



# Post-irradiation creep rupture properties of FBR grade 316 SS structural material

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## Abstract

In order to investigate the effects of neutron irradiation on the creep properties of FBR grade type 316 stainless steel for structural material (316FR), post-irradiation creep rupture tests were conducted in the specimens irradiated in JOYO and JMTR. Below the neutron exposure of around 0.3 dpa, the rupture strength of 316FR almost maintains the same level of unirradiation one, since finely dispersed precipitates should trap the helium bubbles within grains and prevent the helium accumulation at grain-boundaries. However, beyond about 0.3 dpa, a reduction of creep rupture strength and intergranular brittle fracture mode were observed in 316FR. Such phenomena could be attributed to the growth of phosphide precipitates which deteriorates both the trapping capability of helium within the grain and solution strengthening of phosphorus in matrix. These results are applicable to the design of structural materials in fusion reactor. © 1999 Published by Elsevier Science B.V. All rights reserved.

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## 1. Introduction

Structural materials used in a reactor vessel and reactor lower plate of FBR are required to have superior high temperature creep properties and radiation resistance during the reactor life time. Type 304 SS and type 316 SS were applied to the structural materials of FBR.

It was reported that creep life time and ductility of the irradiated type 316 and type 304 SS decrease due to irradiation compared with unirradiated one, and such degradation has been characterized in relation to helium (He) productions, defects and irradiation-induced precipitations [1,2]. In this study, in order to understand degradation mechanism of the post-irradiation thermal creep properties of 316FR, which is candidate for future FBR structural material, the effects of He production and accumulated neutron dose in dpa were investigated as compared with properties of the irradiated type 304 SS.

## 2. Experimental procedures

### 2.1. Tested material

The chemical composition of both 316FR and type 304 SS are listed in Table 1. In 316FR, an appropriate amount of nitrogen was added instead of carbon, since carbide precipitates at the grain-boundaries could deteriorate the long term creep properties. A suitable amount of phosphorus was also added into 316FR. The hot rolled plates of 316FR and type 304 SS were fabricated in a thickness of 50 and 40 mm, from which cylindrical test specimens in diameter of 4 or 6 mm were machined in parallel with the rolling direction.

### 2.2. Irradiation test

Both 316FR and type 304 SS test specimens were irradiated in Experimental Fast Reactor JOYO and Japan Material Testing Reactor (JMTR) of a light water cooled thermal testing reactor in JAERI. An irradiation temperature is ranged 748–848 K. It is characterized that specimens irradiated in JOYO are exposed in higher dpa due to fast neutron dominant, whilst JMTR-irradiated ones are produced much abundant He by the reaction of

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Table 1  
Chemical compositions of 316FR and type 304 stainless steel

	Chemical composition (wt%)									
	C	Si	Mn	P	S	Ni	Cr	Mo	N	B
Type316 (JIS)	0.08 $\geq$	1.00 $\geq$	2.00 $\geq$	0.045 $\geq$	0.030 $\geq$	10.00 ~ 14.00	16.00 ~ 18.00	2.00 ~ 3.00	–	–
316FR (Check)	0.012	0.52	0.86	0.024	0.004	10.59	16.58	2.14	0.08	0.0003
Type304 (Check)	0.05	0.59	0.87	0.027	0.003	8.98	18.47	–	0.022	0.0005

$^{10}\text{B}(n,\alpha)$  or  $^{58}\text{Ni}(n,\gamma)^{59}\text{Ni}(n,\alpha)$  under the thermal neutron environment. The accumulated dpa and He in the specimens were calculated in accordance with ASTM method using nuclear reaction cross section of 'ENDF/B-IV' [3].

### 2.3. Post-irradiation creep test

Creep tests of irradiated specimens have been conducted at 773 and 823 K according to the Japan Industrial Standard Z 2272. Test temperature was set within  $\pm 25$  K for irradiation temperature. Microscopic observations of specimens before and after creep test were carried out by transmission electron microscopy (TEM) and scanning electron microscopy (SEM) for ruptured surface analysis.

## 3. Results of post-irradiation creep test

The results of creep tests demonstrated that creep rupture strength of irradiated both 316FR and type 304 SS was degraded as compared with unirradiated one, although the strength level of 316FR is superior to that of type 304 SS in the material itself.

