Effect of an intermediate heat treatment during a cold rolling on microstructures and mechanical properties of 10Cr-1Mo ODS steel

Hyun Ju Jin*, Ki Baik Kim, Byoung Kwon Choi, Suk Hoon Kang, Sang Hoon Noh, Ga Eon Kim and Tae Kyu Kim
Nuclear Materials Development Division, Korea Atomic Energy Research Institute, Yuseong-gu, Daejeon, Republic of Korea
**Corresponding author: hjin@kaeri.re.kr*

1. Introduction

With a recent accelerated evolution of nuclear reactors combined with ever-increasing world energy demands, Generation IV future nuclear systems have spurred considerable research and developmental interests in several countries with nuclear power reactors because it is believed that the Gen IV improves efficiency, safety, reliability, and proliferation-resistance of nuclear reactors [1]. To achieve them, improved core structural materials with a potential to be applicable at elevated temperature under severe neutron exposure environment are required. Ferritic/martensitic (FM) steels are very attractive for the structural materials of fast fission reactors such as a sodium cooled fast reactor (SFR) owing to their excellent irradiation resistance to a void swelling [2,3], but are known to reveal an abrupt loss of their creep and tensile strengths at temperatures above 600 °C [4]. Accordingly, high temperature strength should be considerably improved for an application of the FM steel to the structural materials of SFR. Oxide dispersion strengthened (ODS) FM steels are considered to be promising candidate materials for high- temperature components operating in severe environments such as nuclear fusion and fission systems due to their excellent high temperature strength and radiation resistance stemming from the addition of extremely thermally stable oxide particles dispersed in the ferritic/martensitic matrix [5-7]. Since the nano-oxide particles can never be subsequently dissolved or refined at any stage of the fabrication route, ODS steels are usually characterized by low ductility and high hardness at room temperature. This low cold workability, which makes the manufacturing more complicated, implies that intermediate softening heat treatments are essential.

This study investigates effects of an intermediate heat treatment during a cold rolling on microstructures and mechanical properties of 10Cr-1Mo FM ODS steel. For this, 10 Cr -1Mo FM ODS steel was prepared by mechanical alloying (MA), hot isostatic pressing (HIP), and hot extrusion process. Hardness measurements were carried out after the cold rolling process and intermediate heat treatments to evaluate the influences of the intermediate heat treatments on the mechanical properties. The microstructures were observed using SEM, electron back-scatter diffraction (EBSD) and transmission electron microscopy (TEM) with energy dispersive spectroscopy (EDS).

2. Experimental procedure

The work presented here was focused on FM ODS steel, the chemical composition of which is given in Table 1. The FM ODS steel, sample A: Fe-10Cr-1Mo and sample in wt% was fabricated by MA and HIP processes.

Table 1. Chemical composition (wt. %) of 10Cr-1Mo ODS FM steel.

Alloy (wt.%)	Fe	Cr	Mo	Mn	V	Ti	C	Y_2O_3
A : 10Cr-1Mo	Bal.	10	1.2	0.5	0.15	0.25	0.13	0.35

Pre-mixed metallic raw powders and yttria powder were mechanically alloyed by a horizontal ball-mill apparatus, CM-08, under a high purity Ar gas (purity in 99.999%) atmosphere. The mechanical alloying was performed at an impeller rotation speed of 300rpm for 40hrs with a ball-to-powder weight ratio (BPWR) of 10:1. MA powders were then placed in an AISI 304 L stainless steel containers. The sealed capsules were degassed at 500 ^{o}C below 5×10^{-3} torr for 1h. The HIP was carried out at 1150 ^{o}C under a pressure of 100 MPa for 4 hr at a heating rate of 5^{o}C/min and followed by furnace cooling. Hipped samples were hot-extruded by a 600 ton capacity press for several seconds with a 6.3: 1 extrusion ratio after annealing in the furnace at 1100 ^{o}C for 2h. The hot-extruded specimen was annealed at 1150 ^{o}C during 4min followed by a slow cooling to obtain a homogenized and softened microstructure. The annealed specimen was cold-rolled with a cross-section reduction ratio of about 10%. After the cold rolling, various intermediate heat treatments were employed, as given in Table 2.

Table 2. Intermediate heat treatment conditions of 10Cr-1Mo ODS FM steel.

