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In-pile creep rupture properties of ODS ferritic steel claddings

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ABSTRACT

In order to examine irradiation effect on creep rupture strength of Oxide Dispersion Strengthened (ODS) steel claddings, the in-pile creep rupture test was conducted using Material Testing Rig with Temperature Control (MARICO)-2 in the experimental fast reactor JOYO. Fourteen creep rupture events were successfully detected by the temperature change in each capsule and the gamma-ray spectrometry of the cover gas. Time to creep ruptures of six ODS steel specimens were identified by means of Laser Resonance Ionization Mass Spectrometry (RIMS), and no irradiation effect on creep rupture strength was confirmed within the irradiation condition in the MARICO-2 test.

1. Introduction

Superior radiation resistance and high temperature capability are essential properties for the cladding of the advanced fast reactor (FR) fuels because they are used in severe conditions such as high temperature environment around 973 K and average burn-up of 150 GWd/t which is relevant to a peak neutron dose as high as 250 dpa. The oxide dispersion strengthened (ODS) ferritic steel is one of the most promising cladding materials for advanced sodium-cooled fast reactors [1,2] as well as being applicable as a low activated fusion reactor materials [3].

In order to investigate the influence of the neutron irradiation on the material properties of ODS steels developed by Japan Atomic Energy Agency (JAEA), the material irradiation test using Core Material Irradiation Rig (CMIR) is executed in the experimental fast reactor JOYO. However, internal creep rupture strength that is the most important property as the fuel claddings can not be obtained by the CMIR test. Therefore, we developed Material Testing Rig with Temperature Control (MARICO)-2 as a new irradiation test device, and executed the in-pile creep rupture test of the ODS steel claddings. In this paper, the results of the in-pile creep rupture test using MARICO-2 are reported.

2. Experimental procedure

2.1. Specimens

The creep rupture tests were conducted using two types of ODS steel claddings; 9Cr-ODS steel (Mm14) and 12Cr-ODS steel (F14). The chemical composition of these ODS steel claddings is listed in Table 1. The basic composition of these ODS steels is Fe–Cr–W–Ti–Y_2O_3. The detail manufacturing process of these ODS tubes is cited in Refs. [4–6].

Schematic drawing of a pressurized tube specimen for internal creep rupture tests is shown in Fig. 1. These specimens were prepared from the ODS steel claddings having the dimension of 6.9 mm outer diameter and 0.4 mm wall-thickness. The cladding tube length of the specimens secured 30 mm or more based on the report that the cladding tube length was necessary more than 4.3 times of the outer diameter not to be affected by the restriction of the joining part. The both end-plugs of specimens were joined by using the pressurized resistance welding method [7]. The hoop stress was set by adjusting the pressure of enclosed helium gas to become the predetermined stress at each test temperature. In addition, for the in-pile creep rupture test, to identify the ruptured specimen, a few cm^3 of a unique blend of stable xenon and krypton tag gas was enclosed.

2.2. Out-of-pile creep rupture test

As reference to the creep rupture strength for both Mm14 and F14 claddings, creep rupture tests were conducted in argon gas atmosphere. These test conditions are shown in Table 2. The test temperature is 923, 973 and 1023 K, and the range of the hoop stress is from 60 to 180 MPa. Rupture of the specimen was identified by detecting the helium gas released from inside of the specimen.

2.3. In-pile creep rupture test

The in-pile creep rupture test was conducted in JOYO using MARICO-2. The structure of the MARICO-2 is shown in Fig. 2. In

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Table 1
Chemical composition of ODS steel claddings.

Composition (wt%)	9Cr-ODS (Mm14)	12Cr-ODS (F14)
C	0.13	0.043
Si	0.009	0.051
Mn	< 0.01	0.09
P	< 0.005	0.005
S	0.003	0.004
Ni	< 0.01	0.08
Cr	8.84	11.37
W	1.97	1.88
Ti	0.20	0.26
Y_2O_3 [*1]	0.350	0.229
Ex.O [*2]	0.096	0.056
N	0.012	0.010
Ar	0.005	0.004

[*1] $Y_2O_3 = 1.27 \times Y$.
[*2] $Ex.O = Total\ O - 0.27 \times Y$.

