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Effect of grain boundaries on dynamic recrystallization behaviour of copper isoaxial bicrystals with ⟨001⟩ tilt boundaries

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Dynamic recrystallization behaviour of copper isoaxial bicrystals with various ⟨001⟩ tilt boundaries was investigated at 1023 and 1073 K at an initial strain rate of $3.33 \times 10^{-4} sec^{-1}$, with special attention paid to the effect of grain boundaries on the recrystallization stress (strain) and the substructure developed during deformation. The grain boundaries in the bicrystals have almost no effect on the stress–strain curves. Flow stress was found to fall abruptly and significantly during deformation, being similar to the flow stress in the single crystals. The stress σ_R (ε_R), just before the stress fall indicates the stress (strain) at which dynamic recrystallization occurs. The growth of recrystallized grains is markedly fast in these bicrystals, suggesting that dynamic recrystallization is controlled by a nucleation process. The value of σ_R in bicrystals with a tilt boundary of 9°, θ9 bicrystals, is almost equal to that in single crystals, but is larger than σ_R in θ23, θ36 and θ43 bicrystals. These results indicate that the θ9 boundary has no effect on the dynamic recrystallization of bicrystals, whereas grain boundaries with tilt angles above 23° accelerate the dynamic recrystallization of these bicrystals. The θ-dependence of σ_R or ε_R is discussed in connection with the stress concentration near the grain boundary.

1. Introduction

Dynamic recrystallization is important from a practical viewpoint, such as in the controlled rolling of metals, and especially of steels. There have, for that reason, been many investigations concerning dynamic recrystallization in polycrystals of pure metals and alloys [1–6]. However, the dynamic recrystallization mechanism has not yet been made clear. This may result from such experimental difficulties as the exact determination of the recrystallization stress (σ_R) and strain (ε_R) at which dynamic recrystallization occurs; the distinguishing of recrystallized grains from the deformed matrix; and the precise determination of the nucleation site for recrystallization.

Recently, the dynamic recrystallization of single crystals has been studied [7–9] with a view to avoiding such experimental difficulties as mentioned above. In single crystals, the flow stress drops markedly just as dynamic recrystallization occurs, enabling σ_R and ε_R to be exactly and easily determined. Furthermore, the newly recrystallized grains in single crystals can be readily distinguished from the deformed matrix even using an optical microscope. Thus, a lot of important information on dynamic recrystallization has been obtained, including the orientation relationship between the recrystallized grains and the matrix metal as well as the stress- and temperature-dependences of the deformation rate in dynamic recrystallization [7–9].

The dynamic recrystallization behaviour of single crystals is therefore significantly different from that of polycrystals: σ_R in single crystals is much larger than the recrystallization stress in polycrystals, meaning that dynamic recrystallization occurs more easily in polycrystals than in single crystals. Grain boundaries, thus, play an important role in the dynamic recrystallization: they are regions at which the recrystallized grains preferentially nucleate and grow. However, there have as yet been no detailed studies of the grain-boundary effect on the recrystallization.

In the present work, we used isoaxial bicrystals of copper with ⟨001⟩ orientation. To use the bicrystals is quite helpful in discussing dynamic recrystallization for the following reasons: in each component single crystal with ⟨001⟩ orientation, four equivalent slip systems are active from the beginning of deformation [10–13], so that the deformation bands, at which recrystallized grains preferentially begin to form, would be almost totally undeveloped. Dynamic recrystallization of the bicrystals is, therefore, expected to occur at the grain boundary. Furthermore, the specimen axis does not rotate during deformation in ⟨001⟩ oriented single crystals [12, 13] so that the crystallographic relationship between the component single crystals in each bicrystal is expected to be maintained during deformation.

The present study aims, therefore, to clarify the grain-boundary effect on the dynamic recrystallization behaviour, such as that of σ_R (ε_R), with particular attention paid to the influence of the tilt angle.

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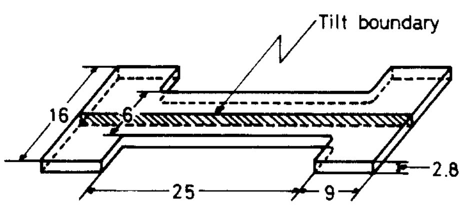

Figure 1 Schematic representation of bicrystal specimens used (dimensions in mm).

