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Ring-tensile properties of irradiated oxide dispersion strengthened ferritic/martensitic steel claddings

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Abstract

The effects of fast neutron irradiation on ring-tensile properties of the oxide dispersion strengthened (ODS) ferritic/martensitic steel claddings with improved microstructural isotropy from recrystallization and α–γ phase transformation were investigated. The samples were irradiated in the experimental fast reactor JOYO using the material irradiation rig at temperatures between 670 and 807 K to fast neutron fluences ranging from 5.0×10^{25} to 3.0×10^{26} n/m^2 ($E > 0.1$ MeV). The post-irradiation ring-tensile tests were carried out on samples with five sets of irradiation conditions. The experimental results showed that strengths were increased by 10% due to irradiation. Microstructural improvement considerably increased elongations for these samples and specimens retained respectable elongation after irradiation. The ring-tensile properties of these ODS steel claddings remain excellent within the fluence level achieved in this study.

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1. Introduction

Japan Nuclear Cycle Development Institute (JNC) has made extensive efforts to develop oxide dispersion strengthened (ODS) ferritic/martensitic steels, which have more swelling resistance than austenitic steels and are expected to have superior creep strength at an elevated temperature of ~973 K [1–5]. The ODS ferritic/martensitic steels are applicable materials not only for long-life cladding materials in fast reactors, but also for the fusion reactor materials due to their excellent properties [6]. Since earlier JNC-developed ODS ferritic steel claddings had strong anisotropy in their mechanical properties and low elongation in the hoop direction at around 673 K due to strong microstructural anisotropy [1], recrystallization and α–γ phase transformation procedures have been applied in the tube manufacturing process of newer ODS ferritic/martensitic steel claddings in order to improve microstructural anisotropy and to allow manufacturing of cladding tubes by the cold-rolling technique [7]. Unfortunately, there are no neutron irradiation data on these new ODS ferritic/martensitic steel claddings although some ion and electron irradiation data exist [8].

The first neutron irradiation test on these ODS ferritic/martensitic steel claddings has been conducted to understand the fundamental irradiation behavior, especially the effectiveness of microstructural improvement on mechanical properties after neutron irradiation. The focus of this study is to evaluate the effects of neutron irradiation on the ring-tensile properties of the ODS ferritic/martensitic steel claddings with improved microstructures.

2. Experimental procedure

2.1. Materials and irradiation conditions

Three ODS steel cladding materials were examined in this study of composition Fe–0.06C–12Cr–2W–0.3Ti–0.24Y_2O_3 (F94 and F95) and Fe–0.12C–9Cr–2W–0.2Ti–0.35Y_2O_3 (M93) (wt%). Argon gas-atomized ferritic

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powder and yttrium oxide powder were mechanically alloyed using a high energy attrition type ball mill. The mechanical alloying was carried out in an argon gas atmosphere for F95 and M93 and in a helium one for F94. These powders were degassed in vacuum, sealed in cans and consolidated by hot extrusion at 1423 K. From these consolidated bars, cladding tubes were manufactured by a two-pass cold-rolling procedure with intermediate and final heat treatments. Recrystallization occurred in the 12Cr-ODS steel during the final heat treatment (1423 K for 1 h) and the α–γ phase transformation occurred in the 9Cr-ODS steel during the final heat treatment by normalizing at 1323 K for 1 h and tempering at 1023 K followed by very slow air cooling; these procedures ensured the tube microstructures were equiaxed. The final tube nominal outer diameter was 9.3 mm and the thickness was 0.6 mm. The chemical composition of these cladding tubes is shown in Table 1.

The 5 mm wide ring-tensile specimens with 1.5 mm wide gauge section were prepared from the cladding tubes using an electro-discharge machine. Fig. 1 shows a schematic drawing of a specimen. These ring-tensile samples were irradiated to five irradiation conditions in the experimental fast reactor JOYO using the material irradiation rig at temperatures between 670 and 807 K to fast neutron fluences ranging from 5.0×10^{25} to 3.0×10^{26} n/m^2 ($E > 0.1$ MeV).

