Evaluation of mechanical properties of helium implanted Inconel X-750 alloys using micro bending test

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1. Introduction

The spacer component in CANDU reactor system manufactured by the X-750 alloy containing high density of nanometer sized gamma (γ`) phase has been known to exhibit intergranular grain boundary (GB) failure after neutron irradiation [1]. It is thought that the GB failure phenomenon seems to be linked with various microstructural changes such as the formation of grain boundary helium bubbles and dislocation loops, and disordering and dissolution of nanometer sized gamma phase (γ`) in matrix. This work focus on mechanical properties change of X-750 alloys after the helium implantation through micro cantilever bending test. The experimental approach in this work is believed to be applied for demonstration of material degradation behavior in irradiated X-750 alloy used for the spacer components.

2. Experimental

2.1 Experimental X-750 alloys

The experimental material in this study is a solution annealed nickel based alloy (Inconel X-750) plate. The chemical compositions of the experimental sample are given in Table 1. Some of X-750 alloy were heat-treated at 730 °C for 16 hours to emulate one of typical microstructures (the formation of γ`) of the annular garter spring spacer component.

Table 1 Chemical composition of the experimental sample

Al	C	Co	Cr	Cu	Fe	
0.72	0.05	0.09	14.85	0.01	6.80	
Mn	**Nb**	**S**	**Si**	**Ta**	**Ti**	**Ni**
0.09	1.03	0.003	0.18	0.01	2.61	Bal.

2.2 Helium implantation

Helium implantation on the Inconel X-750 alloys was performed in the Michigan Ion beam laboratory. Helium ions were used in multiple energies ranges, from 1420 keV to 2820 keV, for the development of uniform radiation damage and implanted ion concentration (~ 1000 appm He) in the ion-implanted samples. The detail helium implantation condition is given in Table 2.

Table 2 Helium implantation condition

Temperature	Helium implantation conditions	
	Energy (keV)	Dose (helium/cm2)
RT	2820 keV	3.1E15
	2640 keV	2.8E15
	2465 keV	2.7E15
	2315 keV	2.4E15
	2170 keV	2.3E15
	2025 keV	2.2E15
	1870 keV	2.1E15
	1720 keV	2.1E15
	1570 keV	2.0E15
	1420 keV	2.0E15

The experimental specimens (as-received and heat-treated) for the helium implantation were prepared by mechanical polishing. The surface of specimens was mechanically wet-polished using SiC sand papers. Fine mechanical polishing was performed with a fine-sized diamond suspension (3 μm and 0.25 μm) and a colloidal silica suspension (0.1 μm).

2.3. Sample preparation for material characterization

We utilized mainly a focused ion beam (FIB) device for fabrication of micro-cantilevers near edge region of the experimental samples. The scanning electron microscopy (SEM) image shown in Fig. 1 represents the micro cantilever for the micro bending test. We obtained the geometric information of the micro-cantilevers through SEM imaging and also conducted crystal orientation analysis with electron backscattered diffraction (EBSD) equipped in SEM.

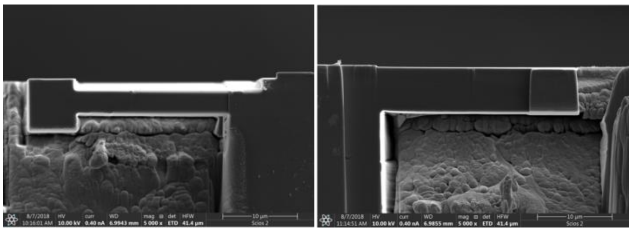

Fig. 1 SEM images of cantilever

2.4 Micro bending test

Micro bending of the micro-cantilevers in the experimental sample was carried out with a nano indenter (UNHT, CSM instruments) installed in KAERI. A berkovich tip was used for the bending test. Loading rate for the micro bending test was set to be 0.5 mN/min. The indentation displacement was limited to be 1-2 μm.

