JOURNAL OF MATERIALS SCIENCE **38** (2003) 1895–1899

The effect of Ga on internal friction of pure Al before and after deformation

Y. SHI, B. CAI, Q. P. KONG*, P. CUI
Key Laboratory of Internal Friction and Defects in Solids, Institute of Solid State Physics, Chinese Academy of Science, Hefei 230031, People's Republic of China
E-mail: qpkong@mail.issp.ac.cn

G. GOTTSTEIN
Institut fuer Metallkunde und Metallphysik, RWTH Aachen, 52056 Aachen, Germany

The grain boundary internal friction of pure Al and the same Al doped with different amounts of Ga (10, 50 and 410 ppm) was measured before and after high temperature deformation at two different modes (static tensile creep and cyclic reverse torsion). It was found that without deformation, the smaller amount of Ga (10 and 50 ppm) slightly increases, but the larger amount of Ga (410 ppm) obviously increases the activation energy. After tensile creep, the peak height for Al-10 ppm Ga and Al-50 ppm Ga decreases to a lower level, but keeps at a higher level after cyclic torsion, than that for pure Al and Al-410 ppm Ga. The results are interpreted in terms of perturbations in the grain boundaries due to slip and grain boundary roughening for stationary boundaries and an enhanced sliding by grain boundary migration. Migration and roughening are promoted by Ga contents up to 50 ppm while the effects are diminished by higher amounts of Ga. This is consistent with previous observations that smaller amounts of Ga increase the grain boundary mobility in pure Al.

1. Introduction

The element Ga is known to have a conspicuous effect in Al. Larger amounts of Ga in Al-polycrystals lead to intergranular embrittlement. Recently, Gottstein, Molodov and Shvindlerman [1–3] found that smaller amounts of Ga (e.g., 10 and 50 ppm) can increase the mobility and thus accelerate the grain boundary migration in pure Al bicrystals with ⟨111⟩ tilt grain boundaries, while large amounts of Ga (≥410 ppm) reduce the mobility. HRTEM examinations showed that such effect is possibly related to the shift of atom position near grain boundary induced by the minor amount of Ga [4].

The internal friction peak associated with grain boundaries [5] is a sensitive tool to detect the change of grain boundaries induced by deformation and impurity. Kong and coworkers [6, 7] investigated the change of the internal friction peak during deformation in pure Al and Cu. It was observed that the internal friction peak varied differently under different test conditions. In pure Al, the internal friction peak decreased significantly during tensile creep, while the decrement was much smaller during cyclic torsion. Microstructure observations revealed that such phenomenon was associated with different evolution of grain boundary structure during different modes of deformation [6].

The present paper attempts to study the grain boundary behavior of pure Al doped with different amounts of Ga before and after deformation by using the grain boundary internal friction peak as a tool.

2. Experimental

The 99.999% pure Al and the same Al doped with different amounts of Ga (10, 50, and 410 ppm) were produced by Pechiney, France. The specimens were cut by spark erosion from cold-rolled sheets with thickness of 2.0 mm, and annealed at 450°C for 1 hour. The average grain sizes for the four kinds of specimens were in the range of 0.8–1.2 mm. The specimens were 55 mm in gauge length and 2.0×1.3 mm^2 in cross section.

Internal friction was measured during both cooling and heating in air with an automatic inverted torsion pendulum at three frequencies (0.3, 1.0 and 3.1 Hz) with temperature changing rate 4 K/min, or at five frequencies (0.03, 0.1, 0.3, 1.0 and 3.1 Hz) with 2 K/min. The strain amplitude for measuring internal friction was 1×10^{-5}. At first, the internal friction was measured prior to deformation, and then after each increment of deformation.

The deformation tests were conducted in situ at 450°C and two different modes as follows. (a) The static creep in tension was tested at 1.5 MPa. (b) The cyclic torsion was performed with a cyclic period of 30 s and reverse torsion angle ±40°, which is equivalent to a

---

[*] Author to whom all correspondence should be addressed.

minimum shear strain $\gamma = \pm 0.83\%$ (or a tensile strain $\pm 0.48\%$) on the surface per cycle.

