

# Effect of cold-work on the radiation-induced deformation of austenitic stainless steels

Johsei Nagakawa<sup>a,b,\*</sup>, K. Ueno<sup>a,b</sup>, Y. Murase<sup>a</sup>, N. Yamamoto<sup>a</sup>

<sup>a</sup> National Institute for Materials Science (NIMS), 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047, Japan

<sup>b</sup> Interdisciplinary Graduate School of Engineering Sciences, Kyushu University, 6-1 Kasuga Koen, Kasuga, Fukuoka 816-8580, Japan

## Abstract

Austenitic stainless steels will be used in ITER as a major structural material. Although, initially supplied in annealed condition, cold-working is inevitable during construction or operation. Martensite would be introduced by cold-work in the case of unstabilized stainless steels. In this study, the effect of cold-work on the irradiation creep was examined under 17 MeV proton irradiation ( $2 \times 10^{-7}$  dpa/s) at 288 °C for stabilized (SUS 316L) and unstabilized (SUS 304) stainless steels. Radiation-induced stress relaxation was also evaluated using the creep data. The stress dependence of irradiation creep was different for the two steels; linear for SUS 316L and quadratic for SUS 304. This difference would influence the stress relaxation, which is faster in SUS 316L. The behavior of SUS 316L is consistent with computer simulation for evenly distributed network dislocations, while that of SUS 304 appears to originate from the dislocations localized in the vicinity of martensite boundaries.

© 2007 Elsevier B.V. All rights reserved.

## 1. Introduction

Austenitic stainless steels will be used in ITER as major structural materials for the blanket and the vacuum vessel. Although, these steels are to be supplied in the annealed condition, cold-working, e.g., from final grinding and polishing, especially in the vicinity of welds, will be inevitable during construction or operation and induce a high density of network dislocations and associated residual stresses.

Moreover, in the case of unstabilized steels like SUS 304, cold-work is known to introduce martensite in the austenite matrix in addition to network dislocations.

Recently, stress corrosion cracking (SCC) has been found in the core internals, particularly in the shroud, of many light water reactors (LWRs) in Japan. These internals were made of SUS 316L which is supposed to be less susceptible to such cracking. Residual tensile stresses on the surface layer introduced by cold-working are now considered to be one of the major causes of the cracking [1]. Radiation-induced deformation, especially stress relaxation, may influence such cracking by reducing the residual stress [2] or by affecting the crack propagation process [3]. This suggests the importance of

\* Corresponding author. Address: National Institute for Materials Science (NIMS), 1-2-1 Sengen, Tsukuba, Ibaraki 305-0047, Japan. Tel.: +81 29 859 2553/2014; fax: +81 29 859 2014.

E-mail address: [NAGAKAWA.johsei@nims.go.jp](mailto:NAGAKAWA.johsei@nims.go.jp) (J. Nagakawa).

the cold-work dependence of radiation-induced deformation in austenitic stainless steels to be used in ITER as well as in LWRs.

Accumulation of radiation-induced deformation (irradiation creep and radiation-induced stress relaxation) data of austenitic stainless steels in the anticipated temperature range for ITER, i.e. about 300 °C, is not sufficient. In particular, the systematic study of the effect of cold-work has scarcely been examined. In this study, the effect of cold-work on radiation-induced deformation was examined and compared for stabilized (SUS 316L) and unstabilized (SUS 304) austenitic stainless steels. Irradiation creep behavior was examined at 288 °C with two levels of cold-work. The stress relaxation behavior under irradiation was evaluated based on the obtained creep data. Finally, the mechanisms of radiation-induced deformation in the two austenitic stainless steels will be discussed.

## 2. Experimental procedure

Irradiation creep tests were performed for the cold-worked materials (thickness reduction of 5% and 25% by rolling) of SUS 304 (Cr: 18.28, Ni: 8.19, Mn: 0.87, C: 0.04, Si: 0.40, P: 0.026, S: 0.003, in wt%) and SUS 316L (Cr: 17.66, Ni: 12.19, Mn: 0.86, Mo: 2.12, C: 0.017, Si: 0.61, P: 0.024, S: 0.001, in wt%) austenitic stainless steels. Cold-worked sheets were punched into a thin-sheet specimen geometry 2.0 mm long, 10 mm wide and 0.15 mm thick. Average grain size after the final annealing (925 °C; 30 min) was 15  $\mu\text{m}$  and 13  $\mu\text{m}$  for SUS 316L and SUS 304, respectively. Yield stresses of the unirradiated SUS 316L measured by tensile testing at 288 °C were 320 MPa for the 5% CW (cold-worked) and 630 MPa for the 25% CW specimen. Those of the unirradiated SUS 304 were 330 MPa for the 5% CW and 660 MPa for the 25% CW specimen.

