# Hardness and Modulus of 410 Martensitic Steel after High Temperature Proton **Irradiation**

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## 1. Introduction

The elevated thermo-mechanical stresses and radiations degrade nuclear power plant materials and emphasize upon careful selection of structural materials [1]. The radioactive service environment generates microscopic defects in the materials such as voids, cavities, stacking faults and interstitial loops etc. [2] and consequently deteriorates the material properties by causing segregation, precipitation, hardening and embrittlement [3].

Due to limitations of Ni-based superalloys, austenitic and ferritic steels such as low thermal conductivity, low weldability, high thermal coefficient, the ferritic martensitic (F/M) steel is used as a structural material in fission-based nuclear power plants. Its applications range from in-core components to piping and ducts [2,4,5,6,7,8]. The solenoid type fuel injector is also manufactured from F/M steel due to its ferromagnetic properties [9].

F/M steels experience irradiation hardening [3] and increase in ductile to brittle transition temperature (DBTT) due to irradiation [10]. Hence irradiation degrades structural component of fission power reactor and put the safe operation at risk [11].

The F/M steel offers several useful characteristics such as good oxidation resistance and creep strength, which drags researchers attention towards its irradiation properties so that its applications can be extended to the nuclear industry [2,5,6,7,12,13].

The present study explains the effects of high temperature proton irradiation of 410 martensitic steel with a fluence of  $1.0 \times 10^{17} \text{ p/cm}^2$  up to irradiation damage of 5.2x10<sup>-3</sup> dpa.

## 2. Methods and Results

#### 2.1 Simulation of irradiation

The irradiation of samples in the reactor is challenging in terms of cost and material handling [4, 12] therefore neutron irradiation is simulated with the help of protons by using proton accelerator [12, 14].

### 2.2 Evaluation of irradiation damage

Due to very low penetration depth, nanoindentation test is used to evaluation the post irradiation mechanical behavior of irradiated samples [12, 14].

#### 2.3 Experimental method

The 410 F/M steel samples were irradiated up to 5.2 x 10<sup>-3</sup> dpa via 3 MV pelletron accelerator at Michigan Ion Beam Laboratory (MIBL). The ion beam accelerator, equipped with a quadrupole focusing unit, is capable of irradiating up to an area covering 100 mm<sup>2</sup>. During irradiation, the temperature of the samples was maintained at 632 Κ with the help of temperature-variable sample holder. The dose was increased up to 1 x 1017 p/cm2. The pre- and postirradiation mechanical behavior of the 410 F/M steel was evaluated via nanoindentation test.

### 2.2 Results

Irradiation hardening is considered to be the primary irradiation defect. In order to evaluate irradiation hardening on the thin lamellas of 410 martensitic steel, the nanoindentation tests where carried out. For the comparison of mechanical properties the same test was performed on unirradiated samples. Fig. 1 (a) and (b) show the depth profiles of hardness and modulus, respectively, of un-irradiated 410 F/M steel samples. The test was repeated at least six times while keeping the test parameters same, so that the reliability of data can be ensured.



Fig. 1. Indentation depth profiles of (a) hardness and (b) modulus, comparison of (d) hardness and (e) modulus at 250-350 nm and 700 nm depth of un-irradiated 410 martensitic steel.

Due to indentation size effect [15], the hardness and modulus values can be seen decreasing with depth. The average values at the depth 250-350nm and 700nm is shown in Fig. 1 (c) and (d).

Fig. 2 shows the depth profiles of the same mechanical parameters i.e. hardness and modulus of the samples irradiated at 632 K up to  $5.2 \times 10^{-3}$  dpa via 3 MeV protons and  $1 \times 10^{17}$  p/cm<sup>2</sup>.



Fig. 2. Indentation depth profiles of (a) hardness and (b) modulus, comparison of (d) hardness and (e) modulus at 250-350 nm and 700 nm depth of 410 martensitic steel irradiated up to  $5.2 \times 10^{-3}$  under the  $10^{17}$  p/cm<sup>2</sup> fluence of 3 MeV protons.

Insignificant variation in hardness and modulus values after irradiation can be seen in Fig. 2.

The hardness and modulus values at 250-350 nm, which were  $4.72\pm0.12$  GPa and  $199.89\pm5.57$  GPa, before irradiation, experienced a slight change due to irradiation and became  $4.56\pm0.17$  GPa and  $204.20\pm16.75$  GPa, respectively. Similar insignificant variation in hardness and modulus values at 700 nm was also observed. Table

1 summarizes the pre-and post-irradiation hardness and modulus data at various indentation depth.

Table I: Comparison of Pre- and Post-Irradiation Average Hardness and Modulus Values at Various Indentation Depths

	Indentation depth 250-350 nm		Indentation depth 700 nm	
	Un- irradiated	Irradiated	Un- irradiated	Irradiated
Hardness (GPa)	4.72 <u>+</u> 0.12	4.57+0.17	3.44 <u>+</u> 0.06	3.73 <u>+</u> 0.17
Modulus (GPa)	199.89 <u>+</u> 5.57	204.20+16.75	154.43 <u>+</u> 6.47	163.71 <u>+</u> 19.31

The variation in mechanical properties of 410 F/M steel after irradiation remained insignificant, therefore the safe operation of a structural component of fission power plants made up of 410 F/M steel can be ensured up to damage level of  $5.2 \times 10^{-3}$  dpa.