3. Results

Fig. 1 shows the internal friction of pure Al doped with different amounts of Ga prior to deformation at 1.0 Hz. The peak height had a little scattering for each kind of specimens (because the grain sizes were very large, close to the linear dimension of cross section of specimens), but the peak temperature was highly reproducible. It can be seen from Fig. 1 that the shape and height of grain boundary internal friction peak does not vary with the amount of Ga, but the peak position shifts to higher temperature as the amount of Ga increases.

Assuming the internal friction peak is superimposed by a background Q_b^{-1} exponentially increasing with temperature, which can be expressed by [8]

$$Q_b^{-1} = A_1 + A_2 \exp(-A_3/kT), \qquad (1)$$

where k is Boltzmann constant, T is absolute temperature, A_1, A_2 and A_3 are constants.

After subtracting the background according to Equation 1 by using a fitting procedure, the resolved net internal friction peak and respective background for each curve in Fig. 1 are shown in Fig. 2.

The internal friction peak occurs at the following condition [5, 8] :

$$2\pi f \tau = 2\pi f \tau_0 \exp(H/kT) = 1, \qquad (2)$$

where f is the frequency, τ is the relaxation time, τ_0 is the pre-exponential factor, and H is the activation energy.

Based on the measurements at different frequencies (0.03, 0.1, 0.3, 1.0 and 3.1 Hz) as shown in Fig. 3, the activation energies H for pure Al doped with 0, 10, 50 and 410 ppm Ga were found to be 1.28, 1.32, 1.33 and 1.49 eV respectively, while the pre-exponential factor of relaxation time τ_0 remains in the same order of magnitude (10^{-14} s). The data indicate that the smaller amount of Ga (i.e., 10 and 50 ppm) slightly increases, but the larger amount of Ga (410 ppm) obviously increases the activation energy of grain boundary internal friction. Obviously, the addition of Ga in Al is indeed involved in the process of grain boundaries.

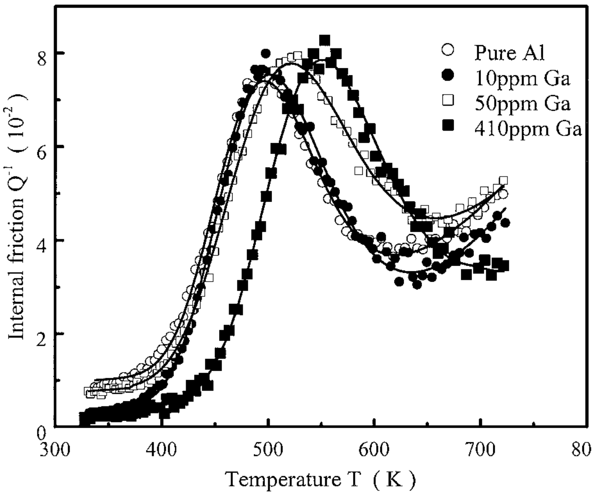

Figure 1 Internal friction Q^{-1} of pure Al doped with different amounts of Ga after annealing at 450°C for 1 h (1.0 Hz).

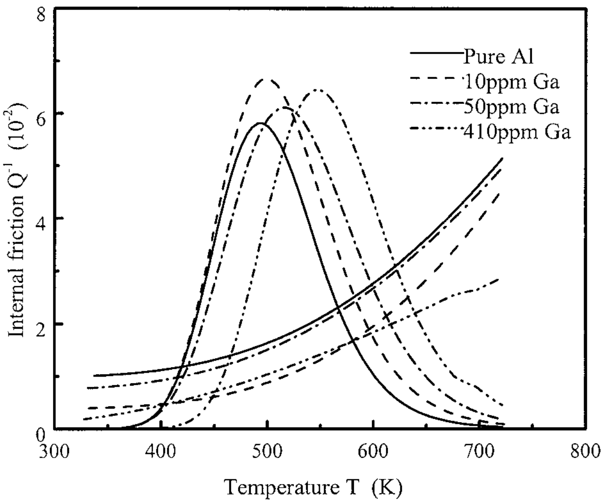

Figure 2 The resolved net peak and background for each internal friction curve in Fig. 1.

