Study on microstructures and tensile properties with addition of V and Sc in ODS ferritic/martensitic steels

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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 compositions to improve the high temperature strength and radiation resistance.

This study investigates effects of addition of V and Sc on microstructures and mechanical properties of 10Cr ODS FM steel. For this, three kinds of 10 Cr ODS FM steels were prepared by mechanical alloying (MA), hot isostatic pressing (HIP), and hot rolling process. Tensile tests were carried out at room temperature and 700 °C to evaluate the influences of V and Sc elements 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 ODS FM steels, the chemical compositions of which are given in Table 1. The ODS FM steels, sample A: Fe-10Cr-1Mo-0.6Mn sample B: Fe-10Cr-1Mo-0.6Mn-0.1V and sample C: Fe-10Cr-1Mo-0.6Mn-0.05Sc in wt% were fabricated by MA and HIP processes. 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 rolled in a fixed rolling direction for a plate shape, which resulted in a final reduction rate of 65%, and then normalized at 1150 °C for 1hr and tempered at 750 °C for 1hr through air cooling respectively. To evaluate mechanical property, according to ASTM E8, tensile tests were conducted at room temperature and 700 °C in air at a strain rate of $3.3\text{x}10^{-4}s^{-1}$. Microstructure of the tempered plate 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.

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

Alloy (wt.%)	Fe	Cr	Mo	Y_2O_3	Mn	V	Sc
A : 10Cr-1Mo-0.6Mn	Bal.	10	1.2	0.35	0.6	-	-
B : 10Cr-1Mo-0.6Mn-0.1V	Bal.	10	1.2	0.35	0.6	0.1	
C : 10Cr-1Mo-0.6Mn-0.05Sc	Bal.	10	1.2	0.35	0.6		0.05

3. Results and Discussions

Fig. 1 shows the tensile test results of the 10Cr ODS FM steels at room temperature and 700 °C. The results clearly show that sample B with V addition has the highest yield and tensile strengths, while sample C with Sc addition demonstrates the lowest yield and tensile strengths at room temperature and 700 °C. The tensile strength of sample B was increased from 1081 to 1097 MPa at room temperature, and from 231 to 245 MPa at 700 °C, but there is not much difference in the elongation of between sample A and B for both temperatures. In the case of sample C, the tensile strength was decreased from 1081 to 1013 MPa at room temperature, and from 231 to 164 MPa at 700 °C. The elongation was slightly decreased from 10.1 to 9.5% at room temperature, but was significantly increased from 29 to 44.5% at 700 °C. These results indicate that the tensile property of ODS FM steel is greatly improved by addition of V, but is adversely affected by Sc addition.

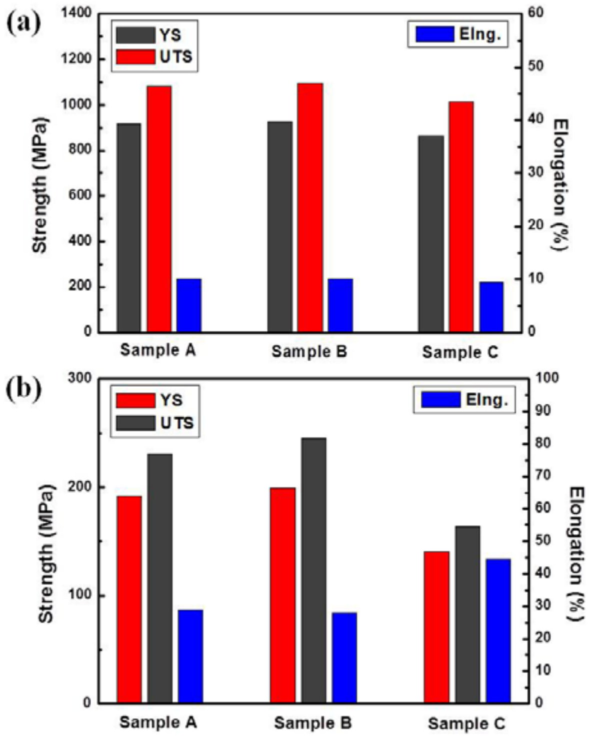

Fig. 1. Tensile test results of 10Cr-1Mo-0.6Mn, 10Cr-1Mo-0.6Mn-0.1V, and 10Cr-1Mo-0.6Mn-0.05Sc ODS FM steels in the temperature (a) RT and (b) 700 °C.

