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Preliminary Study on the Cold Working Process of the High Cr ODS Ferritic Steels for Cladding Tubes

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1. Introduction

Oxide dispersion strengthened (ODS) steel is the most promising structural material for advanced nuclear systems, because of excellent creep and irradiation resistance [1]. This is mainly attributed to uniformly distributed nano-oxide particle with a high density, which is extremely stable at the high temperature and acts as effective obstacles when the dislocations are moving in ferritic/martensitic matrix. Recently, advanced radiation resistant ODS steel (ARROS) has been newly developed and its cladding tube has been successfully fabricated for the in-core structural components in SFR, which has very attractive microstructures to achieve both superior creep and radiation resistances at high temperatures [2]. However, several intermediate heat treatment was essentially required to fabricate the cladding tube with a sufficient dimension. Therefore, not only high strength, but workability of the ODS steel is also very important for structural component productivity.

In this study, cold workability of newly developing high Cr ferritic ODS steels was examined in a laboratory scale. Microstructures as well as mechanical properties were also investigated to determine the optimized condition of the mechanical properties and fabrication processes.

2. Methods and Results

2.1 Experimental procedure

To fabricate high Cr ferritic ODS steels in this study, a commercial stainless steel powder was employed and its chemical composition was Fe(bal.)-22Cr-4.5Al-3Mo-0.5Si-0.02C in wt%. This alloy shows excellent corrosion, oxidation resistance with good phase stability at high temperatures due to the dense formation of passive film, consisted of chromium and aluminum oxides on the surface. Some additional minor elements were incorporated in raw material preparation. Titanium is well known to refine the strengthening dispersoids forming Y-Ti-O type complex oxides with a high number density [3]. To investigate the effect of alloying elements, furthermore, zirconium was also added in 0.6 wt%, tentatively. The chemical compositions of the ferritic ODS steels were summarized in Table I and their fabrication processes could be found in previous study [3].

The ODS steels were fabricated by mechanical alloying (MA) and uniaxial hot pressing (UHP) processes. The MA is essential process that the continuous collision between grinding media and raw powders with a high revolving energy makes the repeated crushes and cold welding of powders, which eventually create the homogenous mixing and alloying in the constitution elements. The commercial alloy powders and some raw powders were mechanically alloyed by a planetary ball-mill apparatus. The atmosphere was thoroughly controlled in ultra-high purity argon (99.9999%) gas. The MA was performed with a ball-to-powder weight ratio of 10:1. The MA powder was then consolidated using UHP at 1150 ºC for 2h at a heating rate of 10ºC/min. The process was carried out in a high vacuum ($<5\times10^{-4}$ Pa) under a hydrostatic pressure of 80 MPa in uni-axial compressive loading mode. After the process, the pressure was relieved and the samples were cooled in the furnace. For microstructural observations, ODS steels were mechanically wet ground and a twin-jet polished to fabricate the thin foil specimens using a solution of 5% $HClO_4$ + 95% methanol in vol. % at 18V with 0.5mA at -40 ℃. The grain morphology and precipitate distributions were observed by a transmission electron microscope. To evaluate the strength, tensile tests were carried out at room temperature and elevated temperatures. The miniaturized and sheet-typed tensile specimens were machined with 5mm of a gauge length, 1.2mm of a width and 0.5mm of a thickness. Tensile tests were performed at room temperature and 700 ℃ at a strain rate of $6.7\times10^{-3}s^{-1}$ in the air. To investigate the cold workability, ODS ferritic steels were cold-rolled in a direction and Vickers hardness tests were also carried out under 4.9N for 10s during the cold rolling process.

Table I: Nominal compositions of the ODS steels (in wt%)

Elements	Alloys	
	ODS1	ODS2
Fe	bal.	bal.
Cr	22.0	22.0
Al	4.5	4.5
Mo	3.0	3.0
Si	0.5	0.5
C	<0.02	<0.02
Ti	0.5	0.5
Zr	-	0.6
Y_2O_3	0.35	0.35

2.2 Microstructure and tensile properties of ODS ferritic steels

Microstructural images of grain morphology on ODS ferritic steels are shown in Fig. 1. All ODS steels showed typically equiaxed ferrite grains because of uniaxial hot pressing process. Both ODS steels with Ti (ODS1) and Zr (ODS2) had quite homogeneous grain distributions as shown in Fig. 1(a) and (b). However, the ODS steel with Zr addition had extremely finer grains than ODS steel with Ti. Mean grain sizes of ODS steels with Ti and Zr were evaluated as 480 and 160nm, respectively.

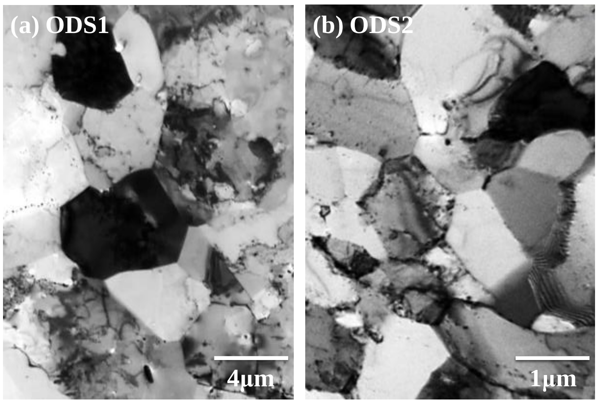

Fig. 1. Grain morphology of the ODS ferritic steels with (a)Ti and (b)Zr additions.