For this purpose, ⟨0 0 1⟩ oriented bicrystals of copper with various tilt boundaries were chosen. The recrystallization behaviour of these bicrystals was compared with that of single crystals with ⟨0 0 1⟩ orientation as well as with that of polycrystals.

2. Experimental procedure

⟨0 0 1⟩ oriented single crystals were made from 99.99% copper in a vacuum by the modified Bridgman method using a split graphite mould. Mother bicrystals were grown from these seed crystals in a similar manner. The growth rate of the bicrystals was $5.6 \times 10^{-6}\,\mathrm{m\,sec^{-1}}$. Bicrystal specimens, the geometry of which is shown in Fig. 1, were cut for tensile tests from the mother bicrystals. After being electrolytically polished to a thickness of about 2.0 mm, the specimens were annealed for 10.8 ksec at 1100 K. The two component crystals, of which each bicrystal specimen is composed, tilt toward each other at an angle, θ, around a common specimen axis of ⟨0 0 1⟩ orientation, as shown in Fig. 2. The θ values of the bicrystals used were 9, 23, 36 and 43°, or 0.157, 0.401, 0.628 and 0.750 radians, respectively. These crystals were lettered according to the θ value in such a way that a bicrystal with a tilt angle of 9° was named a θ9 bicrystal. Bicrystals with the θ value of 45° (0.785 radians) were used in addition to the above bicrystals. The specimen axis orientation and θ value of each bicrystal were checked using the X-ray reflection method. The accuracy was $\pm 1°$ (0.018 radians).

Tensile tests were performed using an Instron type tensile machine (Shimazu IM-100) with rapid cooling equipment. Rapid cooling was performed by N_2 gas jet in order to maintain the microstructure which had developed during deformation at elevated temperatures. Specimens were tested at an initial strain rate of $3.33 \times 10^{-4}\,\mathrm{sec^{-1}}$ at 1023 and 1073 K in a vacuum of about 10^{-2} Pa. Test temperatures were controlled within ± 2 K during deformation.

The bicrystal specimens, unloaded and rapidly cooled just after the flow stress reached σ_R, were cut

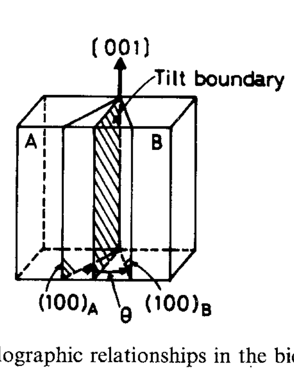

Figure 2 Crystallographic relationships in the bicrystals used.

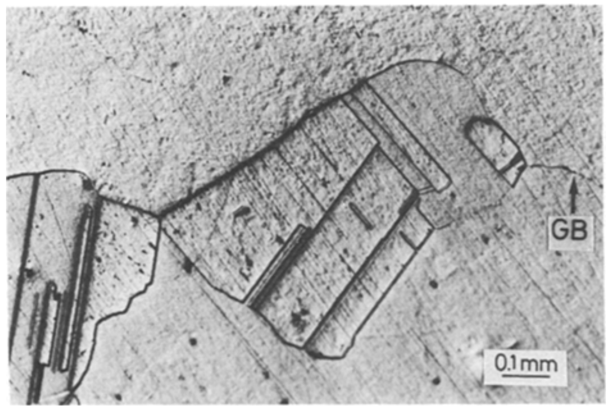

Figure 3 Typical microstructure on a {1 1 1} plane of an isoaxial bicrystal with a ⟨0 0 1⟩ tilt boundary of 23° deformed at 1023 K, indicating recrystallized grains formed on the tilt boundary. GB represents the ⟨0 0 1⟩ tilt boundary.

parallel to a {1 1 1} or {1 1 0} plane using a spark cutter. After the cross-sections were electrolytically polished and etched using the Livingston solution (CH_3COOH, 30 ml; HCl, 45 ml; Br_2, 1 ml; and H_2O, 250 ml), the microstructure was examined using an optical microscope.

