ELSEVIER

Journal of Nuclear Materials 283–287 (2000) 386–390

journal of nuclear materials

www.elsevier.nl/locate/jnucmat

In-pile and post-irradiation creep of type 304 stainless steel under different neutron spectra

Y. Kurata [a,*], Y. Itabashi [b], H. Mimura [b], T. Kikuchi [c], H. Amezawa [b], S. Shimakawa [b], H. Tsuji [a], M. Shindo [d]

[a] Department of Nuclear Energy System, Japan Atomic Energy Research Institute, Tokai Research Establishment, Tokai-mura, Naka-gun, Ibaraki-ken 319-1195, Japan

[b] Department of JMTR, Japan Atomic Energy Research Institute, Oarai-machi, Higashi-ibaraki-gun, Ibaraki-ken 311-1394, Japan

[c] Department of Technology, Japan Atomic Energy Research Institute, Tokai-mura, Naka-gun, Ibaraki-ken 319-1195, Japan

[d] Hot Laboratory, Japan Atomic Energy Research Institute, Tokai-mura, Naka-gun, Ibaraki-ken 319-1195, Japan

Abstract

In addition to post-irradiation creep tests, in-pile creep tests were performed using newly developed technology with in situ measurement under different neutron spectra. The in-pile creep properties of type 304 stainless steel at 550°C appear to depend on neutron spectrum, but a spectral effect on post-irradiation creep properties is not clearly seen. The rupture time of in-pile creep under a high thermal neutron flux condition is the shortest. The order of the rupture time following the high thermal flux condition is post-irradiation creep, in-pile creep with a thermal neutron shield condition and finally creep of unirradiated material, all in increasing order. It is suggested that the acceleration of creep deformation and fracture observed in irradiation creep tests may be related to enhancement of thermal creep in terms of FMD increased under a high thermal neutron flux in addition to increased helium embrittlement.

1. Introduction

Irradiation creep is an important concern in structures under irradiation. In particular, the study of irradiation creep is essential for the core structures of fast breeder reactors and for first wall and blanket structures of fusion reactors. The effect of irradiation on creep properties has been investigated by means of either in-pile/post-irradiation creep tests conducted in fast reactors [1,2] and in mixed-spectrum fission reactors [3,4] or in-beam/post-implantation creep tests using α-particles from a cyclotron [5]. A pressurized tube technique has been mostly employed for in-pile creep tests [1–4,6–9]. Although irradiation creep tests using this method did not provide detailed time-dependent information on creep deformation process at first, considerable information has been obtained using re-irradiation and specimen miniaturization techniques [6–9]. Uni-axial creep tests with in situ measurement of specimen elongation under irradiation can provide much more detailed information of creep deformation behavior. It is thought to be necessary to obtain reliable creep deformation curves under irradiation in order to fully clarify the mechanism of irradiation creep.

Neutrons with different energy levels give rise to different levels of radiation damage. It is, therefore, considered that irradiation creep properties are influenced by neutron spectrum, flux and fluence, as well as irradiation temperature and stress. The influence of dpa and helium produced by irradiation has been discussed under conditions of fusion reactor environment [5,8–10]. In recent studies [9,10], it has been also shown that the formation and growth of helium bubbles can lead to acceleration of the irradiation creep coefficient. The possible direct relationship between irradiation creep and neutron spectrum has not been previously studied.

* Corresponding author. Tel.: +81-29 282 5059; fax: +81-29 282 6489.

E-mail address: ykurata@popsvr.tokai.jaeri.go.jp (Y. Kurata).

0022-3115/00/$ - see front matter
PII: S0022-3115(00)00107-0

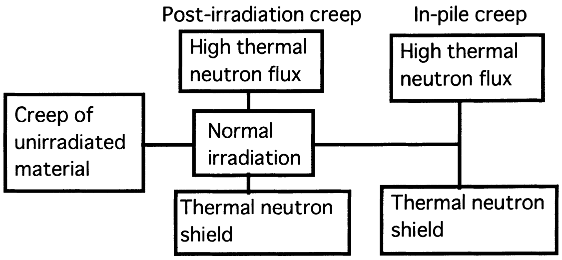

Fig. 1. Total plan of study on in-pile and post-irradiation creep.

