

APT Characterization of Solute Segregation to Individual Dislocations

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Abstract

Solute segregation to individual dislocations may be quantified by atom probe tomography. Dislocations may be observed in field ion images by a change of the normal concentric atom terraces to spirals. Dislocations are evident in three-dimensional atom probe atom maps by enhanced levels of solute along linear features. The magnitude of the solute segregation may be quantified with the use of the maximum segregation envelope method. Solute segregation to dislocations in NiAl, neutron irradiated pressure vessel steels and a mechanically alloyed oxide dispersion strengthened (MA/ODS) ferritic alloy are presented.

Introduction

The characterizations of dislocations are not routinely performed in the atom probe due to their low densities in most materials and the limited volume of analysis. These types of characterization are generally limited to cold worked or mechanically alloyed materials and irradiated materials where the dislocation density is significantly higher.

Atom Probe Field Ion Microscopy

If a dislocation has a Burger's vector component normal to the plane of the specimen surface at its point of emergence, the usual concentric rings in the field ion image will exhibit a spiral, as shown in Fig. 1. In cases where the point of emergence occurs in a high index region, the dislocation may not be evident in the field ion image. The visibility criterion for a perfect dislocation having a Burgers vector **b** and line **l** that converts a stack of planes of normal **n** into a spiral ramp is $\mathbf{n} \cdot \mathbf{l} \neq 0$ and $\mathbf{n} \cdot \mathbf{b} \neq 0$. The mechanical stress imposed on the specimen by the applied electric field used to produce the field ion image seriously influences the configuration of the dislocation. A consequence of this stress is that glissile dislocations will invariably slide out of the apex region of the specimen when the electric field is applied [1,2].

Solute segregation is evident in the field ion image by the presence of brightly imaging atoms near the point of emergence of the dislocation, as shown in Fig. 1 [3]. The

identity of these atoms are determined by single atom catching experiments in the atom probe [2,3]. Character plots from atom probe analyses in a classical probe hole type of atom probe demonstrating zirconium segregation to the dislocation are shown in Fig. 2 [2]. In this zirconium doped NiAl example, local concentrations of 3-5 at. % Zr were measured in the vicinity of the dislocation and 0.007±0.002 at. % Zr was measured in the matrix far from the dislocation [3].

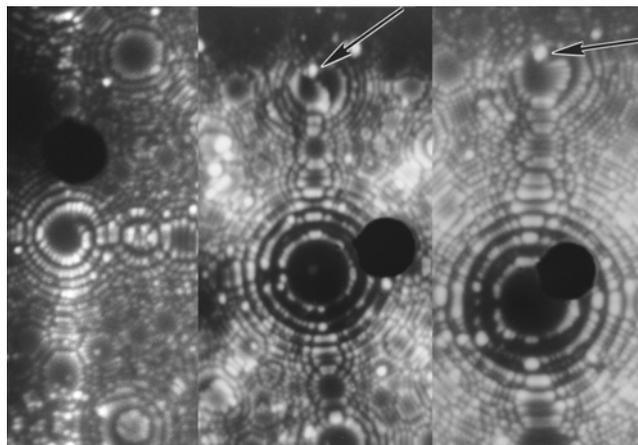


Fig. 1. Field ion images of dislocations in NiAl doped with 0.7 at. % Mo and 0.4% Zr [3]. The arrowed spots were determined to be zirconium atoms by atom probe analyses.

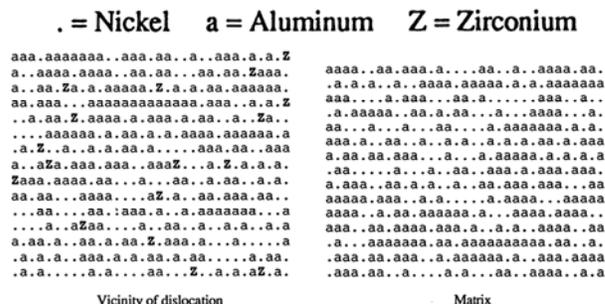


Fig. 2. Character plots from the matrix in the vicinity of and far from a dislocation in NiAl doped with 0.7 at. % Mo and 0.4% Zr. Each symbol represents an individual atom. Zirconium segregation near the dislocation core is evident.

