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# Radiation-induced shear bands enhance softening 316LN austenitic stainless steel

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ABSTRACT

Radiation hardening is usually a major concern for materials used for nuclear reactors. Here, we report that helium (He) radiation could weaken the radiation hardening effect and even soften 316LN austenitic stainless steel by forming shear bands. The experiment results showed that the nanohardness of the steel decreased sharply after radiation by 200 keV He ion to a fluence of $2.3 \times 10^{21}$ ions/m$^2$. Moreover, it is noteworthy that the number and spacing of shear bands can be controlled indirectly by forming the radiation bubbles with appropriate size and distribution, which is hopeful to extend the application of He bubble engineering.



## 1. Introduction

316LN austenitic stainless steel (316LN SS) with an exceptional combination of mechanical and corrosion properties is suggested to produce first wall in an international thermonuclear experimental reactor and primary components in the future generation of nuclear power plant (Kim et al., 2009; Kumar et al., 2017). Compared with 316-type stainless steels, the strength and fatigue resistance of the 316LN SS with 0.12 wt% nitrogen element are improved without reducing the plasticity and toughness (Wang et al., 2016). However, the (n, a)-reactions occurred during the neutron radiation will form definitely large number of He bubbles. These bubbles tend to accumulate along grain boundaries, resulting in macroscopic embrittlement (Trinkaus et al., 2003). Radiation embrittlement of metallic materials is usually linked to hardening of the metals, hence the phrase "Radiation damage" (Sarkar et al., 2014). In the present work, based on nanoindentation experiments, we found that He radiation can actually soften 316LN SS. Nanoindentation results showed that the hardness and Young's modulus of the steel decreased with increasing $He^{2+}$ ion radiation doses from 0.33 dpa (displacements per atom) to 5.40 dpa.

## 2. Material and methods

The material used for the present study was the 316LN SS with average grain size of 146 μm, which was cut from AP1000 primary coolant pipe first and then solution annealed at 1050 °C for 90 min. Chemical composition of the steel is given in Table 1. The samples with a size of $5 \times 5 \times 2.5$ mm$^3$ were irradiated at room temperature with 200 keV $He^{2+}$ ions at fluences ranging from $1.4 \times 10^{20}$ to $2.3 \times 10^{21}$ m$^{-2}$. Before radiation, the specimens were polished to mirror-like with mechanical method. The depth profile of the local damage dose was determined by using SRIM program (Stoller et al., 2013). The simulation result predicts that the damage peak induced by $He^{2+}$ collisions in the steel is ~5.40 dpa at a depth of ~500 nm.

The nanoindentation tests were performed by using Nano Indenter XP (Agilent Technologies Inc.) with a Berkovich type indentation tip at room temperature. The strain rate was 0.05 s$^{-1}$, and the surface approach velocity was 10 nm/s. The continuous stiffness measurement (CSM) technique was used to obtain the hardness (H) as a function of indenter displacement up to 2000 nm. At least six points were tested for each sample in order to ensure the repeatability. The sample morphologies after the nanoindentations were observed by using a SUPRA55 scanning electron microscope (SEM). The microstructural characterization of as-irradiated samples was investigated via a Tecnai F20 transmission electron microscopes (TEM). The TEM samples were prepared using the focused ion beam 'lift-out' technique.

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**Table 1**
Chemical composition of 316LN SS (wt%).

| C | Si | Mn | P | S | Cr | Ni | Mo | Cu | N | Fe |
|---|---|---|---|---|---|---|---|---|---|---|
| 0.01 | 0.24 | 1.3 | <0.02 | <0.004 | 17.18 | 13.12 | 2.23 | 0.12 | 0.12 | Balance |

## 3. Results and discussions

### 3.1. Hardness and modulus measurements

Fig. 1a shows the hardness decreases with the increased indentation depth. The deviation of the repeated test is slight and the experimental results are reproducible. The indentation size effect (ISE) makes the sample difficult to get the credible hardness. To obtain the characteristic hardness values, Nix-Gao model was used in this study, and it is given by (Nix and Gao, 1998): $H = H_0\sqrt{1+\frac{h^*}{h}}$. Where $H$ is the hardness for a given depth of indent, $h$. $H_0$ is the hardness in the limit of infinite indent depth. $h*$ is a characteristic length scale. The hardness fitted curves (solid lines) obtained from the Nix-Gao model are given in Fig. 1b and c. In order to eliminate surface artifacts and soft substrate effect during the hardness test (Manika and Maniks, 2008; Fave et al., 2014), 1/h range of 2–5.6 $\mu m^{-1}$ in the $H^2$ versus 1/h plot was selected for the hardness fitting.

