

Grain Refinement in As-Cast 7475 Al Alloy under Hot Multiaxial Deformation

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Abstract. Effect of pass strain ($\Delta\varepsilon$) on grain refinement was studied in multiaxial compression (MAC) of a coarse-grained 7475 Al alloy at 763 K under a strain rate of $3 \times 10^{-4} \text{ s}^{-1}$. The value of $\Delta\varepsilon$ influences significantly the kinetics of grain refinement. MAC of $\Delta\varepsilon=0.7$ results in a substantial grain refinement in high strain, finally leading to formation of a fine grain structure with a mean size of 7.5 μm and the volume fraction of about 85%. In contrast, MAC of $\Delta\varepsilon=0.4$ leads to formation of a smaller volume fraction of fine grains with the similar grain size. The grain refinement mechanisms operating under hot MAC are discussed in some details.

Introduction

The studies of grain refinement of engineering materials under thermomechanical processing are very important for improvement of several mechanical, chemical and physical properties [1,2]. Extensive investigations [1,2] show that considerable refinement of microstructure can be achieved by imposing very high plastic deformations. Several techniques based on introduction of severe plastic deformation, e.g. torsion under high pressure, equal channel angular extrusion and multiaxial compression (MAC), are now available for attaining of fine grain structures in many metals and alloys. MAC in these various methods seems to be especially attractive, because it is the easiest method without any special device and has great potentiality for scaling up of relatively large samples.

The principle of MAC assumes multiple free forging operations; repeating compression process changing the axis of the applied strain $x \rightarrow y \rightarrow z \rightarrow x \rightarrow \dots$ at each step [2-4]. Redundant plastic strains can be accumulated into the material as it is repeatedly deformed at ambient to moderate temperatures [1]. Since a work piece changes hardly its shape under MAC, many repetitive passes can be undertaken to achieve very high total strains [1-4]. It has been shown recently [3,4] that such strain accumulation applied from various directions becomes very important for evolution of fine grains and so the grain refinement. Grain refinement may be controlled not only by total strains accumulated, but also by a strain per each pass of MAC. No investigation has, however, been performed to examine the effect of step strain on grain refinement under MAC, as far as the authors know.

The aim of the present work was to study any influence of the value of pass strain on grain refinement during MAC in high temperature. The 7475 Al alloy was used as one of the commercial-base materials showing continuous dynamic recrystallization (cDRX) behavior under hot compression [5]. The grain refinement mechanisms operating under hot MAC are discussed in some details.

Experimental

The present authors have studied microstructure evolution taking place during uniaxial hot compression of an as-cast and coarse-grained 7475 Al alloy [5,6]. The present experiments were

conducted by using the same 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 initial microstructure was composed of dendrite lamellas lying parallel to the ingot axis [5]. The boundaries of lamellar grains were rather straight and/or corrugated and the average size of the lamellas was in the range from 1 to 10 μm in longitudinal direction and from 50 to 200 μm in transverse one. Rectangular samples with a starting dimension of 10 x 10 x 13 mm were compressed with changing the loading direction in 90° with pass-to-pass. The samples were compressed in vacuum at T=763K under strain rate of $3 \times 10^{-4} \text{s}^{-1}$ with a powder of boron nitride as a lubricant. The tester enabling true strain rates constant was equipped with a water quenching apparatus. First compression pass was performed along the ingot axis. Pass strain ($\Delta\epsilon$) in each compression was 0.4 and 0.7, and the applied total strains ($\Sigma\Delta\epsilon$) were around 3.5. The metallographic analysis was carried out on a section parallel to the last compression axis of deformed samples using an Olympus PME3 optical microscope.

Results

Mechanical properties. Fig.1 represents typical true stress - true strain (σ - ϵ) curves obtained under MAC with $\Delta\epsilon=0.4$ and $\Delta\epsilon=0.7$ at 763K and at $\dot{\epsilon}=3 \times 10^{-4} \text{s}^{-1}$. These curves are integrated flow curves obtained by each multiaxial compression. It is seen in Fig.1 that both the σ - ϵ curves exhibit a sharp stress peak just after yielding followed by a significant work softening. The flow curve under MAC of $\Delta\epsilon=0.7$ shows larger flow softening and lower flow stresses in high strain than that for $\Delta\epsilon=0.4$. Namely, increasing of $\Delta\epsilon$ results in more clear reduction of flow stress in high strain.

It should be also noted in enlarged picture, Fig. 1(b), that difference between the yield stress at reloading and the flow stress immediately before unloading in each pass was found rather large for MAC of $\Delta\epsilon=0.4$, but negligible small for MAC of $\Delta\epsilon=0.7$. This suggests that any restoration processes during interrupted deformation may hardly take place in the test with $\Delta\epsilon=0.7$ and, in contrast, readily in the test with $\Delta\epsilon=0.4$ [3].

