



## Effects of residual stress on irradiation hardening in stainless steels

N. Okubo\*, Y. Miwa, K. Kondo, Y. Kaji

Japan Atomic Energy Agency, Tokai-mura, Ibaraki-ken 319-1195, Japan

### A B S T R A C T

Effects of residual stress on irradiation hardening were studied in advance for predicting irradiation assisted stress corrosion cracking. The specimens of SUS316 and SUS316L with several % plastic strains, which correspond to weld residual stress, were prepared by bending and keeping deformation under irradiation. Ion irradiations of 12 MeV Ni<sup>3+</sup> were performed at 330, 400 and 550 °C to 45 dpa. No bended specimen was simultaneously irradiated with the bended specimen. The residual stress was estimated by X-ray residual stress measurements before and after the irradiation. The micro-hardness was measured by using nanoindenter. The residual stress did not relax even for the case of the higher temperature aging at 500 °C for the same time of irradiation. The residual stress after ion irradiation up to high dpa, however, relaxed at these experimental temperatures. The irradiation hardening of stressed specimen was obviously lower than that of un-stressed one in case of SUS316L irradiated at 300 °C to 12 dpa.

© 2009 Elsevier B.V. All rights reserved.

### 1. Introduction

Structural materials used in fusion reactor with water cooling system will undergo corrosion in aqueous environment and more irradiation fluence than those in LWR. Irradiation assisted stress corrosion cracking (IASCC) will be induced in austenitic stainless steels exposed in these environments for a long term of reactor operation. The IASCC is considered to be caused mainly in a welding zone, where irradiation hardening, irradiation induced stress relaxation, radiation induced segregation and swelling are occurred with each time (or displacement damage: dpa) dependent behavior. The irradiation hardening and the radiation induced segregation increase with increasing the damage level [1,2]. The swelling increases rapidly above a threshold damage level [3]. On the other hand, the residual stress (tensile and/or compressive stress) induced by the welding decreases with increasing the damage level [4,5]. In order to predict the life time of the structural materials undergoing IASCC, it is necessary to evaluate and simulate the synergistic effect of these irradiation behaviors. In this study, the behavior of irradiation induced stress relaxation and irradiation hardening were investigated, prior to evaluating intricate IASCC.

### 2. Experimental procedures

Specimens used in this study were austenitic stainless steels, SUS316 and SUS316L. Chemical compositions of the specimens are given in Table 1. The specimens were solution annealed at

1030 °C and then water quenched. The specimen size was prepared to be 20.0 mm long, 5.0 mm wide and 0.5 mm thick by mechanical and electrochemical polishing. Specimen holder made of SUS316L with curvature radius of 12.5 mm to give about 2% plastic strain into the 0.5 mm thick specimen was developed in this study. By bending and keeping deformation, the specimen with tensile stress of about 200 MPa, which corresponds to weld residual stress, was prepared for ion irradiation. The bending (stressed) and no-bending (un-stressed) specimens were irradiated simultaneously by using this special holder [6]. The ion irradiations of 12 MeV Ni<sup>3+</sup> were performed at 330, 400 and 550 °C in TIARA facility at JAEA. The surface temperature of the both specimens was measured by two-dimensional infrared pyrometer and kept to be constant under irradiation. The irradiation flux was monitored by beam profile monitor. The peak depth of displacement damage and Ni ion projectile range were calculated to be 2.8 and 3.2 μm, respectively, by SRIM-2000 code [7]. The mean displacement damage, which value was selected at depth of 1.6 μm, was changed from 1 to 45 dpa. The displacement damage rate was set to be about  $2 \times 10^{-3}$  dpa/s. It is noted that no bended (un-stressed) specimen was simultaneously irradiated with the bended (stressed) specimen on the same holder by X–Y ion-beam scanning. The difference of surface temperatures of the both specimens was below 20 °C under the irradiation. The residual stress, which was tensile stress in this study, was estimated by X-ray residual stress measurements before and after the irradiation with keeping the both specimens on the irradiation holder. The measurements were performed by using iso-inclination scanning method ( $2\theta\text{-sin}^2\psi$ ) [8], where  $\theta$  was Bragg angle and  $\psi$  was changed to be 0, 11.25, 22.5, 33.75 and 45°. The micro-hardness was measured by using nanoindenter. The hardening behavior of the irradiated specimens was evaluated

\* Corresponding author.

E-mail address: [okubo.nariaki@jaea.go.jp](mailto:okubo.nariaki@jaea.go.jp) (N. Okubo).

**Table 1**

Chemical compositions of the SUS316 and SUS316L (mass %).