Therefore, a study on in-pile/post-irradiation creep under different neutron spectra has been planned using uni-axial tensile specimens as shown schematically in Fig. 1.

The objective of this paper is to contribute to the understanding of the neutron spectral effect on creep behavior. For this purpose, in-pile creep capsules with uni-axial creep specimens to be irradiated under different neutron spectra were developed for irradiation in Japan Materials Testing Reactor (JMTR).

2. Experimental procedure

2.1. Material and specimen

The tested material was type 304 stainless steel for fast breeder reactor internals such as a core barrel and support plate. The main chemical composition of the tested material is the following: 0.047C, 0.54Si, 0.78Mn, 0.024P, 0.003S, 9.08Ni, 18.52Cr, 0.034N, 0.05Co and 0.0002B in mass %. Creep strength of this material was relatively high. Plates of type 304 stainless steel with a thickness of 40 mm were annealed at 1050°C for 12 min and followed by water cooling. Cylindrical specimens derived from these plates for creep tests of unirradiated material were 6 mm in diameter and 30 mm in gauge length. Smaller specimens with 2 mm in diameter and 10 mm in gauge length were used in post-irradiation and in-pile creep tests.

2.2. Irradiation and creep test conditions

Table 1 lists the irradiation conditions. Values of dpa and helium production were calculated by analysis of fluence monitor wires. Values of helium production in this table were calculated ones. However, the helium production in 93M-33A capsule was measured by chemical analysis and the accuracy of the calculation was confirmed. Normal irradiation without spectral tailoring in a fuel zone was performed for (1) 93M-33A. Under the conditions of (3) 94M-1A and (5) 94M-3A, thermal neutrons were shielded using cadmium. In contrast, a high thermal neutron flux was obtained using graphite under the conditions of (2) 93M-37A and (4) 94M-2A. Although a damage level of 2 dpa was expected after 8 cycle irradiation under the conditions of in-pile creep of (4) 94M-2A and (5) 94M-3A, irradiation of 94M-2A and 94M-3A was finished in 1 cycle and 2 cycles, respectively, because of specimen rupture.

Fig. 2 shows a schematic diagram of an irradiation creep capsule, 94M-2A. Different levels of stress can be loaded on two creep specimens using bellows pressurized by helium gas. The elongation of each creep specimen was measured using a linear variable differential transformer. A high thermal neutron flux was obtained with graphite in this capsule. The temperature of the specimen was measured using a thermocouple on the gauge length portion of the specimen, and controlled at 550°C using the electric heater installed in the capsule. The temperature of the specimen was normally controlled at 550 ± 1°C, and at 550 ± 3°C, even in case of large temperature fluctuations sometimes produced by irradiation of other capsules.

Out-of-core creep tests of both unirradiated and irradiated material were performed at 550°C. Irradiation temperatures of specimens for post-irradiation creep ranged between 485°C and 556°C. In-pile creep tests with high thermal neutron flux were performed at 550°C using stress levels of 245 and 284 MPa. Under the thermal neutron shield condition, the in-pile creep test of the lower specimen was performed at 550°C and 245 MPa because the temperature of the upper specimen could not reach 550°C.