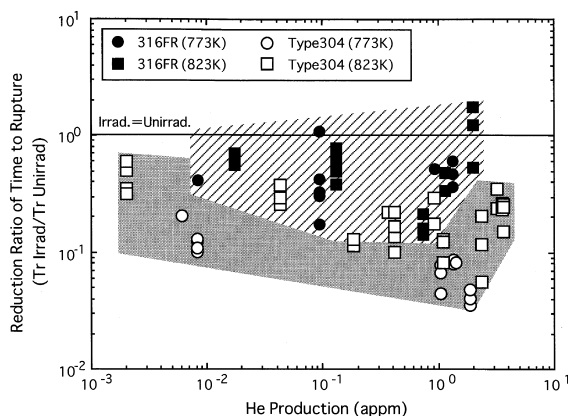


Fig. 1. Reduction ratio of creep rupture time of 316FR and type 304 SS as a function of helium production.

The effects of neutron irradiation on the creep strength degradation can be characterized in terms of the amount of He produced and dpa parameters [4–6]. Figs. 1 and 2 show the reduction ratio of creep rupture time of irradiated to unirradiated ones at 773 and 823 K for 316FR and type 304 SS. The creep rupture time gradually decreases with increasing of both He production and dpa for type 304 SS. However, there is not such a clear correlation in 316FR, except for neutron accumulation beyond around 0.3 dpa where the creep rupture strength in 316FR decreases with accumulated dpa.

It was also demonstrated that post-irradiation creep rupture elongation of type 304 SS decreased due to irradiation, especially in JMTR irradiated specimens. On the other hand, only JOYO irradiated specimens degrades in 316FR, which corresponds to the irradiated specimens beyond neutron accumulation of 0.3 dpa.

## 4. Discussion

In order to investigate the characteristic feature of the creep rupture properties of the irradiated 316FR, microstructure of the irradiated specimens has been studied by means of TEM and SEM. Fig. 3 compares

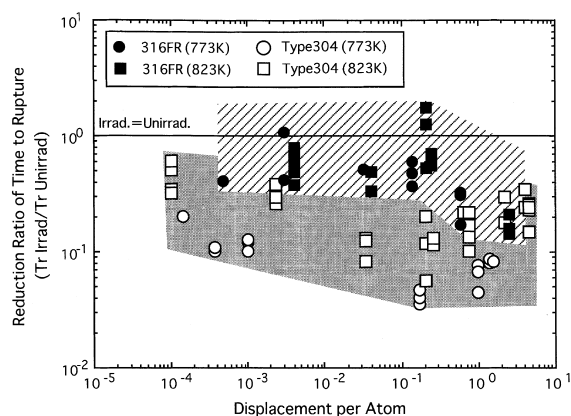


Fig. 2. Reduction ratio of creep rupture time of 316FR and type 304 SS as a function of dpa.

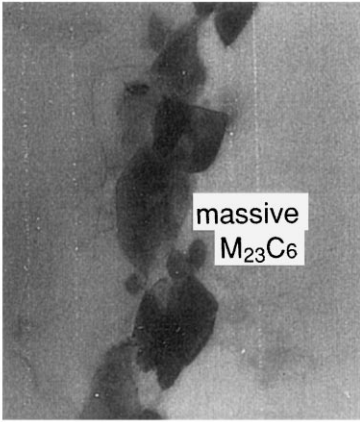
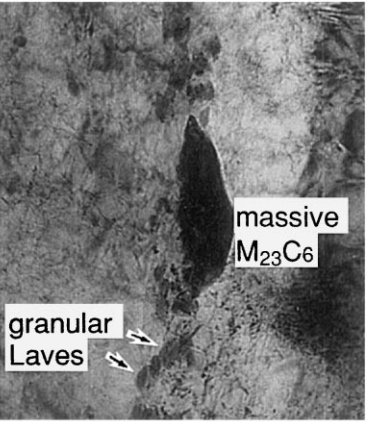
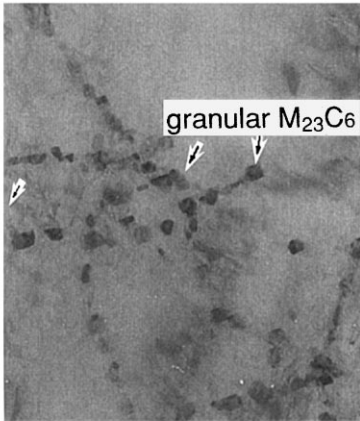
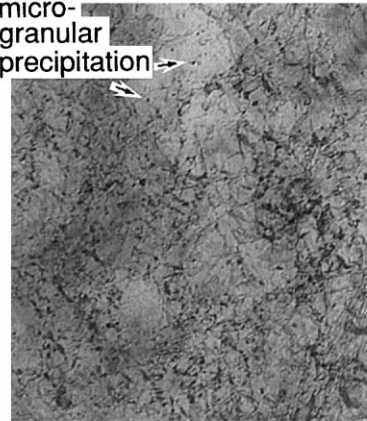
<p>Inter granu lar</p>	 <p>massive <math>M_{23}C_6</math></p>	 <p>massive <math>M_{23}C_6</math></p> <p>granular Laves</p>
<p>Trans granu lar</p>	 <p>granular <math>M_{23}C_6</math></p>	 <p>micro- granular precipitation</p>
<p>— — 100nm</p>	<p><b>Type 304 SS</b> 779K, 0.23dpa, He:2.0appm</p>	<p><b>316FR</b> 811K, 0.21dpa, He:2.4appm</p>