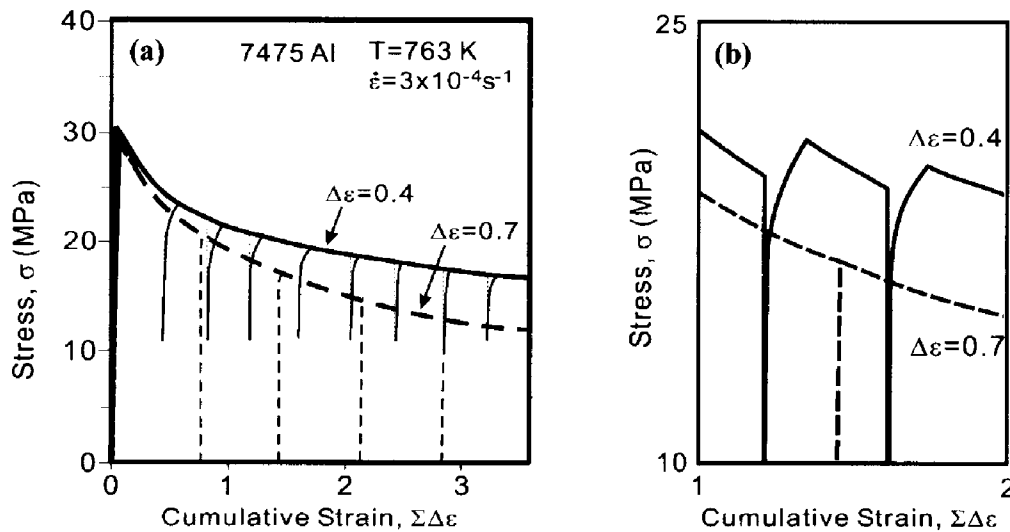


Fig. 1. Effect of pass strain ($\Delta\epsilon$) on true stress - true strain curves under multiaxial compression of 7475 Al alloy at 763K and $\dot{\epsilon}=3 \times 10^{-4} \text{s}^{-1}$.

Microstructural development. Fig. 2 shows typical optical micrographs of the 7475Al alloy multiaxially deformed up to $\Sigma\Delta\varepsilon \approx 3.5$ with $\Delta\varepsilon=0.4$ and 0.7 at 763K and at $3 \times 10^{-4} \text{ s}^{-1}$. It is clearly seen that grain refinement takes place frequently during high-temperature MAC and the fine-grained structures are evolved very non-uniformly through the whole area. Under MAC of $\Delta\varepsilon=0.4$, the fraction of new grains is about 50% and there are many original coarse grains remained. In contrast, under MAC of $\Delta\varepsilon=0.7$, the volume fraction of fine grains is significantly large and 80-85%. In the central regions of Fig. 2(b), new fine grains are formed almost fully. This suggests that larger pass strain under MAC can promote the occurrence of grain refinement, resulting in lower flow stresses in high strain (see Fig.1). Let us consider the effect of pass strain on microstructural characteristics in more details.

Fig. 3 represents strain dependence of the volume fraction of new grains, V_{rex} , developed by MAC with $\Delta\varepsilon=0.4$ and 0.7 . It is remarkable to note in Fig. 3 that new grains are formed more rapidly by increase in pass strain under MAC. On the other hand, no difference was observed between the size of new fine grains developed under MAC of $\Delta\varepsilon=0.4$ and 0.7 . The average grain size was around $7.5 \mu\text{m}$ at $\Sigma\Delta\varepsilon=3.5$. This suggests that changes in $\Delta\varepsilon$ play a relatively minor role in the average grain size developed under MAC.



Fig. 2. Microstructures developed in 7475 Al under MAC at 763K under a strain rate of $3 \times 10^{-4} \text{ s}^{-1}$. Specimens were deformed to a cumulative strain of about 3.5 with (a) $\Delta\varepsilon=0.4$ and (b) $\Delta\varepsilon=0.7$. Regions with dark color are composed of new fine grains with an average size of $7.5 \mu\text{m}$. The last compression axis is vertical.