Table 2
Out-of-pile creep rupture test conditions and results.

Material	Temperature (K)	Hoop stress (MPa)	Number of hoop stress level
9Cr-ODS	923	140–160	2
	973	90–170	12
	1023	60–90	5
12Cr-ODS	973	130–180	14
	1023	74–120	8

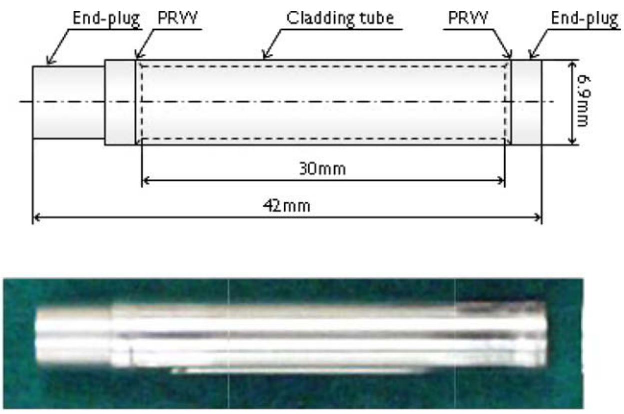

Fig. 1. Schematic drawing of an internal creep rupture specimen.

the test assembly, there are fifteen irradiation capsules that consist of three types: temperature control, on-line temperature monitor and off-line temperature monitor. The in-pile creep rupture tests were executed by mainly using the temperature control type capsules. The temperature control type capsules loaded with specimens have a double wall thermal insulated structure and are filled with sodium in order to maintain isothermal conditions. The temperature inside the capsule is measured with a thermocou-