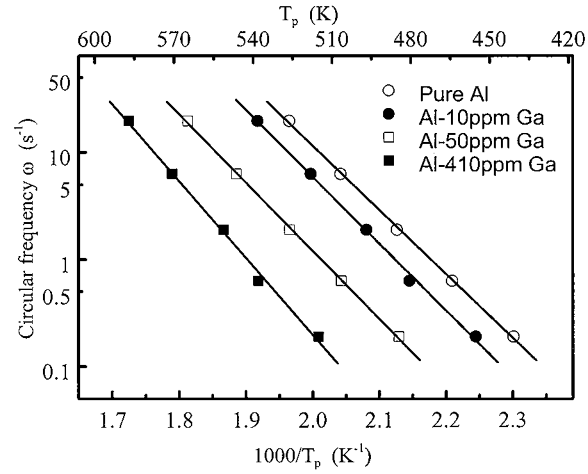

Figure 3 Semi-logarithmic plots of circular frequency ω ($2\pi f$) versus reciprocal peak temperature $1/T_p$ for Al doped with different amounts of Ga.

It is to be pointed out that the activation energy and pre-exponential factor obtained by internal friction in polycrystalline Al is a statistical result of anelastic sliding of all the grain boundaries with various types and misorientations [5], which may not be identical to those obtained by migration measurement in Al bicrystals having a single boundary with a specific type and specific misorientation.

Fig. 4 gives an example of the internal friction of Al-50 ppm Ga after different strains of static creep at 450°C and 1.5 MPa. Similar results were also obtained for Al with 0, 10, and 410 ppm Ga. It can be seen from Fig. 4 that the peak height decreases obviously as the creep strain increases.

Fig. 5 gives another example of internal friction of Al-50 ppm Ga after different cycles of reverse torsion at 450°C. Similar results were also obtained for Al doped with 0, 10, and 410 ppm Ga. The internal friction peak

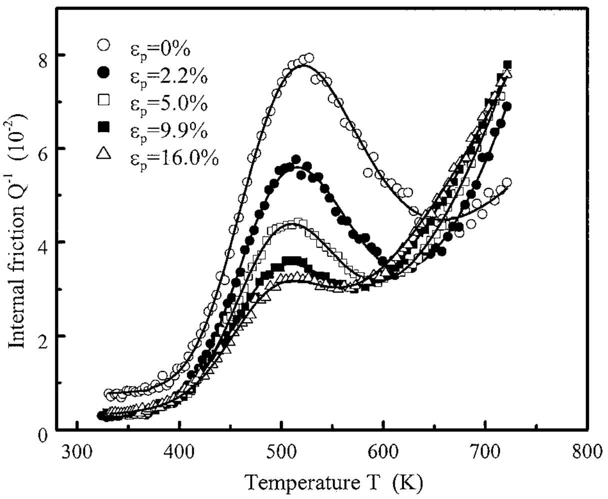

Figure 4 Internal friction of Al-50 ppm Ga after different strains ε_p of static creep at 450°C and 1.5 MPa (1.0 Hz).

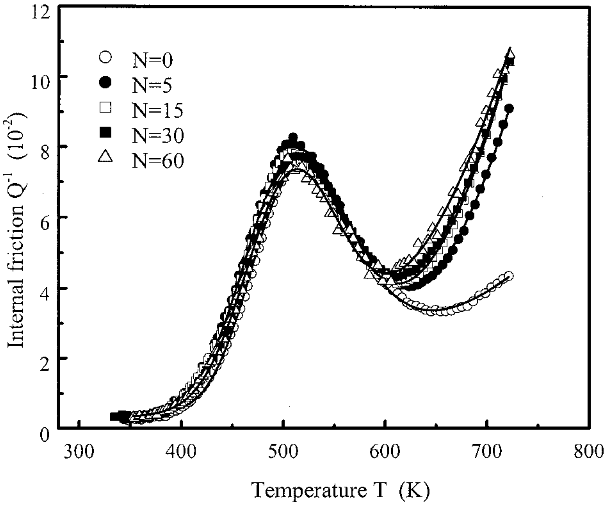

Figure 5 Internal friction of Al-50 ppm Ga after different cycles *N* with torsion angle ±40° at 450°C (1.0 Hz).