3. Result and discussion

Fig. 2 shows typical radiation damage profile and concentration of residual implanted ions after helium implantation, calculated by the Stopping Range of Ions and Matter (SRIM) computer program [2]. It is found that the uniform layer for radiation damage and implantation is located in the region ranging from 2 ~ 5 μm in depth. The radiation damage is calculated to be approximately 0.1 dpa and the implanted He concentration is also estimated to be approximately 1000 appm in the layer.

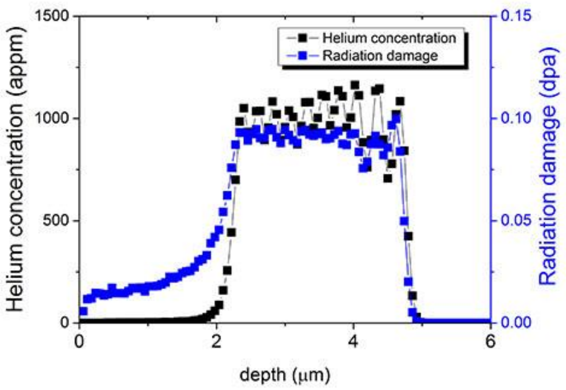

Fig.2 Radiation damage and helium concentration calculated by SRIM

The cantilevers for the micro bending test were fabricated with uniform implanted layer ranging from 2 μm to 5 μm in depth. Load to displacement data of the helium implanted X-750 alloys were obtained by micro-bending test. The applied load obtained from the data was used to calculate the engineering stress (σ) [3].

$$\sigma = \frac{4Fy}{wh^2} \quad \text{(Eq.1)}$$

where F is the applied load, y is the distance to indenting point (moment arm), w is the width of cantilever beam, and h is its thickness. The resolved shear stresses are estimated using the maximum Schmid factors. The Schmid factor for each micro-cantilever was estimated with relationship between the transverse crystal direction (tensile direction under micro-bending) measured by the EBSD analysis and the slip system of face centered cubic material. Fig.3 shows stress - displacement profiles of helium implanted X-750 alloys by micro bending test. The critical resolved shear stress (CRSS) can be determined in the profile of Fig. 3. Note that the average CRSS of the micro-cantilever samples before helium implantation was measured to be 300 MPa for as-received X-750 alloy and, 500 MPa for heat-treated X-750 alloy respectively.

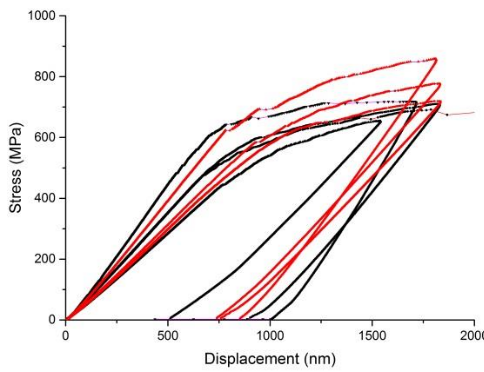

Fig.3 Stress - displacement profiles of the as-received (black) and the heat-treated (red) X-750 alloys after helium implantation by micro bending test

After the multiple energies helium implantation at room temperature, the increase in CRSS value is measured to be approximately 200 MPa for the as-received sample and 100 MPa for the heat-treated X-750 sample respectively. It is expected that the significant hardening is caused by helium bubble formation in the matrix. The hardening of the heat-treated X-750 alloy is measured to be lower. The ordering phase as one of significant hardening contributors is known to become disordered during low temperature irradiation [4]. Hence, the low hardening phenomenon seems to be due to the disordering process of γ`.

4. Conclusions

We performed the micro bending test for evaluation of mechanical properties change of Inconel X-750 alloy after helium ion irradiation. The micro bending test indicates that the helium implantation cause significant hardening and the hardening of X-750 alloy by the helium implantation is dependent to the initial microstructure. Further microstructural characterization will be investigated by TEM examination to better understand the effects of irradiation on microstructure and mechanical properties of X-750 alloy.

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