For comparison, 11Cr-ODS ferritic steel cladding (1DS) which was manufactured into tubes by warm-working [1] was used. Their chemical composition is also shown in Table 1. The ring-tensile samples of 1DS claddings were irradiated separately in JOYO using the core material irradiation rig at temperatures between 667 and 812 K to fast neutron fluences ranging from 4.5×10^{25} to 3.6×10^{26} n/m^2 ($E > 0.1$ MeV).

2.2. Ring-tensile tests

The ring-tensile test is used to measure strength and elongation in the hoop direction of cladding tube specimens to duplicate thermal and mechanical stresses under internal pressure conditions in actual fuel pins. It is very important to examine the mechanical properties of JNC ODS ferritic/martensitic steel claddings in the hoop direction because of characteristic microstructural anisotropy [1,9].

The ring-tensile tests were carried out in the air using a screw-driven tensile testing machine and all tests were conducted at a cross-head speed of 1.67×10^{-3} mm/s (with an initial strain rate of 8.3×10^{-4}/s). The test temperatures were 673, 723, 773, 798 and 823 K to match the irradiation temperatures. Yield strength was determined by the 0.2% offset method. Uniform elongation and total elongation were obtained from the engineering stress–strain curve.

Table 1
Chemical composition of F94, F95, M93 and 1DS cladding tubes (wt%)

	C	Si	Mn	P	S	Ni	Cr	W	Ti	Y_2O_3[a]	Excess O[b]	N	Ar	Fe	Remarks
F94	0.058	0.03	0.049	0.004	0.004	0.025	11.78	1.93	0.30	0.24	0.04	0.010	0.0003	Bal.	Mechanically alloyed in helium, as-heat treatened
F95	0.056	0.03	0.048	0.003	0.004	0.025	11.72	1.92	0.31	0.24	0.04	0.010	0.0038	Bal.	Mechanically alloyed in argon, as-heat treatened
M93	0.12	0.02	0.036	0.003	0.004	0.022	8.99	1.94	0.20	0.35	0.06	0.010	0.0033	Bal.	Mechanically alloyed in argon, as-heat treatened
1DS	0.09	0.05	0.03	0.003	0.002	0.15	10.98	2.67	0.40	0.40	0.119	0.014	–	Bal.	Mechanically alloyed in argon, as-warm worked

[a] Estimated from Y content with assumption that Y exists as Y_2O_3 (Y content×1.27).

[b] Estimated from total oxygen content minus oxygen coupled with Y_2O_3 (total oxygen minus Y content×0.27).

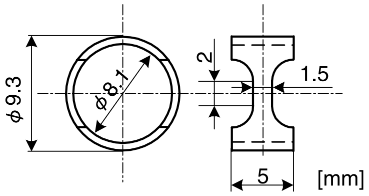

Fig. 1. Dimensions of ring-tensile specimen.

3. Experimental results

This was the first mechanical tests on irradiated ODS ferritic/martensitic steel claddings having microstructures improved by recrystallization or α–γ phase transformation. Ring-tensile tests must also be used for unirradiated samples because the deformation of ring-tensile specimens may differ from those of the usual uniaxial tensile tests, especially at the beginning of the tests. Yield strength of irradiated samples as a function of test temperature is shown in Fig. 2 along with yield strength of unirradiated samples. For unirradiated samples, yield strength decreased with increasing test temperature. Strengths increased in the order F94 < F95 < M93 for these test temperatures and the strength of 1DS was similar to that of M93. It is expected that strength levels for these materials would correspond with Y_2O_3 content. After irradiation, yield strengths of irradiated F94, F95 and M93 claddings were modestly higher (less than 10%) than those of the unirradiated specimens at all test temperatures up to 798 K due to irradiation hardening. Irradiation hardening of 1DS was larger for the irradiation conditions examined. The trends in ultimate tensile strength (UTS) were similar to those for yield strength as a function of test temperature.