Sample ID	Intermediate heat treatment (IHT) conditions		
	Heat ID	Temp.(°C)	Time (hr)_Cooling type
A: 10Cr-1Mo	IHT 1	1050°C	4min_FC
	IHT 2	1150°C	4min_FC
	IHT 3	780°C	1hr_AC
	IHT 4	1050°C X 780°C	(1hr_AC) X (1hr_AC)

To evaluate the effect of the intermediate heat treatment during the cold rolling on mechanical property, hardness measurements were conducted by using a microhardness tester under a load of 0.5kgf.

Microstructure of the specimen was characterized by an optical microscopy. Samples for the optical microscopy were mechanically wet ground and chemically etched in 5% aqua regia solution for 10min. The grain morphology was observed by SEM and EBSD after the electro-polishing in a 5% $HClO_4$ + 95% methanol solution in vol. % at 18V with 0.5mA at -50 °C. To examine the size distribution and the elemental analyses on the precipitates, TEM observation with EDS was carried out. For this, the carbon extraction replicas were prepared by means of a mechanical polishing, etching with a mixed solution of 93 vol.% water, 5 vol.% nitric acid and 2 vol.% fluoric acid, a carbon coating, and removing the replicas by electrochemical etching with a mixed solution of 90 vol.% methanol and 10 vol.% hydrochloric acid.

3. Results and Discussions

The workability of steels can be estimated by hardness measurement during the cold rolling followed by the heat treatments. Fig.1 shows the effects of the cold rolling and intermediate heat treatment on the Vickers microhardness of the specimen. The hardness of the mother plate was measured to be about 335 Hv, and it increased up to 393 Hv after 10% cold rolling. After normalizing at 1050 ^{o}C and 1150 ^{o}C for 4 min followed by furnace cooling at 5ºC/min (designated as IHT1 and IHT 2), the hardness was measured to be 333 and 332 Hv, respectively. On the other hand, the hardness was identified to be 365 Hv after tempering at 780 ºC for 1h, air cooling at 150ºC/min. In the case of IHT 4, which was normalized at 1050 ^{o}C for 1h and tempered at 780 ºC for 1h, air cooling at 150ºC/min, the hardness value was similar to IHT3. From these results, it was found that the IHT1 and/or IHT2 heat treatments are proper to avoid any damage during the fabrication route.

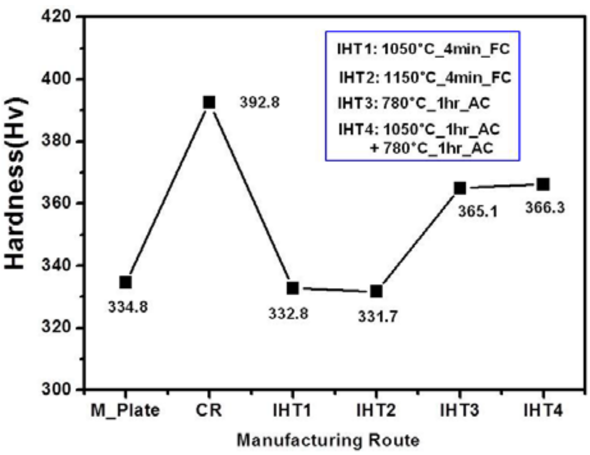

Fig. 1. Effects of the cold rolling and intermediate heat treatments on the Vickers microhardness of 10Cr FM ODS steel.

Optical microstructures of FM ODS steels after the hot extrusion and the cold rolling are shown in Fig. 2. The results clearly show that the specimen after the hot extrusion and cold rolling consists of fine equiaxed grains and a portion of elongated grains along with the extrusion and rolling direction. Considering the fact that the temperature is increased from room temperature to 1150^{o}C for the HIP process, it is considered that fine equiaxed grains are martensite and the elongated grains parallel to the rolling direction are delta-ferrite, which reside untransformed without transforming into gamma during the rise of temperature. In Fig. 2(b), deformed microstructures were observed and the grain sizes were refined after the cold rolling.

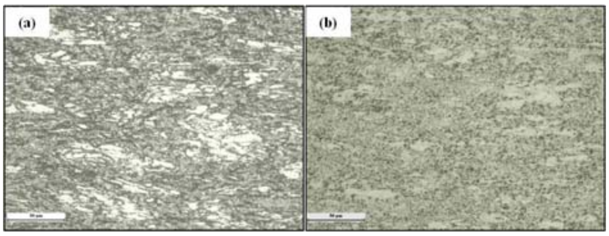

Fig. 2. Optical micrographs of (a) hot-extruded and (b) cold-rolled 10Cr-1Mo FM ODS steels.