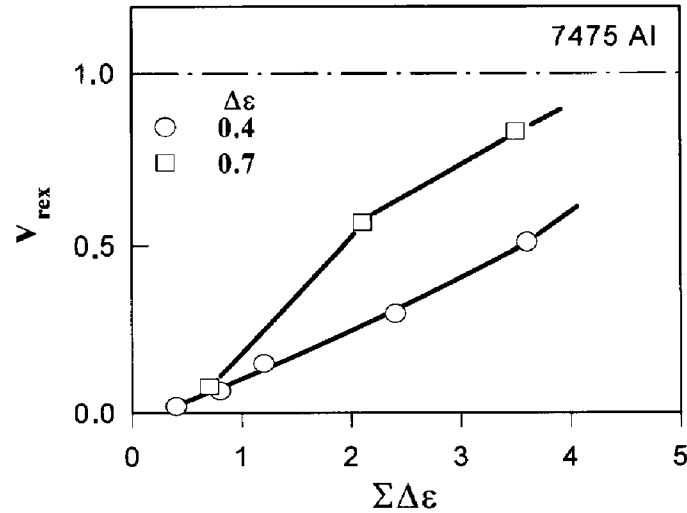


Fig.3. Strain dependence of the volume fraction of new fine grains developed in 7475 Al alloy during MAC at $T=763\text{K}$ under a strain rate of $3 \times 10^{-4} \text{s}^{-1}$.

Discussion

Flow softening. The present results above mentioned show that multiaxial compression (MAC) can lead effectively to grain refinement accompanied with lower flow stresses in high strain. The mechanisms of flow softening taking place during single-pass hot deformation was studied in the same Al alloy in details elsewhere [5]. It is shown in [5] that grain boundary sliding (GBS) takes place in the initial coarse-grained dendritic structure even in early deformation, and frequently in fine-grained regions developed in large deformation. It is discussed in [5] that work softening can be resulted from formation of fine grains followed by localization of plastic flow in fine-grained regions. The same discussion can be applied to the present results, i.e. the operation of GBS and significant increase in the volume fraction of new grains developed under hot MAC are the main factors providing rapid decrease in flow stresses in high strain (Fig.1). It can be clearly seen that σ - $\Sigma \Delta \epsilon$ relationships under MAC are qualitatively in agreement with the results of V_{rex} (Figs. 2 and 3), i.e. increasing of V_{rex} can bring about decrease in flow stress. This will be discussed latter again.

Grain refinement processes. Grain refinement taking place under conventional hot compression of the present alloy can result from a series of strain-induced continuous reactions; that is continuous dynamic recrystallization (cDRX) [5,6]. Fig. 4 shows typical microstructures evolved after first pass compression of the Al alloy, i.e. at (a) $\epsilon=0.4$ and (b) $\epsilon=0.7$. Such structural changes can be explained as the following. The uniaxial deformation at the earlier stages brings about formation of geometrically necessary dislocation boundaries with low- to medium angle misorientations, which are recognized sometimes as deformation bands [5,6,7]. These bands are visible in polished and etched surface of specimen by using polarized optical microscopy [6], as can be seen in Fig.4. The misorientation and the number of such deformation bands increase with increase in strain, leading to their gradual transformation into conventional high angle boundaries and finally local development of a fine grain structure in high strain.

The same structural mechanism can be operative during MAC. During MAC with a pass strain of 0.7, high density deformation bands with moderate to high angle misorientations can be developed more clearly accompanied with evolution of fine grains along the direction of maximum

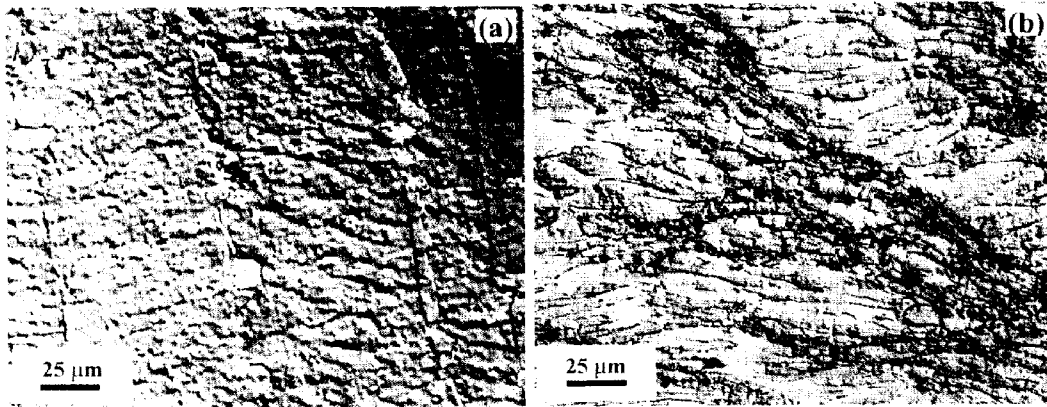


Fig. 4. Microstructures evolved in 7475 Al alloy under single-pass deformation at $T=763\text{K}$ under a strain rate of $3 \times 10^{-4} \text{s}^{-1}$: (a) $\epsilon=0.4$; (b) $\epsilon=0.7$. Polarized light. Compression axis is vertical.

of shear stress (Fig. 4(b)). Under repetitive deformation with changing of the compression axis, such deformation bands can be developed in various directions and so intersected with each other, finally followed by rather rapid evolution of fine grains in high strain. The present data also show that the kinetics of grain refinement is dependent on the pass strain, $\Delta\epsilon$. Several factors controlling development of new grains under MAC with increasing $\Delta\epsilon$ will be discussed here.

