Effects of Thermal Aging on the Mechanical Properties of FeCrAl-ODS Alloy Claddings

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The FeCrAl-ODS alloy claddings were manufactured and Vickers hardness, ring tensile tests and transmission electron microscopy (TEM) observations of these claddings were performed to investigate the effects of thermal aging at 450°C for 5,000 and 15,000 h. The age-hardening of all FeCrAl-ODS alloy cladding was found. In addition, the significant increase in tensile strength was accompanied by much larger loss of ductility. It was suggested that this age-hardening behavior was attributed to the (Ti, Al)-enriched phase (β' phase) and the α' phase precipitates (content of Al is <7 mass%). In comparison with FeCrAl-ODS alloys with almost same chemical compositions, there was significant age-hardening in both alloys. However, the extrusion bar with no-recrystallized structures was keeping good ductility. It was suggested that this different behavior of reduction ductility was attributed to the effects of grain boundaries, dislocation densities and specimen preparation direction. [doi:10.2320/matertrans.MT-M2021057]

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1. Introduction

Oxide dispersion strengthened (ODS) steel has been studied for use as potential fuel cladding materials in fast reactors (FRs) owing to their excellent resistance to swelling and their high-temperature strength.^{1–5} Therefore, Japan Atomic Energy Agency (JAEA) has selected ODS steel claddings for use in the Japan Sodium-cooled Fast Reactor (JSFR) to achieve higher burnup with a higher coolant temperature.⁶ On the other hand, zirconium (Zr)-based alloys have exhibited a number of desirable features as a cladding material for light water reactors (LWRs) in steady-state operation for about half century. However, in light of the event occurred at the Fukushima Daiichi nuclear power plant in March 2011, there has been an increased research effort into accident tolerant fuel (ATF) cladding materials, especially, to reduce the burden on the emergency core cooling system (ECCS) during severe accidents by decreasing the rate and total amount of heat generated from cladding oxidation in high temperature steam.⁷ Therefore, development of ATF cladding materials, such as FeCrAl alloy,^{8–10} surface-coated zircaloy^{11–13} and SiC/SiC composite,^{14,15} has been worldwide studied. FeCrAl-ODS alloys have been also developed in one of ATF cladding materials^{4,16–19} that attempt to deal with these challenges in current LWRs due to their high temperature strength and oxidation resistance in the presence of high temperature steam. On the other hand, it is well-known that the decomposition of the ferritic phase to Fe-rich phase (α -phase) and Cr-rich phase (α' -phase) in the temperature range of 280–500°C, which is called “475°C embrittlement”, occurs in high-Cr (above 13 mass%) ferritic, martensitic^{20,21} and duplex steels.^{22–25} It is very important to evaluate this aging embrittlement because the mechanical properties are degraded during the long-time thermal aging at 300–500°C. There are, however, no published reports on FeCrAl-ODS alloys cladding tube manufacturing and me-

chanical properties of the recrystallized cladding after thermal aging, although there are many published reports on plates and bars.^{17,26,27}

In this study, cladding tube manufacturing by means of Pilger mill rolling and subsequent recrystallization heat-treatment were characterized as a function of Cr, Al and Zr contents. In addition, the thermal aging behavior of the recrystallized FeCrAl-ODS alloy claddings at 450°C for 5,000 and 15,000 h was investigated by Vickers hardness, ring tensile tests and transmission electron microscopy (TEM).

2. Experimental Procedure

2.1 Manufacturing process

The manufacturing process of the FeCrAl-ODS alloy cladding tubes is shown in Fig. 1. Nine mechanical alloyed powders in parameter of Cr, Al, Zr and oxygen contents were manufactured in Ar-gas atmosphere by using a 10 kg attritor. Excess oxygen (Ex. O) was controlled by Fe₂O₃ powder addition during mechanical alloying, where Ex. O was estimated by subtracting the oxygen present in Y₂O₃ from the total amount of oxygen. Mechanical alloyed powders were sealed in 76 mm diameter cans, and degassed at 400°C in 0.1 Pa vacuum for 2 h. With these materials, raw bars in 25 mm diameter were produced by hot-extrusion at elevated temperature of 1,150°C. The chemical composition of the raw bars is shown in Table 1, where SP1 does not contain Al, and SP8, SP9, SP10 and SP15 contain Zr.

