Identification of Radiation-Induced Segregation in Ion-irradiated Stainless Steel 316 using Atom Probe Tomography

Gyeong-Geun Lee [*], Hyung-Ha Jin, Kunok Chang, Junhyun Kwon
Nuclear Materials Division, Korea Atomic Energy Research Institute,
111, Daedeok-daero 989beon-gil, Yuseong-gu, Daejeon, 305-353, Korea
[*]Corresponding author: gglee@kaeri.re.kr

1. Introduction

Various kinds of defects are produced by the irradiation of energetic particles onto a structural material. The defect fluxes such as mobile vacancies and self-interstitial atoms cause the diffusion of the solute atoms in the matrix. The preferential interaction of the solute with defects induces the enrichment or depletion of the solutes at the defect sinks such as the grain boundaries, and surfaces. These phenomena are generally known as radiation-induced segregation (RIS) [1].

Transmission electron microscopy (TEM) and energy dispersive X-ray spectroscopy (EDS) are generally used as basic analysis techniques to obtain a concentration profile of RIS arising from irradiation. However, the resolution of EDS is limited due to beam broadening, and the overlapping of the probed volume with the matrix prohibits a quantitative analysis of the concentration changes.

In the current work, we introduced atom probe tomography (APT) to analyze RIS in SS 316. Various types of radiation-induced defects were identified and the compositional characteristics were quantitatively provided from a wide view point. The measured concentrations were compared with values in the literature [2,3]. This work can provide a fundamental understanding of the RIS behavior in ion-irradiated SS 316.

2. Methods and Results

2.1 Experiments

The test sample was taken from a solution-annealed plate made of SS 316. The chemical composition of the plate is given in Table 1. A small plate of 10×10×2.5 mm^3 was prepared for an ion irradiation test. The specimen was ground with 1200 mesh SiC paper, and finally polished with paste of a 0.25-μm diamond suspension.

Table I: Composition of stainless steel 316

Cr	Ni	Si	Mo	Mn	P	C	S	Fe
16.7	11.1	0.59	2.0	1.3	0.05	0.047	0.01	Bal.

Ion irradiation was carried out using a tandem ion accelerator at the Korean Institute of Geoscience and Mineral Resources. The ion source was Fe^{4+}, and the acceleration voltage was 2 MeV. The temperature of the specimen was ~400℃ and the irradiation time was 4 h. The total ion fluence was estimated to be 5.6×10^{15} ions/cm^2. The peak dose of the irradiation was estimated to be 12 dpa at a depth of 1.7 μm using the SRIM code with full cascade evaluation mode [4]. The region at a depth of 1 μm below the irradiated surface was selected for the APT analysis. The dose of the specimen was estimated to be about 4 dpa.

Atom probe needles were prepared using a Helios Nanolab 650 dual beam SEM/FIB instrument from FEI. The detailed procedure of the preparation of APT is described elsewhere [2]. Fig. 2 shows an APT needle micromachined by means of a focused ion-beam (FIB) technique. APT measurements were carried out using a laser-assisted, wide angle atom probe from CAMECA.

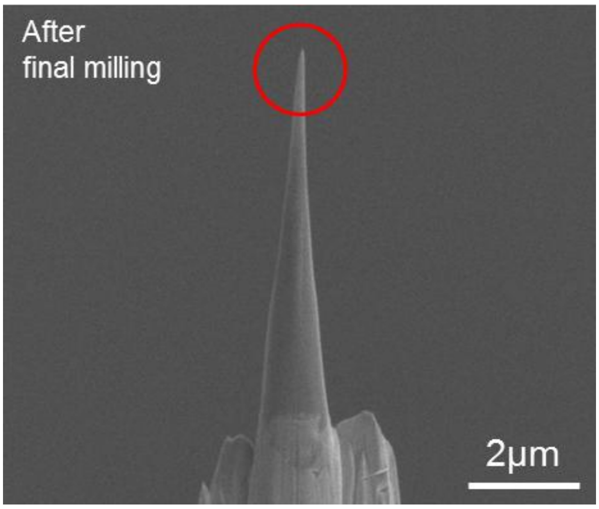

Fig. 1. Micrographs of an APT specimen prepared using the focused ion beam technique.

The corresponding element-specific atomic positions within the 3D volume were reconstructed using the Tap3Ddata program from CAMECA, and complemented by the analysis program developed at the Korea Atomic Energy Research Institute.

