Microhardness and microstructure of Ferritic-Martensitic ODS steel tube fabricated by a pilger process

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1. Introduction

Sodium cooled fast reactor (SFR) is one of the fourthgeneration nuclear systems with efficiency, safety, and reliability. To achieve this system, it is necessary to develop the improved structural material having both high strength and irradiation resistance at high temperatures [1]. Ferritic-martensitic (FM) steels are attractive for the structural materials of the future generation nuclear systems owing to excellent thermal conductivity and good swelling resistance. Unfortunately, the available temperature range of FM steel is limited up to 650°C [2]. Accordingly, high temperature strength should be improved for an application of the FM steels to the structural materials of SFR. FM ODS (oxide dispersion strengthened) steel is the most promising structural material for SFR because of excellent creep and irradiation resistance by uniformly distributed nano-oxide particles which is extremely stable at the high temperature [3-5].

investigates This studv microhardness and microstructure of FM ODS steel tube fabricated by a pilger process. For this, 10Cr-1Mo FM ODS steel tube was prepared by mechanical alloying (MA), hot extrusion, pilgering and heat treatment (HT). Hardness measurement was carried out for mother tube, pilgering and HT to evaluate the effects of tube fabrication the mechanical property. process on The microstructures were observed using electron backscatter diffraction (EBSD) and transmission electron microscope (TEM).

2. Experimental procedure

The work presented here is focused on Fe (bal.)-10Cr-1Mo (in wt.%) FM ODS steel. The chemical composition of the sample is presented in Table 1.

Metallic powders and yttria powder were mechanically alloyed by a horizontal ball-mill apparatus, CM-08, under high purity Ar gas atmosphere. The mechanical alloying was performed at an impeller rotation speed 240rpm for 48hr with a ball-to-powder weight ratio (BPWR) of 10:1. MA powders were then placed in a carbon steel container. The sealed capsules were degassed at 400°C below $5x10^{-4}$ Pa for 3hr. The hot extrusion was carried out by a 600 ton capacity press for several seconds with 6.3:1 extrusion ratio after annealing in the furnace at $1100 \degree$ for 2hr.

Mother tube of FM ODS steel fabricated by mechanical process after annealing in the furnace at 1150° for 1hr followed by a furnace cooling (OD : 18.50mm, thickness : 1.25mm). The thin ODS steel tube was made by means of a pilger cold rolling process with a cross-section reduction ratio of about 21% (OD : 15.88mm, thickness : 1.15mm). After pilger rolling, FM ODS steel was heat-treated at 1150°C for 1h and slowly cooled because our previous study [6] revealed that the heat treatment performed in the austenitic region at 1150°C for 1h followed by furnace cooling were proper to guarantee safe fabricating for FM ODS steel.

Table 1. Chemical compositions of FM ODS steel (wt.%)

Fe	С	Cr	Mo	Ti	Mn	v	Y ₂ O ₃
Bal.	0.15	10	1	0.20	0.50	0.10	0.25

Hardness Electron Back-Scatter measurement, Diffraction (EBSD) and Transmission Electron Microscope (TEM) are used to characterize the mechanical properties and microstructure of FM ODS steel tube fabricated by a pilger process. Samples are taken at different steps of the fabrication route. Hardness measurement were conducted under 0.5Kg f loading. EBSD scans were performed with a FE-SEM (JSM-7100F) on cross-sections relative to the extrusion or rolling direction. Samples for EBSD were mechanically wet ground and electro-polished in 10% HClO₄ + 90% ethanol at -39° C with 20.5V. TEM helped to illustrate the precipitate of FM ODS steel tubes. TEM observations were carried out on a JEM-2100 JEOL.

3. Results and Discussion

To evaluate the workability of FM ODS steel, hardness measurement during tube fabrication was carried out. Fig. 1. shows Vickers microhardness of FM ODS steel tube fabricated by a pilger process. The hardness of the mother tube was measured to be about 244 Hv, and it increased up to 287 Hv after pilgering. Due to heat treatment at 1150° for 1hr followed by a furnace

cooling, the hardness of the tube was indicated to be 241 Hv. $\,$

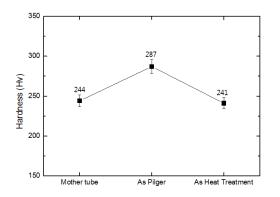


Fig. 1. Hardness change in the course of FM ODS steel tube fabrication.

EBSD images on microstructure of mother tube, pilgering and heat treatment (HT) were shown in Fig. 2. The EBSD image indicates that the tube after pilgering consists of elongated grains along with the rolling direction. On the other hand, the heat-treated tube shows a homogenization and grain growth at 1150° C for 1hr followed by a slow cooling. This observation suggests that furnace cooling heat treatment from the austenization temperature lead to the very large ferrite grains by diffusional transformation.

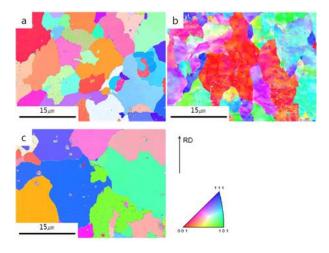


Fig. 2. EBSD image of FM ODS steel tube (a) mother tube (b) pilgering and (c) heat treatment (HT).

TEM micrographs of the FM ODS steel tube fabricated by the pilger process show carbides and oxide particles (Fig. 3.). In the case of heat treatment tube, the size of carbides was larger than mother and/or pilgered tube. It is considered that the coarse carbides form by the considerable internal stress due to the rolling process and slow cooling. On the contrary, oxide particles are uniformly dispersed in the matrix during the tube fabrication process.

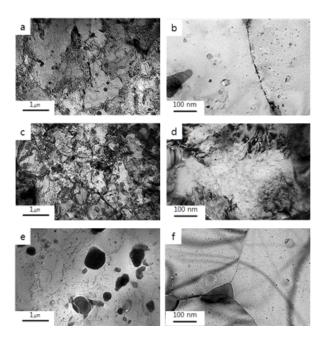


Fig. 3. Precipitates in FM ODS steel tube (a, b) mother tube (c, d) pilgering and (e, f) heat treatment (HT).

3. Conclusions

In this study, microhardness and microstructure of ferritic-martensitic ODS steel tube fabricated by a pilger process was investigated. The FM ODS steel tube after pilgering indicated high hardness value by the high density dislocation due to cold rolling. On the other hands, heat treatment tube at 1150° C for 1hr followed by a furnace cooling showed the very large ferrite grains and coarse carbides, leading to the softening of the strength for the FM ODS steel tube. It is believed that these results will be helpful in the development of FM ODS steel tube.

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REFERENCES

[1] T.K. Kim, S.H. Kim, Journal of Nuclear Materials, Vol. 411, pp. 208, 2011.

[2] B. Raj, M. Vijayalakshmi, Comprehensive of Nuclear Materials, Vol. 4, pp. 97-121, 2012

[3] S. Ukai, M. Harada, H. Okada, M. Inoue, S. Nomura, S. Shikakura, K. Asabe, T. Nishida, M. Fujiwara, J. Nucl. Master., Vol. 204, pp. 65, 1993.

[4] D. K. Mukhopadhyay, F. H. Froes, D. S. Gelles. J.

Nucl. Master., Vol. 258-263, pp. 1209, 1998.

[5] S. Ukai, Comprehansive of Nuclear Materials, Vol. 4, pp. 241-271, 2012

[6] H. J. Jin et al., Transactions of Korean Nuclear Society Spring Meeting, P04I10, 2015