Table 1
Irradiation conditions

Capsule name	Damage by thermal neutron (dpa)	Total damage (dpa)	Helium production (appm)
(1) 93M-33A, Post-irradiation test (normal irradiation, fuel zone)	2.87×10^{-3}	2.19	2.93
(2) 93M-37A, Post-irradiation test (high thermal neutron flux)	1.78×10^{-2}	1.51	11.0
(3) 94M-1A, Post-irradiation test (thermal neutron shield)	1.09×10^{-5}	0.97	0.9
(4) 94M-2A, In-pile creep (high thermal neutron flux)			
Upper	7.34×10^{-4}	5.62×10^{-2}	0.64
Lower	2.18×10^{-4}	1.67×10^{-2}	0.21
(5) 94M-3A, In-pile creep (thermal neutron shield)	2.74×10^{-6}	0.21	0.20

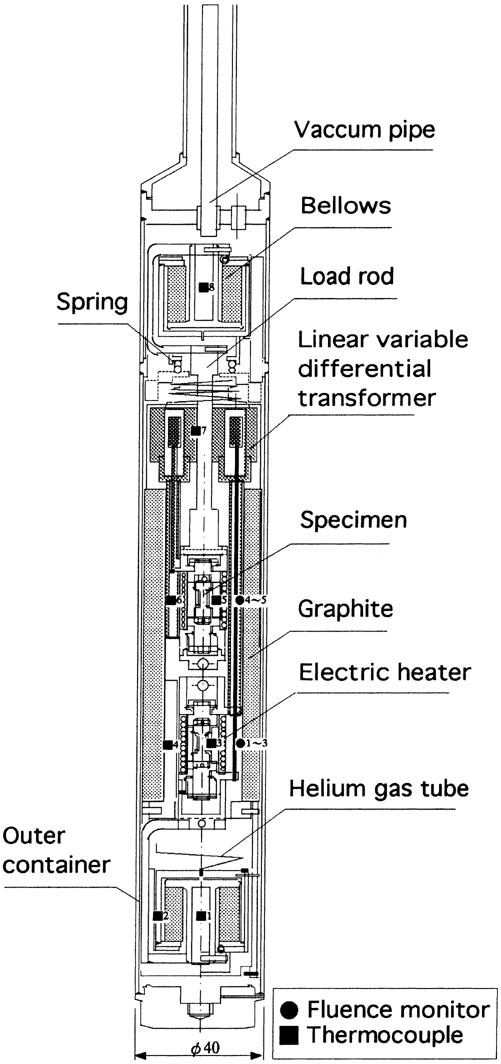

Fig. 2. Schematic diagram of irradiation creep capsule, 94M-2A under a high thermal neutron flux condition.

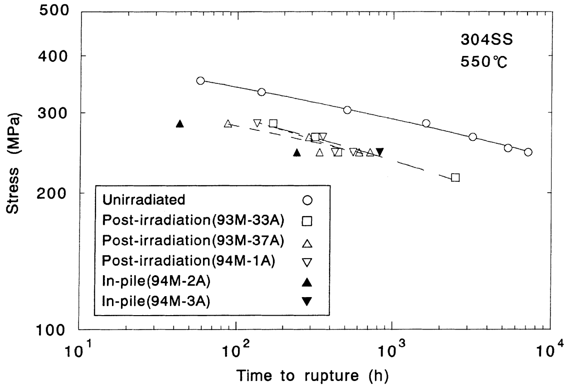

Fig. 3. Effect of neutron irradiation on time to rupture of type 304 stainless steel.

