Journal of Alloys and Compounds, 182 (1992) L15–L18
JALCOM 169

Letter

Low temperature recovery of gadolinium

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(Received September 20, 1991)

The anomalously low temperature recovery behaviour of gadolinium filings has been the subject of several previous studies in this school. It was first noticed by Nellist [1] whilst examining the faulting behaviour of gadolinium and Gd–Ce alloys. It was found that annealing at temperatures as low as 100 °C produced significant sharpening of lines on X-ray Debye–Scherrer films, which is indicative of recovery occurring in the specimen. Recovery in metals of commercial purity is usually associated with a T/T_m of not less than about 0.4. Gadolinium melts at 1313 °C and a 100 °C anneal represents a T/T_m of only 0.2.

A subsequent investigation by Druce [2] showed recovery to be occurring at room temperature over a period of days. This investigation also showed the importance of powder particle size on the extent of recovery observed (finer powder was found to recover more quickly) and that filing in air rather than in a vacuum or under a solvent reduced the rate of recovery. This suggests that the free surface of the powder is important in its recovery behaviour and that a surface oxide layer could retard the recovery process.

Outside the work performed in this school, Milstein and Zyvoloski [3] mention that cold-rolled material softened over 3 months, indicating that recovery had occurred during this time.

The study presented here used X-ray diffraction and transmission electron microscopy (TEM) to examine the recovery behaviour of gadolinium. Powder for the X-ray studies was obtained from bulk material by filing under 1,1,1-trichloroethane using a diamond-coated file. The solvent was used to cool the material and help prevent oxidation. Once dried, the material was stored in a vacuum desiccator. Powder was produced from bulk material in both deformed and annealed states. The TEM specimen was prepared from rolled foil by spark machining followed by dimpling and ion beam thinning. It was viewed initially immediately after removal from the ion beam thinner and

0925-8388/92/$5.00

then again a few weeks later. Like the powders, the sample was stored in a vacuum desiccator to minimize oxidation.

The results from computerized X-ray diffractometry on a single sample of powder over a period of 2.5 weeks are shown in Fig. 1. The degree of line sharpening is measured by the full width at half-maximum (FWHM) height of the diffractometer peaks and is shown for the low angle $(10\bar{1}1)$, $(10\bar{1}0)$ and (0002) reflections. The same trend of sharpening peaks was also clearly visible in Debye–Scherrer films taken of other powder samples from both deformed and annealed bulk materials.

The electron micrographs in Fig. 2 show direct evidence of recovery occurring in gadolinium over a period of weeks. Figure 2(a), taken immediately after thinning, shows a very high density of dislocations evident in the structure. Figure 2(b) (the same sample a few weeks later), although it has oxidized badly, reveals a recovered structure with dislocation subboundaries (double arrow) and dislocation-free subgrains.

The recovery process involves the rearrangement and annihilation of dislocations forming subgrains and dislocation cell walls by polygonization. Such structures were clearly seen by TEM. The arrangement of dislocations by the process of climb to form subgrain boundaries involves the movement of vacancies in the lattice and is thus usually associated with elevated temperatures. It would appear that in gadolinium, at least in a surface layer, the diffusion of vacancies is activated at room temperature, allowing recovery to take place.

Much of the work done on recovery in gadolinium points towards the process being chiefly a surface phenomenon. The importance of particle size found in earlier studies provides evidence for this, as does the lack of any recovery structures present in the foil material when first observed by TEM. It was only once the foil had been thinned that recovery could occur and be observed in the microscope. From a consideration of the mass absorption coefficient of gadolinium it is found that 99% of the information obtained