Fig. 3. Comparison of precipitates of irradiated 316FR and type304 SS at JMTR by transmission electron microscopy (after creep test).

the precipitates at grain boundary and in matrix after creep tests of the irradiated 316FR with those of type 304 SS in TEM micrographs. Irradiation condition is about 0.2 dpa and 2 appm He for both materials. There are blocky type grown carbide ( $M_{23}C_6$ ) at the grain-boundary and granular type carbide ( $M_{23}C_6$ ) within the grain for type 304 SS. On the other hand, finely granulated carbide and finely needle shaped phosphide precipitates are transgranularly observed for 316FR, although there are grown carbide precipitates at the restricted part of the grain-boundaries.

Fig. 4 shows He bubbles formed at the grain-boundary in type 304 SS and 316FR. Before the creep test, He bubbles exist similarly in both 316FR and type 304 SS specimens. After creep test, however, type 304 SS specimen contains larger amount of accumulated He bubbles

at the grain-boundaries, whilst there is no change in the He bubbles population at grain-boundaries in 316FR specimen. These results suggest that decrease of creep rupture strength of type 304 SS specimen due to irradiation should be attributed to the He bubbles accumulation at the grain-boundaries during the creep test. In the case of 316FR, fine precipitates within the grain could trap He bubbles and prevent them to accumulate at the grain-boundaries. As a result, grain-boundaries sliding or cracking does not easily occur in 316FR specimen. That is reason why the creep rupture strength does not easily deteriorate in 316FR even at higher He production of 2 appm.

In 316FR specimens with higher damage accumulation which were indicated to be decreased in creep rupture strength shown in Fig. 2, grown needle shaped

