

Mechanical property evaluation of heat treated Inconel X-750 alloy using small-scaled mechanical testing

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1. Introduction

Inconel X-750 alloy is widely used in the various component parts (springs, pins and bolts) in reactor core of pressurized water reactors and CANDU reactors. Since the components are relatively small in size and have geometric complexity in shape, it is difficult to measure its mechanical properties by conventional methods. Moreover, it is apparent that high energy neutron irradiation produces radioactive isotopes. To overcome the problem, we have been utilizing a small-scale mechanical testing method in the study on material degradation by neutron irradiation. Armstrong et al. [1, 2] showed the feasibility of micro-bending test among small scaled mechanical tests in nuclear material field, presenting experimental results for mechanical behavior of nuclear materials. The experimental approach is believed to be applied for demonstration of mechanical behavior change in irradiated X-750 alloy. In this work, we aim to provide experimental data on microstructure and mechanical property of fresh Inconel X-750 alloy prior to irradiation through micro-bending test and TEM observation. The preliminary experimental data presented in this work will be used for further investigation on interpretation of fracture behavior change in the Inconel X-750 alloy after neutron or ion irradiation.

2. Experimental

2.1 Experimental X-750 alloy

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. Typical heat treatment, so-called No 1 temper, was performed at 730 °C for 16 hours to emulate typical microstructures of the annular garter spring spacer component.

2.2. Sample preparation for micro-compression test

The experimental specimens 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).

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. Sample preparation for micro-compression test

The experimental specimens 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). Mirror surfaces could be obtained on the samples after the final polishing with the colloidal silica suspension. After the fine polishing, we utilized a focused ion beam (FIB) device for fabrication of micro-cantilevers near edge region of the experimental sample. The scanning electron microscopy (SEM) image shown in Fig. 1 represents the fabricated micro cantilever. We obtained the geometric information of the micro-cantilevers.

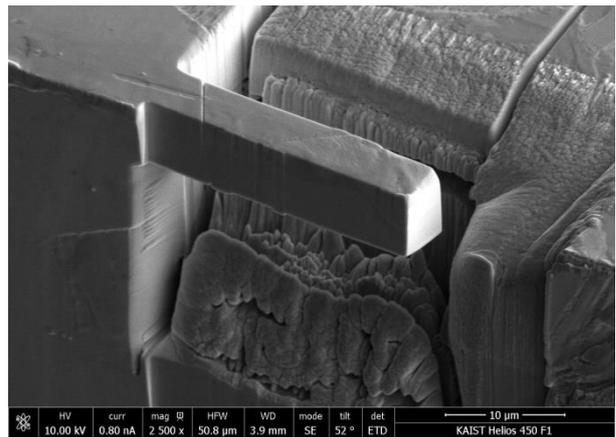


Fig. 1 SEM image of micro cantilever in the X-750 sample

2.3. Micro bending test and TEM analysis

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/sec. The indentation displacement was limited to be around 2 μm . Microstructural changes in the heat treated X-750 alloy were analyzed through analytical transmission electron microscopy (TEM). A Jeol 2100F equipped with energy dispersive spectrometer (EDS) and electron energy loss spectrometer (EELS) in KAERI was utilized for the microstructural analysis.

3. Result and discussion

Load to displacement data by micro-bending test is shown in Fig. 2. 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. The critical resolved shear stress (CRSS, τ_c) was determined to be a value of shear stress at which the stress is no longer proportional to strain in the stress-displacement curves. The average CRSS of the micro-cantilever samples is measured to be approximately 416 MPa with standard deviation of 28 MPa.

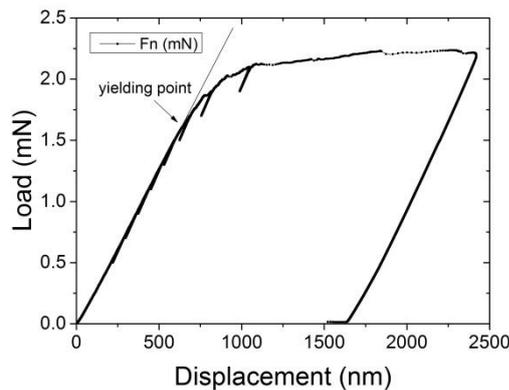


Fig. 2 Typical load to displacement data by micro-bending test

The typical microstructure observed in the No 1 temper heat treated X-750 alloy is shown in Fig. 3. The STEM-EDS analysis indicates that γ' precipitates are composed with Ni, Ti and Al and are distributed uniformly in the matrix. The average diameter of the γ' precipitate is measured to be approximately 20 nm. The number

density of the γ' precipitate is estimated to be approximately $\sim 3 \times 10^{22} \text{ m}^{-3}$.

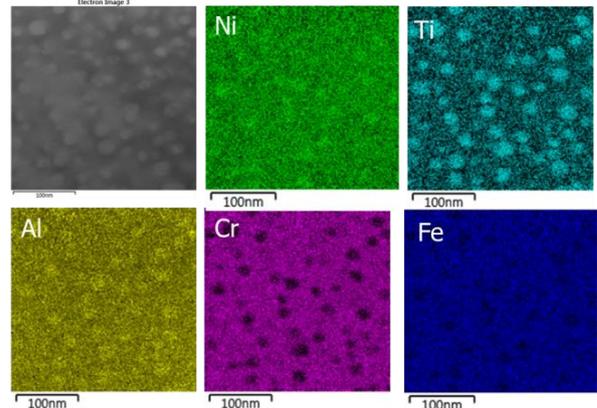


Fig. 3 Typical TEM-EDS mapping result in the heat treated X-750 alloy

4. Conclusions

The current study was intended to obtain mechanical and microstructural information of the heat treated X-750 alloy prior to ion irradiation. The average resolved shear stress measured by the micro bending test indicates that the heat treated X-750 alloy has high strength. Microstructural characterization by TEM shows the high strength of heat treated X-750 alloy is caused by the high density of fine γ' precipitates. This preliminary result will be used as base information to understand the effects of irradiation on microstructure and mechanical properties of X-750 alloy.

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