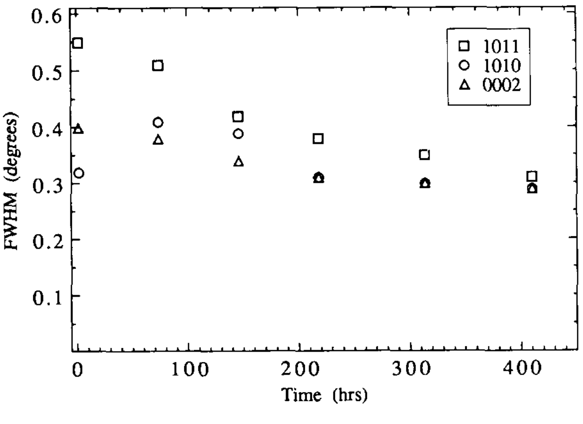
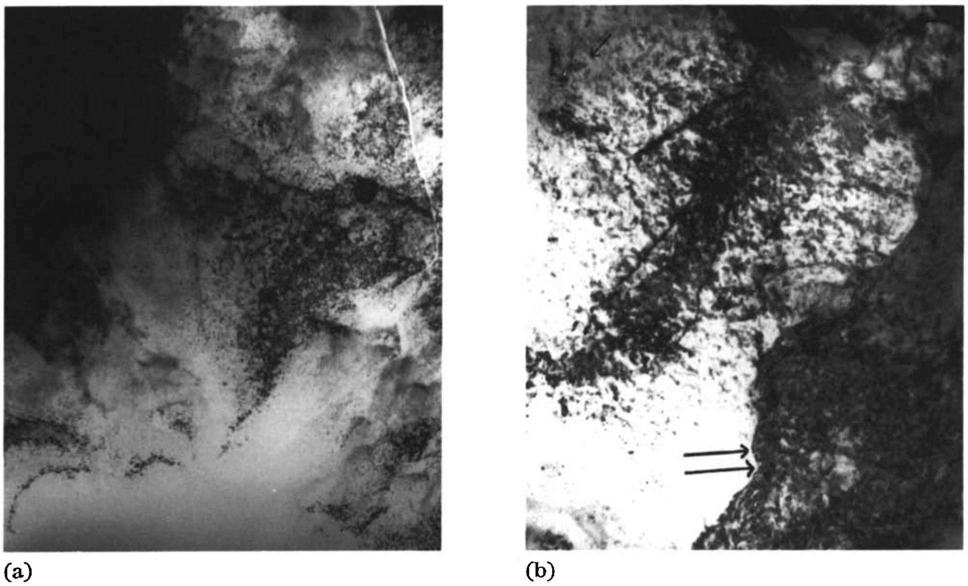


Fig. 1. Change in diffractometer peak heights with time.



(a) (b)

Fig. 2. Transmission electron micrographs before and after recovery. (a) Before recovery. A heavily dislocated structure is seen (Philips 420T, 100 kV). (b) The same sample some weeks later. The dislocations have recovered into subgrain boundaries, one of which is indicated by a double arrow. Despite being kept in a desiccator, the foil has oxidized badly and this is the origin of the considerable contrast between the dislocations. Single arrow, $g = 11\bar{2}0$ (JEOL 4000 FX, 350 kV). In both cases the bar represents 50 nm. The two areas are *not* the same.

from X-ray diffraction results comes from a depth of only 17 μm and does not therefore give any evidence that recovery is occurring beneath a relatively thin surface layer.

It is not clear why recovery should occur preferentially at the surface. Possibly grinding deformation is more violent in the surface regions. Low temperature oxidation might also be responsible for injecting vacancies. The phenomenon appears to be specific to gadolinium: terbium, for example, shows no similar effect [2].

From the work done so far the following conclusions can be drawn.

(1) Filed gadolinium undergoes substantial recovery near its surface over a period of days.

(2) Recovery can also be observed directly in thin films by TEM over a similar period of time. The process proceeds by polygonization and the formation of subgrains and dislocation cell walls.

(3) The injection of vacancies into the structure could provide a driving force for enhanced diffusion to occur, leading to dislocation mobility.

Acknowledgment

The work described formed part of an undergraduate project carried out in the School of Metallurgy and Materials, University of Birmingham.

References

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