Materials Letters 122 (2014) 301–305

Contents lists available at ScienceDirect

Materials Letters

journal homepage: www.elsevier.com/locate/matlet

Direct observation of solute–dislocation interaction on screw dislocation in a neutron irradiated modified 316 stainless steel

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ARTICLE INFO

Article history:
Received 22 October 2013
Accepted 19 January 2014
Available online 25 January 2014

Keywords:
Defects
Segregation
Nuclear materials
Atom probe
Radiation damage

ABSTRACT

A three-dimensional atom probe (3DAP) technique was used to observe the three-dimensional solute distribution around screw dislocations in neutron-irradiated modified 316 austenitic stainless steel. Si, Ni, and P were observed to get enriched around the cores of screw dislocations, while Fe, Cr, Mn, and Mo were depleted in the same areas. Si was segregated to a narrow region in the core for which, it was concluded that Si was trapped in the initial stage of segregation. Other enriched elements segregated to some edge features such as kinks and/or jogs formed as a result of Si trapping. The results suggest that solute–dislocation interaction of screw dislocations was similar to radiation-induced segregation, which is affected by the volume size factor of solute atoms.

1. Introduction

It has been widely accepted that dislocations act as sinks for point defects and defect clusters. Especially, edge dislocations are controlled by irradiation defects due to the interstitial bias of the sink [1,2]. Edge dislocations have hydrostatic stress fields around their core; therefore, it absorbs the point defects easily. While, there are no hydrostatic stress fields around the cores of screw dislocations. Thus, it is presumed that a driving force for moving point defects to the core of a screw dislocation does not exist. Because of these differences in the stress fields around the cores, the sink strength for a point defect is thought to be weaker than that for an edge dislocation.

However, the mechanism for the screw dislocation acting as a sink for point defects can be found indirectly, e.g., it is known that the absorption of either interstitials or vacancies at a screw dislocation changes it to a helical dislocation having partial edge component [3,4]. Therefore, the core of the screw dislocation is considered to form kinks or jogs with an edge component, which shows interstitial bias. Such kinks or jogs would enhance the transformation from screw to helical dislocation. Irradiation softening is caused by such increase in the edge component. This increase results in a large degree of softening in bcc metals because the Peierls stress rapidly increases at low temperature [5,6]. In this way, screw dislocations are considered to have a significant influence on the formation of defects during irradiation, and the resulting mechanical properties increases edge components as a result of solute–dislocation interactions.

Using conventional methods, it is difficult to analyze solute atoms trapped at the core of dislocations, and it is also difficult to clarify which solute atom is dominant in the solute–screw dislocation interactions. In a recent study, we succeeded in clearly visualizing in three-dimension, the solute segregation to the extended edge dislocations and proved solute segregation to stacking faults and Shockley partial dislocations in a neutron irradiated modified 316 stainless steel by using three-dimensional atom probe (3DAP) [7]. In this study, we apply the method to liner screw dislocations to discuss the solute–screw dislocation interaction under neutron irradiation.

2. Experimental

For this study, we used modified 316 (Fe–16Cr–14Ni–2.5Mo–0.25P–0.004B–0.1Ti–0.1Nb–0.8Si–0.05C wt.%) austenitic stainless steel. The alloy was solution-annealed at 1353 K for 4 min before irradiation. Neutron irradiation was conducted in the experimental fast reactor JOYO at 862 K with a neutron fluence of 1.12×10^{27} n/m^2 (≥ 0.1 MeV), which corresponds to approximately 56 dpa (displacements per atom). Needle-shaped specimens for the 3DAP analysis were prepared from irradiated specimens using the focused ion beam (FIB) lift-out technique [8].

The 3DAP specimen preparations were conducted in a FIB (FIB, SMI-2050; SII) using a 30 keV Ga^+ ion beam. After the FIB milling,

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http://dx.doi.org/10.1016/j.matlet.2014.01.109
0167-577X

the in situ lift-out technique was used to transfer the milled stainless steel tip onto a needle-like W tip prepared by electropolishing. After bonding the tips by W deposition, the stainless steel tip was further thinned using FIB and a transmission electron microscope (TEM) to include tangled dislocations around the top of the tip, which did not include precipitates, grain boundaries or twins.

The 3DAP observations were performed using a Cameca Instruments Inc. laser-assisted local electrode 3DAP (LEAP-3000XHR). To reduce the probability of tip fracture during 3DAP evaporation of the atomic layers, we used field evaporation with a laser pulse instead of an electric field pulse. The 3DAP observation conditions were as follows: laser energy of 0.5–0.6 nJ, a laser-pulse repetition rate of 250 kHz, a DC bias voltage of 4–7 kV, and a specimen temperature of 55 K. The DC voltage was automatically controlled to keep a constant evaporation rate of 0.4% at constant energy of the laser pulse. The Ga^+-irradiated regions by FIB around the top of the thinned tip were fractured before 3DAP observation at a DC voltage range of 2–4 kV. The computer program IVAS3.6.1 (Cameca Instruments Inc.) was used to analyze the data. Orientations of the screw dislocations were defined with high accuracy on the basis of {111} planes of stacking faults, which were observed while mapping the same atom. Details of the methods were explained in our previous paper [7].