Irradiation experiments were carried out with 17 MeV protons using the cyclotron accelerator at National Institute for Materials Science. A broadened proton beam was further wobbled vertically at 10 kHz in order to more evenly irradiate the whole area of the specimen gage. The proton beam reached the specimen through a slit of 10 mm high and 3 mm wide, which was just in front of the specimen. The atomic displacement rate averaged through the thickness direction was  $2 \times 10^{-7}$  dpa/s according to the calculation using the TRIM 91 code for the present experimental conditions, and

its variation along the thickness direction was less than 15% [4].

The tensile irradiation creep apparatus has a high-sensitivity laser strain-measurement system with a resolution of 0.0097  $\mu\text{m}$ . Load control is maintained by adjusting the vacuum inside of a drum, sealed with a thin metal diaphragm, through a precise control of a Piezo valve following the feedback from a load cell. Details of the apparatus are described in Ref. [4]. During experiments, the specimen temperature was held at 288 °C with accuracy of  $\pm 0.15$  °C by controlling the Joule heating of the specimen, following feedback from a type K thermocouple of 0.1 mm diameter attached to the specimen by spot-welding. Specimen temperature was also monitored by an infrared pyrometer with a spot size of 1 mm diameter. Emissivity of the specimen was adjusted so as to equalize the pyrometer indication with the thermocouple temperature, while heating the specimen by a high temperature jet of helium.

## 3. Results and discussion

Fig. 1 shows the measured irradiation creep rates of the 5% CW specimens. The creep rates had a stress exponent of 1.2 and 1.9 for SUS 316L and SUS 304 steel, respectively, at the lower stress region where thermal activation does not induce a measurable creep rate at 288 °C in both steels. Further, cold-work to 25% did not change the stress dependence as shown in Fig. 2, though the magnitude was considerably reduced in SUS 316L, but only slightly in SUS 304. From these results, a difference in the stress dependence of

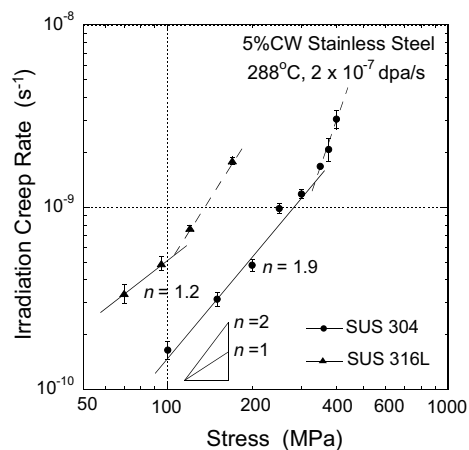


Fig. 1. Stress dependence of irradiation creep rate for 5% CW materials.

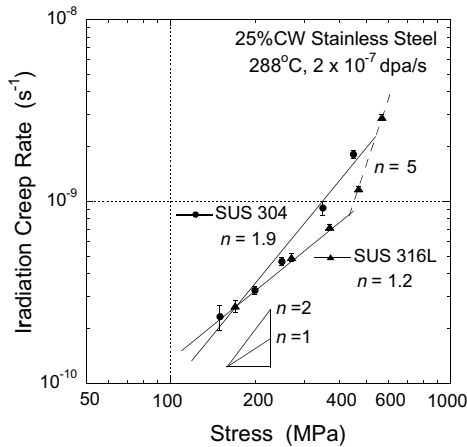


Fig. 2. Stress dependence of irradiation creep rate for 25% CW materials.

radiation-induced creep is clearly demonstrated by the two stainless steels, i.e. almost linear in SUS 316L and quadratic in SUS 304.