Atom Probe Tomography

The introduction of the three-dimensional atom probe and in particular the local electrode atom probe has facilitated the analysis of dislocations due to their significantly larger volumes of analysis [4]. With these instruments, the atomic coordinates and the elemental identities of atoms in a small volume of analysis are determined. Dislocations are revealed from the local enrichment of solute, as shown in Figs. 3 and 4 for an as-prepared mechanically alloyed (MA) oxide dispersion strengthened (ODS) ferrite steel and a neutron irradiated pressure vessel steel weld, respectively.

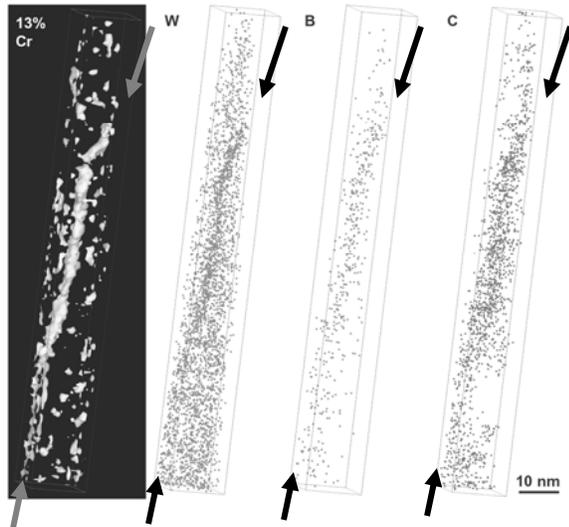


Fig. 3. Cr-isoconcentration surface and W, C and B atom maps showing solute segregation at a dislocation in an as-prepared MA/ODS 12YWT alloy.

Table 1. Solute concentrations in the ferrite matrix and in the vicinity of a dislocation in an as-prepared MA/ODS 12YWT alloy. The balance is iron.

at. %	Alloy	Ferrite	Dislocation	Enrichment
Cr	13.3	13.0	15.9 ± 0.16	1.2
W	0.92	0.78	1.21 ± 0.05	1.6
Ti	0.46	0.09	0.44 ± 0.03	4.9
Y	0.13	0.03	0.16 ± 0.02	5.3
O	0.19	0.11	0.53 ± 0.03	4.8
C	trace	0.18	1.38 ± 0.05	7.7
B	trace	0.05	0.44 ± 0.03	8.8
N	trace	0.15	0.17 ± 0.02	1.1

The composition of the solute-enriched region around the dislocation may be estimated with the maximum separation envelope method [4]. In the MA/ODS steel analysis shown in Fig. 3, the extent of the solute enriched region is defined by a C + B atom maximum separation distance of 1.5 nm. The results shown in Table 1 indicate that both interstitial and substitutional solutes are enriched near the dislocation compared to the ferrite matrix.

Phosphorus enrichment (>240X) was measured around the dislocation in the neutron irradiated pressure vessel steel. Ultrafine copper-enriched precipitates were observed on the dislocation (grey arrow) and in the interior of the ferrite matrix.

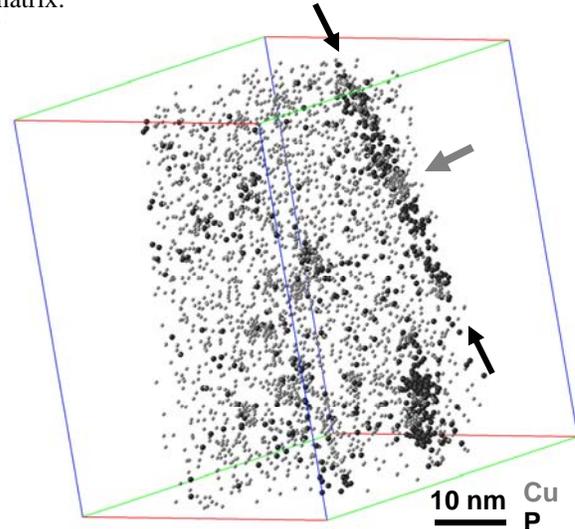


Fig. 4. Atom map of a phosphorus decorated dislocation and ultrafine (~3 nm diameter) copper-enriched precipitates in a neutron irradiated (fluence = $5 \times 10^{23} \text{ n m}^{-2}$ ($E > 1 \text{ MeV}$), $T = 288^\circ \text{C}$) JRQ pressure vessel steel.

Conclusions

Solute segregation to and precipitation on individual dislocations may be characterized by atom probe tomography. Atom probe tomography revealed that both interstitial and substitutional elements segregate to dislocations.

Acknowledgements

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References

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