In IHT1 and IHT2, it can be seen that very large ferrite grains with a low fraction of martensite were observed as shown in Fig. 3(c, d). Since the cooling rate of 5ºC/min from the normalizing temperature is very slow, the ferrite grains grew over 10μm. This observation suggests that the furnace cooling heat treatment from the austenization temperature leads to the very large ferrite grains by diffusional transformation, resulting in the softened FM ODS steel at room temperature. In the case of IHT3 and IHT4, finely dispersed carbides along the grain boundaries were observed due to the tempering heat treatment at 780ºC. IHT3 consists of coarsened grains, compared to IHT4, but its size was smaller than IHT1 and/or IHT2. In IHT4, it has a typical tempered martensite structure consisting of very fine martensitic grains due to the normalizing heat treatment. Based on the results that we made, the IHT1 and/or IHT2 heat treatments during the cold rolling would be effective to make 10Cr-1Mo FM ODS steel softened in order to guarantee safe forming during the fabrication route.

4. Conclusions

In the present study, the effects of an intermediate heat treatment during a cold rolling on microstructures and mechanical properties of 10Cr-1Mo FM ODS steel were investigated. The FM ODS steels were manufactured by the MA, HIP and hot-extrusion processes. Intermediated heat treatments including furnace cooling, tempering, and normalizing-tempering with air cooling were performed after the cold rolling. Slow cooling from the austenitic domain achieves a softened ferritic structure with larger grains, compared to tempering, and normalizing-tempering with air cooling, which can be easily further cold working. It is believed that these preliminary results will be helpful in the development of advanced FM ODS steel.

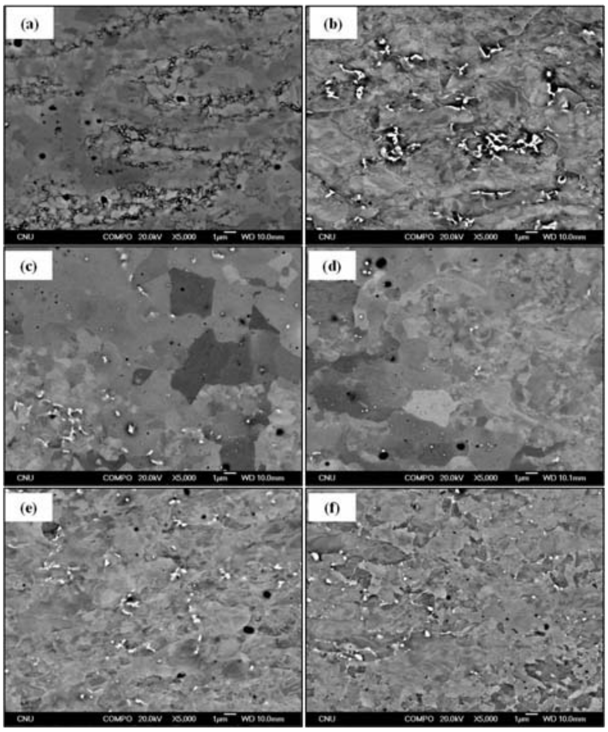

Fig. 3. SEM micrographs of the grain morphology for 10Cr FM ODS steels in different intermediate heat treatment conditions are presented: (a) hot-extruded, (b) cold rolled, (c) IHT1, (d) IHT2, (e) IHT3, and (f) IHT4

ACKNOWLEDGEMENT

This work was supported by the National Research Foundation of Korea (NRF) grant funded by the Korea government(2012M2A8A1027872)

REFERENCES

[1] T. K. Kim and S. H. Kim, J. Nucl. Mater. 411 (2011) 208–212.
[2] F.A. Garner, M.B. Tgoloczko, B.H. Sencer, J. Nucl. Mater. 276 (2000) 123.
[3] R.L. Klueh, A.T. Lelson, J. Nucl. Mater. 371 (2007) 37.
[4] T.R. Allen, J. Gan, J.I. Cole, M.K. Miller, J.T. Busby, S. Shutthanandan, S. Thevnthasan, J. Nucl. Mater. 375 (2008) 26.
[5] S. Ukai, M. Harada, H. Okada, M. Inoue, S. Nomura, S.Shikakura, K. Asabe, T. Nishida, M. Fujiwara, J. Nucl. Mater. 204 (1993) 65.
[6] D.K. Mukhopadhyay, F.H. Froes, D.S. Gelles, J. Nucl. Mater. 258–263 (1998) 1209.
[7] S. Ukai, Comprehensive of Nuclear Materials, pp. 241-271, Elsevier, Holland (2012).