The raw bars were machined and drilled to mother tubes of 18 mm diameter, 3 mm thickness and about 180 mm length for claddings. Cold-rolling using a pilger mill was repeated four times resulting in final claddings with dimensions of 8.5 mm outer diameter and 0.5 mm wall thickness. The reduction ratio by the pilger mill was approximately 45% for each pass. Intermediate recovery heat treatment was carried out to soften the cold-rolling tube, and the final heat treatment for recrystallized structures was conducted at 1,150°C for 1 h.

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Fig. 1 Cladding tube manufacturing process for all ODS alloys.

2.2 Evaluation tests

Optical microstructure observation and hardness measurements were carried out at the stage of manufacturing process by cold-rolling and heat-treatment in order to assess the amount of work hardening or softening, and grain morphology changes.

Thermal aging was carried out for the recrystallized claddings at 450°C for 5,000 and 15,000 h in vacuum capsules at less than 10–4 Pa pressure to prevent specimen's oxidation. Micro-Vickers hardness tests were conducted on the center of wall thickness of claddings with a load of 500 gf and 10 seconds dwelling time for each aged cladding, and the average value was used to evaluate the hardness evolution.

The ring tensile test was used to investigate thermal aging effects on tensile properties in the hoop direction of claddings. It is important to examine the mechanical properties in the hoop direction, because recrystallized ODS alloy claddings have microstructural anisotropies. A drawing of the ring tensile test specimen is shown in Fig. 2. Ring tensile tests were carried out in air using a screw-driven tensile test machine at a constant strain rate of 8.3×10^{-4} s⁻¹. The test temperatures were room temperature (RT) and

Fig. 2 Schematic drawing of ring tensile test specimen.

Table 1 Chemical compositions of manufactured ODS alloy claddings (mass%).

Material	Lot	C	Cr	W	Ti	Al	Y	Zr	O	N	Ar	Y ₂ O ₃ ^{a)}	ZrO ₂ ^{b)}	ExO ^{c)}	Fe
12Cr-2W-ODS	SP1	0.027	11.78	1.9	0.23	0	0.19	<0.01	0.13	0.006	0.006	0.241	—	0.079	Bal.
15Cr-5Al-ODS	SP4	0.031	14.16	—	0.5	4.57	0.37	<0.01	0.22	0.005	0.006	0.470	—	0.120	Bal.
15Cr-7Al-ODS	SP7	0.031	14.18	—	0.51	6.44	0.37	<0.01	0.22	0.006	0.006	0.470	—	0.120	Bal.
15Cr-7Al-0.4Zr-0.07ExO-ODS	SP8	0.033	14.66	—	0.50	6.39	0.37	0.35	0.19	0.004	0.005	0.470	0.47	0.090	Bal.
15Cr-7Al-0.4Zr-0.14ExO-ODS	SP9	0.030	14.70	—	0.50	6.38	0.36	0.36	0.25	0.005	0.005	0.457	0.49	0.153	Bal.
15Cr-7Al-0.4Zr-0.21ExO-ODS	SP10	0.029	14.76	—	0.50	6.40	0.37	0.37	0.32	0.004	0.005	0.470	0.50	0.220	Bal.
15Cr-7Al-0.2Zr-0.12ExO	SP15	0.03	15.07	—	0.51	6.99	0.4	0.19	0.24	0.004	0.006	0.508	0.25	0.132	Bal.
14Cr-7.5Al-0.5Ti-0.12ExO	SP18	0.03	14.1	—	0.52	7.49	0.39	<0.01	0.21	0.005	0.005	0.4953	—	0.1047	Bal.
10Cr-7Al-ODS	SP19	0.025	10.21	—	0.46	7.05	0.4	<0.01	0.22	0.004	0.006	0.508	—	0.112	Bal.

a) Estimated Y concentration with assumption that Y exists as Y₂O₃.

b) Estimated Zr concentration with assumption that Zr exists as ZrO₂.

c) Estimated by subtracting the oxygen present Y₂O₃ from total amount of oxygen in the steel.