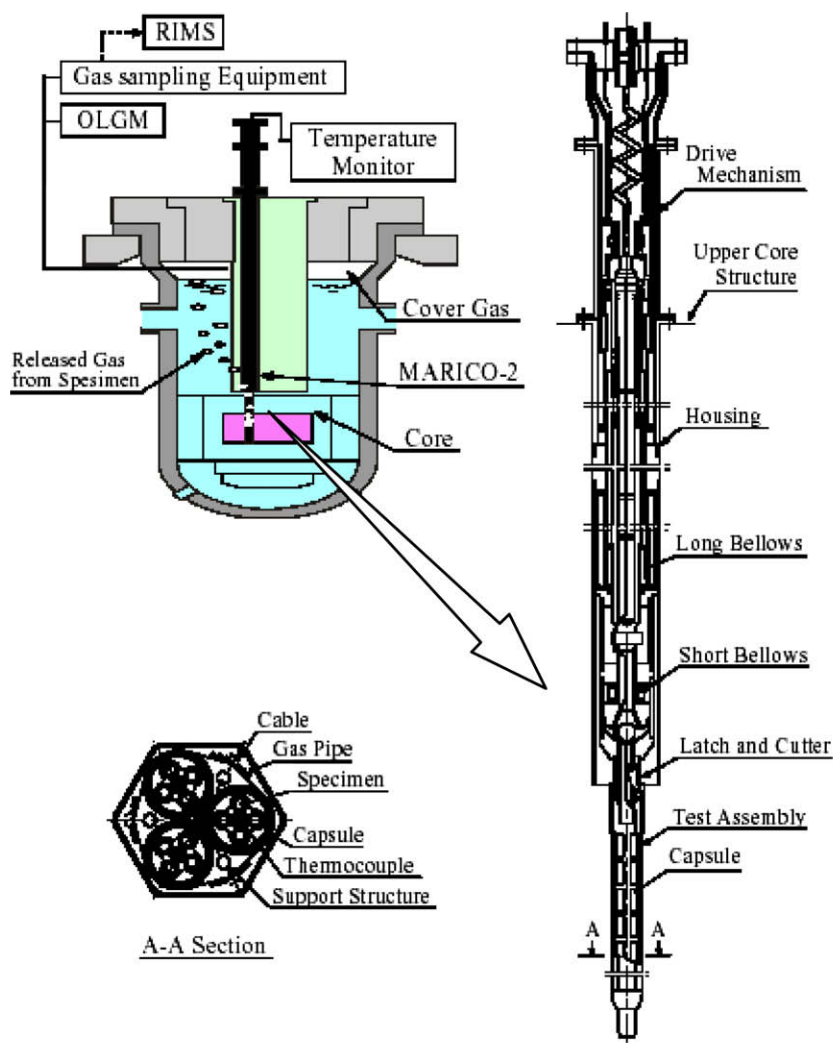

Fig. 2. Structure of MARICO-2.

Table 3
In-pile creep rupture test conditions.

Material	Temperature (K)	Hoop stress (MPa)	Number of hoop stress level
9Cr-ODS (Mm14)	973	75–155	9
	998	45–93	6
12Cr-ODS(F14)	973	109–137	3
	1023	75–114	6

ple. The temperature is then controlled by changing the mixture ratio of argon and helium gases enclosed in the thermal insulated gas gap between the capsule's double wall stainless tubes. The specimens can be kept at a constant temperature with a precision of ±4 K by controlling the heat flow across the gap.

As shown in Table 3, twenty-four pressurized tube specimens made from both Mm14 and F14 claddings were irradiated at temperatures of 973, 998 and 1023 K. The range of the hoop stress is from 45 to 155 MPa. To determine by identifying the ruptured specimen, the following two methods were employed in the MARICO-2. First of all, the temperature change with the gas release from the ruptured specimen is detected with the thermo-couple installed on each capsule. And then, released gases are transferred to the argon cover gas region illustrated in Fig. 2. The cover gas is measured by means of gamma-ray spectrometry using the Online Gamma-ray Monitor (OLGM) and Laser Resonance Ionization Mass Spectrometry (RIMS). The example of creep rupture detection is shown in Fig. 3. The temperature change more than 10 K using thermocouples indicated the capsule which contains the ruptured specimen. The OLGM measured the gamma-rays emitted from