(i). *GBS*. During hot MAC, GBS can frequently take place especially in the regions fragmented by deformation bands [6]. Grain rotation accompanied by inhomogeneous GBS in fragmented regions may accelerate transformation of low- to moderate angle boundaries of deformation bands to high angle ones [6,8]. In contrast, these can hardly operate along lower angle boundaries and result in slow transformation of low-angle boundaries into high-angle ones, as shown in Fig. 4(a). This is one of the reasons why a smaller volume fraction of fine grains are developed under MAC of $\Delta\epsilon=0.4$ (Figs. 2 and 3).

(ii). *Static restoration taking place between passes*. It is known [9] that an assembly of higher misoriented (sub)boundaries can be very stable, whereas lower misoriented substructure is intrinsically less stable, and so may be rapidly recovered during the period of passes. As it has been above mentioned, each interrupted flow curve with $\Delta\epsilon = 0.4$ indicates that rather high softening resulted from static recovery occurring at each deformation pass [3]. Such a static annealing effect, in contrast, appears quite small under interrupted test with $\Delta\epsilon = 0.7$ (Fig.1). This suggests that dislocation substructures evolved by previous deformation are more recovered under MAC with smaller $\Delta\epsilon$ and then fine grains with high-angle boundaries can be developed slowly even in high cumulative strain.

It is concluded from the present results and discussion that a value of $\Delta\epsilon$ can significantly influence grain refinement during MAC. Quite a few data on the effect of $\Delta\epsilon$ under MAC have been reported in previous literatures and additional experiments will be required to discuss this effect. It is significant to recognize here that formation of a fine-grained structure under MAC can be dependent not only on a total accumulated strain, but also each pass strain.

Summary

The influence of the value of pass strain ($\Delta\epsilon$) in multiaxial compression (MAC) was studied in a coarse-grained and as-cast 7475Al alloy at a temperature of 763K under a strain rate of $3 \times 10^{-4} \text{s}^{-1}$.

The main results are summarized as follows.

1. The cumulative flow curves integrated by flow curves for each compression exhibit a sharp peak stress just after yielding followed by significant work softening up to high cumulative strains of about 3. Increasing of $\Delta\varepsilon$ leads to more clear reduction of flow stress in high strain.
2. MAC can result in a substantial grain refinement in as-cast 7475 Al alloy. Under MAC, deformation bands can be developed in various directions and so intersected with each other, followed by increasing in their number and misorientation and, finally, establishing arrays of fine equiaxed grains in high strain. This mechanism of grain refinement is similar to continuous dynamic recrystallization.
3. Increasing of pass strain under MAC can accelerate the occurrence of grain refinement. Under MAC with higher pass strain, high-density deformation bands with moderate to high angle misorientations can be developed more clearly. They are more stable under reheating between passes and can more readily support grain boundary sliding (GBS). Accordingly, they can more rapidly transform into high angle boundaries during deformation accompanied with evolution of new fine grains. As a result, larger fraction of new grains is developed under MAC with higher $\Delta\varepsilon$.

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References

- [1] F.J. Humphreys, P.B. Prangnell, J.R. Bowen, A. Gholinia, C. Harris: *Phil. Trans. R. Soc. London* Vol. A357 (1999), p. 1663
- [2] R.Z. Valiev, R.K. Islamgaliev, I.V. Alexandrov: *Progr. Mater. Sci.* Vol. 45 (2000), p. 103
- [3] A. Belyakov, T. Sakai, H. Miura, R. Kaibyshev, *ISIJ International* Vol. 39 (1999), p. 592
- [4] R.M. Imaev, G.A. Salishchev, O.N. Senkov, V.M. Imaev, M.R. Shagiev, N.K. Gabdullin, A.V. Kuznetsov, F.H. Froes: *Mater Sci Eng.* Vol. A300 (2001), p. 263
- [5] R. Kaibyshev, O. Sitdikov, A. Goloborodko, T. Sakai: *Mater Sci Eng.* Vol. 344 (2003), p. 348
- [6] O. Sitdikov, A. Goloborodko, R. Kaibyshev, T. Sakai: *Proc. of TMS* (2003) (in press)
- [7] B. Bay, N. Hansen, D.A. Hughes, D. Kuhlmann-Wilsdorf: *Acta Met. Mater.* Vol. 40 (1992), p. 205.
- [8] X. Yang, H. Miura, T. Sakai: *Mater. Trans.*, Vol. 43 (2002), p. 2400
- [9] F.J. Humphreys: *Acta Mater.* Vol.45 (1997), p. 4231