Fig. 3 Vickers hardness history of cladding tube manufacturing (a) with only Al and (b) with both Al and Zr.

450°C. Yield strength (YS) was determined as 0.2% offset proof stress. Uniform elongation (UE) and total elongation (TE) were obtained from engineering stress-strain curves. Fracture features and cross-sectional views of the fracture surfaces were observed with a Hitachi S-3400N scanning electron microscope (SEM).

For microstructural observations by transmission electron microscopy (TEM) after thermal aging, specimens were sliced from claddings and mechanically thinned to about 70 μm ; and then 3 mm disks in diameter were punched from the sheets. The thin foils for TEM were prepared using a conventional twin jet electro polisher in an electrolytic solution of $\text{HClO}_4\text{:CH}_3\text{OH:C}_6\text{H}_{14}\text{O}_2 = 1\text{:}10\text{:}6$. The Sample of claddings (lot SP19) after thermal aging at 450°C for 5,000 h were observed using a FE-TEM (FEI Talos-F200X) operated at 200 kV.

3. Results

3.1 Hardness changes at cladding manufacturing

The results of hardness change induced by cold-rolling (CR) and heat-treatment (HT) in the course of cladding manufacturing are shown in Fig. 3. In these processes, there are two key factors: keeping hardness less than 400 Hv for next CR and producing the recrystallized structure at the final HT for cladding products. All recovered structures gave hardness under 400 Hv. Hardness and HT temperatures of FeCrAl-ODS alloys with or without Zr at each pass were much lower than SP1 without Al and Zr, as shown in Fig. 3. The typical optical microstructure changes before and after the final HT for recrystallization are shown in Fig. 4, which indicate that there is no significant change in the degree of recrystallization for all alloys, but that the recrystallization

Fig. 4 Typical optical microstructures before and after final heat treatment: (a) SP1, (b) SP7, (c) SP8 and (d) SP19 with the same magnification.

temperature of SP1 without Al is about 100°C higher. It is known that the Al addition in FeCrAl-ODS alloy causes a decrease in strength due to the formation of coarsened yttrium (Y)–Al oxide particles with decreasing number density instead of finely distributed Y–Ti oxide particles in ODS alloy without Al.²⁹⁾ Therefore, it is considered that decrease of hardness, HT and recrystallization temperatures are attributable to the decrease in the pinning force by the coarsened oxide particles due to enlargement of space distance between oxide particles.

3.2 Mechanical property changes by thermal aging

Vickers hardness values and the change of age-hardening ($\text{Shift in Hv}(\Delta\text{Hv}) = \text{Hv}_{\text{aged}} - \text{Hv}_{\text{as-received}}$) as a function of aging time are shown in Figs. 5(a), (c) and (b), (d), respectively. There is no significant difference in Vickers hardness of SP1 before and after thermal aging. It is suggested that oxide particle and matrix in the 12Cr-ODS alloy without Al and Zr (lot SP1) are stable during thermal aging. On the other hand, the increase of age-hardening is observed for all FeCrAl-ODS alloys and the aging time of saturation for age-hardening is almost 5,000 h. In the FeCrAl-ODS alloys with same Cr content (15Cr), the increase of age-hardening in FeCrAl-ODS alloys with Zr (lot SP8, 9, 10, 15) is much higher than those of FeCrAl-ODS alloys without Zr (lot SP4, 7, 18).

Figures 6(a) and (b) show typical engineering stress-strain (SS) curves of FeCrAl-ODS alloys (lot SP7 and SP9) tested at RT and 450°C before and after thermal aging at 450°C for 15,000 h, respectively. In Fig. 6, closed and open symbols, and solid and dashed lines indicate aged and as-received data, respectively. At RT, a few FeCrAl-ODS alloy with Zr are breached without plastic deformation or breached immediately after UTS, namely did not have plastic instability, as

Fig. 5 Two properties of as a function of aging time at 450°C: (a)–(c) Vickers hardness and (b)–(d) Shift in Vickers hardness.