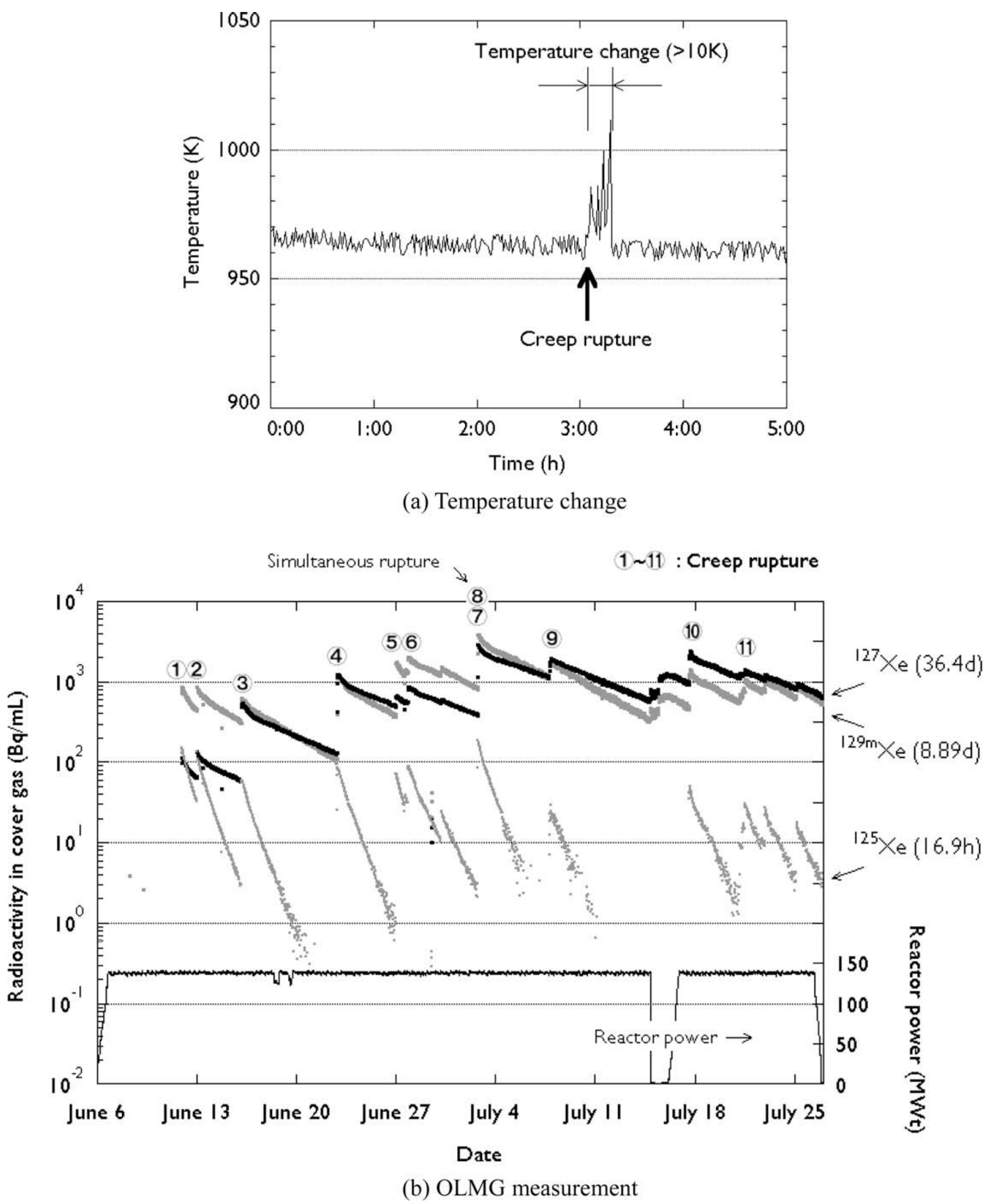

Fig. 3. In-pile creep rupture detection by temperature change and OLGM measurement.

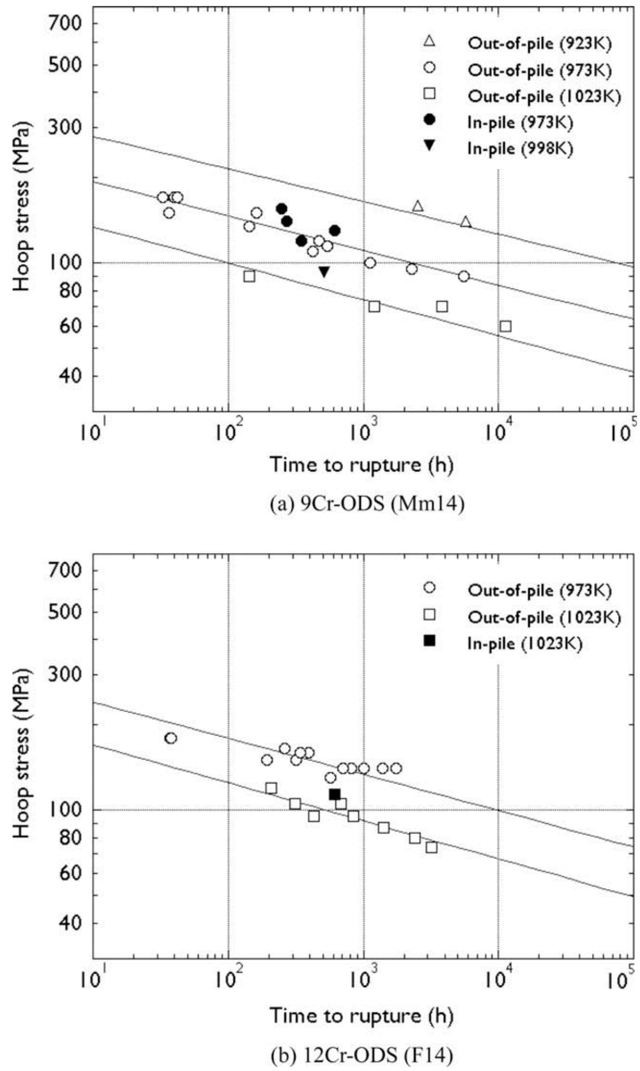

Fig. 4. The results of in-pile and out-of-pile creep rupture tests.

