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Influence of heat treatment conditions on microstructures and mechanical properties of ferritic-martensitic ODS steel

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1. Introduction

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 [1,2], but are known to reveal an abrupt loss of their creep and tensile strengths at temperatures above 600 °C [3]. 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 [4-6]. To develop an advanced ODS steel for core structural materials for next generation nuclear reactor system applications, it is important to optimize its heat treatment conditions to improve the high temperature strength and radiation resistance.

This study investigates effects of heat treatment conditions on microstructures and mechanical properties of FM ODS steel. For this, 10Cr -1Mo FM ODS steel was prepared by mechanical alloying (MA), hot isostatic pressing (HIP), and hot extrusion process. Hardness measurements were carried out after heat treatments to evaluate the influences of 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

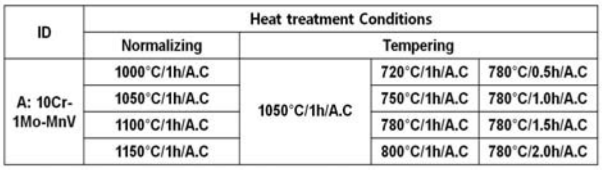
The work presented here was focused on FM ODS steel, the chemical composition of which is given in Table 1. The FM ODS steel, sample A: Fe-10Cr-1Mo and sample in wt% was fabricated by MA and HIP processes.

Table 1. Chemical composition (wt. %) of 10Cr-1Mo FM ODS 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 °C below 5×10^{-3} torr for 1h. The HIP was carried out at 1150 °C under a pressure of 100 MPa for 4 hr at a heating rate of 5°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 °C for 2h. After the hot- extrusion, various heat treatments were employed, as given in Table 2.

Table 2. Normalizing and tempering conditions employed for 10Cr-1Mo ODS FM steel in the present study.



ID	**Heat treatment Conditions**			
	Normalizing	**Tempering**		
A: 10Cr-1Mo-MnV	**1000°C/1h/A.C**	**1050°C/1h/A.C**	**720°C/1h/A.C**	**780°C/0.5h/A.C**
	1050°C/1h/A.C		**750°C/1h/A.C**	**780°C/1.0h/A.C**
	1100°C/1h/A.C		**780°C/1h/A.C**	**780°C/1.5h/A.C**
	1150°C/1h/A.C		**800°C/1h/A.C**	**780°C/2.0h/A.C**

Microstructure of the annealed specimen was characterized by SEM and EBSD. Samples for EBSD were prepared by 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

To determine temperature regimes for different phase domains for 10Cr-1Mo FM ODS, differential scanning calorimetry(DSC) technique was employed. A typical DSC profile obtained during heating of the specimen, employing a rate of 10 K min^{-1} is shown in Fig. 1. It was found that onset temperature of phase transformation from α to γ in 10Cr-1Mo FM ODS was 960°C as the result of DSC analysis, as shown in Fig. 1.

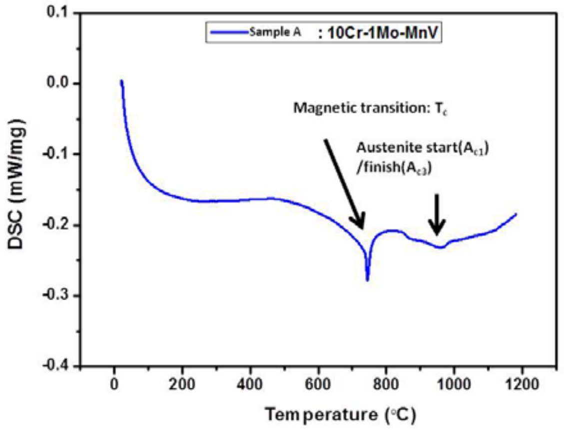


Fig. 1. DSC spectrum of as-extruded 10Cr-1Mo FM ODS steel heated at 10K/min

Based on the results of DSC analysis, as presented in Table 2, after various heat treatment processes, hardness measurement were performed to evaluate the effects of heat treatment conditions on mechanical properties of 10Cr-1Mo FM ODS steel. Fig. 2 shows the variations of Vickers hardness with (a) normalizing temperatures, (b) tempering temperatures, and (c) tempering time of 10Cr-1Mo FM ODS steel. In Fig. 2(a) showing the results of hardness measurement with normalizing temperature, it was found that the hardness value at 1050 ^{o}C was higher than other temperature regions. In the case of Fig. 2(b) describing the results of hardness measurement with tempering temperature, the maximum hardness value was found to be about 400 Hv at 780 ^{o}C. In addition, the optimized tempering time was found to be 1.0h at 780 ^{o}C after normalizing at 1050 ^{o}C. These results suggest that the optimized heat treatment for 10Cr-1Mo FM ODS steel to enhance the mechanical property is normalized at 1050 ^{o}C for 1h and tempered at 780 ^{o}C for 1h.

