

## Application of proton irradiation for study on radiation damage of reactor pressure vessel steel

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### 1. Introduction

Radiation hardening (RH) or embrittlement (RE) of reactor pressure vessel (RPV) is considered as one of detrimental degradations against integrity of pressurized water reactor (PWR) for the long term operation [1]. In-depth analyses on radiation induced microstructural change of RPV steels has been progressed using advanced microstructural characterization techniques such as analytical TEM and 3D-APT to demonstrate radiation hardening mechanism of RPV steel in commercial nuclear reactors [2, 3]. It is evident that accurate trend on radiation hardening and embrittlement of the RPV will be predicted for nuclear plant life extension. In this work, radiation hardening behavior of commercial RPV steel has been investigated through three dimensional atom probe tomography (3D-APT) and micro-mechanical testing (Indentation, micro-indentation). Irradiated experimental samples were prepared through proton irradiation to emulate neutron irradiation effect. The indentation techniques were conducted to evaluate yield strength increase by the proton irradiation indirectly. Analytical microstructure analyses were used for identifying sources to generate radiation hardening of the proton-irradiated RPV steel.

### 2. Experimental

#### 2.1 Materials

The composition of the experimental RPV steel is given in Table 1. Before the proton irradiation, mechanical polishing with colloidal silica suspension was conducted for damage-free surface.

Table 1 Chemical compositions of the test alloy (wt%)

C	Mn	Ni	Si	Cr	Mo	Cu	Fe
0.19	1.42	0.86	0.04	0.14	0.51	-	Bal.

#### 2.2 Proton irradiation

Proton irradiations were conducted at the Michigan Ion beam Laboratory in the University of Michigan. The General Ionex Tandatron accelerator was used for the proton irradiations. The energy of proton used for the irradiation was 2 MeV. The proton irradiation were conducted at a temperature of  $300 \pm 5$  °C. These steels were irradiated up to 0.55 dpa at 300 °C. Three irradiated samples were prepared for the 3D-APT analysis and hardness measurements. The calculated

radiation damage (dpa) is shown in Fig. 1. The calculation was performed with full cascade mode in the SRIM code [4]. In the calculation, the displacement energy was set to be 40 eV [5]. According to Fig. 1 of SRIM calculation result, the radiation damage remained constant up to around 15  $\mu\text{m}$  in depth. There was a steep rise at 18 ~ 20  $\mu\text{m}$  in depth.

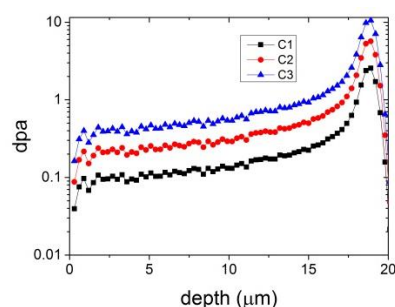


Fig. 1 Radiation damage profiles by calculation of SRIM code

#### 2.3. The preparation of analytical sample

To gain information on radiation hardening and radiation induced microstructure changes of proton-irradiated RPV steel, cross-sectional experimental samples were prepared through the following procedure. First, the proton-irradiated surface of irradiated samples was bonded with a dummy bar of un-irradiated RPV steel. Mechanical polishing on the bonded sample was performed with fine-sized diamond suspensions (3 and 0.25  $\mu\text{m}$ ) and a colloidal silica suspension. After the final polishing with the colloidal silica suspension, slight etched surface can be obtained. The purpose of this procedure was to inspect radiation hardening behavior as a function of radiation damages (dpa) conducting micro-mechanical test (nano-indentation) in the proton irradiated layer. We also prepared analytical samples for 3D-APT experiment from the cross-sectional samples using FIB milling.