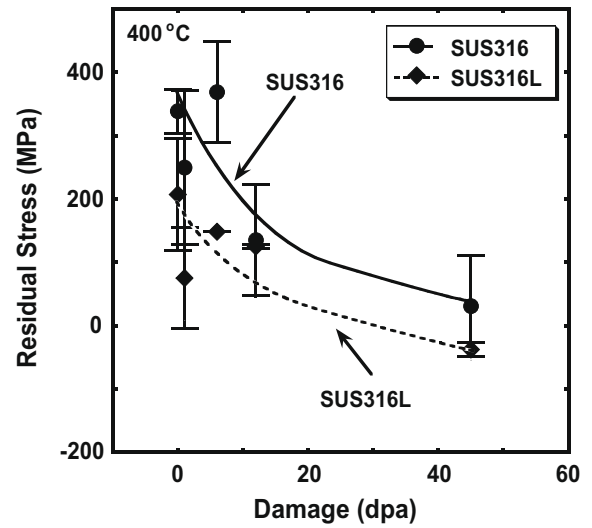
	C	Si	Mn	P	S	Ni	Cr	Mo
SUS316	0.05	0.43	0.83	0.026	0.001	10.05	16.13	2.07
SUS316L	0.008	0.40	0.82	0.025	0.0011	12.86	17.72	2.30
		N	Co	B	V	Al	Fe	
SUS316	–	–	–	–	–	–	Bal.	
SUS316L	<0.001	0.031	<0.001	<0.0001	0.002	0.009	Bal.	

by indentation depth of 300 nm in consideration of the damage depth of about 3 μm. The micro-hardness was calculated from mean values of 10 points per each specimen.

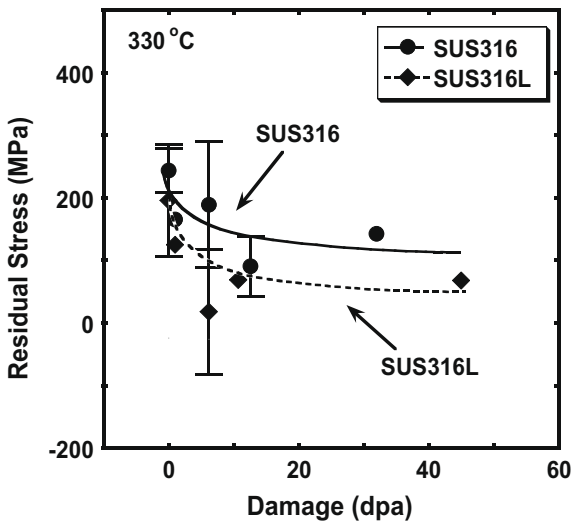
**3. Results and discussion**

The residual tensile stress of bended (stressed) specimen was evaluated to estimate the dpa dependence on the irradiation induced stress relaxation. The behavior of irradiation induced stress relaxation changed for each irradiation temperature under bending deformation. The initial residual stress of SUS316 and SUS316L before irradiation was measured to be about 200–300 MPa, which was comparable to weld residual stress in commonly stainless steels. The dpa dependence of SUS316 and SUS316L on the irradiation induced stress relaxation for 330 °C irradiation is shown in Fig. 1. The initial residual stress of 200 MPa decreased gradually with increasing dpa. The residual stress was about 100 MPa around 10 dpa and did not disappear completely even in the case of 45 dpa irradiation. The relaxation behaviors of both specimens are similar, though the initial residual stress of SUS316 was slightly higher than that of SUS316L. The residual stress after ion irradiation relaxed at these experimental temperatures in SUS316L. By using the irradiation holder, the thermally stress relaxation was evaluated without irradiation. The residual stresses for SUS316 specimens after thermal aging at 330 °C were 244, 232, 214 and 161 MPa for the same time of the irradiation to 0, 1, 6 and 45 dpa, respectively. In the case of SUS316L, the residual stresses were 156, 157, 167 and 68 MPa, respectively. Accordingly, the net stress relaxation induced by the irradiation at 330 °C above 10 dpa was from 50 to 60 MPa for SUS316 and 60 to 70 MPa for SUS316L. The behavior of stress relaxation induced by the irradiation at 400 °C is shown in Fig. 2. Though the initial residual stress of SUS316 is slightly higher than that of SUS316 shown in Fig. 1,

