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Effects of helium production, displacement damage on mechanical properties and surface acoustic wave in austenitic stainless steels and martensitic steel

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ABSTRACT

The effects of helium production, displacement damage on mechanical properties and microstructures were investigated in some specimens such as austenitic stainless steels of SUS304 and SUS316 and a high chromium martensitic steel of HCM12A steels irradiated in a high fluence irradiation facility (HIT) of the University of Tokyo. The simultaneous dual ion (nickel plus helium ions) irradiations were adopted to simulate nuclear irradiation environments such as fast reactor and fusion reactor at HIT to 1 or 10 dpa and to 0, 1, 10, and 100 appm-He at 500, 550, and 600 °C. Thin foils for a transmission electron microscopy (TEM) were prepared with a focused ion beam (FIB) micro-sampling system. After the irradiation, the microstructures were observed by a transmission electron microscope and the irradiation hardening was measured by nano-indentation technique. In this study, the efficient of non-destructive measurement method of surface acoustic wave (SAW) excited by laser beam on the detection of radiation hardening behavior was also examined. Irradiation hardening was observed and the increment was tended to increase with dpa and He/dpa, which was especially effective in the condition of 100 appmHe/dpa even at 550 °C. Non-linear effect on amplitude of the excited SAW was also observed on the ion irradiated materials, and the propagation velocity of SAW was tended to be increased with irradiation dose. It was found that the behaviors of SAWs results were correlated with the changes of mechanical properties.

1. Introduction

Reactor vessels and core internals in nuclear power plants are continuously exposed to radiation such as neutrons and gamma-rays and relatively mild. The exposure durations are relatively long and these structures are difficult to be replaced. The proper management of irradiation damages on the structure materials is important to ensure the health of long-life nuclear plants. Irradiation dose is known to be related to irradiation hardening and embrittlement [1–14]. Transmutation product, helium, can be also affected on mechanical properties and microstructural changes. Therefore, irradiation dose and transmutation helium content are recognized as key parameters to be strongly influenced on the mechanical properties. Austenitic stainless steel and high-chromium martensitic steel are considered as the structural materials in nuclear power plants. It is very important to evaluate the irradiation damage and helium content for the life of the power plants.

In fast breeder reactor (FBR) plant, a design life of 60 years is planned in Japan. The irradiation damage and the transmutation product helium content in the structural materials of the FBR plants is assumed up to about 1 dpa and about 30 appm under the operation at 550 °C [15]. In some recent studies, the effects of irradiation damage and helium content on the mechanical properties were examined [15–18], and the relationships between irradiation damage and non-destructive tests were investigated [19–23]. The former studies were done by reactor irradiation such as JOYO and JRR-3 M and cyclotron helium implantations. The later studies were performed by magnetic flux density measurement and surface acoustic wave measurement.

Non-destructive, non-contact diagnostic methods for nuclear materials are crucial to avoid radioactive contamination. Non-destructive methods are beneficial in that they do not disturb the specimen

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regardless of the physical distance of the measurement. Non-contact methods ensure that the specimen continues functioning throughout the measurement process. For a diagnostic system to evaluate mechanical properties of material surfaces, surface acoustic wave (SAWs) are an ideal non-contact method. SAWs, are also known as Rayleigh waves, propagate at about 0.93 the velocity of transverse wave. SAWs propagate about one wavelength near the surface of the medium. The SAWs propagation velocity Vr [24] is given approximately by the following Eq. (1):

$$Vr = 0.93Vs = 0.93(G/\rho)^{0.5} \qquad (1)$$

where Vs is the velocity of the bulk wave (shear wave), G is the shear modulus and ρ is the density of the medium. The calculated values on 316 austenitic stainless steel (ρ = 8.0 g/cc, G = 77.0 GPa) and HCM12A martensitic steel (ρ = 7.84 g/cc [25], G = 84 GPa [26])) are Vs (316) = 2.85 × 10^3 m/s, Vs (HCM12A) = 3.0 × 10^3 m/s. Here, supplemental factors, such as temperatures and crystal directions are not taken into account. The vibration velocity in the normal direction of SAWs increases linearly as the excited laser power density increases [19,20].

In this study, the effects of helium production, displacement damage on mechanical properties and microstructures in austenitic stainless steels and martensitic steel were investigated as a function of displacement per atom (dpa), helium content, and He/dpa by mechanical tests and non-destructive diagnostic measurements.