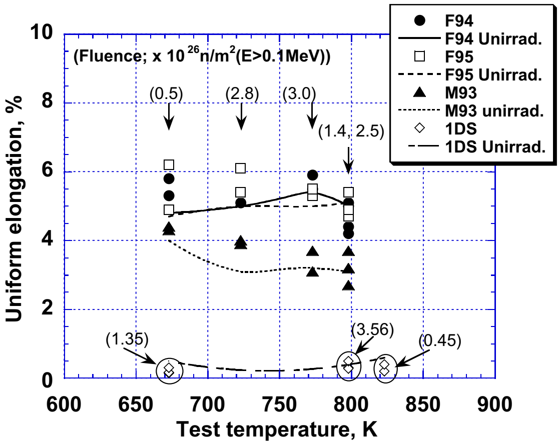

Fig. 3. Uniform elongation of ODS ferritic/martensitic steel claddings before and after irradiation.

Fig. 3 plots uniform elongation before and after irradiation as a function of test temperature. Uniform elongation for unirradiated F94 and F95 cladding was almost the same at all test temperatures and uniform elongation of M93 was lower in relation to strength. Uniform elongation in the hoop direction for all three claddings was more than 3% at these test temperatures although that of 1DS was particularly low (less than 1%) due to its microstructural anisotropy [1,9]. Fig. 3 indicates that there was no significant change in uniform elongations for F94, F95 and M93 between before and after irradiation. Total elongation as a function of test temperature is shown in Fig. 4. Before irradiation, total elongation for F94 and F95 increased similarly with increasing test temperature with F95 higher than F94 at all test temperatures. Total elongation for unirradiated M93 was almost as high as that of F95, but it decreased with increasing test temperature from 673 to 773 K and increased noticeably at 798 K. After irradiation, total elongations for both F94 and M93 decreased remark-

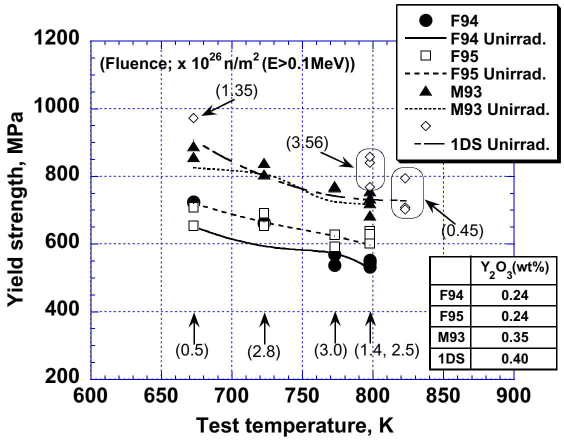

Fig. 2. Yield strength of ODS ferritic/martensitic steel claddings before and after irradiation.

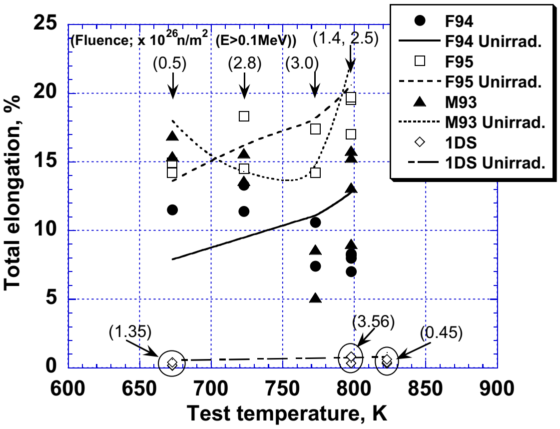

Fig. 4. Total elongation of ODS ferritic/martensitic steel claddings before and after irradiation.

ably at test temperatures higher than 773 K, although there was no obvious change for F95. Total elongations generally ranged from 5% to 20% in this study, so it seemed that respectable ductility was retained and no irradiation embrittlement occurred under these irradiation conditions.

There were no significant effects of fluence on strength and elongation under the irradiation conditions in this study.