Fig. 6 Typical engineering stress-strain curves at (a) RT and (b) 450°C before and after thermal aging.

shown in Fig. 6(a). The UTS and TE obtained from other SS curves tested at RT and 450°C are summarized in Figs. 7(a)–(d) and (e)–(h), respectively. The significant age-hardening between 50 and 300 MPa at RT and 450°C was accompanied by much larger loss of ductility, except for SP1 and SP19, as shown in Fig. 7. The tendency of decrease in TE was corresponding to the increase in tensile strength.

Compared to tensile properties at RT and 450°C, there was no significant change of increase in tensile strength, but reduced ductility at RT was larger than that at 450°C. On the other hand, it is well known that the criterion for plastic deformation is 1.0% to meet the conditions of no systematic fuel rod damage in LWRs.³⁰⁾ In this study, the TEs of almost all FeCrAl-ODS alloy claddings were higher than this criterion even after thermal aging tests.

Figure 8(a)–(b) and (c)–(d) show the typical results obtained after tensile failure of FeCrAl-ODS alloy claddings (lot SP7 and SP9), which were aged at 450 for 15,000 h, tested at RT and 450°C, respectively. At RT, the fracture surface showed cleavage like facets with river patterns and the fracture mode appears to be brittle fracture as shown in Figs. 8(a) and (b). In contrast, at 450°C, the fracture mode appears to be dimpled rupture and these observations indicated that fracture occurred in ductile mode at 450°C. The tendency of observations was corresponding to SS curves as shown in Fig. 6.

4. Discussion

As-shown in the previous section, the Vickers hardness and tensile strength increased as compared with that of as-received FeCrAl-ODS alloy claddings, namely, age-hardening of all FeCrAl-ODS alloy occurred. P. Dou *et al.* reported that α' phase precipitation in 15Cr-ODS steels could be completely suppressed when the content of Al was equal to or higher than 7 mass%, but significant precipitation of (Ti,Al)-enriched phase (β' phase) occurred in all the Al alloyed 15Cr-ODS steels.²⁷⁾ The β' phase corresponds to the B2-ordered FeAl phase with the Ti substituting for the Al.²⁸⁾ Therefore, in this study, it is suggested that the age-hardening of FeCrAl-ODS alloys (lot SP4: 15Cr-5Al) is due to both α' and β' phase precipitations, and that the age-hardening in the other 15Cr-7Al-ODS alloys is attributed to only the β' phase

Fig. 7 Tensile properties tested at (a)–(d) RT and (e)–(h) 450°C.

precipitation, including hardening of SP19 (10Cr-7Al) even at only 10 mass% Cr.

To investigate the precipitation behavior of the α' and β' phases in our FeCrAl-ODS alloys, TEM observations were performed for SP19 aged at 450°C for 5,000 h. The macroscopic bright-field (BF) image of Fig. 9(a) shows typical recrystallized structure with the elongated grains. The arrow in Fig. 9(a) indicates ring tensile direction, which is perpendicular to the elongated grain direction. High-magnification BF and dark-field images of the square, which are indicated in Fig. 9(a), are shown in Fig. 9(b) and (c), respectively. A kind of extra spots besides BCC matrix reflection are observed in the electron diffraction patterns of aged SP19 as shown in Fig. 9(b). In Figs. 9(b) and (c), only fine oxide particles are distributed in the matrix and no precipitates such as ordered phase, which contributes to age-hardening, are observed. Furthermore, the high-angle annular dark-field (HAADF) image with corresponding its EDS element maps is shown in Fig. 10. The Cr-enriched α' phase

precipitate cannot be found and oxide particles are identified as Y–Al–O and Y–Ti–O according to these element maps. Furthermore, fine Ti-enriched precipitates, which are smaller size than oxide particles, are also observed. It is difficult to identify a chemical form of these precipitates; however, these Ti-enriched precipitates is considered to be (Ti, Al)-enriched β' phase based on Dou's microstructural results.²⁷⁾

