

## Micro Compression Test in Proton Irradiated Austenitic Stainless Steel

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### 1. Introduction

Pouchon et al. [1], Hosemann et al. [2] and Shin et al. [3, 4] showed the feasibility of micro compression test in nuclear material field, presenting experimental results for various structural materials such as oxide dispersed strengthened steels (ODS), ferritic/martensite steel (FMS) and silicon carbide (SiC), which have been considered as candidate materials of structural components for advanced nuclear reactor system. Evaluation of mechanical property change under neutron or particle irradiation has also been promised as a key issue for the integrity of nuclear reactor or the development of advance nuclear reactor system. In this work, the micro compression test using micro-scaled pillars was conducted to evaluate the yield strength change in proton irradiated layer of austenitic stainless steel.

### 2. Experimental

#### 2.1. Information of proton-irradiated stainless steel

The experimental material in this study is typical austenitic stainless steel having the chemical composition shown in Table 1.

Table 1 Chemical composition of the experimental sample

	Cr	Ni	P	Mo	Mn	Si	S	C	Fe
wt%	16.7	10.8	0.1	2.0	1.3	0.59	0.001	0.047	Bal.

The experimental specimens were prepared by mechanical and electro-chemical polishing for proton irradiation. The surface of specimens was mechanically wet-polished using SiC sand papers and then electro-polished for 15 ~ 30 seconds in a 50% phosphoric acid, 25% sulfuric acid and 25 % Glycerol at room temperature. Proton irradiation was performed with the General Ionex Tandetron accelerator at the Michigan Ion Beam Laboratory at the University of Michigan. The energy of proton used for the irradiation was 2 MeV. The proton irradiation were conducted at a temperature of  $360 \pm 10$  °C. The damage profile for the austenitic stainless steel calculated by the SRIM is shown in Fig. 1. The radiation induced damage by the proton irradiations was expected to be formed up to a

depth of 20  $\mu\text{m}$ . The proton irradiated samples contained plateau radiation damage up to 15  $\mu\text{m}$ .

#### 2.2. Sample preparation for micro-compression test

After irradiation, the samples were cut in half using diamond high speed saw. The proton irradiated samples were bonded with dummy austenitic stainless steel samples using G1-epoxy. Use of dummy sample is to maintain flat edge including radiation damage layer in the proton irradiated samples during following mechanical polishing. FIB milling was done for the fabrication of micro pillars on these determined areas. We prepared micro pillars with diameters of 5 and 10  $\mu\text{m}$  through the FIB milling.

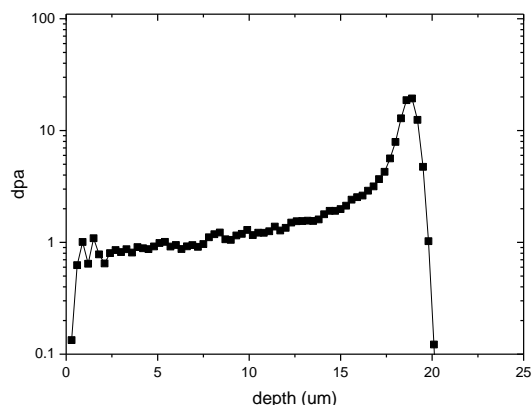


Fig. 1. Radiation damage calculated by SRIM (Full damage mode) [5].

#### 2.3. Micro compression test

Micro compression of the micropillars in the proton irradiated sample was carried out with a nanoindenter (NHT<sup>2</sup>, CSM instruments). A diamond flat punch tip with 20  $\mu\text{m}$  diameter was used for the compression test. Loading rate for the micro compression test was set as 1 mN/sec. The indentation depth was limited to be around 10 ~ 20 % strain for the micro pillars.