Fig. 1d shows the change trends of hardness and modulus values for the irradiated steel. When the damage dose reached 0.70 dpa, the radiation hardening effect of the 316LN SS was weakened. This weaken effect was enhanced when the damage dose reached 2.82 dpa. With increasing damage dose up to 5.40 dpa, the hardness of the sample decreased to 1.62 GPa from 2.47 GPa of the sample irradiated 2.82 dpa. As shown in the Damage dose-Modulus curve, the change trends of the elastic modulus were consistent with the hardness. Chakoumakos et al. (1991) showed that the elastic modulus of $ZrSiO_4$ decreased linearly with the increased α-decay dose. Yang et al. (2007) indicated that the existence of nano-bubbles resulted in a decreased elastic modulus for single crystalline materials. Therefore, the softening effect found in this study may be related to the bubbles introduced by the radiation. To analyze the softening behavior of the irradiated samples, the indentation morphologies were observed by SEM.

### 3.2. Microstructural analysis

Fig. 2a shows the force diagram of metallic material with a Berkovich type indentation tip. The normal stress and shear force can be described as Sneddon (1965):

$$\sigma_{zz}(r,0) = \frac{1}{2}E_r \cot\alpha \cdot ar\cosh\left(\frac{a}{r}\right) \quad (1)$$

$$\sigma_{rz}(0,z) = \frac{E_r a^2 \cot\alpha}{2(a^2+z^2)} \quad (2)$$

where $a$ is the contact radius, α is the half cone angle of the indenter, $E_r$ is reduced modulus. As shown in Fig. 2b, no slip step or shear band was found in the as-received sample during in the indentation. When irradiated at 0.33 dpa, the steel showed highly localised slip steps with regular spacing (Fig. 2c). Outside the indentation, the

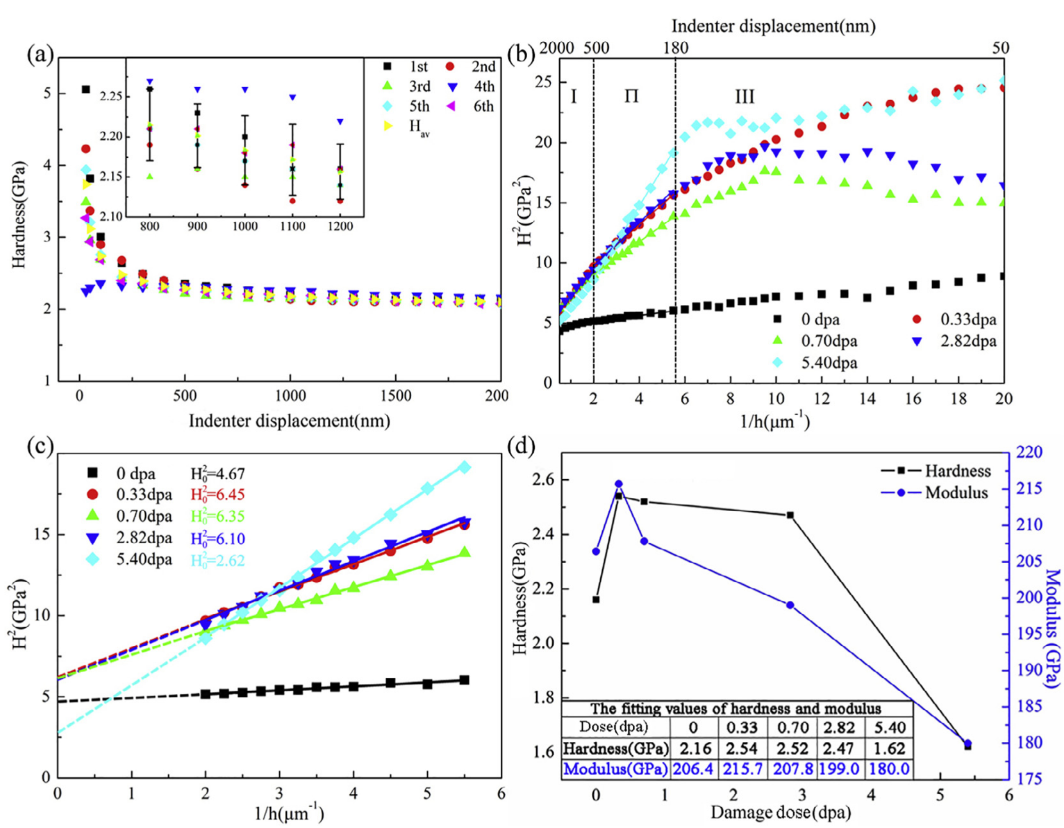


**Fig. 1.** Results from the nanoindentation on the 316LN SS. (a) The deviation of the repeated tests for as-received steel; (b) Squared hardness versus reciprocal indent depth; (c) The magnified image of area II from (b); (d) The fitting values of hardness and modulus.