Stress relaxation reflects a reduction of elastic stress associated with the strain conversion from elastic to plastic while the total strain is kept constant. From the irradiation creep data obtained in the present experiments, radiation-induced stress relaxation was evaluated. Creep rates, including that of the present irradiation creep, can be expressed in a form of power law. Then, stress relaxation can be expressed by the equations,

$$\sigma/\sigma_0 = \exp(-Ect); \quad n = 1, \quad (1)$$

$$\frac{\sigma}{\sigma_0} = \exp \left[ -\frac{1}{n-1} \times \ln \{ 1 + (n-1)EC\sigma_0^{n-1}t \} \right]; \quad (2)$$

$n > 1,$

where  $\sigma$  is the stress,  $\sigma_0$  is the initial stress,  $E$  is the elastic modulus,  $C$  and  $n$  are the coefficient and the stress exponent of the power-law creep equation, respectively [2]. Calculated stress relaxation versus dose is shown in Fig. 3 for the 5% CW and the 25% CW specimens. Stress relaxation predicted for SUS 316L is faster than that for SUS 304 particularly in the 5% CW condition. This difference results from the stress exponent  $n$  of the two steels.

As was shown above, radiation-induced deformation behavior was different between the cold-worked specimens of SUS 316L and SUS 304. The increase in interstitial migration energy with decreasing Ni content has been reported by Dimitrov and Dimitrov [5]. Since radiation-induced deformation results from the migration of intersti-

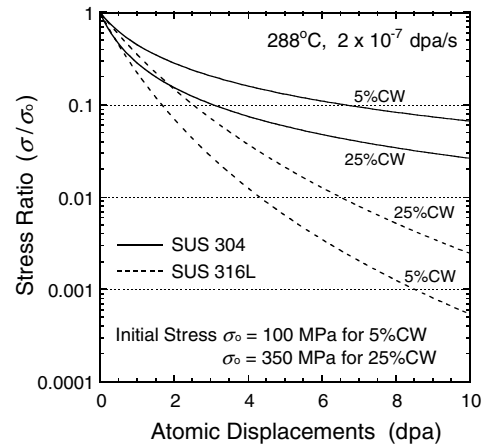


Fig. 3. Calculated stress relaxation curve for 5% CW and 25% CW materials.

tial atoms under the influence of applied stress, differences in Ni content between SUS 316L and SUS 304 may cause different deformation behavior. Hence, numerical calculation was performed for two different interstitial migration energies, that is, 0.92 eV for SUS 316L and 1.02 eV for SUS 304, as estimated from Ref. [6]. This calculation is based on the stress-influenced kinetics of point defects and simultaneous operation of all major irradiation creep mechanisms competing for the point defects. The calculation was performed for the austenitic steels with evenly distributed network dislocations. Details of the calculation were described previously [2,6]. Fig. 4 shows the calculated stress dependence of irradiation creep rates for the present experimental condition with very high network dislocation density. In both cases, the stress exponent  $n$  is slightly larger than unity and the magnitude of the creep rates do not differ very much. Both  $n$  and the magnitude coincide with the observed values of 25% CW specimen in the case of SUS 316L, but not for SUS 304. Therefore, the difference in interstitial migration energy should not be the origin of the observed difference in radiation-induced deformation.

In general, the stress dependence of the creep rate reflects the rate-controlling mechanism. In many cases, irradiation creep rate increases linearly with stress at lower stresses. Such a linear dependence is usually regarded as indicative of the climb motion of network dislocations by SIPA: stress-induced preferred absorption of point defects by dislocations [7] or due to SIPN: stress-induced preferred nucleation of dislocation loops [8]. This linear depen-

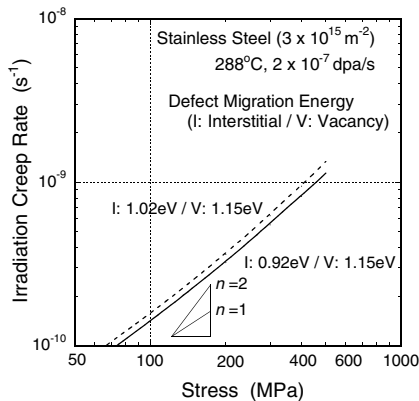


Fig. 4. Calculated stress dependence with two different interstitial migration energies.