### 3. Result and discussion

Fig. 2 present shear stress-strain curves of the proton irradiated sample. Since most micro pillars in this

compression test showed yielding phenomenon above 2.5 % strain, the critical resolved shear stress (CRSS,  $\tau_c$ ) was determined to be a value of shear stress at 2.5 % strain in the shear stress-strain curves. The proton irradiated sample showed  $\sim 230$  MPa in  $\tau_c$  with small standard deviations. Since a value of CRSS in austenitic stainless steel was found to be  $\sim 100$  MPa in the un-irradiation condition [6, 7], the values of increase in CRSS for the proton irradiated sample can be estimated to be  $\sim 130$  MPa.

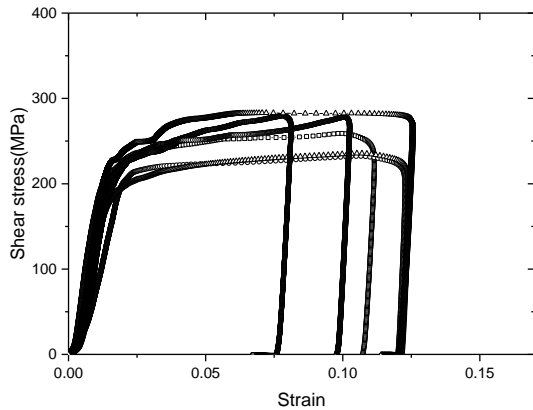


Fig. 2. Shear stress-strain curves of the proton irradiated sample

Yield strength of the proton irradiated austenitic stainless steel can also be evaluated based on the value of  $\tau_c$  measured experimentally in this work. A classical relationship between uniaxial yield strength and the resolved shear stress in poly crystal can be expressed as following equation.

$$\sigma_y = M \cdot \tau_c \quad (\text{Eq.1})$$

where  $M \sim 3.06$  is the Taylor factor [8] and  $\sigma_y$ , yield strength. The value of yield strength of the proton irradiated austenitic stainless steel is evaluated to be  $\sim 700$  MPa according to the Eq. 1. The value is well consistent with experimental data of yield strength of bulk austenitic stainless steel by neutron irradiation [9, 10] and proton irradiation [11].

#### 4. Conclusions

Micro compression test have been used to measure the mechanical properties of commercial austenitic stainless steel. Significant increase in resolved shear stress after the proton irradiation was measured by the micro compression test with various micro-pillars. Also evaluated is the yield strength of the proton irradiated austenitic stainless steel by the micro compression test using small micro-pillars.

#### REFERENCES

- [1]M.A. Pouchon, J. Chen, R. Ghisleni, J. Michler, W. Hoffelner, *Exp. Mech.* 50 pp 79-84, 2010.
- [2]P. Hosemann, J.G. Swadener, D. Kiener, G.S. Was, S.A. Maloy, N. Li, *J. Nucl. Mater.* 375, pp 135-143,2008.
- [3]C. Shin, H.-H. Jin, H. Sung, D.-J. Kim, Y.S. Choi, k. Oh, *Exp. Mech.* 50, pp 79-84, 2010.
- [4]C. Shin, S. Lim, H.-H. Jin, P. Hosemann, J. Kwon, *J. Nucl. Mater.* 444, pp 43-48, 2014.
- [5]J.F. Ziegler, J.P. Biersack, U. Littmark, *The Stopping and Range of Ions in Solids*, Pergamon, New York, 1985.
- [6]G. Monnet, M.A. Pouchon, *Mater. Lett.* 98, pp 128-130, 2013.
- [7]I. Karaman, H. Sehitoglu, H.J. Maier, Y.L. Chumlyakov, *Acta Mater.* 49, pp 3919-3933, 2001.
- [8]U.F. Kocks, *Metall. Trans.* 1, pp 1121,1970.
- [9]G.R. Odette, G.E. Lucas, *J. Nucl. Mater.* 179-181, pp 572-576, 1991.
- [10]K. Tangri, P. Schiller, , *J. Nucl. Mater.* 155-157, pp 1296-1300, 1988.
- [11]G.S. Was, J.T. Busby, T. Allen, E.A. Kenik, A. Jenssen, S.M. bruemmer, J. Gan, A.D. Edwards, P. M. Scott, P.L. Andresen, *J. Nucl. Mater.* 300, pp 198-216, 2002.