^{125}Xe, ^{127}Xe, ^{129m}Xe, etc., which were the activated tag gas nuclides used to detect the creep rupture of specimens.

3. Results and discussions

3.1. In-pile creep rupture detection

The MARICO-2 test was carried out for the JOYO MK-III 3rd to 6th operation cycles from April 2006 until May 2007. The cumulative irradiation time reached 196 EFPDs, and it corresponds to maximum neutron dose of 20 dpa. Fourteen creep rupture events were successfully detected by the temperature change in each capsule and the gamma-ray spectrometry of the cover gas. Of these creep rupture events, the time to ruptures of six ODS steel specimens ruptured before the simultaneous rupture was identified by means of RIMS. The time to rupture after the simultaneous rupture will be identified.

3.2. In-pile creep rupture property

The in-pile creep rupture strengths of Mm14 and F14 are shown in Fig. 4(a) and (b) by comparison with that of out-of-pile test. In these figures, abscissa is time to rupture in hours and ordinate represents the hoop stress in MPa. As for Mm14, the time to rupture of out-of-pile test covers from 17 to 11 348 h. As shown in Fig. 4(a), the creep rupture strength clearly decreases with an elevated temperature. The in-pile creep rupture data of up to the time to rupture of 614 h ($\approx$2.0 dpa) at 973 K and 512 h ($\approx$3.1 dpa) at 998 K were obtained in MARICO-2 test, the strength reduction due to the neutron irradiation was not recognized for Mm14 under this irradiation condition as shown in Fig. 4(a). As for F14, the time to rupture of out-of-pile test covers from 37 to 3199 h. The creep rupture strength of F14 decreases with an elevated temperature as shown in Fig. 4(b). The in-pile creep rupture strength reduction due to the neutron irradiation of F14 also was not confirmed though the data obtained in MARICO-2 test was only one point of the time to rupture of 611 h ($\approx$3.1 dpa) at 1023 K as well as Mm14.

As already reported in Refs. [8–10], the tensile properties of ODS steel claddings irradiated up to 15 dpa at 793 K in JOYO were considerably maintained and showed the excellent performance. From the result of transmission electron microscope (TEM) observation of ODS steel claddings having same specification as Mm14 and F14, the oxide particles and microstructure were confirmed to be stable after neutron irradiation. Especially, the oxide particles were finely and uniformly distributed in the matrices after the neutron irradiation up to neutron dose of 15 dpa as shown in Fig. 5. Thus, there is no creep rupture strength reduction in ODS steel claddings because the oxide particles are stable under the irradiation condition. In addition, the creep rupture strength reduction was not confirmed under 973 K stagnant sodium immersion condition at time to exceed 10 000 h [11]. From these results, the irradiation effect on creep rupture strength of ODS steel claddings will be very small for a longer time.

On the other hand, in the in-pile creep rupture test of 20% cold-worked modified 316 stainless steel (PNC316) conducted using Material Open Test Assembly (MOTA) in Fast Flux Test Facility (FFTF), the creep rupture strength reduction due to the neutron irradiation was already recognized the time to rupture of 300 h ($\approx$2.5 dpa) at 878 K. Such strength reduction in PNC316 could be