In Fig. 2, EBSD observations of the grain morphology of ODS FM steels with addition of V and Sc are presented: (a) sample A of 10Cr-1Mo-0.6Mn as reference, (b) sample B of 10Cr-1Mo-0.6Mn-0.1V, and (c) sample C of 10Cr-1Mo-0.6Mn-0.05Sc. It was found that all samples consist of fine equiaxed grains and a small portion of elongated grains along with the hot rolling direction. Since the temperature is increased from room temperature to 1150°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. When a portion of fine equiaxed grains are compared among samples, it is observed that the portion of fine equiaxed grains in sample B are slightly reduced, yet an overall grain size is refined, however, the addition of Sc to 10Cr ODS FM steel lead to a considerable decrease of fine equiaxed grains, compared with sample A and B. These results suggest that a grain refining effect is enhanced by addition of V, but is diminished by addition of Sc in ODS FM steels.

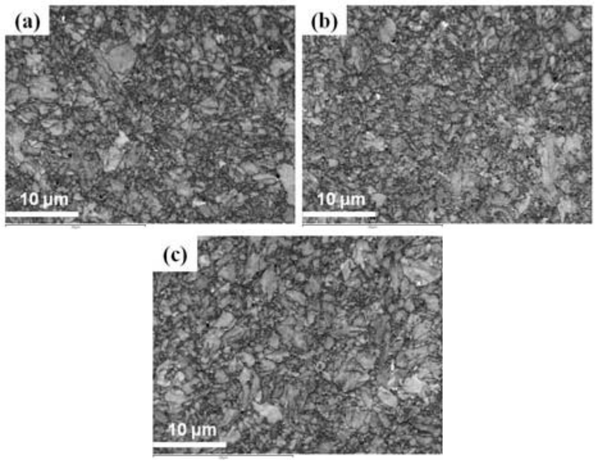

Fig. 2. EBSD images of (a) 10Cr-1Mo-0.6Mn, (b) 10Cr-1Mo-0.6Mn-0.1V, and (c) 10Cr-1Mo-0.6Mn-0.05Sc ODS FM steels.

Fig. 3 shows the SEM micrographs of grain morphologies and precipitates for 10Cr ODS FM steels: (a) sample A, (b) sample B, and (c) sample C. It can be seen that all samples have martensite structures with finely dispersed carbides along the grain boundaries, as well as delta-ferrite structures. Also, it is found that a number of precipitates are increased with addition of V and Sc, meaning the carbide formation is promoted by addition of V and Sc. However, it should be noted that sample C reveals the precipitate coarsening which has an adverse influence on high temperature strength, as well as an abrupt increase of precipitates, resulting in the excessive extraction of carbons from the matrix.

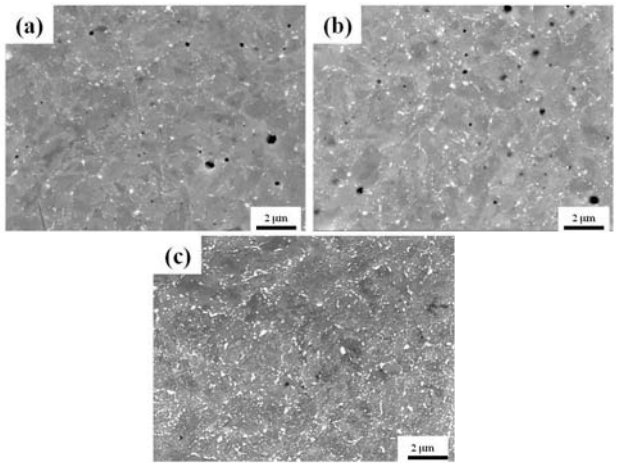

Fig. 3. SEM micrographs of (a) 10Cr-1Mo-0.6Mn, (b) 10Cr-1Mo-0.6Mn-0.1V, and (c) 10Cr-1Mo-0.6Mn-0.05Sc ODS FM steels.