3. Results and discussion

Fig. 3 shows a comparison of rupture time among post-irradiation creep, in-pile creep under different neutron spectra and creep of the unirradiated material. The rupture time of in-pile creep under a high thermal neutron flux condition (94M-2A) is the shortest. The rupture time of in-pile creep and post-irradiation creep decreases in comparison with that of the unirradiated material. The relative order is in-pile creep under a high thermal neutron flux condition (94M-2A), post-irradiation creep under different neutron spectra (93M-33A, 93M-37A and 94M-1A), in-pile creep under a thermal neutron shield condition (94M-3A) and creep of the unirradiated material, in increasing order. The irradiation under different neutron spectra produced damage levels of about 1–2 dpa, and the helium production ranged from about 1 to 11 appm in the specimens of the post-irradiation tests (Table 1). There was no clear effect of neutron spectrum observed in the post-irradiation tests. The scatter in the rupture time of the unirradiated material was about ±20%. One of the causes of the large scatter in the rupture time of the post-irradiation creep may be the variety of irradiation temperatures ranged between 485°C and 556°C. It is notable that the rupture time of in-pile creep appears to change depending on the neutron spectrum. Additional tests are, however, required to confirm the conclusion because the finding is based on three in-pile test data alone. Lovell et al. [1] and Gilbert et al. [2] reported that the rupture time of in-pile creep in EBRII fast spectrum for cold-worked type 316 stainless steel was longer than that of post-irradiation creep. In contrast, Wassilew et al. [3,4] reported that the rupture time of in-pile creep in the BR2 mixed-spectrum reactor for type 1.4970 stainless steel was shorter than that of post-irradiation creep. Our results may explain the difference between their results.

Fig. 4 shows a comparison of creep curves obtained by in-pile creep under different neutron spectra, post-irradiation creep and creep of the unirradiated material. The creep curve after normal irradiation is shown as a typical example of the creep curve of post-irradiation. Although the in-pile creep deformation under a thermal neutron shield condition is only a little larger than that of the unirradiated material, the tertiary creep stage starts early under this condition. The in-pile creep deformation under a high thermal neutron flux condition is large from the early stage in comparison with that of the others. All creep curves of post-irradiation tests at 550°C and 245 MPa are shown in Fig. 5 together with creep curves of in-pile tests and unirradiated material. The creep curves of post-irradiation tests vary strongly even among specimens irradiated in the same capsule, e.g., three specimens of 93M-37A and two specimens of

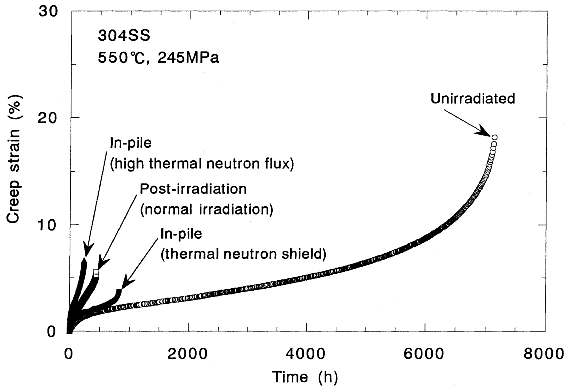

Fig. 4. Effect of neutron irradiation on creep curves of type 304 stainless steel.

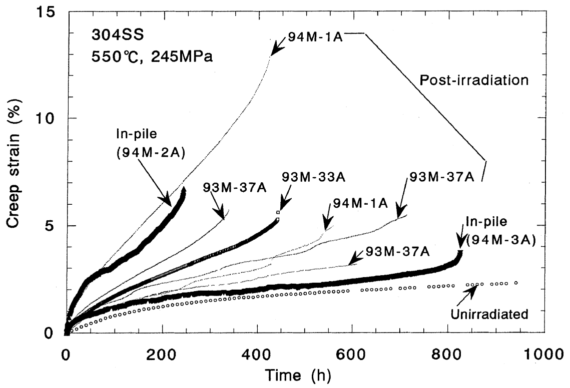

Fig. 5. Comparison of creep curves of in-pile (94M-2A [high thermal flux] and 94M-3A [thermal shielded]), post-irradiation (93M-33A, 93M-37A and 94M-1A) tests and unirradiated material.

94M-1A. For this reason, the creep rate of post-irradiation tests also has values scattered with a wide range. Fig. 6 shows a comparison of the steady-state creep rates. The steady-state creep rate of post-irradiation creep increases in comparison with that of the unirradiated material although it has a large scatter band. The steady-state creep rate of in-pile creep under a thermal neutron shield condition is a little larger than that of the unirradiated material. In contrast, the steady-state creep rate of in-pile creep under a high thermal neutron flux condition increased by a factor above 10 in comparison with that of the unirradiated one and lies at the upper scatter band of the post-irradiation creep.