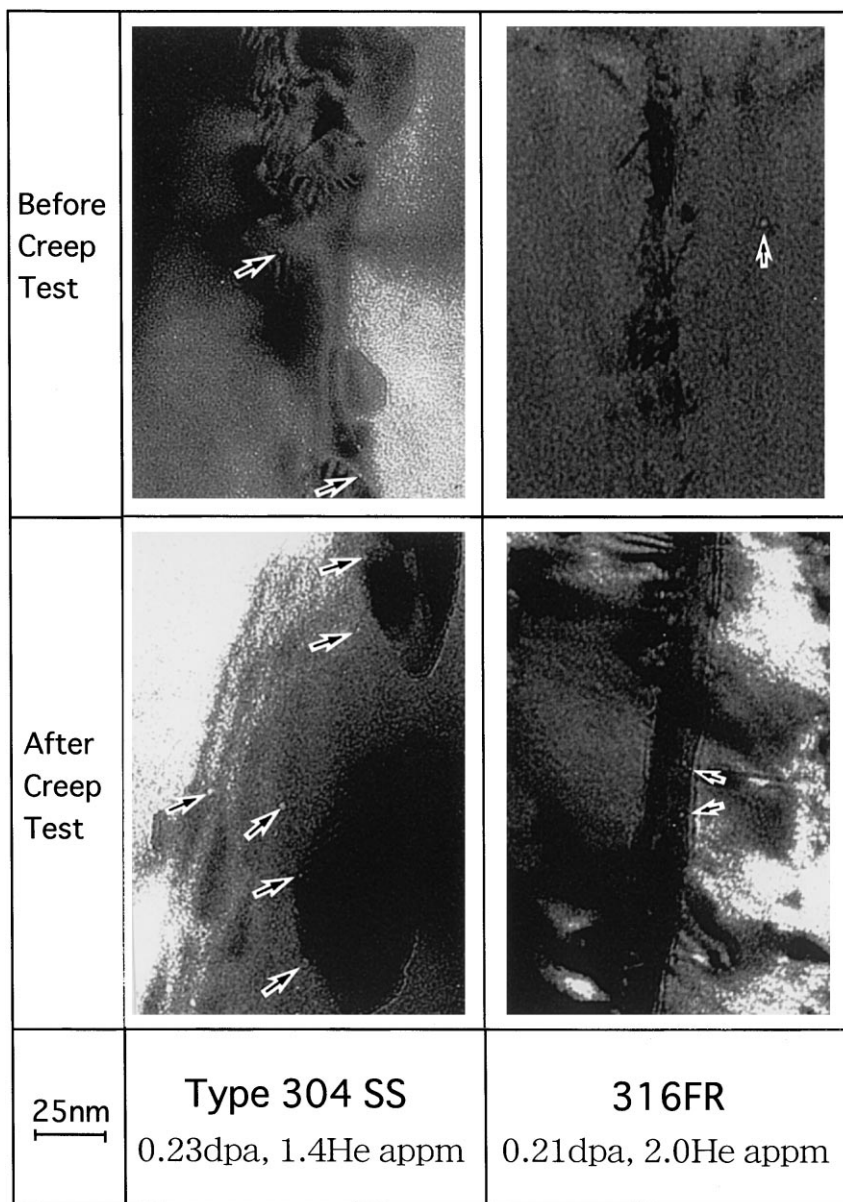


Fig. 4. Transmission electron micrographs of Helium bubbles formed at the grain-boundary of type 304 SS and 316FR.

phosphide precipitates were observed in the grains and  $M_6C$  precipitates decorate along the grain-boundaries as shown in Fig. 5. These precipitates are well known to be formed by the radiation induced solute segregation (RIS), and could be observed just beyond the neutron exposure level of about 0.3 dpa. Therefore, it is considered that a slight degradation of the creep rupture strength of 316FR beyond 0.3 dpa could be ascribed to following three aspects; accelerated He accumulation at grain-boundaries owing to less He trapping capability for growth phosphides, increase of grain-boundary

fracture sensitivity due to decorated  $M_6C$  precipitates along grain-boundaries, and deteriorated phosphorous solution strengthening by decreased phosphorous content in matrix.

Those behavior was interpreted from a viewpoint of fracture surface and ductility. Fig. 6 represents SEM fracture morphologies of creep rupture specimens of irradiated 316FR and the results of image analysis of SEM fracture surface. It is evidently indicated that the fracture mode changes from the transgranular ductile manner to the intergranular brittle one with decreasing

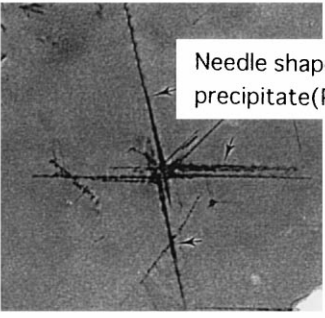
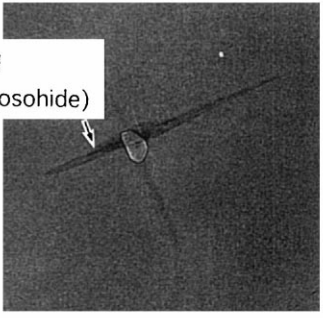
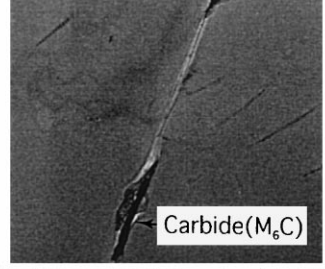
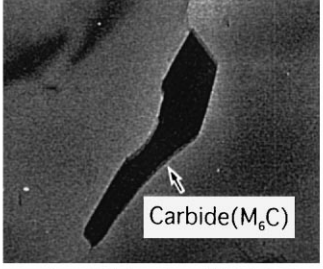
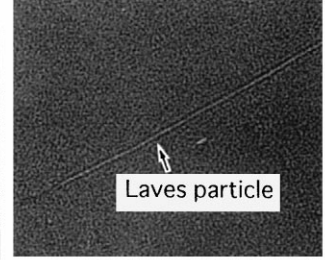
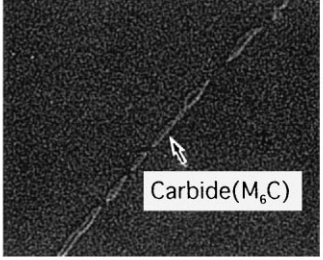
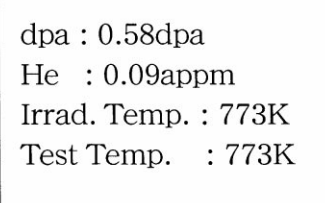
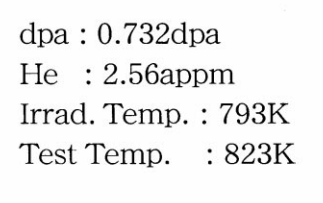
Trans granular	TEM		
			