4. Discussion

4.1. Strength and irradiation hardening

The experimental results show that the increase in yield strength and UTS of F94, F95 and M93 caused by irradiation hardening is moderate at lower temperatures (~673 K) and that strengths of irradiated samples are almost as same as those of unirradiated samples at 773 and 798 K. This indicates that oxide dispersion suppresses immoderate irradiation hardening at lower temperatures (~673 K), although 9Cr–1MoVNb steel without oxide dispersion showed considerable increase in strengths caused by irradiation hardening at 663 K [10]. In addition, it is suggested that oxide dispersion restrains irradiation-enhanced softening in these materials at temperatures higher than 798 K since the irradiated 11Cr-ODS ferritic steel claddings showed obvious increase in burst strength at irradiation temperatures up to 878 K [9]. For 12Cr-ODS steels, the strength of F95 is relatively higher than that of F94. The difference between these materials is basically only the mechanical alloying gas used (Ar for F95 and He for F94), but the effect of such gases on strength is not yet understood.

In order to evaluate the work hardening, the ratio of yield strength and UTS as a function of test temperature is shown in Fig. 5. The ratio of strength in 1DS cladding is very high (more than 0.9) in spite of irradiation because 1DS was originally very hard [1]. On the other hand, the ratios of F94, F95 and M93 claddings range from 0.76 to 0.9 and there is no large change caused by irradiation, in particular the ratio for F95 decreases after irradiation at temperatures below 773 K. It should be emphasized that these ODS steels with improved microstructure retained considerable workhardenability after irradiation and this resulted in respectable uniform elongation. Experimental data obtained in this study suggest that irradiation causes quite small effects on mechanical properties in the irradiation conditions examined in this study.

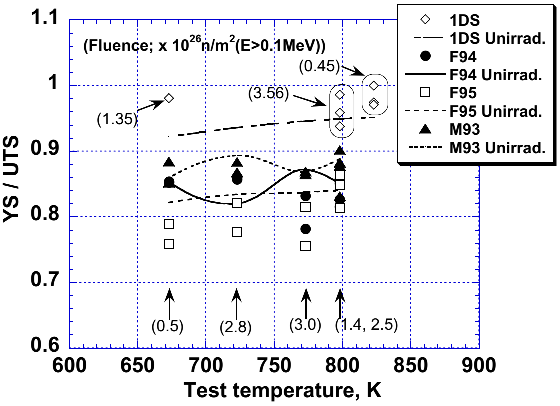

Fig. 5. The ratio of yield and ultimate tensile strength of ODS ferritic/martensitic steel claddings.

4.2. Ductility and microstructural improvement

As shown in Figs. 3 and 4, the hoop direction elongation for F94, F95 and M93 claddings was improved significantly by the microstructural improvement and it was retained sufficiently after irradiation. This indicates that the microstructural improvement by recrystallization or α–γ phase transformation is quite effective in maintaining the well-balanced mechanical properties, especially strength and ductility for ODS steel cladding, not only for as-received conditions but also following irradiation. On the other hand, as-mentioned above, the total elongation for F94 and M93 decreased at higher temperatures. From TEM observations on these claddings, it can be suggested that the decrease in total elongation for M93 is related to coarsening of $M_{23}C_6$ type carbides and precipitation of Laves phase or MC type carbides above 773 K [11]. Unfortunately, not enough information has been obtained yet to explain the cause of the difference in total elongation between F94 and F95. TEM observations indicated no remarkable changes in microstructures during irradiation, such as dislocations and precipitates, could be observed in F94, F95 and M93 claddings [11]. In addition, it has also been reported that the distribution and structure of Y_2O_3 oxide particles were stable during irradiation [12]. These microstructural data suggest that the oxide particles would be effective sinks for irradiation defects and this results in the suppression of irradiation hardening. Therefore, from these results and microstructural considerations, it is expected that the current JNC ODS ferritic/martensitic steel claddings with improved microstructure will show excellent mechanical properties at high fluence, where the microstructures, including the oxide distribution and structure, are stable.

5. Conclusions

Post-irradiation ring-tensile tests were conducted on ODS ferritic/martensitic steel cladding with microstructures improved by recrystallization or α–γ phase transformation. The experimental results showed that irradiation increased strength by 10%. Heat treatment increased elongation which was retained at respectable levels after irradiation. It is expected that these excellent properties following irradiation are due to the stability of the microstructures which suppress irradiation hardening during irradiation at least within the fluence level in this study.

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