Although there is the qualification of a small number of in-pile test data and a large scatter of post-irradiation creep properties, the in-pile creep deformation appears to be accelerated under a high thermal neutron flux condition. It is, therefore, interesting to speculate on what might cause such an acceleration of creep.

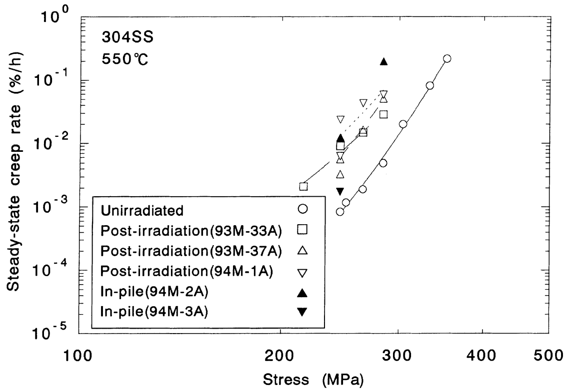

Fig. 6. Effect of neutron irradiation on the steady-state creep rate of type 304 stainless steel.

According to the theory of irradiation creep, the value of the stress exponent, n is as follows: $n = 1$ for stress induced preferred absorption (SIPA) mechanism [11,12], $n = 2$ for the preferred absorption glide (PAG) mechanism [12] and $n < 1$ for the stress induced preferential loop nucleation (SIPN) mechanism [13,14]. The stress exponent obtained from in-pile creep under a high thermal neutron flux condition is the high value of about 18 and similar to that of thermal creep of this material as shown in Fig. 6. Furthermore, the values of the creep coefficient obtained from the in-pile creep of this study were above 1×10^{-4} MPa^{-1} dpa^{-1} and much greater than $\sim 1 \times 10^{-6}$ MPa^{-1} dpa^{-1} which was expected in irradiation creep [15,16]. For these reasons, the creep rate is in the thermal creep regime. It is, therefore, suggested that the cause of the acceleration of the creep rate during irradiation is enhancement of thermal creep under a high thermal neutron flux condition. In addition, the acceleration of the creep rate is not caused by the helium bubble effect on irradiation creep [9,10].

Helium can also influence thermal creep behavior as helium embrittlement. Although helium bubbles at grain boundaries may accelerate the creep rate, helium bubbles within grains act as pinning centers for dislocations and obstacles of dislocation movement [17,18]. As shown in Figs. 4 and 5, however, the creep rate of the in-pile test under a high thermal neutron flux condition is accelerated even at the early stage where helium bubbles develop within grains. Therefore, it is difficult to consider that the acceleration of the creep rate before a tertiary creep stage is caused by helium produced during irradiation. Rupture ductility is decreased in creep tests under irradiation as shown in Fig. 4. One of the causes of reduction of the rupture time is helium embrittlement through early onset of a tertiary creep stage and ductility loss [17,18]. Recently, the concept of freely migrating defects (FMD) was presented [19] and has been used for analysis of radiation damage [19–23]. When crystalline materials are irradiated by fast neutrons, the numbers of

FMD are smaller than those of dpa as a result of cascade damage [19–23]. In contrast, numbers of FMD for iron-base alloys are almost equal to those of dpa in case of irradiation of thermal neutrons [21]. If 1.5% of the calculated number of defects can escape from energetic displacement cascades and become freely-migrating [23], it becomes almost the same level as that of FMD produced by thermal neutrons in case of the in-pile creep under high thermal neutron flux (Table 1). The total FMD is, therefore, increased twice. On this basis, it is plausible that irradiation under this condition enhances thermal creep in terms of FMD produced by recoil from thermal neutron captures.