2.2 APT analysis

Fig. 2 shows a TEM micrograph of the damaged matrix region located at a depth of 1 μm below the surface. Dislocation loops and black disks are clearly observed. The loop size is measured to be 20–30 nm, and the loops are arranged along the special orientations. TEM observations can identify the shape and density of the irradiation defects, but compositional changes at the irradiation defects cannot be measured quantitatively. This is why APT needed to identify the irradiation defects.

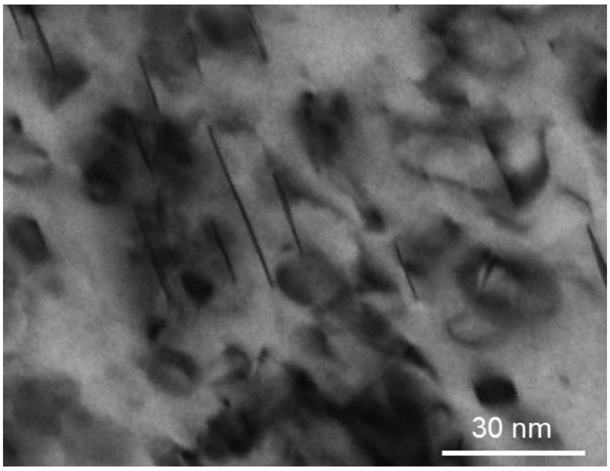

Fig. 2. TEM micrograph of the damaged matrix region at 1 μm in depth from the surface.

Fig. 3 shows atomic maps of the elements in the APT specimen. Because of the large number of atoms, the enrichment of Ni and depletion of Cr are not evident. In contrast, Si is enriched, clearly at the irradiation defects. To obtain a detailed view of the Si enrichment, the atomic map of Si was rotated and various forms of Si enrichment were found, specifically torus-shaped, disk-shaped, and rounded clusters, as well as 1D linear features. The torus-shaped and disk-shaped clusters are similar to the TEM observation presented in Fig. 2.

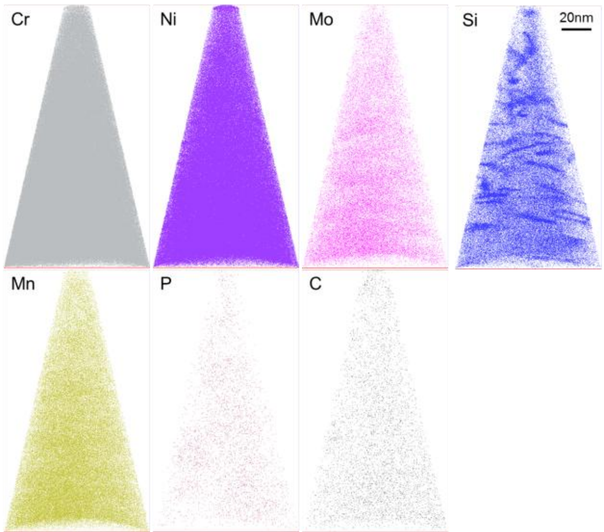

Fig. 3. Atomic maps of various solutes in ion-irradiated SS 316. The Si map clearly shows enrichments at various irradiation defects.

3. Summary

In this study, an APT analysis of RIS in ion-irradiated SS316 was performed. Various types of irradiation defects were observed. Si atoms are located at the core structures of dislocation loops and clusters. It is believed that there are strong Si-interstitial interactions during irradiation. The APT analysis was well suited for discovering the structure of irradiation defects and the quantitative analysis of RIS in irradiated specimens.

REFERENCES

[1] G.S. Was, "Fundamentals of Radiation Materials Science," Springer, New York (2007).

[2] A. Etienne, B. Radiguet, N.J. Cunningham, G.R. Odette, and P. Pareige, "Atomic scale investigation of radiation-induced segregation in austenitic stainless steels," J. Nucl. Mater. vol. 406, pp.244-250, (2010).

[3] T. Toyama, Y. Nozawa, W. Van Renterghem, Y. Matsukawa, M. Hatakeyama, Y. Nagai, A. Al Mazouzi, and S. Van Dyck, "Grain boundary segregation in neutron-irradiated 304 stainless steel studied by atom probe tomography," J. Nucl. Mater. vol. 425, pp.71-75 (2012).

[4] J.F. Ziegler, J.P. Biersack, U. Littmark, "The Stopping and Range of Ions in Solids," Pergamon, New York (1985).