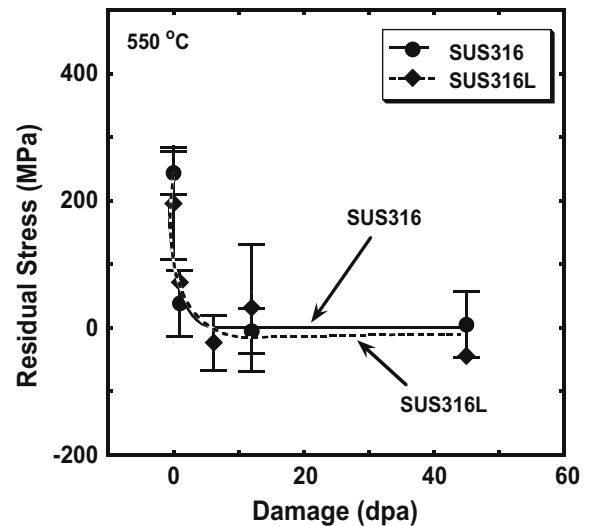
the residual tensile stress of both SUS316 and SUS316L relax gradually with increasing dpa. In the case of 400 °C irradiation at 45 dpa, the residual tensile stress became 0 MPa and almost all relaxed for both of SUS316 and SUS316L. The behavior of stress relaxation induced by the irradiation at 550 °C is shown in Fig. 3. In this case, the difference of relaxation behavior was not clear between SUS316 and SUS316L. The residual stress did not relax completely even for the case of the higher temperature aging at 500 °C



**Fig. 2.** Dependence of irradiation induced stress relaxation on irradiation damage at 400 °C.



**Fig. 1.** Dependence of irradiation induced stress relaxation on irradiation damage at 330 °C.



**Fig. 3.** Dependence of irradiation induced stress relaxation on irradiation damage at 550 °C.

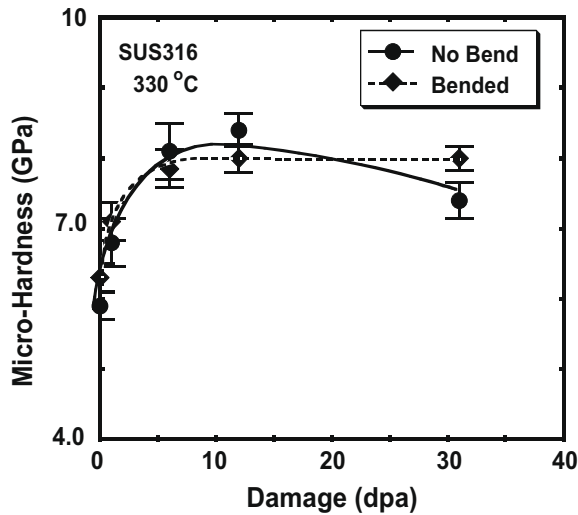


Fig. 4. Dependence of micro-hardness on irradiation damage at 330 °C for SUS316.

for the same time of the 6 dpa irradiation. However, most of the residual tensile stress was relaxed at 1 dpa and the residual stress was 0 MPa above 6 dpa.

The dependence of micro-hardness on the irradiation damage was evaluated for SUS316 and SUS316L with and without bending deformation. Fig. 4 shows the behavior of irradiation hardening of SUS316 at 330 °C. In both case of bended (stressed) and no bend (un-stressed) specimens, the hardness increases immediately up to 6 dpa and saturates around 12 dpa. On the other hand, distinct difference of hardening behavior was appeared for the case of SUS316L irradiated at 330 °C as shown in Fig. 5. The hardness of no bend specimen increases immediately and saturates around 12 dpa. The hardness of bended specimens, however, slightly become lower than that of no bend specimen at 6 dpa and increase slowly with increasing dpa, resulting in comparable hardness with no bend specimen at 45 dpa. The suppression of irradiation hardening was about 20% for the case of 12 dpa. Fig. 6 shows that irradiation hardening of bended specimen is suppressed at 12 dpa in comparing with that of no bend specimen for the irradiation at 400 °C.

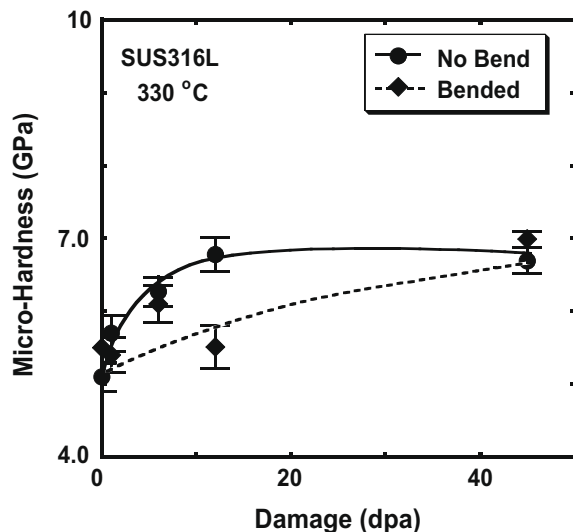


Fig. 5. Dependence of micro-hardness on irradiation damage at 330 °C for SUS316L.