4. Conclusions

The technology of in-pile creep tests under different neutron spectra was developed, and uni-axial irradiation creep tests and post-irradiation creep tests were performed to clarify effect of neutron spectrum on creep properties of type 304 stainless steel. The results obtained were as follows:

1. The creep rupture time of in-pile creep under a high thermal neutron flux condition is the shortest. The order of the rupture time following the above is post-irradiation creep under different neutron spectra, in-pile creep under a thermal neutron shield condition and, finally the unirradiated material in increasing order. The in-pile creep properties at 550°C appear to depend on neutron spectrum, but a spectral effect on post-irradiation creep properties is not clearly observed.
2. The steady-state creep rate of in-pile creep under a thermal neutron shield condition is a little larger than that of the unirradiated material. The steady-state creep rate of in-pile creep under a high thermal neutron flux condition and post-irradiation creep increases in comparison with that of the unirradiated material.
3. It is suggested that the cause of the acceleration of creep deformation and fracture observed in irradiation creep tests may be related to enhancement of thermal creep in terms of FMD formed under a high thermal neutron flux in addition to increased helium embrittlement.

Acknowledgements

The authors are thankful to the staff of the JMTR for technical support of irradiation and post-irradiation experiments.

References

[1] A.J. Lovell, B.A. Chin, E.R. Gilbert, J. Mater. Sci. 16 (1981) 870.
[2] E.R. Gilbert, B.A. Chin, Nucl. Technol. 52 (1981) 273.
[3] C. Wassilew, K. Anderko, L. Schaefer, in: J. Poirier, J.M. Dupouy (Eds.), Proceedings of the International Conference on Irradiation Behaviour of Metallic Materials for Fast Reactor Core Components, Ajaccio, France, 1979, p. 419.
[4] C. Wassilew, W. Schneider, K. Ehrlich, Radiat. Eff. 101 (1986) 201.
[5] H. Schroeder, P. Batflasky, J. Nucl. Mater. 103&104 (1981) 839.
[6] F.A. Garner, M.L. Hamilton, R.J. Puigh, C.R. Eiholzer, D.R. Duncan, M.B. Toloczko, A.S. Kumar, Eff. Rad. Mater., ASTM-STP 1175 (1993) 631.
[7] M.B. Toloczko, F.A. Garner, J. Nucl. Mater. 212–215 (1994) 509.
[8] M.L. Grossbeck, J.A. Horak, J. Nucl. Mater. 155–157 (1988) 1001.
[9] F.A. Garner, M.B. Toloczko, M.L. Grossbeck, J. Nucl. Mater. 258–263 (1998) 1718.
[10] C.H. Woo, F.A. Garner, J. Nucl. Mater. 271&272 (1999) 78.
[11] P.T. Heald, M.V. Speight, Philos. Mag. 29 (1974) 1075.
[12] L.K. Mansur, Philos. Mag. A 39 (1979) 497.
[13] R.V. Hesketh, Philos. Mag. 7 (1962) 1417.
[14] A.D. Brailsford, R. Bullough, Philos. Mag. 27 (1973) 49.
[15] K. Ehrlich, J. Nucl. Mater. 100 (1981) 149.
[16] F.A. Garner, in: Nuclear Materials, Part 1, Materials Science and Technology: A Comprehensive Treatment, vol. 10A, VCH, 1994, pp. 419–543 (Chapter 6).
[17] H. Trinkaus, H. Ullmaier, Philos. Mag. A 39 (1979) 563.
[18] H. Trinkaus, J. Nucl. Mater. 118 (1983) 39.
[19] L.E. Rehn, J. Nucl. Mater. 174 (1990) 144.
[20] L.E. Rehn, R.C. Birtcher, J. Nucl. Mater. 205 (1993) 31.
[21] H.L. Heinisch, J. Nucl. Mater. 178 (1991) 19.
[22] S.J. Zinkle, B.N. Singh, J. Nucl. Mater. 199 (1993) 173.
[23] A. Mueller, V. Naundorf, M.-P. Macht, J. Appl. Phys. 64 (1988) 3445.