dence is observed for SUS 316L as indicated in Figs. 1 and 2, and it has been supported by the numerical calculation [6]. On the other hand, the stress exponents for both 5% CW and 25% CW SUS 304 were about 2 at lower stresses as indicated in Figs. 1 and 2. In SUS 304 stainless steel, martensite is introduced in the gamma phase matrix by cold-working. Irradiation creep of martensitic stainless steels has generally been evaluated at very high temperatures and data around 300 °C are not available. However, the difference in martensite contents (volume ratio) between 5% CW and 25% CW SUS 304, measured by X-ray analysis, was quite remarkable, 5% and 77%, respectively. Despite such a large difference in martensite content, stress dependence was almost the same between the two cold-worked SUS 304s. Thus, it is hard to accept that the observed quadratic stress dependence in the cold-worked SUS 304 is simply due to the martensite itself, even if the stress dependence in martensite is quadratic or higher.

Mansur proposed a PAG (glide of network dislocations enabled by SIPA-induced climb) model, which gives a quadratic stress dependence, for materials with high density of network dislocations distributed evenly along the three axis directions [9]. However, his analysis indicates that SIPA climb overwhelms PAG at lower stresses and the SIPA-dominant region with  $n = 1$  expands to higher stresses as the network dislocation density increases. Transmission electron microscopy in the present study showed that the distribution of network dislocations in SUS 304 was localized and tangled in the vicinity of martensite boundaries, in contrast with the evenly distributed dislocations in the cold-

worked SUS 316L. Therefore, it is difficult to apply Mansur's model, which assumes evenly distributed network dislocations, directly to the cold-worked SUS 304. In Mansur's PAG model, network dislocations overcome the obstacles, which he considers as the network dislocations themselves, by the SIPA-climb. Once dislocations are released, they will bow out by glide and produce strain. At the same time, the climb process itself can induce a large amount of strain and this must be the reason why the SIPA climb mechanism dominates and gives a linear stress dependence even in the heavily cold-worked SUS 316 [6]. On the other hand, in the cold-worked SUS 304, the high density of tangled network dislocations cannot climb easily but can glide out of the tangles into the gamma phase areas, where network dislocations are relatively scarce, and move large distances. Thus, a glide mechanism, analogous to PAG but with some modification, would dominate the radiation-induced creep of cold-worked SUS 304 and give a quadratic stress dependence in the lower stress region.

#### 4. Conclusions

The effect of cold-work on the irradiation creep of SUS 316L and SUS 304 austenitic stainless steel was investigated at 288 °C under 17 MeV proton irradiation (damage rate:  $2 \times 10^{-7}$  dpa/s). Stress relaxation was also evaluated using the observed creep data. The results are summarized as follows:

- (1) Stress dependence of irradiation creep rates was different between the two steels; linear in SUS 316L and quadratic in SUS 304. This difference influenced the stress relaxation, which was faster in SUS 316L.
- (2) Behavior of the cold-worked SUS 316L is consistent with the existing irradiation creep models based on the evenly distributed network dislocations, while that of the cold-worked SUS 304 appears to originate from glide of dislocations localized in the vicinity of martensite boundaries.

#### Acknowledgements

This work was financially supported by the Budget for Nuclear Research of the Ministry of Education, Culture, Sports, Science and Technology,

based on the screening and counseling by the Atomic Energy Commission.

The authors thank Drs M. Obata and H. Kitsunai of NFD for providing the stainless steels.

## References

- [1] Editorial Committee on Nuclear Safety Research Results, Progress of Nuclear Safety – 2003, JAERI-Review 2004-010, Japan Atomic Research Institute (JAERI), (2004).
- [2] J. Nagakawa, *J. Nucl. Mater.* 225 (1995) 1.
- [3] J. Nagakawa, Y. Murase, N. Yamamoto, T. Fukuzawa, *J. Nucl. Mater.* 283–287 (2000) 391.
- [4] K. Ueno, J. Nagakawa, Y. Murase, N. Yamamoto, *J. Nucl. Mater.* 329–333 (2004) 602.
- [5] C. Dimitrov, O. Dimitrov, *J. Phys. F* 14 (1984) 793.
- [6] J. Nagakawa, K. Ueno, *J. Nucl. Mater.* 329–333 (2004) 597.
- [7] P.T. Heald, M.V. Speight, *Philos. Mag.* 29 (1974) 1075.
- [8] R.V. Hesketh, *Philos. Mag.* 7 (1962) 1417.
- [9] L.K. Mansur, *Philos. Mag. A* 39 (1979) 497.