#### 3. Conclusions

The neutron irradiation of  $3.5 \times 10^{18} \text{ n/cm}^2$  was simulated via 3 MeV proton irradiation up to  $10^{17} \text{ p/cm}^2$ . Post irradiation hardness and modulus, determined by nanoindentation were compared with the same mechanical characteristics of unirradiated material. Insignificant variation in mechanical behavior due to irradiation damage was found which ensures the reliable utilization of 410 F/M steel up to the  $5.2 \times 10^{-3}$  dpa in fission power plants.

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#### REFERENCES

 R. J. Kurtz, A. Alamo, E. Lucon, Q. Huang, S. Jitsukawa, A. Kimura, R. L. Klueh, G. R. Odette, C. Petersen, M. A. Sokolov, P. Spatig, and J. W. Rensman, Recent Progress Toward Development of Reduced Activation Ferritic/Martensitic Steels For Fusion Structural Applications, Journal of Nuclear Materials, Vol. 386–388, p. 411–417, 2009.
 Y. Chen, Irradiation Effects of HT-9 Martensitic Steel, Nuclear Engineering and Technology, Vol. 45, p. 311–322, 2013.

[3] J. L. Straalsund, R. W. Powell, and B. A. Chin, An Overview of Neutron Irradiation Effects in LMFBR Materials, Journal of Nuclear Materials, Vol. 108–109, p. 299–305, 1982.
[4] S. A. Maloy, T. J. Romero, P. Hosemann, M. B. Toloczko, and Y. Dai, Shear Punch Testing of Candidate Reactor Materials After Irradiation in Fast Reactors and Spallation

Environments, Journal of Nuclear Materials, Vol. 417, p. 1005-1008, 2011.

[5] B. H. Sencer, J. R. Kennedy, J. I. Cole, S. A. Maloy, and F. A. Garner, Microstructural Analysis of an HT9 Fuel Assembly Duct Irradiated in FFTF to 155 dpa at 443 C, Journal of Nuclear Materials, Vol. 393, p. 235–241, 2009.

[6] A. Sarkar, A. H. Alsabbagh, and K. L. Murty, Investigation of Microstructure and Mechanical Properties of Low Dose Neutron Irradiated HT-9 Steel, Annals of Nuclear Energy, Vol. 65, p. 91–96, 2014.

[7] O. Anderoglu, T. S. Byun, M. Toloczko, and S. a. Maloy, Mechanical Performance of Ferritic Martensitic Steels for High Dose Applications in Advanced Nuclear Reactors, Metallurgy Material Transaction A, Vol. 44, p. 70–83, 2012.

[8] Y. Hamaguchi, H. Kuwano, H. Kamide, R. Miura, and T. Yamada, Effects of Proton Irradiation on the Hardening Behavior of HT-9 Steel, Journal of Nuclear Materials, Vol. 133–134, p. 636–639, 1985.

[9] J.-W. Lee, S. Surabhi, S.-G. Yoon, H. J. Ryu, B.-G. Park, Y.-H. Cho, Y.-T. Jang, and J.-R. Jeong, Study on Proton Radiation Resistance of 410 Martensitic Stainless Steels Under 3 MeV Proton Irradiation, Journal of Magnetics, Vol. 21, p. 183–186, 2016.

[10] P. Chakraborty and S. B. Biner, Parametric Study of Irradiation Effects on the Ductile Damage and Flow Stress Behavior in Ferritic-Martensitic Steels, Journal of Nuclear Materials, Vol. 465, p. 89–96, 2015.

[11] Y. E. Kupriiyanova, V. V. Bryk, O. V. Borodin, A. S. Kalchenko, V. N. Voyevodin, G. D. Tolstolutskaya, and F. A. Garner, Use of Double and Triple-Ion Irradiation to Study the Influence of High Levels of Helium and Hydrogen on Void Swelling of 8–12% Cr Ferritic-Martensitic Steels," Journal of Nuclear Materials, Vol. 468, p. 264–273, 2015.

[12] P. Hosemann, C. Vieh, R. R. Greco, S. Kabra, J. A. Valdez, M. J. Cappiello, and S. A. Maloy, Nanoindentation on Ion Irradiated Steels, Journal of Nuclear Materials, Vol. 389, p. 239–247, 2009.

[13] A. F. Rowcliffe, J. P. Robertson, R. L. Klueh, K. Shiba, D. J. Alexander, M. L. Grossbeck, and S. Jitsukawa, Fracture Toughness and Tensile Behavior of Ferritic–Martensitic Steels Irradiated at Low Temperatures, Journal of Nuclear Materials, Vol. 258–263, p. 1275–1279, 1998.

[14] X. H. Li, J. Lei, G. G. Shu, and Q. M. Wan, A Study on the Microstructure and Mechanical Property of Proton Irradiated A508-3 steel, Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms, Vol. 350, p. 14–19, 2015.

[15] L. Jing, D. Hui, S. Guo-gang, W. Qiang-mao. Study on the Mechanical Properties Evolution of A508-3 Steel under Proton Irradiation, Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms, Vol. 338, p. 13–18, 2014.