Although age-hardening in high Cr steels generally causes an increase in tensile strength and a decrease in tensile elongation, P. Dou *et al.*²⁷⁾ reported that the significant age-hardening at RT in bar type tensile specimens of FeCrAl-ODS alloys (extrusion bars) in parallel with the extrusion direction was accompanied by a rather small loss of ductility with a reduction in TE less than 3% as shown in Fig. 11. For comparison, the results of SP4 and SP7, which have almost same chemical compositions with Dou's alloys, were also given in Fig. 11. In addition, the change of the hardness due to thermal aging is also shown as the ΔH_v value of both alloys. The difference between our materials and Dou's alloys

Fig. 8 Typical SEM fracture images of SP7 and SP9 claddings after ring tensile tests at RT (a)–(b) and 450°C (c)–(d).

are with or without recrystallized structures. However, in this study, the significant age-hardening was accompanied by much larger loss of ductility as shown in Fig. 11. The tendency of decrease in TE corresponds to the increase in tensile strength. It is considered that both α' and β' phase precipitations occurred in Dou's and our FeCrAl-ODS alloys after aging because there wasn't significant difference tendency in increase of Vickers hardness of both alloys. Therefore, it is suggested that recrystallized structures such as claddings are more sensitive to α' and β' phase precipitations with hard and brittleness than no-recrystallized structures

such as extrusion bars. No-recrystallized structures such as extrusion bars consist of small grains with fine oxide particles and high dislocation density, but recrystallized structures consist of large grains with only fine oxide particles. It is considered that α' and β' phase precipitations would be preferential path for crack propagation, but many grain boundaries would act as barriers to this crack propagation in no-recrystallized structures, which resulted in keeping ductility. Moreover, in general, ODS alloy strength is weaker perpendicular to the extrusion direction, particularly for high-Cr ODS alloys, although structure of high Cr-ODS alloys is

Fig. 9 TEM images of SP19 aged at 450°C for 15,000 h: (a) macroscopic bright-field (BF) image, (b) BF image and (c) dark-field image of oxide particles in the matrix.

Fig. 10 HAADF-STEM image with its corresponding EDS elemental maps of SP19 aged at 450°C for 15,000 h.

Fig. 11 Comparison with bar type tensile²⁷⁾ and ring tensile data in FeCrAl-ODS alloys with almost same chemical compositions.

fully recrystallized to overcome this strength anisotropy. In addition, it is known that the effects of specimen preparation direction are greater in tensile elongation rather than in tensile strength, namely, elongation of T-direction is smaller than that of L-direction.³¹⁾ Therefore, it is also suggested that decrease in ring tensile elongation (T-direction) of FeCrAl-ODS alloy claddings are attributed to the specimen preparation direction, compared to that of bar type tensile specimens (L-direction). It is suggested that this different behavior of reduction ductility in FeCrAl ODS alloys with

almost same chemical compositions due to age-hardening was attributed to the effects of grain boundaries, dislocation densities and specimen preparation direction.

5. Conclusions

The manufacturing and mechanical properties of the recrystallized FeCrAl-ODS alloy claddings were investigated after thermal aging at 450°C for 5,000 and 15,000 h. The results of this study are summarized as follows:

- (1) There is no significant difference in the degree of recrystallization for all alloys with Al and Zr or without Al, but the recrystallization temperature of SP1 without Al is about 100°C higher. It is suggested that the results are attributable to the formation of coarsened Y–Al oxide particles with decreasing number density in Al-contained ODS alloys instead of finely distributed Y–Ti oxide particles.
- (2) The age-hardening of the recrystallized FeCrAl-ODS alloy claddings were characterized. The significant increase in tensile strength is accompanied by much larger loss of ductility. It is considered that this age-hardening behavior is attributed to the (Ti, Al)-enriched β' phase precipitate and the α' phase precipitate for Al content <7 mass%.
- (3) In comparison with FeCrAl-ODS alloys with almost same chemical compositions, there was significant age-hardening in both alloys. However, age-hardening

was accompanied by much larger loss of ductility in recrystallized claddings, although extrusion bar was keeping good ductility. It is suggested that this different behavior of reduction ductility was attributed to the effects of grain boundaries, dislocation densities and specimen preparation direction.

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