3. Results and discussion

Fig. 3 shows the typical microstructure of a θ23 bicrystal deformed at 1023 K, unloaded and rapidly cooled just after measuring σ_R, which was observed on a {1 1 1} plane. It was found that the recrystallized grains containing twins form on the already present tilt boundary (GB in the figure), indicating that the boundary is the preferred nucleation site for dynamic recrystallization.

Fig. 4 shows a typical stress–strain curve of a θ23 bicrystal deformed at 1023 K at an initial strain rate of $3.33 \times 10^{-4}\,\mathrm{sec^{-1}}$. One can easily see a drastic stress drop at a strain of 0.37, which is where dynamic recrystallization occurs. Dynamically recrystallized grains were identified using an optical microscope, as shown in Fig. 3. The flow stress oscillates after the marked stress fall. These characteristics in the stress–strain curves have also been shown in single crystals of copper, silver and nickel [7–9]. Thus, the generation of dynamic recrystallization in bicrystals can be clearly detected in terms of the change in flow stress on

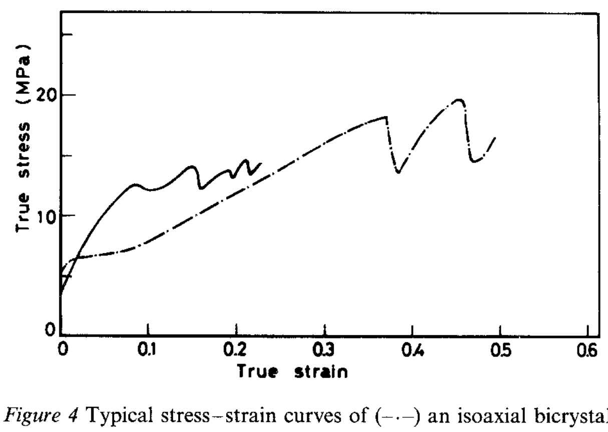

Figure 4 Typical stress–strain curves of (–·–) an isoaxial bicrystal with a ⟨0 0 1⟩ tilt boundary of 23°, and (——) that of a polycrystal, deformed at 1023 K.

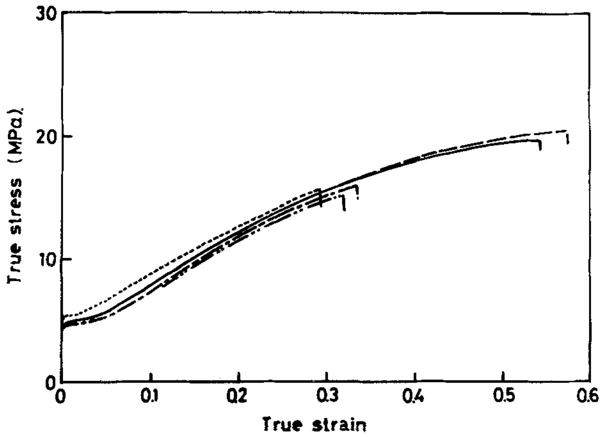

Figure 5 Stress–strain curves of isoaxial bicrystals having various ⟨0 0 1⟩ tilt boundaries and an ⟨0 0 1⟩ oriented single crystal, deformed at 1073 K: (—–) θ9, (-·-) θ23, (-··-) θ36, (---) θ43, (——) single crystal.

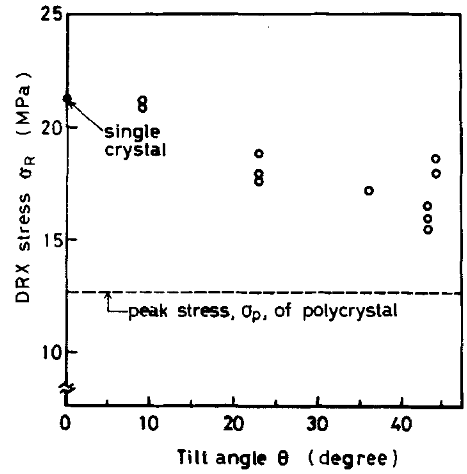

Figure 6 Dynamic recrystallization (DRX) stress as a function of tilt angle for isoaxial bicrystals with ⟨0 0 1⟩ tilt boundaries deformed at 1023 K. The horizontal line indicates the peak stress in a polycrystal deformed at 1023 K.

the stress–strain curve, giving reliable values for σ_R and ε_R.