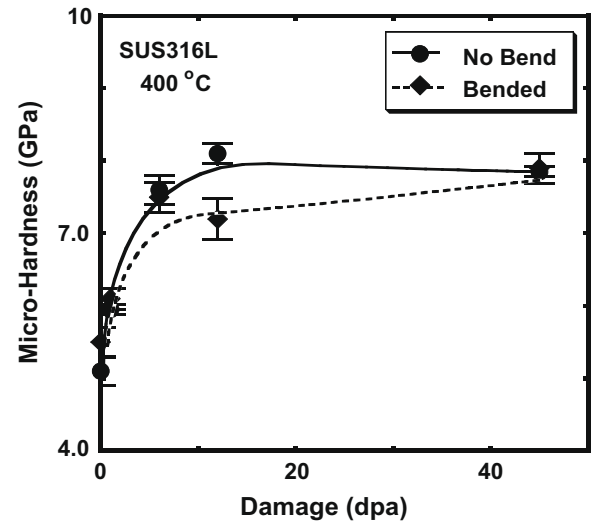


Fig. 6. Dependence of micro-hardness on irradiation damage at 400 °C for SUS316L.

The hardness of both specimens with and without bending was not so changed after irradiation at 550 °C. In this case, the hardness of specimens irradiated at 1–6 dpa slightly increased below 10% and the hardness was constant to be initial hardness up to 45 dpa. In the bended specimens, it is considered that irradiation induced point defects form dislocation loops along vertical direction of stress. The growth of loops and/or absorption of frenkel defects could cause climbing and gliding of dislocation and then, the stress could relax. This mechanism may be explained by stress induced preferred nucleation of dislocation loop (SIPN) [9] or stress-induced preferred absorption of point defects by dislocations (SIPA) [10] because dislocation loops were preferentially formed at relatively low irradiation temperature (300–400 °C). Microstructural evaluation by TEM is essential issue for interpreting the mechanism of hardness reduction.

#### 4. Conclusion

Effects of residual stress on irradiation hardening in SUS316 and SUS316L were investigated, prior to predicting and estimating IAS-CC. The behavior of irradiation induced stress relaxation was evaluated for irradiation damage and temperature under bending deformation. It was evident that irradiation hardening was reduced for the bending (stressed) specimens compared to the no bend (un-stressed) specimens for SUS316L irradiated at 300 and 400 °C.

#### Acknowledgement

Present study is the result of 'New evaluation method of material degradation considering synergetic effects of radiation damage' entrusted to Japan Atomic Energy Agency by the Ministry of Education, Culture, Sports, Science and Technology of Japan (MEXT).

#### References

- [1] J.T. Busby, J. Gan, M. Daniels, G.S. Was, S.M. Brummer, D.J. Edwards, in: Proceedings of Ninth International Conference of Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors, 1998, pp. 1089–1097.
- [2] K. Chatani, Y. Kitusnai, M. Kodama, S. Suzuki, Y. Tanaka, S. Ooki, S. Tanaka, T. Nakamura, in: Proceedings of 12th International Conference of Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors, 2005, pp. 349–355.
- [3] R.A. Weiner, A. Boltax, J. Nucl. Mater. 68 (1977) 141.

- [4] Y. Kaji, Y. Miwa, T. Tsukada, M. Kikuchi, S. Kita, M. Yonekawa, J. Nakano, H. Tsuji, H. Nakajima, *J. Nucl. Mater.* 307–311 (2002) 331.
- [5] Y. Ishiyama, K. Nakata, M. Obata, H. Anzai, S. Tanaka, T. Tsukada, K. Asano, in: *Proceedings of 11th International Conference of Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors, 2003*, pp. 920–929.
- [6] K. Kondo, Yukio Miwa, Nariaki Okubo, Yoshiyuki Kaji, Takashi Tsukada, in: *Proceedings of 13th International Conference of Environmental Degradation of Materials in Nuclear Power Systems, 2007*, submitted for publication.
- [7] J.F. Ziegler, J.P. Biersack, SRIM 2003 (Stopping and Range of Ion in Materials). Available from: <http://www.srim.org>.
- [8] L.E. Depero, M. Zocchi, *J. Appl. Cryst.* 24 (1991) 928–930.
- [9] R.V. Hesketh, *Philos. Mag.* 7 (1962) 1417.
- [10] P.T. Heald, M.V. Speight, *Philos. Mag.* 29 (1974) 1075.