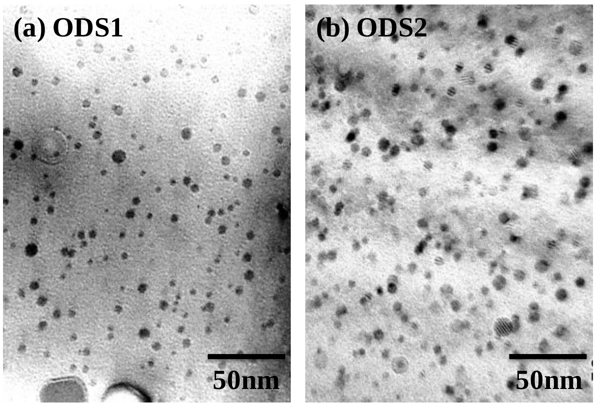

Fig. 2. Oxide particles in a micro-grain of the ODS ferritic steels with (a)Ti and (b)Zr additions.

In Fig.2, bright field TEM images showing the nano-oxide particle distributions in a micro-grain of the ODS ferritic steels were presented. The ODS1 showed very fine and uniform distribution of oxide particles with a mean diameter of 5.2 nm. In contrast, ODS2 had slightly coarser oxide particles than ODS1. Mean diameters were evaluated as 6.7 nm. Analysis results of the chemical elements by the TEM-EDS revealed that fine oxide particles in the micro-grains of ODS1 were composed of Y-Ti-O and Y-Si-O complex oxides. ODS2 showed different oxide particles in the micro-grains. Y-Zr-O complex oxides were mainly observed. Interestingly, oxide particles in the ODS2 with higher number density were observed than those in ODS1 as shown in Fig. 2. It is estimated that Zr additions in the ODS ferritic steel leads to the significant decrease of the grain size, but and higher number density of oxide particles.

In Fig. 3, tensile properties of the ODS ferritic steels was summarized. Slightly increase on the yield strength at room temperature and 700 °C was estimated by the Zr addition. It seemed that microstructural differences on the grain size and particle number density were not so critical to the tensile properties. More detailed estimation will be performed to investigate the mechanical properties on the ODS ferritic steels.

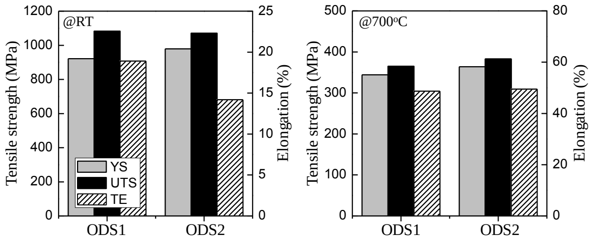

Fig. 3. Tensile properties of ODS ferritic steels at RT and 700 °C.

2.3 Cold rolling tests of ODS ferritic steels for a tubing process

Tubing processes such as piercing, drawing, and pilgering are essential process for fabrication of tube components. The ARROS thin-walled tube was successfully fabricated and a pilgering process was very effective technique [2]. To simulate the tubing process of ODS ferritic steels, preliminary cold rolling test was conducted and their hardness changes were investigated. Outward appearances of cold rolled ODS steels were shown in Fig. 4. As increase the thickness reduction rate from 40% to 90%, no cracking and tearing were observed in the edge of each specimen. In Fig. 5, hardness changes of ODS ferrtic steels during the cold rolling processes was presented. Hardness of both ODS steels were about 350Hv, however, these were slightly increased to 460Hv. In spite of high hardness during the process, very thin sheets with a thickness of 0.4mm were successfully fabricated as shown in Fig. 4.

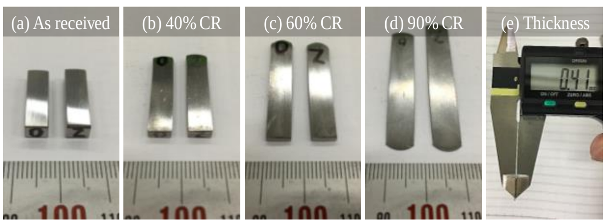

Fig. 4 Outward appearances of cold rolled ODS ferritic steels and thickness measurement.

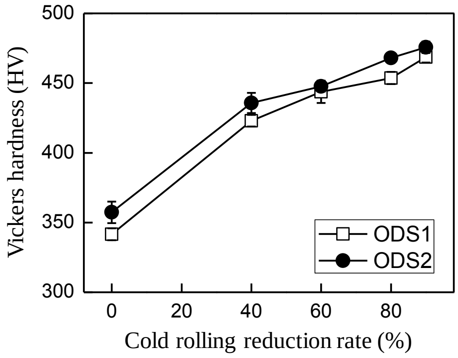

Fig. 5 Hardness changes during cold rolling process of ODS ferritic steels.

3. Conclusions

This study examined characterization and cold working processes of newly developing high Cr ODS ferritic steels for tube components of advanced nuclear systems. It is estimated that ODS ferrtic steel with Zr additions showed favorable microstructure and tensile properties. Very thin sheets of ODS ferritic steels with a thickness of 0.4mm could be successfully fabricated by cold rolling processes with a thickness reduction rate, 90%.

Acknowledgements

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REFERENCES

[1] T.K. Kim, S. Noh, S.H. Kang, J.J. Park, H.J. Jin, M.K. Lee, J. Jang, and C.K. Rhee, Nuclear Engineering and Technology, Vol.48, p.572, 2016.
[2] S. Noh, Y.C. Kim, H.J. Jin, B.K. Choi, S.H. Kang, T.K. Kim, Transactions of the Korean Nuclear Society Spring Meeting, 2016.
[3] S. Ukai, T. Nishida, H. Okada, T. Okuda, M. Fujiwara, and K. Asabe, Journal of Nuclear Science and Technology, Vol.34, p. 256, 1997.