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# Burst properties of irradiated oxide dispersion strengthened ferritic steel claddings

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#### Abstract

The effects of fast neutron irradiation on the burst properties of oxide dispersion strengthened (ODS) ferritic steel claddings which were previously manufactured by warm working as the first trial cladding tube manufacturing in Japan Nuclear Cycle Development Institute, were investigated. The samples were irradiated in the experimental fast reactor JOYO using the core material irradiation rig (CMIR) at temperatures between 723 and 878 K to fast neutron fluences ranging from 2.1 to  $4.2 \times 10^{26}$  n/m<sup>2</sup> (E > 0.1 MeV). The burst tests were conducted on a total of four irradiation conditions. The result of burst tests showed that the burst strength of the irradiated claddings was higher than that of unirradiated at the test temperatures up to 873 K and that the diametrical strain just before rupture of irradiated specimens was almost similar to unirradiated one. It was suggested that there was no irradiation embrittlement under the irradiation conditions examined.

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## 1. Introduction

Japan Nuclear Cycle Development Institute (JNC) has made efforts extensively to develop the oxide dispersion strengthened (ODS) ferritic steels, which have more swelling resistance than austenitic steels and are expected to have superior creep strength at elevated temperature of 973 K [1-5]. The ODS ferritic steels are prospective for the long-life cladding materials of fast reactor as well as for the fusion reactor materials due to these excellent characteristic features [6]. Therefore, it is very important to understand the effect of fast neutron irradiation not only on its mechanical properties but also on its microstructures, especially irradiation hardening, irradiation induced embrittlement, dislocation structures, and the stability of oxides and their distribution in order to confirm its superior performance during its service life. But unfortunately, there is very few public data about irradiated ODS ferritic steel claddings, except the microstructural and tensile data of DT2203Y05 irradiated to high doses in the Phenix reactor [7].

The irradiation tests and post-irradiation examinations have been conducted vigorously on the ODS ferritic steel claddings to know their basic irradiation behavior in JNC. The focus of this study is to evaluate the effect of fast neutron irradiation on the burst properties of the previous JNC ODS ferritic steel claddings, which were tube-manufactured by using warm-drawing and warm-rolling technique in 1990 [1].

#### 2. Experimental

## 2.1. Materials and irradiation conditions

The materials examined in this work are two kinds of ODS ferritic steel claddings, which are Fe– $0.05C-13Cr-3W-0.5Ti-0.35Y_2O_3$  (1DK) and Fe– $0.12C-11Cr-3W-0.5Ti-0.5Y_2O_3$  (1DS) (wt%). The argon gas atomized ferritic powder and yttrium oxide powder were mechanically alloyed using a high-energy attrition type ball

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mill and these powders were degassed in vacuum, sealed in can, and consolidated by the hot extrusion at 1423 K. From these consolidated bars, cladding tubes were manufactured by using warm-drawing and warm-rolling techniques [1]. The nominal outer diameter of cladding is 7.5 mm and nominal thickness is 0.4 mm in final products. The chemical composition of 1DK and 1DS cladding tubes is shown in Table 1. The tensile tests on both as-received claddings in longitudinal direction indicated that the yield strength (YS) and ultimate tensile strength (UTS) of 1DK cladding were relatively higher than those of 1DS in temperatures up to 873 K, and that the total elongation of 1DK was a little lower than that of 1DS in relation to strengths [1]. But generally, it is considered that both materials have similar mechanical properties in as-received condition.

The cladding tube specimens (75 mm in length) were irradiated in the experimental fast reactor JOYO using the core material irradiation rig (CMIR) at temperatures between 723 and 878 K to fast neutron fluences ranging from 2.1 to  $4.2 \times 10^{26}$  n/m<sup>2</sup> (E > 0.1 MeV).

#### 2.2. Burst test

The burst tests were carried out using a machine that was specially installed in the hot cell of Materials Monitoring Facility in Oarai Engineering Center. The specimen was plugged in one side and connected to the high-pressure pipe (6.35 mm in diameter) to load the internal pressure of high-purity argon gas from out side of the hot cell. Then, the specimen was inserted in the electric furnace horizontally.

Specimen was firstly heated up to the test temperature and pressurized internally at a ramp rate of  $3.3 \times 10^{-1}$  MPa/s. The rupture stress was measured and the outer diameter at a midpoint in longitudinal direction of the specimen was also measured continuously by using the laser micrometer during test. The test temperatures were 723, 823, 853 and 873 K, which were in accordance with the irradiation temperatures of specimens. The burst tests were conducted in air, but argon gas was flowed around the specimen to prevent an undesired surface oxidation from occurring on the specimen during tests.

# 3. Experimental

The test results of post-irradiation burst tests of each specimen are listed in Table 2 with the irradiation and test conditions. The burst tests on the unirradiated specimens of both materials were also carried out as shown in Table 2. Figs. 1 and 2 show the rupture hoop stress and the diametrical strain just before rupture as a function of test temperature, respectively.

