7,10–15,17,22)] Various shearing directions appear during MDF and so the number of repeated compression passes can be more useful for evolution of equiaxed grains.[13)] The stable grain size is, therefore, developed more rapidly during MDF with smaller pass strain, *i.e.* $\Delta\varepsilon = 0.4$ (Fig. 7(c)).

4.2 Effect of pass strain

The data in Figs. 3 and 7 indicate that fine grain formation during MDF can be dependent on not only total strain accumulated, but also each pass strain, $\Delta\varepsilon$. It has been mentioned in Fig. 7 that increase in $\Delta\varepsilon$ from 0.4 to 0.7 under MDF conditions results in evolution of smaller stable grain size and also promotes more rapid increase in the volume fraction and average misorientation of new grains. Let us discuss here the effect of pass strain on such microstructural development in details.

Figure 8 represents typical OIM microstructures developed in remained original grains at strains of around 2 during MDF of $\Delta\varepsilon = 0.4$ and 0.7. The color and the thickness of boundaries have the same meanings as those in Fig. 4. It can be seen in Fig. 8 that the density of deformation-induced boundaries is much higher and the average size of new evolved grains is smaller in MDF of $\Delta\varepsilon = 0.7$ compared with those in MDF of $\Delta\varepsilon = 0.4$. This suggests that, as larger magnitude of local strains should be introduced during MDF of $\Delta\varepsilon = 0.7$, the density and misorientation of dislocation subboundaries become larger, finally resulting in more rapid evolution of new finer grains. There may be several factors accelerating development of new grains during MDF with increasing $\Delta\varepsilon$.

(i). GBS. New grain evolution can be assisted by operation of GBS especially in the regions fragmented by microshear bands, as discussed in 4.1. MDF with higher $\Delta\varepsilon$ can introduce more frequently the boundaries with higher misorientation angles and then grain rotation may occur with higher rate in fragmented regions. This should additionally accelerate transformation of low- to moderate angle boundaries to high angle ones *e.g.*[13,15,17,23,24)] This is one of the reasons why the volume fraction of fine grains is

