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To cite this version:
Z. Lai, H. Nordén. THE ENHANCED CONCENTRATION OF Ni AT STRUCTURAL DISLOCATIONS IN A GRAIN BOUNDARY IN W. Journal de Physique Colloques, 1988, 49 (C6), pp.C6-341-C6-345. <10.1051/jphyscol:1988659>. <jpa-00228156>

HAL Id: jpa-00228156
https://hal.archives-ouvertes.fr/jpa-00228156
Submitted on 1 Jan 1988

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THE ENHANCED CONCENTRATION OF Ni AT STRUCTURAL DISLOCATIONS IN A GRAIN BOUNDARY IN W

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Abstract - The enhanced concentration of Ni at a grain boundary in Ni-treated W has been investigated using atom probe field ion microscopy and the measured concentrations have been related to structural observations by field ion microscopy and transmission electron microscopy.

1 - Introduction

It is well known that diffusion along grain boundaries generally proceeds more rapidly than in the lattice at temperature appreciably below the melting point. As a result, the fast grain boundary diffusion can produce a net mass transport along boundaries [1, 2] which can dramatically influence the kinetics of microstructural changes and the properties of materials.

Therefore the grain boundary diffusion plays a crucial role in metallurgical processing and in many applications of materials. However, the diffusion of atoms in grain boundaries is very complex and may in many cases depend on the structure of the grain boundary [3]. Theoretical studies indicate that dislocations can be the preferred paths for vacancy type diffusion in grain boundaries and we have previously reported experimental observations of the enrichment of impurities to the extrinsic grain boundary dislocations.

In present experiment, an atom probe field ion microscope (APFIM) with its near atomic spatial resolution was used to investigate the chemistry of a grain boundary containing an array of structural dislocations.

2 - Experimental

The starting material was cold drawn, doped tungsten wire with a diameter of 0.16 mm. The surface contamination was first removed by electropolishing in a 25% solution of an Agfa photo developer (Neutol S) in water using 10 V AC at room temperature.

The thus cleaned wire was plated in an industrial chloride sulfate nickel bath at 60 °C to form nickel layer about 30 μm thick. The Ni-plated samples were then annealed in high vacuum at 1000 °C for 50 h. The heat treatment conditions were chosen so as to produce a partly recrystallized wire where the central core had been penetrated by Ni but still retained its original fiber structure.

Finally, the needle-shaped specimens with a tip diameter of about 50 nm were prepared by electropolishing in such a way that the specimen apex contained a grain boundary from this pre-recrystallization zone, which might contain Ni.

TEM was used both to monitor the final stage of specimen preparation and to select suitable samples. TEM was also used in combination with FIM to determine the relative orientations of the grains and also to determine position and orientation of the grain boundary.

An atom probe field ion microscope operated at liquid nitrogen temperature was used for imaging and also for the analysis of the composition at different positions at the grain boundary.

(1) On leave from Institute of Metal Research, A.S., Shenyang, P.R. China
3 - Results

Geometry of the grain boundary

The electron micrographs, Figs. 1a - b show two projections, 90° apart, of the needle-shaped specimen after field evaporation. Fig. 1a shows the topography of a grain boundary intersecting the tip of the specimen. The boundary, which runs along the <110> - axis of the specimen appears fairly straight and uniform in this projection. The position of the only bent part of the boundary coincides with an area with low contrast in Fig. 1b.

In this projection, Fig. 1b, when the specimen is viewed perpendicular to the grain boundary it exhibits an array of straight lines. The lines are roughly parallel to the <110> direction and their mean spacing is about 4 nm. The image contrast was rather insensitive to the imaging conditions and complete extinction of the contrast could never be achieved.

A field ion micrograph of the specimen is shown in Fig. 2. The grain boundary intersects the surface close to the central <110> pole and regularly spaced dislocations, three of which have been indicated by arrows, are seen along the boundary. The relative spacing of these grain boundary dislocations agrees well with the spacing of the straight lines seen in the electron micrograph, Fig. 1b.

The results from electron diffraction analysis of the apex region are summarized in Fig. 3. The two grains are aligned along the fiber axis with a relative tilt of about 77° around the <110> axis plus an additional 3° twist around an axis perpendicular to the grain boundary plane. The grain boundary plane is close to (225) in grain A and (334) in grain B.

Chemistry of the grain boundary

In order to determine the distribution of impurity atoms analyses were performed at three positions in the grain boundary region and also at one point away from the boundary for reference. The results of these analyses are given in Table 1.

In Analysis 1 the aperture was initially placed close to the image of the boundary and kept fixed. A continuous change in Ni concentration was recorded as successive layers were removed from the specimen and the image of the boundary and the dislocation shifted across the analyzing aperture. Image gas was introduced and FIM images were recorded at several stages of the analysis and the results are shown in Fig. 4.

It was noticed that it was only when the image of a dislocation was within the detecting aperture that Ni ions were detected. The projected area of the analyzing aperture, \( A_p \), was \(-1.4 \text{ nm}^2\), and the Ni concentration corresponded to about 2.5 Ni atoms per atomic layer along the dislocations in the grain boundary.