Chemica	d composi	tion of I	DK and	IDS claddi:	ng tubes (	(wt%)										
	C	Si	Mn	Р	S	Cr	ïŻ	Мо	M	Ti	Co	в	z	O (Excessive O <sup>a</sup> )	$\mathbf{Y}_2\mathbf{O}_3^{\mathrm{b}}$	Fe
1DK	0.045	0.019	0.013	< 0.005	0.001	12.87	0.16	Ι	2.81	0.52	I	Ι	0.0152	0.186(0.113)	0.34	Balance
1DS	0.09	0.05	0.03	0.003	0.002	10.98	0.15	< 0.01	2.67	0.40	0.05	0.0003	0.014	0.204 (0.07)	0.40	Balance
<sup>a</sup> Estin	nated froi	n total o	xygen coi	ntent minu.	s oxygen (	coupled w	ith $Y_2O_3$	(total oxy	gen minu:	s Y conter	$11 \times 0.27$ ).					

Table 1

<sup>b</sup> Estimated from Y content with assumption that Y exists as  $Y_2O_3$  (Y content  $\times 1.27$ )

Table 2							
Irradiation	condition	and t	he re	sult c	of the	burst	tests

Experiment	Specimen no.	Material	Irradiation temperature (K)	Fluence $(\times 10^{26} \text{ n/m}^2)^a$	Test temperature (K)	Rupture hoop stress (MPa)	Diametrical strain (%) <sup>b</sup>	Rupture type
JOYO/	S1	1DS	723	3.6	723	1071	0.48	Violent
CMIR	S2	1DS	853	3.4	853	757	0.46	Violent
	K1	1DK	815	4.2	823	983	0.78	Violent
	K2	1DK	878	2.1	873	801	2.19	Violent
Unirradiated	S1C	1DS	_	_	723	1002	0.54	Violent
	S2C	1DS	_	_	853	719	0.70	Violent
	S3C	1DS	_	_	773	971	0.67	Violent
	K1C	1DK	_	_	823	867	0.86	Violent
	K2C	1DK	_	_	873	740	2.54	Violent

All burst tests were conducted at a pressurizing rate of  $3.3 \times 10^{-1}$  MPa/s.

 $^{a}E > 0.1$  MeV.

<sup>b</sup> Diametrical strain just before rupture.



Fig. 1. Rupture hoop stress of 1DK and 1DS ODS ferritic steel claddings.



Fig. 2. Diametrical strain just before rupture of 1DK and 1DS ODS ferritic steel claddings.

For unirradiated materials, the rupture hoop stress (burst strength) of 1DK cladding was higher than that of 1DS in relation to their tensile properties as mentioned above. The diametrical strains just before rupture of both claddings were very low (below 2.5%) in general, especially less than 1% at temperatures ranging from 723 to 853 K. There is no considerable difference in diametrical strain between 1DK and 1DS. It is considered that these lower diametrical strains were due to their characteristic microstructure so called 'bamboo structure' of 1DK and 1DS claddings, where grains were elongated extremely in longitudinal direction by warm-drawing and warm-rolling in tube manufacturing [1].

After irradiation, rupture hoop stresses of irradiated 1DK and 1DS claddings were obviously higher than those of unirradiated specimens in all test temperatures up to 873 K due to irradiation hardening. Irradiation hardening of 1DK seemed to be more evident than that of 1DS under irradiation conditions examined. Diametrical strains just before rupture of irradiated specimens were a little lower than those of unirradiated specimens in each temperature, but no considerable decrease in diametrical strains caused by neutron irradiation was observed under irradiation conditions examined.

#### 4. Discussion

#### 4.1. Burst strength and irradiation hardening

The result of burst tests showed that the increase in burst strength of 1DK and 1DS caused by irradiation hardening occurred in test temperatures up to 873 K. It is indicated that the irradiation hardening of these ODS ferritic steels will appear in temperatures around 873 K, although it is well known that the increase in strength



Fig. 3. The ratio of burst strength, YS and UTS of irradiated claddings to those of unirradiated.

due to irradiation hardening disappears in temperature range more than about 773 K for austenitic steels such as 316 stainless steels [8]. Ferritic steel claddings without oxide dispersion strengthening also showed similar temperature dependence in irradiation hardening to austenitic stainless steels. It is suggested that the ODS ferritic steels would keep the increase in strength due to irradiation hardening at higher temperatures about one hundred degrees than austenitic stainless steels or ferritic steels without oxide dispersion strengthening. Fig. 3 shows the ratio of burst strength before and after irradiation as a function of test temperature. In addition, the ratios of tensile strengths before and after irradiation of DT2203Y05 claddings irradiated in Phenix [7] were also represented in Fig. 3. DT2203Y05 that is a 13% Cr ferritic alloy strengthened by the fine dispersion of yttrium and titanium oxides, is one of the most well known ODS ferritic steels for fast reactor fuel claddings. DT2203Y05 is prepared by the mechanical alloying method from elementary metallic elements and oxides powders in the same way as 1DK and 1DS. As shown in Fig. 3, the ratio of YS is higher than the ratio of UTS of DT2203Y05 claddings in lower temperatures because the increase in strengths caused by the irradiation hardening is more obvious generally on YS than on UTS.