varies seemly not much with the increase of torsion cycle. After carefully subtracting the background, a small variation of the net internal friction peak can be detected (see Fig. 7).

It is interesting to note that the static creep leads to an obvious decrease of the peak, while the effect of cyclic torsion is not so obvious. It means that in static creep, extensive perturbation of grain boundaries is produced, suppressing the anelastic sliding of grain boundaries; while in cyclic torsion, the perturbation of grain boundaries is mostly recovered during reverse torsion, facilitating the anelastic sliding. The present work further confirms the observations of pure Al in our previous paper [6].

Fig. 6 shows the normalized peak height $Q_{\mathrm{md}}^{-1}/Q_{\mathrm{mb}}^{-1}$ (i.e., the peak height after deformation divided by that before deformation) versus static creep strain ε_p for pure Al doped with different amounts of Ga. It can be seen that the $Q_{\mathrm{md}}^{-1}/Q_{\mathrm{mb}}^{-1}$ decreases with increasing strain for all the four kinds of specimens. Nevertheless, at the same creep strain ε_p, the $Q_{\mathrm{md}}^{-1}/Q_{\mathrm{mb}}^{-1}$ for Al-10 ppm Ga and 50 ppm Ga decreases to a lower level than that for pure Al and Al-410 ppm Ga. It means that more perturbation of grain boundaries is produced during static creep in the specimens for Al-10 ppm Ga and 50 ppm Ga.

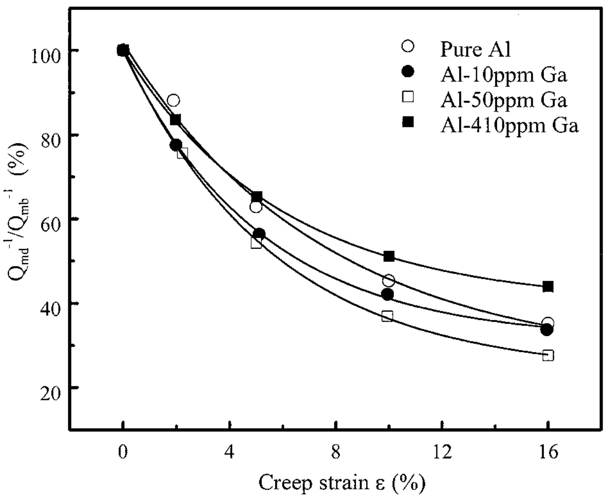

Figure 6 Normalized peak height $Q_{\mathrm{md}}^{-1}/Q_{\mathrm{mb}}^{-1}$ as a function of strain ε_p for static creep at 450°C and 1.5 MPa.

Fig. 7 shows the normalized peak height $Q_{\mathrm{md}}^{-1}/Q_{\mathrm{mb}}^{-1}$ as a function of torsion cycles *N* and the cumulative equivalent tensile strain ε_p for pure Al doped with different amounts of Ga. The equivalent tensile strain per cycle was calculated according to Blum and coworkers [9]:

$$\gamma = w\vartheta/L, \quad \varepsilon_p = \gamma/3^{1/2} \qquad (3)$$

where γ is the shear strain, w is the radial distance from the torsion axis, ϑ is the torsion angle, L is the gauge length of the specimen, and ε_p is the equivalent tensile strain. The $3^{1/2}$ is the conversion factor from torsion to tension. According to Equation 3, the minimum shear strain at the specimen surface is 0.83%, and the equivalent tensile strain is 0.48% per cycle. The cumulative equivalent tensile strain is 0.48% times the number of torsion cycles *N*.