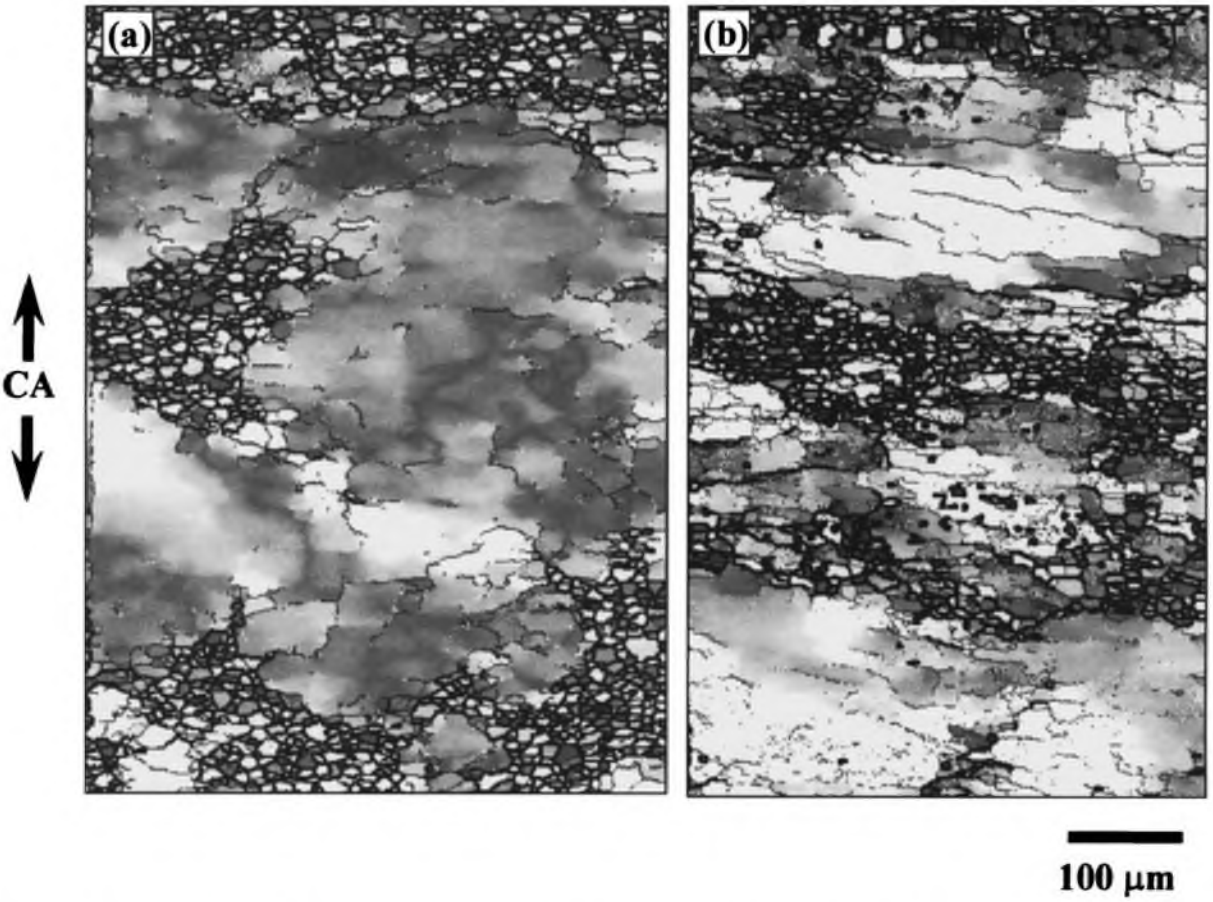

Fig. 8 Typical OIM pictures showing the microstructures developed in remained original grains in 7475 Al alloy. (a) $\Sigma\Delta\varepsilon = 2.4$, $\Delta\varepsilon = 0.4$ and (b) $\Sigma\Delta\varepsilon = 2.1$, $\Delta\varepsilon = 0.7$. The color and the thickness of lines have the same meaning as those in Fig. 4. CA is the last compression axis.

developed faster during MDF of $\Delta\varepsilon = 0.7$ (Figs. 3, 7 and 8).

(ii). Static restoration taking place between inter-pass. It is known[25] that a (sub)grain structure composed of higher misoriented (sub)boundaries can be more stable compared with lower misoriented one and so may be less recovered during the period of inter-pass time in MDF. Interrupted flow curves in $\Delta\varepsilon = 0.7$ indicates that quite small softening results from static recovery occurring at each deformation pass (Fig. 2). Such a static annealing effect, in contrast, appeared rather large during interrupted test with $\Delta\varepsilon = 0.4$.[14] This suggests that dislocation substructures evolved by previous deformation are more recovered under MDF with smaller $\Delta\varepsilon$ and then new grains with high-angle boundaries can be developed more slowly at moderate to high cumulative strains.

On the other hand, the characteristics of grain structures developed during MDF (*i.e.* Θ_{ave} and V_{rex} in Fig. 7) become similar at severe large strains irrespective of $\Delta\varepsilon$. It is interesting to note that V_{rex} approaches a saturation value below 1 even at high strains. Parts of relatively small original grains may remain stable because deformation accompanied by GBS takes place frequently in fine-grained regions. The volume fraction and size of remained original grains decrease and conversely the regions of new fine grains surrounding them increase with repeated MDF. Large strain gradients may, therefore, be hardly developed in these remaining grains because of frequent operation of GBS and dynamic recovery in the adjacent fine-grained regions. Such a stable grain structure is evolved at $\varepsilon \geq 3.5$ during MDF of $\Delta\varepsilon = 0.7$, while it can be observed only at $\varepsilon \approx 6$ during MDF of $\Delta\varepsilon = 0.4$.