### 3. Result and discussion

#### 3.1 Radiation induced hardening

Nano hardness change profile can be plotted as a function of radiation damage value (dpa) as shown in the fig. 2(a) based on the nano-hardness changes measured by 5 mN indentations. It is apparent that the magnitude of radiation hardening increases gradually with radiation damage value (dpa). There is no sudden

change in hardness value up to 0.8 dpa. The radiation hardening resulting from proton-irradiation is also presented in terms of yield strength increase in Fig. 2(b). The yield strength increase of proton-irradiated RPV steels in Fig. 2(b) was calculated from hardness measurement using the following relation [7] :

$$\Delta\sigma_y(MPa) = 3.06 \Delta H_v \left( \frac{kg}{mm^2} \right)$$

The yield strength changes of RPV steel measured after neutron irradiations are also plotted in the Fig. 2(b), which agree with those measured in this work.

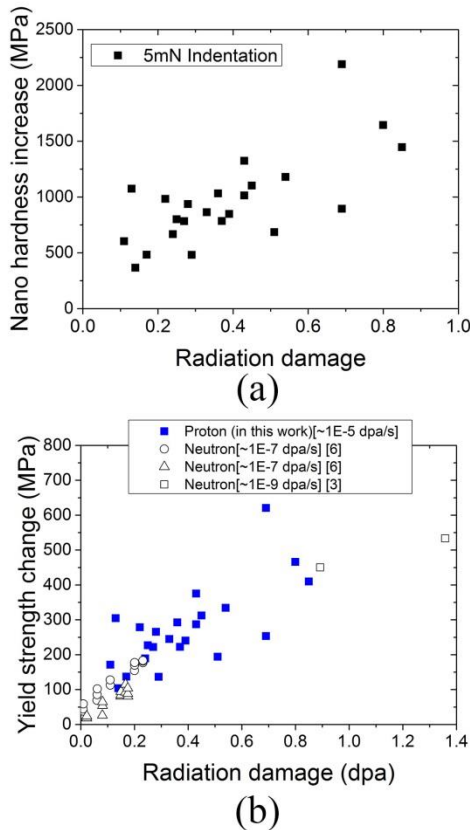


Fig. 2 Hardness change profile (a) and yield strength increase profile (b) as a function of dpa

### 3.2. Radiation induced microstructural changes

In the proton irradiated RPV steels, radiation induced clusters are observed in the matrix through 3D-APT examination. They were seen as Mn-Ni-Si enriched clusters according to the 3D-APT analyses. The spatial distribution of solute cluster was found to be homogeneous at high fluence. Solute cluster density is sensitive to dose. Average radius of the solute cluster is increased with dose up to about 0.5 dpa. The characteristic of solute clusters analyzed in this work are compared with those developed by neutron irradiation as shown in Fig. 3.

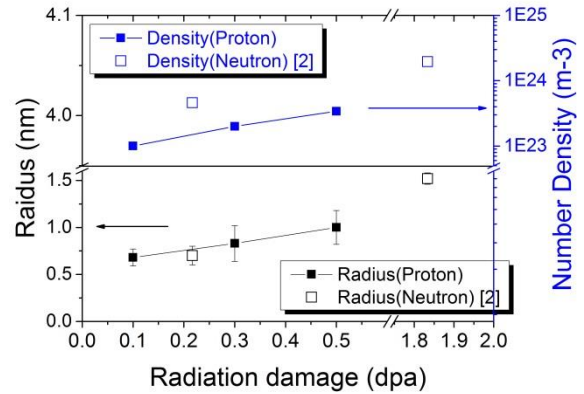


Fig. 3 Average radius and number density of solute cluster in proton-irradiated RPV steels searched by APT analysis

### 4. Conclusions

Radiation hardening behavior of proton-irradiated RPV steel was investigated through nano-indentation measurement and 3D-APT characterization.

- (1) Nano hardness measurements showed that hardness changes gradually increased with radiation damage value (dpa).
- (2) APT analyses indicated that the formation of Mn-Ni-Si enriched cluster play key role in developing radiation hardening of the proton-irradiated RPV steel.
- (3) There is little difference in the magnitude of hardening and the characteristics of solute cluster between proton-and neutron-irradiated RPV steel.

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