For comparison, the stress–strain curve of a polycrystal with an average grain diameter of 0.62 mm, deformed at 1023 K at an initial strain rate of $2.08 \times 10^{-4}\,\text{sec}^{-1}$, is also given in Fig. 4. The flow stress of the polycrystal oscillates after the peak stress, σ_p, at the peak strain, ε_p. Many investigators [3, 4, 6] have reported that the dynamic recrystallization of polycrystals begins at a strain of about $0.7\varepsilon_p$. Therefore, there is no direct correlation between ε_p (σ_p) and the generation of dynamic recrystallization, but they are, nevertheless, important parameters in dynamic recrystallization. σ_p (ε_p) was found to be smaller than σ_R (ε_R), indicating that dynamic recrystallization occurs more easily in polycrystals than in bicrystals.

Fig. 5 shows the influence of the tilt angle, θ, of the grain boundary on the stress–strain curve of bicrystals deformed at 1073 K at an initial strain rate of $3.33 \times 10^{-4}\,\text{sec}^{-1}$. Because these specimens were unloaded and rapidly cooled just after the flow stress reached σ_R to study the substructure, their stress–strain curves after the stress drop are not shown in the figure. For comparison, the stress–strain curve of a ⟨0 0 1⟩ oriented single crystal is also given in the figure. Some important results are obtained from Fig. 5: the flow stresses in all bicrystals are equal to each other at strains up to about 0.3; there is no difference in flow stress between single crystals and bicrystals; the stress–strain curve over all strains and σ_R (ε_R) of the θ9 bicrystal is consistent with those of the single crystal; σ_R (ε_R) in θ23, θ36 and θ43 bicrystals is much smaller than in the θ9 bicrystal and the single crystal; values of σ_R in the θ23, θ36 and θ43 bicrystals are almost the same. The flow stress in isoaxial bicrystals with a single slip orientation was found to be higher than that in single crystals [14, 15]. The difference in these stresses originates mainly from an elastic- or plastic-strain discrepancy in two component crystals due to a discontinuity in the amount of slip in the primary slip system at the grain boundary. On the other hand, however, the flow stress in isoaxial bicrystals with a multiple slip orientation, such as ⟨1 1 1⟩ or ⟨0 0 1⟩ orientation, is similar to that in single crystals [14–16]. This is consistent with the results with the present bicrystals. Thus, the grain boundary has almost no effect on the flow stress in isoaxial bicrystals with a multiple slip orientation. The consistency of σ_R in the θ9 bicrystal and the single crystal indicates that the grain boundary has no influence on the recrystallization. This may be because there are few locally deformed regions where recrystallization is generated in preference to other regions.

Fig. 6 shows the effect of the tilt angle of the grain boundaries on σ_R for bicrystals deformed at 1023 K. One can easily see that σ_R in the θ9 bicrystal is close to that in the single crystal, as mentioned above. Furthermore, σ_R in the θ23, θ36 and θ43 bicrystals is smaller by approximately 5 to 7 MPa than in the θ9 bicrystal and the single crystal, but there is little difference in the values of σ_R in the θ23, θ36 and θ43 bicrystals. A similar effect of tilt angle on ε_R was found in the present bicrystals, as shown in Fig. 7. Such an analogy in the θ-dependences of σ_R and ε_R is attributed to the correlation between σ_R and ε_R, as shown in Figs 4 and 5. The horizontal lines in Figs 6 and 7 represent the peak stress, σ_p, and the peak strain, ε_p, in the polycrystal. σ_p (ε_p) is smaller than σ_R (ε_R) in the bicrystals. This may result from the absence of triple points of grain boundaries and other types of boundary, which are

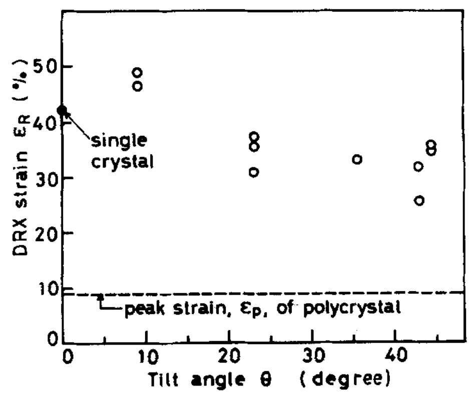

Figure 7 Dynamic recrystallization (DRX) strain as a function of tilt angle for isoaxial bicrystals with ⟨0 0 1⟩ tilt boundaries deformed at 1023 K. The horizontal line indicates the peak strain in a polycrystal deformed at 1023 K.