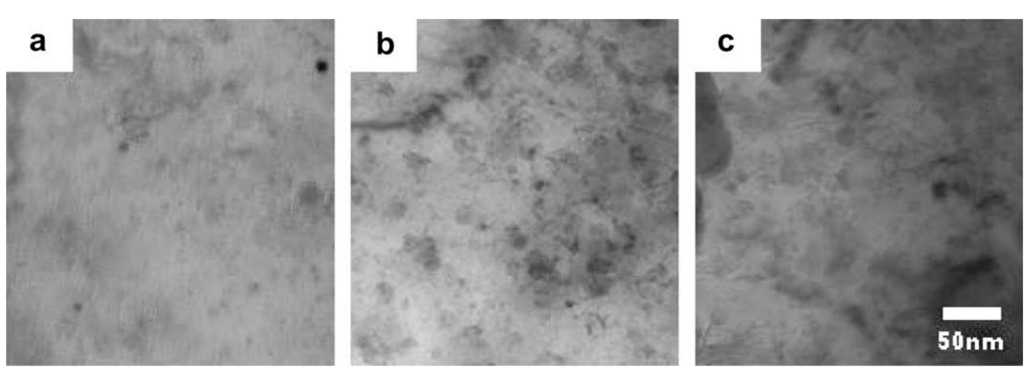

Fig. 5. Bright field TEM micrographs of 9Cr-ODS steel claddings, (a) unirradiated, (b) 2.5 dpa at 670 K and (c) 7.0 dpa at 807 K.

mainly attributed to the earlier recovery of dislocation structure introduced by cold-working. The coarsening of MC precipitates by the radiation-induced solute segregation may be indirectly associated with the early dislocation recovery [12]. The in-pile creep rupture strength reduction was not confirmed in the ODS steels as described above. This result suggests that the dislocation recovery and the coarsening of MC precipitates will not influence the creep rupture strength of the ODS steels. These suggestions will be confirmed by post-irradiation examination.

4. Conclusion

In order to examine irradiation effect on creep rupture strength of the ODS steel claddings, the in-pile creep rupture test was conducted using MARICO-2 in JOYO. Fourteen creep rupture events were successfully detected by the temperature change in each capsule and the gamma-ray spectrometry of the cover gas, and time to ruptures of six ODS steel specimens identified the ruptured time by means of RIMS. As a result, no irradiation effect on creep rupture strength of ODS steel claddings was confirmed within the irradiation condition in MARICO-2 test.

References

[1] H. Niwa, K. Aoto, M. Morishita, Current Status and Perspective of Advanced Loop Type Fast Reactor in Fast Reactor Cycle Technology Development Project, GLOBAL 2007, September 9–13, Boise, Idaho, 2007.
[2] T. Kaito, S. Ohtsuka, M. Inoue, Progress in the R&D Project on Oxide Dispersion Strengthened and Precipitation Hardened Ferritic Steels for Sodium Cooled Fast Breeder Reactor Fuels, GLOBAL 2007, September 9–13, Boise, Idaho, 2007.
[3] D.K. Mukhopadhyay, F.H. Froes, D.S. Gelles, J. Nucl. Mater. 258–263 (1998) 1209.
[4] S. Ukai, S. Mizuta, T. Yoshitake, T. Okuda, M. Fujiwara, S. Hagi, T. Kobayashi, J. Nucl. Mater. 283–287 (2000) 702.
[5] S. Ukai, S. Mizuta, M. Fujiwara, T. Okuda, T. Kobayashi, J. Nucl. Sci. Technol. 39 (2002) 778.
[6] S. Ukai, T. Okuda, M. Fujiwara, T. Kobayashi, S. Mizuta, H. Nakashima, J. Nucl. Sci. Technol. 39 (2002) 872.
[7] M. Seki, K. Hirako, S. Kono, Y. Kihara, T. Kaito, S. Ukai, J. Nucl. Mater. 329–333 (2004) 1534.
[8] Y. Yoshitake, Y. Abe, N. Akasaka, S. Ohnuki, S. Ukai, A. Kimura, J. Nucl. Mater. 329–333 (2004) 342.
[9] S. Yamashita, N. Akasaka, S. Ohnuki, J. Nucl. Mater. 329–333 (2004) 377.
[10] N. Akasaka, S. Yamashita, T. Yoshitake, S. Ukai, A. Kimura, J. Nucl. Mater. 329–333 (2004) 1053.
[11] E. Yoshida, S. Kato, J. Nucl. Mater. 329–333 (2004) 1393.
[12] S. Ukai, S. Mizuta, T. Kaito, H. Okada, J. Nucl. Mater. 278 (2000) 320.