2. Experimental

In this study, the examination specimens were mainly SUS304 and SUS316, and HCM12A (a high chromium martensitic steel), and the chemical compositions used in this study are given in Table 1. The specimens for the ion irradiation were mechanically polished by the following series: SiC paper up to #4000, and 3 μm diamond powder, and then 0.3 and 0.05 μm alumina powder, and finally completed by electrolytic surface finish. The 20 mm × 5 mm square plates were mainly used for ion irradiation, and the area of 10 mm × 5 mm was measured for the SAW velocity. Ion irradiation was conducted at HIT facility [27] operated by the University of Tokyo. 4.0 MeV Ni^{3+} ions for 316FR steel and SUS304 steel or 2.8 MeV Fe^{2+} ions for HCM12A steel were ejected from the Van de Graff accelerator, and He^+ ions were ejected from the 1 MV Tandem accelerator. The helium injection ratio to dpa (He/dpa) was set to (0–100) appm He/dpa at the depth between 500–700 nm from the incident surface with an energy degrader for 1 MeV He^+ ions [28]. The both ions were irradiated onto the target simultaneously. A Ni foil energy degrader was used for helium. The conditions of HIT ion irradiation are given in Table 2, and the temperatures were ranged from 500 °C to 600 °C. The displacement damage was calculated by the TRIM code assuming a 40 eV average displacement threshold energy. The typical damage rate was about 4 dpa/h.

An FIB processor (Hitachi FB-2000A) equipped with a micro-sampling system was used for ion machining and specimen manipulation. After the FIB machining, electro-polishing for a short period (tens of milliseconds) was applied to remove the FIB damage. The microstructures of the irradiated specimens were observed by a transmission electron microscope (TEM), HF-2000, operated at 200 kV. The irradiated specimens were indentation-tested at a load of 7 mN using UMIS-2000 (CSIRO, Australia) ultra-micro-indentation testing system. The direction of indentation was chosen to be parallel to the ion-beam axis, or normal to the irradiated surface. The indenter tip was triangular pyramid the Berkovich. The micro-hardness (H) was analyzed in the manner outlined by Oliver and Pharr [29] and Mencik and Swain. The indentation load was controlled at 7 mN, and the incident depth was about 250 nm. The indentation depth was basically determined from Kato's analysis between indentation depth and micro-hardness in Austenitic stainless steel irradiated by dual ion beams [30]. The indentation data for each measurement were obtained about 30 points, and the average value was evaluated.

Table 2
HIT ion irradiation conditions.

He-appm/dpa	Temperature (°C)	316FR	SUS304	HCM12A
		Displacement damage (dpa)		
0	500	1, 10	–	1, 3, 10
0	550	1, 3, 10	1, 3, 10	10
0	600	1,10	–	–
1	500	1, 10	–	1, 10
1	550	1, 3, 5, 10	1, 10	10
1	600	1,10	–	–
10	500	1, 10	10	10
10	550	1, 3, 5, 10	1, 10	1, 10
10	600	1,10	–	–
100	500	–	–	–
100	550	1	–	–
100	550	10	10	10
100	600	–	–	–
10,000	550	0.005	–	–

In this study, a laser-induced and laser-detected surface acoustic wave (SAW) was adopted as a non-destructive diagnostic system as given in Fig. 1. In the system, the SAWs were excited on the surface of specimen by irradiation of second harmonic wave of pulsed YAG laser (532 nm, 4 ns). The maximum pulse power of this system is 100 mJ, and it was applied up to 60 mJ for the measurement in this study. The SAWs were detected by a laser Doppler vibrometer using optical heterodyne method. The SAWs propagation velocity and the SAWs vibration velocity along the normal direction of the surface were measured to investigate mechanical properties of the substrates. Change of the shear modulus was detected in the annealing substrates. Non-linear effect on amplitude of the excited SAW was observed on the ion irradiated materials. The SAWs were detected by a laser Doppler vibrometer using optical heterodyne method. The behaviors of SAWs results were correlated with the changes of mechanical properties. Typical waveform of SAW is shown in Fig. 2. The horizontal axis is time, and the vertical axis is normal surface velocity in the vertical direction towards laser detection in m/s. The upper waveform is 0.5 mm further from the laser excitation point to the direction point than lower one. The irradiated pulse laser can produce SAWs, various modes of bulk waves and air shock waves on the specimen surface. SAWs exhibit approximately the same waveform for each pulse. Bulk waves exhibit different waveforms for each pulse, which can be isolated from SAWs by averaging 64-accumulations. Further, the waveform of the shock wave due to the air

Table 1
Chemical compositions used in this study (wt.%).

Material	C	Si	Mn	P	S	Cu	Ni	W	Cr	Mo	V	Nb	N	B
316FR	0.01	0.59	0.84	0.026	0.003	0.26	11,19	–	16.87	2.23	0.08	–	0.08	0.0006
SUS304	0.05	0.60	0.87	0.026	0.002	0.09	8.94	–	18.59	–	0.05	–	0.022	0.0005
HCM12A	0.11	0.27	0.64	0.016	0.002	1.02	0.39	1.89	10.83	0.30	0.19	0.054	0.063	0.0031