The ratios of burst strength of 1DK and 1DS claddings ranged from 1.05 to 1.13 and most increased at 823 K. Although, in the case of the tensile properties of DT2203Y05 claddings, the ratios of yield and UTSs are higher than that of burst strength of 1DK and 1DS in temperature range up to about 850 K, it is noticed that the irradiation hardening of DT2203Y05 claddings showed similar temperature dependence to that of 1DK and 1DS claddings taking into account that the fluences of DT2203Y05 were much higher than those of 1DK and 1DS. Therefore, it is considered that the extension of temperature range where the strengths were increased by the irradiation hardening is the characteristic irradiation behavior of these ODS ferritic steels.

The microstructural evolution of 1DK and 1DS claddings during irradiation has also been investigated on a total of four irradiation conditions (1DK; 815 K/  $4.2 \times 10^{26}$  n/m<sup>2</sup>, 878 K/2.1 × 10<sup>26</sup> n/m<sup>2</sup>, 1DS; 723 K/  $3.6 \times 10^{26}$  n/m<sup>2</sup>, 853 K/3.4 × 10<sup>26</sup> n/m<sup>2</sup>) [9]. 1DK and 1DS claddings showed almost same microstructure with longitudinally elongated subgrain structures before irradiation. No remarkable change in microstructures of 1DK and 1DS claddings during irradiation was observed except the precipitation of Laves phase on grain boundaries after irradiation. It is suggested that there is no significant difference in network dislocation density between before and after irradiation and that the distribution of finely dispersed oxide particles is relatively stable under irradiation conditions examined. Although not enough information has yet been obtained to explain the cause for the extension of temperature range where the strength increase caused by irradiation hardening occurred from the TEM observation results mentioned above, the formation of Frank loops or any cluster of point defects which TEM observation could not find, and the effect of oxide dispersion strengthening may be considered as the most responsible factors for the irradiation hardening of 1DK and 1DS ODS ferritic steels in higher temperatures. In addition, it may be possible to suggest that the grain boundary is strengthened by Laves phase precipitation and that the higher strength of irradiated specimen comes from such strengthening effect of grain boundary in higher temperatures.

#### 4.2. Ductility and irradiation embrittlement

Although the diametrical strain of 1DK and 1DS claddings was obviously lower than the elongation in longitudinal direction due to their characteristic microstructure originally [1], no significant decrease in diametrical strain was observed after irradiation. This result suggests that the ductility of 1DK and 1DS claddings is not decreased significantly by the irradiation under the irradiation conditions examined. The ratio of diametrical strain just before rupture (diametrical rupture strain) before and after irradiation as a function of test temperature is shown in Fig. 4. The ratio of total elongation of DT2203Y05 claddings before and after irradiation in the tensile tests [7] was also shown in Fig. 4 in the same way as Fig. 3. Fig. 4 indicates that the decrease in ductility after irradiation of 1DK and 1DS is smaller than that of DT2203Y05, although it is necessary to take into account that the neutron fluences of DT2203Y05 claddings was much higher than that of 1DK and 1DS claddings. In addition, it was reported that the miniaturized Charpy impact property of 1DS specimen



Fig. 4. The ratio of diametrical rupture strain and total elongation of irradiated claddings to those of unirradiated.

(1.5 mmW  $\times$  1.5 mmH  $\times$  20 mmL, with V-notch) which was also irradiated in the JOYO using CMIR under the irradiation condition somewhat similar to that of burst test specimens in this study, was excellent [10].

Although the microstructural analysis indicated that the precipitation of  $\alpha'$  and  $\chi$  phase was considered as the main factor of the significant irradiation embrittlement of DT2203Y05 claddings irradiated in the Phenix [7], neither of  $\alpha'$  nor  $\chi$  phase precipitation were observed in 1DK and 1DS claddings [9]. From the viewpoint of the microstructure, the chemical compositions of 1DK and 1DS were designed in order to prevent  $\alpha'$  and  $\chi$  phase from precipitating, for example, they had no Molybdenum and had Chromium less than 12 wt% to suppress  $\chi$ phase and  $\alpha'$  precipitation, respectively [11]. On the other hand, it seems that Laves phase observed on the grain boundaries in 1DK and 1DS after irradiation had no noticeable effect on the irradiation embrittlement in this study. Therefore, from these results and microstructural consideration, it is suggested that the irradiation embrittlement of 1DK and 1DS ODS ferritic claddings was not significant under irradiation conditions examined.

# 5. Conclusions

The post-irradiation burst tests were conducted in order to investigate the effect of fast neutron irradiation on the mechanical properties of the ODS ferritic steel claddings which were manufactured by warm-drawing and warm-rolling as the first trial cladding tube manufacturing in JNC. The result of burst tests indicated that the burst strength of the irradiated claddings was higher than that of unirradiated at the test temperatures up to 873 K and that the diametrical rupture strain after irradiation was almost similar to that of unirradiated claddings. No remarkable deterioration of the burst property caused by the irradiation was observed and this suggested that there was no irradiation embrittlement in 1DK and 1DS ODS ferritic steel claddings under irradiation conditions examined.

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