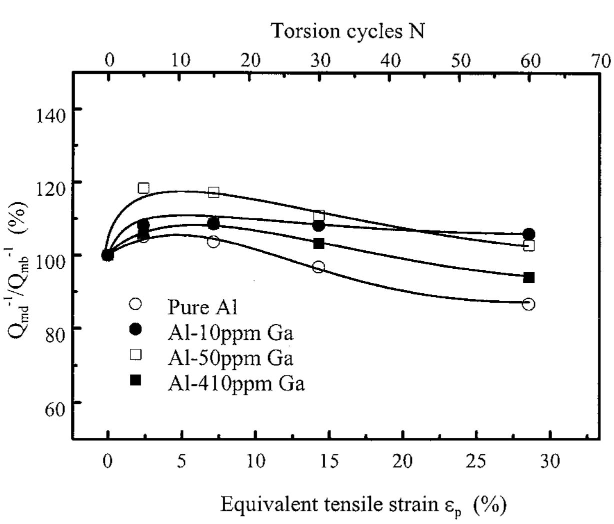

Figure 7 Normalized peak height $Q_{\mathrm{md}}^{-1}/Q_{\mathrm{mb}}^{-1}$ as a function of torsion cycles *N* with torsion angle ±40° and the cumulative equivalent tensile strain ε_p at 450°C.

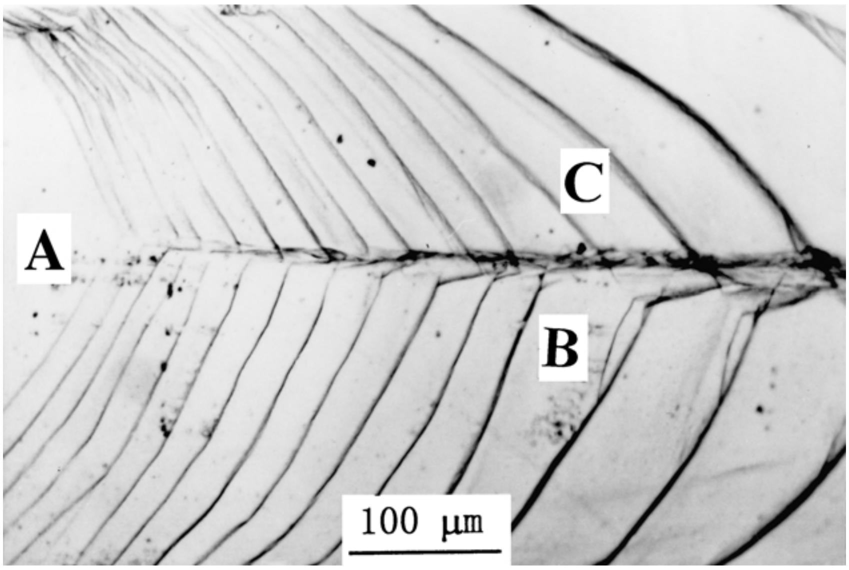

Figure 8 Three high angle boundaries meeting at a triple junction after 15 cycles of reverse torsion at 450°C in Al-10 ppm Ga.

It can be seen from Fig. 7 that the $Q_{\mathrm{md}}^{-1}/Q_{\mathrm{mb}}^{-1}$ seems to increase a little during the initial cycles, then decreases during further cycles for all the four kinds of specimens. However, the $Q_{\mathrm{md}}^{-1}/Q_{\mathrm{mb}}^{-1}$ for Al-10 ppm Ga and Al-50 ppm Ga keeps higher than that for pure Al and Al-410 ppm Ga. It means that during the cyclic torsion, there occurs more extent of recovery in the specimens for Al-10 ppm Ga and Al-50 ppm Ga.