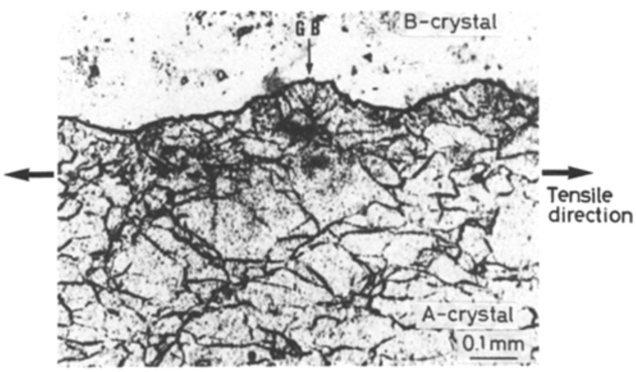

Figure 8 A typical microstructure near the grain boundary on a {0 0 1} plane parallel to the specimen axis in a bicrystal with ⟨0 0 1⟩ tilt boundary of 43°, deformed at 1023 K. GB indicates the ⟨0 0 1⟩ tilt boundary.

preferred nucleation sites in polycrystals, in the present bicrystals. It is emphasized that the θ-dependence of σ_R (ε_R) has not previously been reported, though this dependence is very important and interesting from the point of view of dynamic recrystallization.

Grain growth in copper bicrystals was found to be as rapid as that in single crystals, in which newly recrystallized grains occupy almost the whole cross-section as soon as one or more recrystallized nuclei are generated at σ_R. This fact suggests that σ_R relates to the nucleation, not to the growth of a new grain. Thus, the dynamic recrystallization of the present bicrystals is controlled by a nucleation process. The θ-dependence of σ_R (ε_R) in Fig. 5 (Fig. 6) therefore indicates the ease with which dynamic recrystallization occurs.

Fig. 8 shows a typical microstructure on a {0 0 1} plane parallel to the specimen axis for a θ43 bicrystal deformed at 1023 K and cooled just after the onset of dynamic recrystallization. This microstructure was formed in a region other than that of recrystallization. In the A-crystal, the observed cross-section is a {0 0 1} plane parallel to the specimen axis, while the B-crystal shows a {1 1 0} plane parallel to the specimen axis. We can observe sub-boundaries in the A-crystal, but not in the B-crystal, as shown in Fig. 8. The existing grain boundary, which was straight before deformation, is now wavy. This indicates that the tilt boundaries migrate before the onset of dynamic recrystallization during deformation at elevated temperatures. No marked deformation bands were found to form, this resulting from the activation of multiple slip in ⟨0 0 1⟩ oriented crystals.

As can be seen in Fig. 8, the subgrains are smaller in the vicinity of less than about 150 μm from the grain boundary (GB) than in the regions further away. This may be ascribed to the presence of the tilt boundary. The influence of the grain boundary on the substructure formed during deformation extends over the region near the boundary, but not over regions further away. It is well known that the subgrain size depends upon stress [17]. Therefore, the difference in subgrain size indicates that the stress is higher in the vicinity of the grain boundary than in the region further away, meaning that there is a stress concentration in the region near the grain boundary in the present bicrystals. Similar subgrain sizes near the boundary were observed in the θ23 and θ36 bicrystals. On the other hand, the subgrain size is almost unchanged even in the vicinity of the grain boundary in the θ9 bicrystals. Thus, the grain boundary gives no stress concentration near the boundary in the θ9 bicrystals, yielding values of σ_R (ε_R) close to σ_R (ε_R) in single crystals. In the θ23, θ36 and θ43 bicrystals, the stress concentration near grain boundaries causes the generation of dynamic recrystallization at stresses and strains lower than in the θ9 bicrystals.