In Analysis 2, the compositions at the positions of two of the dislocations marked with arrows were analyzed. An average concentration of about 6 at.% Ni was obtained for both positions, shown in Fig. 5a. The analyzed area, \( A_p \), was \(-2.4 \text{ nm}^2\) and 19 Ni ions out of a total of 200 ions were detected, which corresponds to about 3.2 Ni atoms per atomic layer along the dislocations.

Analysis 3 was made at a position symmetrically between two dislocations in the boundary. Only 2 Ni ions were detected in a total of 451 ions (Fig 5b). The average concentration of nickel here is 0.4 at.%. If the boundary width is taken to be 1 atomic layer, an upper limit of the concentration of Ni in the boundary between dislocations is 0.15 atom per atomic layer, which is much lower than in the GBD zones.

Analysis 4 was taken from the tungsten matrix to be used as a reference. No Ni ions were found in a total of 509 ions which shows that the bulk concentration of Ni is low.

4 - Discussion

Grain boundary dislocations

The formation of the observed grain boundary dislocations can be interpreted in terms of the plane-matching model [4] when a single set of planes of indices (hkl) in each grain adjoining the boundary is
parallel, or nearly parallel, and no significant lattice matching of the two crystal exists. When these
planes are nearly parallel, and the indices \((hkl)\) are fairly low, then the boundary energy can be reduced
by introducing relaxation in the boundary structure which appears in the form of grain boundary
dislocations.

In the present experiment, the boundary is a high angle \(<110>\) tilt boundary, with a small twist
misorientation, \(\Delta \alpha = 3^\circ\) perpendicular to the boundary plane. One set of grain boundary dislocations
should then form a set of parallel lines with a spacing given by

\[
d = d_{hkl} \left\{ \frac{1}{2 \sin (\Delta \alpha / 2)} \right\}^{-1}
\]

The calculated mean spacing, 4.2 nm, is in good agreement with the experimentally observed value of
about 4 nm.

The presence of Ni at the boundary

The nickel present in the grain boundary could either have been present in the material and segregated
from the interior of the tungsten grains or penetrated the material along the boundaries from the surface
nickel plating. No detectable amounts of nickel were found in the bulk so it is reasonable to assume that
the nickel had penetrated the material via the grain boundaries.

In the previous studies, we have reported high local concentrations of nickel along lattice dislocations
[5] and also along extrinsic dislocations in grain boundaries [6]. In the present study a similar enrichment
of nickel atoms has been found at intrinsic grain boundary dislocations, and it is reasonable to assume that
a similar transport mechanism is active in all cases.

Following the plane-matching model the boundary consists of patches of perfect crystal interrupted by
grain boundary dislocations around which the mis-match strain is localized. These strained regions can
then serve as fast diffusion paths for the impurities.

It is worth pointing out that the amount of nickel found at the intrinsic dislocations is about same as
that previously measured at lattice screw dislocations and extrinsic grain boundary dislocations.

5 - Conclusions

1. A grain boundary with an array of structural dislocations has been analyzed
   using an atom probe field ion microscope in combination with transmission electron microscopy.
2. The grain boundary was a large angle \(<110>\) tilt boundary of about \(77^\circ\) with
   an additional small twist of about \(3^\circ\). The boundary plane is \(225\) for grain A
   and \(334\) for grain B. The mean spacing of the dislocations is about 4 nm.
3. The Ni distribution in the grain boundary is not uniform. There were about
   3 Ni atoms per atomic layer along the dislocations as compared to about 0.15
   Ni atoms per atomic layer in grain boundary areas between the dislocations.
4. No Ni was detected in W matrix.

6 - Acknowledgement:

This work was financially supported by the Swedish Natural Science Research Council (NFR).

7 - References

   (1977) 175.
Table 1
Maximum Ni concentrations at different positions

<table>
<thead>
<tr>
<th>Analysis no.</th>
<th>No. of Ni ions</th>
<th>Total no. of ions</th>
<th>Ni atoms per layer</th>
<th>Analyzed position</th>
</tr>
</thead>
<tbody>
<tr>
<td>2a</td>
<td>23</td>
<td>270</td>
<td>2.9</td>
<td>at dislocation</td>
</tr>
<tr>
<td>2b</td>
<td>19</td>
<td>200</td>
<td>3.2</td>
<td>at dislocation</td>
</tr>
<tr>
<td>3</td>
<td>2</td>
<td>452</td>
<td>0.1</td>
<td>between dislocations</td>
</tr>
<tr>
<td>4</td>
<td>0</td>
<td>509</td>
<td>&lt;0.01</td>
<td>bulk</td>
</tr>
</tbody>
</table>

Fig. 1. Two projections, $90^\circ$ apart of the field evaporated specimen

a) The topography of the grain boundary viewed almost edge on

b) The grain boundary dislocations viewed from a direction perpendicular to the boundary plane.

Fig. 2. Field ion micrograph of the same specimen. Some of the grain boundary dislocations are indicated by arrows.

Fig. 3. A diagram of the relative tilt orientation of the two grains as determined by electron diffraction analysis (table)
Fig. 4. The amount of Ni measured during different stages of an evaporation sequence. The analyzing aperture was fixed while the grain boundary position gradually changed:
(a) and (d) outside the boundary.
(b) at a grain boundary dislocation.
(c) between grain boundary dislocations.

Fig. 5. The measured amount of Ni:
(a) At the position of dislocation
(b) Between dislocations.