4.3 Flow softening during MDF

It is generally believed that main restoration mechanism operating during cDRX is principally dynamic recovery and so work softening can hardly take place during hot deformation.[11,12,22] The present data show, however, that grain refinement taking place due to cDRX is accompanied with significant work softening at moderate to high strains, followed by apparent steady state in flow stresses and also in grain structure. When hot deformation is mainly controlled by GBS, flow stress (σ) depends on grain size (d) developed under steady state flow conditions and σ *vs* d can be represented by $\sigma = Cd^k$, where C, $k > 0$.[26] This suggests that smaller d evolved can lead to lower σ. It is discussed in detail[14,15] that work softening can be resulted from frequent operation of GBS in the fine-grained regions accompanied with increase in V_{rex}, which corresponds virtually to decrease of the mean grain size in the whole microstructure.

The quantitative dependencies between flow softening, $\Delta\sigma = \sigma_p - \sigma$, and microstructure development, V_{rex}, during MDF of $\Delta\varepsilon = 0.4$ and 0.7 are shown in Fig. 9. Here σ_p is the

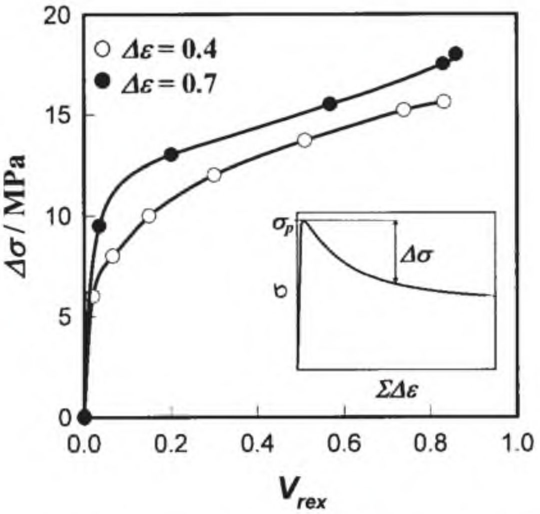

Fig. 9 Relationship between flow softening, $\Delta\sigma$, and volume fraction of new fine grains, V_{rex}, developed in coarse-grained 7475 Al alloy during hot MDF with strain step of 0.4 and 0.7.

peak stress and σ is the instantaneous flow stress in integrated flow curves. It is seen that $\Delta\sigma$ increases first rapidly, then at a medium rate and finally at a lower constant rate with increasing V_{rex} irrespective of $\Delta\varepsilon$. It is remarkable to see in Fig. 9 that $\Delta\sigma$ in $\Delta\varepsilon = 0.7$ is always larger than that in $\Delta\varepsilon = 0.4$ at an equivalent V_{rex}. Such a larger softening taking place in MDF of $\Delta\varepsilon = 0.7$ can result from smaller grain size evolved (Fig. 7(c)). It is concluded, therefore, that more prominent flow softening appearing during MDF with larger pass strain can result from more rapid development of new grains with smaller size (Figs. 2 and 7).

5. Summary

The influence of pass strain ($\Delta\varepsilon$) in multidirectional forging (MDF) was studied in a 7475Al alloy at a temperature of 490°C and at a strain rate of $3 \times 10^{-4}\,s^{-1}$. The main results obtained by MDF of $\Delta\varepsilon = 0.4$ and 0.7 are summarized as follows.

(1) The integrated flow curve exhibits a significant work softening at moderate strains, followed by steady-state like flow at large strains. With increasing in $\Delta\varepsilon$, flow stress decreases more rapidly and approaches faster a lower steady-state flow stress.

(2) Deformation bands including microshear bands are developed in various directions during hot MDF and so intersect each other, resulting in continuous grain fragmentation. Further deformation leads to increase in the number and misorientation of these boundaries and finally almost full development of fine equiaxed grains in high strain. This grain refinement mechanism can be similar to continuous dynamic recrystallization.

(3) With increasing $\Delta\varepsilon$ from 0.4 to 0.7, higher density deformation and/or microshear bands with larger misorientation angles are developed accompanied with evolution of finer grains at moderate strains and finally approach faster to a stable grain structure in high strain. The characteristics of such strain-induced grains, *i.e.* the average misorientation and the volume fraction, however, become almost the same at severe large strains irrespective of $\Delta\varepsilon$.

Acknowledgements

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