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Effect of microstructure on radiation induced segregation and depletion in ion irradiated SS316 steel

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1. Introduction

Irradiation assisted stress corrosion cracking (IASCC), void swelling and irradiation induced hardening are caused by change of characteristics of material by neutron irradiation, stress state of material and environmental situation. It has been known that chemical compositions varies at grain boundary (GB) significantly with fluence level and the depletion of Cr element at GB has been considered as one of important factors causing material degradation, especially, IASCC in austenitic stainless steel [1-2]. However, experimental results of IASCC under PWR condition were directly not connected with Cr depletion phenomenon by neutron irradiation [3]. Because the mechanism of IASCC under PWR has not yet been clearly understood in spite of many energetic researches, fundamental researches about radiation induced segregation and depletion in irradiated austenitic stainless steels have been attracted again [4-5]. In this work, an effect of residual microstructure on radiation induced segregation and depletion of alloy elements at GB was investigated in ion irradiated SS316 steel using transmission electron microscope (TEM) with energy dispersive spectrometer (EDS)

2. Experimental

2.1 Experimental material

A commercial SS316 stainless steel was used in this study. The chemical composition of the SS316 steel was given in Table 1. To change residual microstructure of the SS316 steel with minimum changes in grain shape, the ingot was cut into pieces and cold-rolled three times at room temperature. The cold-rolling procedure is shown in Figure 1.

Table I: Chemical composition of investigated SS316 steel

	Cr	Ni	Si	P	Mo	Mn	C	S	Fe
wt%	16.9	10.4	0.53	0.0026	2.1	1.03	0.57	0.001	bal.

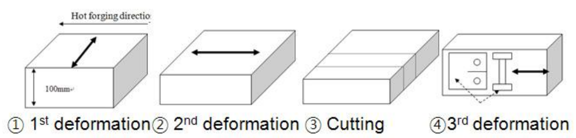

Figure 1 Schematic diagram for cold rolling procedure.

2.2 Fabrication of ion irradiated TEM sample

Atomic displacement damage was introduced by 8MeV Fe^{4+} ions accelerated with an ion accelerator in the Korea Institute of Geoscience & Mineral Resources (KIGAM). The experimental samples were irradiated at the fluence in the order of 5.8×10^{15} ion/cm^2. Irradiation temperature is about 673K~773K. Figure 2 illustrated the thermal history in SS316 steel during ion irradiation the TRIM calculation result about damage profile by ion irradiation. From the TRIM calculation, ion irradiated damage was expected to about 3~5 dpa at 1um depth. Thin foils for TEM observation were prepared with a focused ion beam (FIB) micro-processing device and low energy ion miller. A TEM specimen was fabricated using a FIB micro-processing device to obtain TEM specimen with a specific region. TEM observation was performed by using a 200keV JEOL 2000FXII microscope and JEOL 2100F microscope.

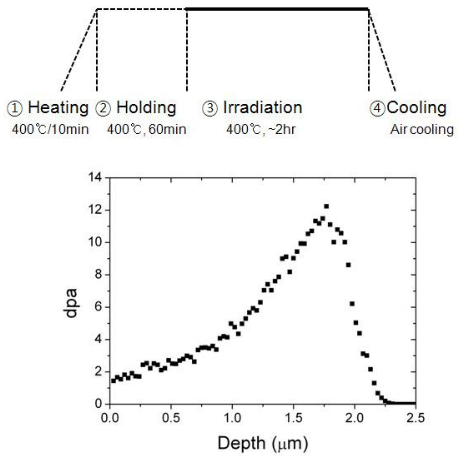

Figure 2 TRIM calculation results

2.3 Quantitative measurement of chemical composition

For precise chemical analysis at GB, proper tilting operation was performed to set the GB plane perpendicular to beam direction. INCA system by Oxford Inc. was used for identification of the chemical compositions at GB using EDS. In quantitative analysis, Cr, Ni, Mo, Si, P and Fe were only considered.

3. Results

It was found that the cold rolled SS316 steel has very narrow interfaces in large grain. They were expected to be caused by the formation of stacking fault and mechanical twin during deformation. In as-received SS316 steel, dislocation density was measured to 1E14 $/m^2$. As a result of cold rolling, the dislocation density of SS316 steel was increased up to 3E15 $/m^2$. Figure 3 shows TEM micrographs of three grain boundaries in as-received SS316 steel and corresponding EDS results in vicinity of GB. It was found that there was typical radiation induced segregation and depletion at GB in ion irradiated SS316 steel. The Cr element at GB was depleted from 20wt% to 14wt% and the Ni element at GB was enriched up to 22wt% .

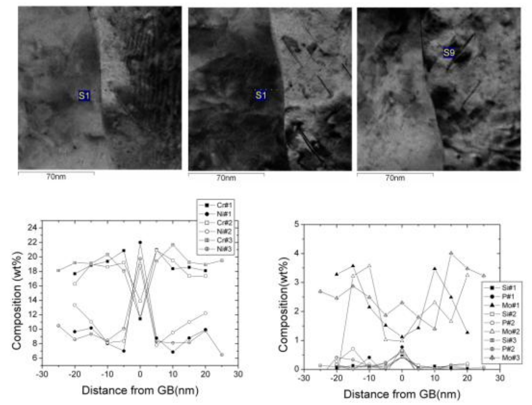

Figure 3 TEM micrographs of three grain boundaries in as-received SS316 steel and corresponding EDS results measured at regular intervals of 5nm in vicinity of GB and with a spot size of 1nm.

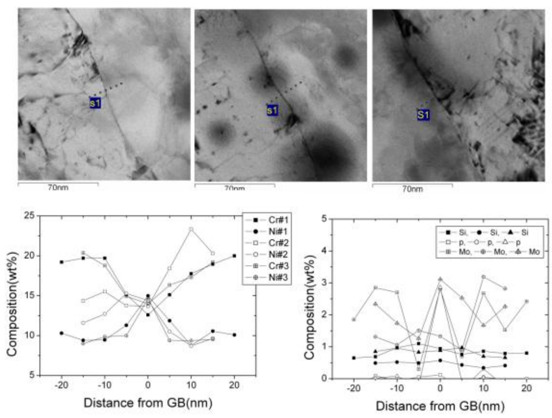

Figure 3 TEM micrographs of three grain boundaries in cold rolled SS316 steel and corresponding EDS results.

In cold rolled SS316 steel with higher dislocation density, Cr depletion and Ni segregation at GB were also observed by TEM-EDS as shown in Figure 4. However, the amounts of Ni segregation and Cr depletion at GB were lower compared with EDS results of as-received SS316 steel.

It seems reasonable to suppose that byproducts such as dislocations, stacking faults and mechanical twins in austenitic stainless steel subjected to deformation are stronger sink sites for annihilation of mobile defects that play key role in movement of alloying element from/to GB under ion bombardment. If threshold value of Cr depletion and Ni enrichment were existed for IASCC initiation, proper control of residual microstructure give rise to slow down irradiation induced degradation phenomena.

4. Conclusions

TEM observation was performed to investigate radiation induced segregation and depletion at grain boundary in SS316 steel. Under an Fe ion irradiation, radiation induced segregation and depletion were developed both in as-received and cold rolled SS316 steels. They were decelerated in cold rolled SS316 steel due to higher population of dislocation, stacking faults and mechanical twins.

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