Inter granular	TEM		
	SEM		
TEM : $\frac{10\text{nm}}$ SEM : $\frac{1\ \mu\text{m}}$		dpa : 0.58dpa He : 0.09appm Irrad. Temp. : 773K Test Temp. : 773K	dpa : 0.732dpa He : 2.56appm Irrad. Temp. : 793K Test Temp. : 823K

Fig. 5. Precipitates in irradiated 316FR at JOYO (After creep test).

time to rupture. The 316FR with intergranular brittle fracture mode corresponds to the specimen irradiated beyond the neutron exposure of 0.3 dpa. Therefore, this behavior should be caused by the intergranular fracture due to accumulation of He bubble at the grain boundary [7].

### 5. Conclusion

Post-irradiation creep rupture strength in 316FR and type 304 SS deteriorates compared to the unirradiated state. The mechanism of such strength degradation

should be different in both materials. It was considered that decreasing of the creep rupture strength in type 304 SS was caused by the He bubbles accumulation at grain-boundaries. As for 316FR, fine precipitates trap He bubbles within grains and prevents the He accumulation at grain-boundaries. Damage accumulation accelerates  $M_6C$  precipitate formation at the grain-boundaries and the phosphide precipitate growth, which deteriorate the He trapping capability and phosphorous solution strengthening in the matrix. Those mechanisms should attribute to the decrease of the post-irradiation creep rupture strength and the grain boundaries brittle fracture mode of 316FR beyond around 0.3 dpa.

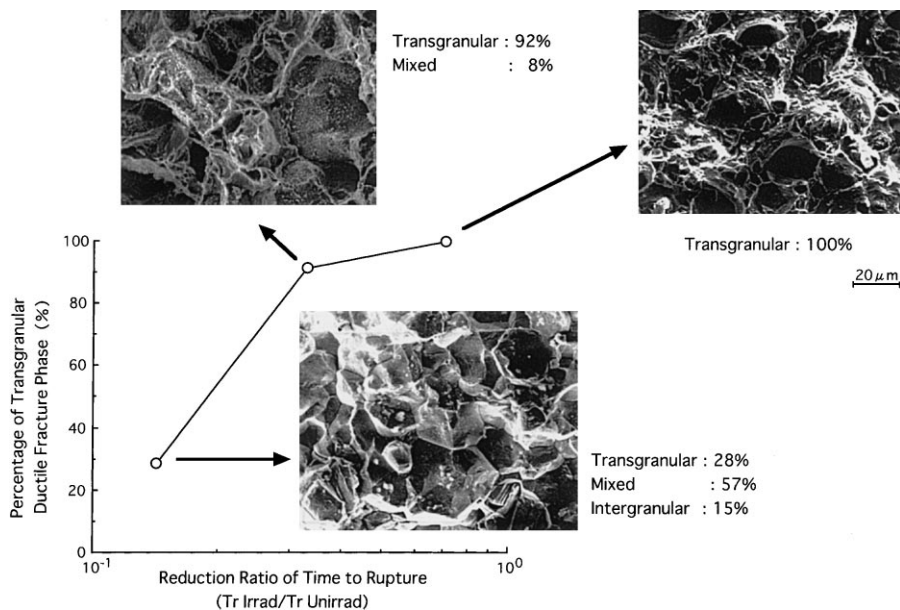


Fig. 6. Relationship of irradiated 316FR at JOYO between percentage of transgranular fracture and reduction ratio of time to rupture with SEM morphologies.

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