The activation energy and peak width of the internal friction peak after different strains for each kind of specimen were found to be essentially unchanged as compared with those before deformation. According to Nowick and Berry [8], the change of normalized relaxation strength versus creep strain or torsion cycle should be similar to that of $Q_{\mathrm{md}}^{-1}/Q_{\mathrm{mb}}^{-1}$ in Figs 6 and 7, and hence not shown here.

SEM and EBSD showed that most grain boundaries in all the four kinds of specimens are general high angle boundaries. After static creep, the surface steps caused by the grain boundary sliding and the slip traces of lattice dislocations were observed; while after cyclic torsion, the migration steps appeared often.

Fig. 8 gives an example of post deformation surface observation, showing three boundaries meeting at a triple junction in a specimen of Al-10 ppm Ga undergone cyclic torsion at 450°C for 15 cycles. The misorientations and rotation axes of boundaries BA and CA determined by EBSD are $34.9^\circ\langle 432\rangle$ and $31.4^\circ\langle 432\rangle$ respectively. The migration directions are respectively along B → A and C → A. The number of migration steps is equal to the number of reverse torsion cycles. The misorientation and rotation axis of boundary BC is $34.1^\circ\langle 100\rangle$, which is close to $\Sigma 5$ ($36.87^\circ\langle 100\rangle$). No obvious migration steps of boundary BC could be observed, perhaps because its orientation is parallel to torsion axis (horizontal) and it is nearly special boundary.

4. Discussion

The internal friction peak observed in this study can be associated with energy dissipation due to grain boundary sliding [5]. The larger the extent of grain boundary sliding during vibration, the higher the internal friction peak or relaxation strength.

The relaxation strength is affected by both the material chemistry and a preceding deformation, i.e., by static creep or cyclic torsion. Interestingly, the response to deformation is not monotonous with increasing impurity level, rather, the change of the relaxation strength with deformation is reversed if the Ga content exceeds 50 ppm (Figs 6 and 7). Moreover, the change of relaxation strength behaves quite differently for the two different deformation modes. While the relaxation strength strongly decreases with increasing creep strain, it rises after initial cycles of torsion and then slightly decreases with further cycles.

For an interpretation of this behavior, one has to consider the effect of deformation on the dissipative mechanisms that causes damping. If this is entirely attributed to grain boundary sliding and deformation is understood to cause glide steps in the boundary due to run in dislocations, then the relaxation strength has to decrease monotonously with increasing deformation [6]. In fact, this is observed after static creep, although differently for different amounts of Ga.

The behavior of the sample after cyclic torsion comes unexpected from this point of view, since its relaxation strength first increases with increasing number of cycles, i.e., growing cumulative cyclic strain, before it slightly decreases. The increase is more pronounced the higher the Ga content up to a Ga concentration of 50 ppm. As known from many investigations on high temperature low cycle fatigue [10, 11], grain boundaries migrate during each cycle to arrange in a position more favorable for grain boundary sliding (e.g., Fig. 8). Trace amounts of Ga up to a level of 50 ppm have been found to enhance grain boundary mobility [1–3] and as obvious from Fig. 7 to facilitate grain boundary sliding. The degradation of relaxation strength by high amounts of Ga (410 ppm) goes along with the experimental finding that such amount of Ga also reduces the grain boundary mobility. Since grain boundary migration during cyclic deformation is most pronounced during the first cycles and diminishes with high number of cycles [10, 11], the accommodation of glide steps becomes less perfect at high cumulative cyclic strains and thus degrades the relaxation strength, as measured (Fig. 7).