The TEM/EDS results of the precipitates and oxide particles taken from the replica samples of 10Cr ODS FM steels are exhibited in Fig. 4. In sample A, the precipitate was found to be mainly $M_{23}C_6$ (M=Fe, Cr, Mo, and Mn) formed in the grain boundaries. In the case of sample B, it was found that V was dissolved in

$M_{23}C_6$ (M=Fe, Cr, Mo, and Mn). The precipitate of sample C was also $M_{23}C_6$ (M=Fe, Cr, Mo, and Mn) with Sc solid solution. Based on these observations, it is considered that the carbide formation is instigated by V addition, resulting in the grain refining effect. This result enables the conclusion that the improvement of tensile properties seen in sample B is attributed to the microstructure change by addition of V. However, the addition of Sc incurred deterioration in tensile property due to the excessive precipitation and precipitate coarsening. Lastly, the complex oxides were identified to be Y-Cr-Ti-Zr-O/Cr-Ti-Zr-O in sample A, Y-Cr-Ti-Zr-O/Y-Cr-Ti-V-O in sample B, and Y-Cr-Ti-Sc-O/Y-Cr-Ti-Zr-Sc-O in sample C, respectively.

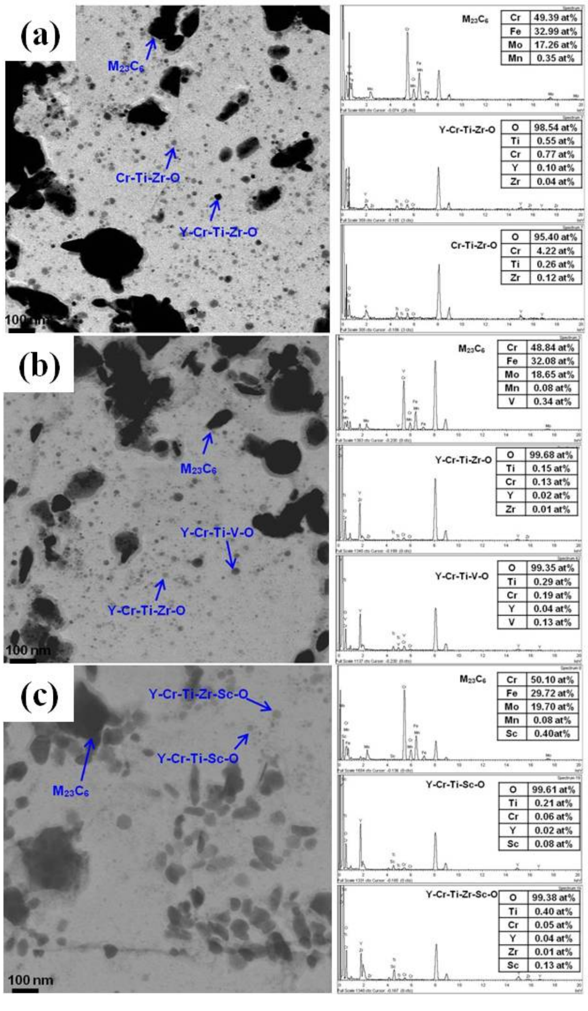

Fig. 4.TEM/EDS results of precipitates and oxide particles in (a) 10Cr-1Mo-0.6Mn, (b) 10Cr-1Mo-0.6Mn-0.1V, and (c) 10Cr-1Mo-0.6Mn-0.05Sc ODS FM steels.

4. Conclusions

This study investigated the effects of addition of V and Sc on the microstructure and tensile properties of ODS FM steels. The ODS FM steels were fabricated by the MA, HIP and hot-rolling processes. The addition of V led to the grain refining with a relatively higher density of precipitates, resulting in the improvement of tensile properties. The addition of Sc made the tensile properties worsened owing to the occurrences of the excessive precipitation and precipitate coarsening. It is believed that these preliminary results can be useful in development of advanced ODS steel.

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