4. Conclusions

To clarify the dynamic recrystallization mechanism, the effect of grain boundaries on the dynamic recrystallization behaviour was studied with copper isoaxial bicrystals with various ⟨0 0 1⟩ tilt boundaries. The crystals were tested in tension at 1023 and 1073 K at an initial strain rate of $3.33 \times 10^{-4}\,\text{sec}^{-1}$. The stress–strain curves of these crystals are similar to those of ⟨0 0 1⟩ oriented single crystals, indicating that the presence of such grain boundaries has almost no effect on the flow stress. In all bicrystals used in this investigation, an abrupt drop in flow stress was observed, this being analogous to that in single crystals. Thus, the recrystallization stress, σ_R, and the strain, ε_R, can be exactly and easily evaluated from the stress–strain curve in the present bicrystals. The value of σ_R (ε_R) in bicrystals with a tilt angle of 9°, the θ9 bicrystals, is consistent with that in the single crystal. In contrast, dynamic recrystallization in θ23, θ36 and θ43 bicrystals occurs at stresses much smaller than in the θ9 bicrystal or the single crystal, indicating that the tilt boundaries in these bicrystals make dynamic recrystallization easier. However, the value of σ_R in the bicrystals is larger than that of the peak stress, σ_P, in the polycrystal. The grain boundary was found to become wavy during deformation due to grain boundary migration. In the θ23, θ36 and θ43 bicrystals, subgrains are smaller in size in the vicinity of the grain boundary than further away, though this change was not detected in the θ9 bicrystals. These results show the presence of stress concentration in the region near the boundary in the θ23, θ36 and θ43 bicrystals, but its absence in the θ9 bicrystals. The θ-dependence of σ_R or ε_R can therefore be explained in terms of the stress concentration near the grain boundary.

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References

1. M. J. LUTON and C. M. SELLAR, *Acta Metall.* **17** (1969) 1033.
2. R. A. PETKOVIC, M. J. LUTON and J. J. JONAS, *ibid.* **27** (1979) 1633.
3. T. MAKI, S. OKAGUCHI and I. TAMURA, in Proceedings of the 7th International Conference on the Strength of Metals and Alloys, Vol. 2, edited by H. J. McQueen, J. -P. Bailon, J. I. Dickson, J. J. Jonas and M. G. Akben, (Pergamon Press, Oxford, 1985) p. 923.
4. T. KISHI, K. SUZUKI and T. ENDO, *Jpn J. Inst. Metals* **46** (1982) 842.
5. E. FURUBAYASHI and M. NAKAMURA, *Tetsu-to-Hagane* **68** (1982) 543.
6. T. SAKAI and J. J. JONAS, *Acta Metall.* **32** (1984) 189.
7. P. J. T. STUIJIE and G. GOTTSTEIN, *Z. Metallkde* **279** (1980) 279.
8. G. GOTTSTEIN and U. K. KOCKS, *Acta Metall.* **31** (1983) 175.
9. P. KARDUCK, G. GOTTSTEIN and H. MECKING, *ibid.* **31** (1983) 1525.
10. U. F. KOCKS, *ibid.* **6** (1958) 85.
11. T. TAKEUCHI, *J. Phys. Soc. Jpn* **40** (1976) 741.
12. S. KIKUCHI, K. TOMITA, Y. MOTOYAMA and M. ADACHI, *J. Jpn Inst. Light Metals* **30** (1980) 480.
13. J. TAKADA, S. MIYAWAKI, K. KAMATA and M. ADACHI, *Trans. Jpn Inst. Metals* **25** (1984) 784.
14. R. S. DAVIS, R. L. FLEISHER, J. D. LIVINGSTON and B. CHALMERS, *Trans. AIME* **209** (1957) 136.
15. S. MIURA, K. HAMASHIMA and K. T. AUST, *Acta Metall.* **28** (1980) 1591.
16. S. MIURA and Y. SAEKI, *ibid.* **26** (1978) 93.
17. S. KIKUCHI and M. YAMAGUCHI, in Proceedings of the 7th International Conference on the Strength of Metals and Alloys, Vol. 2, edited by H. J. McQueen, J. -P. Bailon, J. I. Dickson, J. J. Jonas and M. G. Akaben (Pergamon Press, Oxford, 1985) p. 899.

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