During monotonic static creep, no pronounced grain boundary migration occurs to compensate for glide steps in the boundary. Consequently, the relaxation strength is bound to decrease with increasing creep strain. Surprisingly, this degradation of relaxation strength is the stronger the higher the boundary mobility so that the minimum internal friction occurs for the alloy with 50 ppm Ga, while the alloy with 410 ppm Ga shows even higher internal friction than pure Al (Fig. 6). It appears that the enhancement of mobility in stationary boundaries of crept samples results in more perturbation to boundary sliding. Such a phenomenon may be related to grain boundary roughening. In fact a grain boundary roughening transition was recently proposed as a precursor of grain growth and observed in grain boundaries of various metals [12]. We surmise that such grain boundary roughening is the possible reason for the gradual suppression of grain boundary sliding with Ga contents increasing up to 50 ppm in stationary boundaries. This is because higher mobility in stationary boundaries may enhance the roughening, and lead to a lower relaxation strength [13]. Higher Ga concentrations obviously degrade grain boundary mobility and thus presumably suppress grain boundary roughening and, therefore, enhance relaxation strength. Nevertheless, it is stressed that this roughening effect is only superimposed to the strong decrease of the relaxation strength after static creep due to the generation of glide steps in the boundary by crystallographic slip in the stationary boundary.

5. Summary and conclusions

The grain boundary internal friction of pure Al and the same Al doped with different amounts of Ga (10, 50 and 410 ppm) was measured before and after high temperature deformation at two different modes, i.e., static tensile creep and cyclic reverse torsion.

Without deformation the peak temperature and the activation energy were increased, slightly by smaller amounts of Ga (10 and 50 ppm), but markedly by a larger amount of Ga (410 ppm).

Static creep led to an obvious decrease of the peak, while the effect of cyclic torsion was not so obvious. It means that in static creep anelastic sliding of grain boundaries was suppressed, while in cyclic torsion anelastic sliding was facilitated. The present work further confirms the observations of pure Al in our previous paper.

SEM and EBSD revealed most grain boundaries in all specimens as general high angle boundaries. After static creep, slip traces of lattice dislocations were observed to intersect the grain boundaries, after cyclic torsion migration steps were seen on the surface.

The different behavior of relaxation strength after cyclic torsion compared to static creep and its enhancement by minor Ga additions (≤ 50 ppm) was attributed to coupled grain boundary sliding and migration during cyclic deformation and a roughening transition in the stationary boundaries of crept samples.

Acknowledgements

This project has been jointly supported by the National Natural Science Foundation of China and the Deutsche Forschungsgemeinschaft.

References

1. G. GOTTSTEIN, D. A. MOLODOV and L. S. SHVINDLERMAN, *Interface Science* **6** (1998) 7.
2. D. A. MOLODOV, U. CZUBAYKO, G. GOTTSTEIN and L. S. SHVINDLERMAN *Acta Mater.* **46** (1998) 553.
3. G. GOTTSTEIN and L. S. SHVINDLERMAN, "Grain Boundary Migration in Metals: Thermodynamics, Kinetics, Applications" (CRC Press, Boca Ratom, 1999).
4. W. HU, D. A. MOLODOV, B. SCHÖNFELDER, L. S. SHVINDLERMAN and G. GOTTSTEIN, *Interface Science* **8** (2000) 335.
5. T. S. KÊ, *Metall. Mater. Trans.* A **30** (1999) 2267 (a review).
6. Q. P. KONG, B. CAI and G. GOTTSTEIN, *J. Mater. Sci.* **36** (2001) 5429.
7. Q. P. KONG and Y. DAI, *Phys. Stat. Sol.* (a) **118** (1990) 431.
8. A. S. NOWICK and B. S. BERRY, "Anelastic Relaxation in Crystalline Solids" (Academic Press, Inc., New York, 1972).
9. W. BLUM, Q. ZHU, R. MERKEL and H. J. MCQUEEN, *Z. Metallkd.* **8** (1996) 1.
10. S. CHEN and G. GOTTSTEIN, *Acta Metall.* **36** (1988) 3093.
11. S. WEISS and G. GOTTSTEIN, *Mater. Sci. Tech.* **14** (1998) 1169.
12. DUK YONG YOON, in Proceedings JFMSS-7, edited by T. Watanabe *et al.* (2002) in press.
13. R. RAJ and M. F. ASHBY, *Metall. Trans.* **2** (1971) 1113.

Received 31 July 2002
and accepted 21 February 2003