Optical microstructure and SEM micrograph of 10Cr-1Mo FM ODS steel which was normalized at 1050^{o}C for 1h and tempered at 780 ºC for 1h with air cooling are shown in Fig. 3. The results clearly show that martensite structures with finely dispersed carbides along the grain boundaries, as well as delta-ferrite structures are observed. 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. Based on the comparisons with different heat treatment conditions that we made, coupled with the increase of martensite structures, increased number of density of precipitates enables the conclusion that a proper heat treatment plays an important role in the improvement of mechanical properties, as depicted in Fig.2.

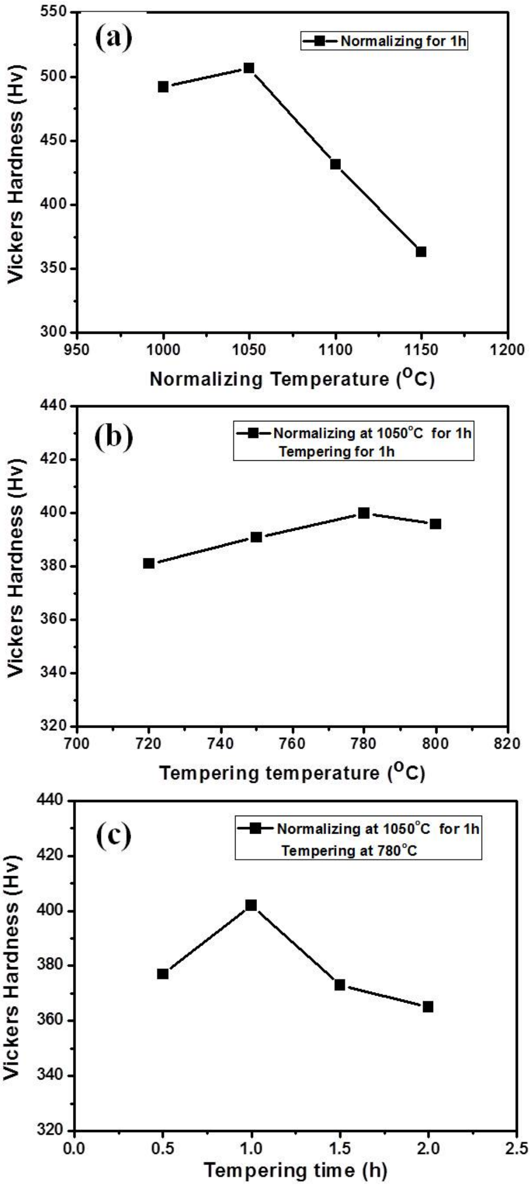


Fig. 2. Variation of Vickers hardness with (a) normalizing temperatures, (b) tempering temperatures, and (c) tempering time of 10Cr-1Mo FM ODS steel.

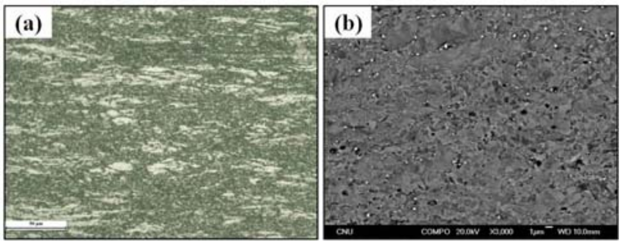


Fig. 3. (a) OM and (b) SEM micrographs of 10Cr-1Mo FM ODS steel which was normalized at 1050 ^{o}C for 1h and tempered at 780 ºC for 1h, air cooling.

4. Conclusions

This study investigated the effects of heat treatment conditions on microstructures and mechanical properties of FM ODS steel. The FM ODS steels were fabricated by the MA, HIP and hot-extrusion processes. The optimized heat treatment condition for 10Cr-1Mo FM ODS was determined to enhance the mechanical property. It is believed that these preliminary results can be useful in development of advanced FM ODS steel.

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