3. Results and discussion

Fig. 1(a) shows the typical three-dimensional atom map of Si before irradiation. In the atom map Si atoms are distributed homogeneously. Since there was no linear or planer enrichment of solute atoms in approximately 20 million atoms, we assumed that significant segregation of solute atoms did not occur in dislocations, before irradiation. Fig. 1(b)–(e) shows Si atom maps of an irradiated specimen consist of two irradiated regions (Regions 1 and 2) and two different viewpoints. Regions 1 and 2 were obtained from the same needle-like specimen. Therefore, both atom maps show the same orientation. Fig. 1(b) and (c) are the atom maps viewed from the [101] direction. Two defects were observed (marked by red and yellow circles), which were the areas of Si enrichment. Fig. 1(c) and (e) shows atom maps viewed from the [0$\bar{1}$0] direction. The defects are linear and extended in the [10$\bar{1}$] direction. Therefore, the linear defects are screw dislocations, and we named them as SD 1 and SD 2 (Fig. 1).

Fig. 2 shows two-dimensional elemental mapping when SD 1 (Fig. 2(a)) and SD 2 (Fig. 2(b)) were observed from the [101] direction. White dotted lines indicate {111} planes, which cross the dislocation core. While the undersized atoms (smaller than Fe) such as Ni, Si, and P were found to concentrate near the dislocation core, the oversized atoms (larger than Fe) such as Cr, Mo, and Mn were observed to be depleted. Moreover, the host atom was depleted. A high Si concentration zone was located within a 2 nm radius of the dislocation core, whereas a Ni-rich zone was located within a 3–4 nm radius. These atoms were arranged in almost concentric circles. This suggested that the SD 1 hardly had long edge component such as jogs. In the case of SD 2, while Fe and Cr were depleted along {111} planes, Ni was found to concentrate in the same plane.

Fig. 3 shows the one-dimensional composition profiles of Fe, Ni, Cr, Si, Mn, Mo, Nb, P, Ti, and C around a screw dislocation core, obtained from the elemental analysis along the arrow shown in the box of 4 nm × 20 nm in Fig. 3(c) and (f). Si profiles show narrow peaks around distances of 10 nm, while the Ni profiles show broad peaks. These differences in elemental distributions around the dislocation core suggested that Si enrichment occured during the initial stage of segregation. P was enriched near the dislocation core and depleted the dislocation core. On the basis of the solute atom distribution, it can be concluded that the screw dislocations had not extended far; hence, it can beconsidered to be the predominant sink for interstitial atoms. In the SD 2, the concentrations of Si and Ni around dislocation core were lower than that of SD 1 and showed a sub peaks to left side of the main

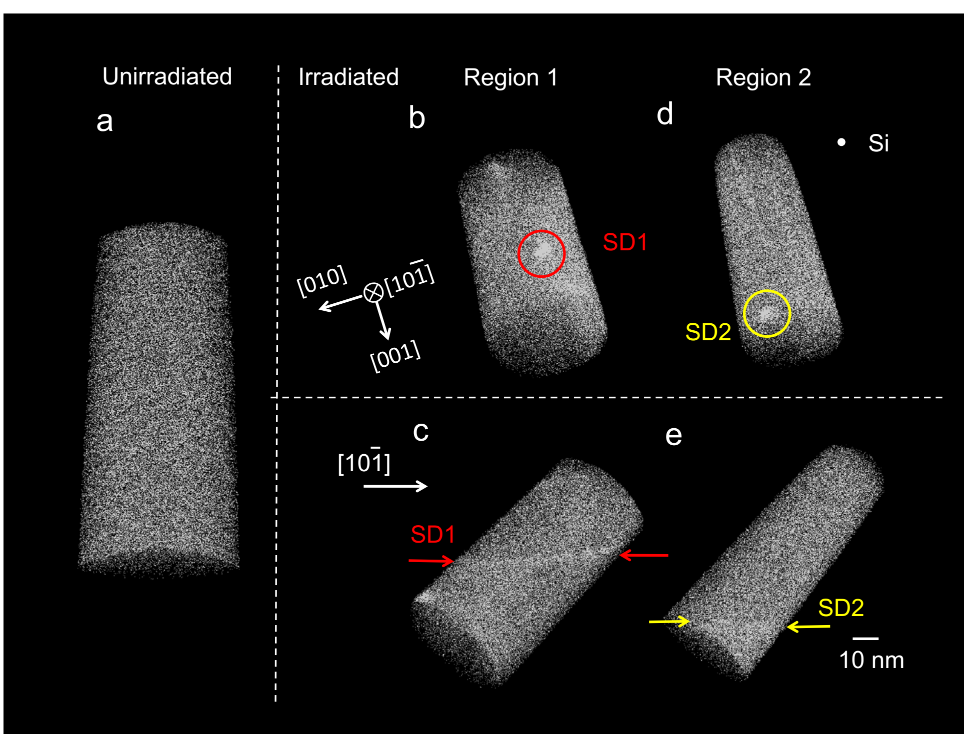

Fig. 1. Atom maps of dislocations in an unirradiated (a) and neutron-irradiated and (b–e) PNC316 authentic stainless steel. The white dots are Si atoms. All other atoms are hidden for clarity. Si distributions in Region 1 (b and (c) and Region 2 (d and e). The view point of (b) and (d) is [10$\bar{1}$] and (c) and (e) is [0$\bar{1}$0]. In these Si atom maps, two screw dislocations SD 1 (marked in red circle and arrows) and SD 2 (marked in yellow circle and arrows) were detected. The two screw dislocation lines are along the [10$\bar{1}$] direction. (For interpretation of the references to color in this figure caption, the reader is referred to the web version of this paper.)