Effects of Yttrium Oxide Addition on Microstructure and Tensile Properties of 15Cr Ferritic Steels

Ji-Eun Choi^{a, b*}, Sanghoon Noh^a, Byung-Kwon Choi^a, Chang-Hee Han^a, Suk Hoon Kang^a, Ki-Baik Kim^a, Sun-Ig Hong^b, Tae Kyu Kim^a

, Tae Kyu Kim^a *^a Nuclear Materials Division, Korea Atomic Energy Research Institute, Daejeon, Korea ^bAdvanced material engineering, Chungnam National University, Daejeon, Korea *Corresponding author: cje335@kaeri.re.kr*

1. Introduction

Next generation nuclear systems require improved core structural materials to realize high efficiency and safety. Ferritic/martensitic steels are very attractive for the structural materials of fast fission reactors and fusion reactors, because of a higher swelling resistance than austenitic stainless steels. However, conventional ferritic/martensitic steels have limited thermal creeprupture strengths at temperatures above 823K. To achieve higher operating temperatures, highly elevated temperature strength is essentially required for ferritic /martensitic steels. Oxide dispersion strengthened (OD S) ferritic steel is considered one of the most promising candidate structural materials for next-generation nuclear systems because of its excellent elevated temperature strength, irradiation resistance, and good compatibility [1,2]. These excellent properties of ODS ferritic steels are attributed to uniformly distributed nano-oxide particles in the ferritic steel matrix [3]. Therefore, it is very important to understand the roles of scanning nano-oxide particles in ODS steels for application to the core structural materials of next generation nuclear systems. This study was conducted to understand the microstructural and mechanical property differences between ODS ferritic steel and conventional ferritic steel. Through these characterizations, the fundamental effects of nano-oxide particles on the microstructure and tensile properties of ODS ferritic steel were discussed.

2. Methods and Results

2.1 Experimental procedure

ODS ferritic steel used in this study is Fe-15Cr-1Mo- $0.3Ti-0.35Y_20_3$ in wt%. For a comparison of the microstructural and tensile properties, conventional ferritic steel was also fabricated using the powder metallurgy in the same chemical composition without Ti and Y_20_3 . Detailed chemical compositions of the materials are summarized in Table 1. Two materials were fabricated through mechanical alloying (MA), hot isostatic pressing (HIP) and hot rolling methods.

Table I: Chemical composition of the materials

					$(wt. \%$	a
	Fe		Mo	m:		
ODS steel	Bal.			U.J		
Ferritic steel	Bal.			$\overline{}$	$\overline{}$	S

Metallic raw powders and Y203 powder were mechanically alloyed using a horizontal ball mill apparatus, CM20, under an ultra-high purity Ar gas (purity in 99.9999%) atmosphere at 240 rpm for 48h with a ball-to-powder weight ratio of 15:1. After mechanical alloying, the particle distribution was measured by a laser diffraction scattering method using a particle size analyzer. Scanning electron microscopy (SEM) was utilized to observe the surface morphology of the MA powders. The MA powders were sieved and charged in stainless a steel capsule. The Sealed capsules were then degassed at 400° C below $5x10^3$ torr for 3h. The HIP was carried out at 1150°C for 3h under 100MPa at a heating rate of 5°C/min and following furnace cooling. Hot rolling at 1150°C was done in a fixed rolling direction for the plate shape, which resulted in a total reduction rate of 75% in thickness. Two materials were finally heat treated through annealing at 1150°C for 1h, followed by air cooling. The microstructures were observed using field emission electron microscopy (FE-SEM) and transmission electron microscopy (TEM). The SEM samples were prepared through mechanical polishing and electrolytic polishing with a mixed solution of 10vol. % perchloric acid and 90vol.% ethanol. Thin foil samples for TEM observations were prepared by jet polishing at 40°C. Carbon extraction replica samples were also prepared to analyze the precipitates. Specimens for mechanical property evaluations were taken out in the rolling direction. Tensile tests were carried out at room temperature and 700°C.

2.2 Effect of Y2O³ on the microstructures of the 15Cr ferritic steels

Fig. 1 shows the microstructures of the ferritic steels taken by FE-SEM. The grain size of ODS steel was relatively the finer than conventional ferritic steel without Y_20_3 . The mean grain size and aspect ratio of the two materials were calculated through an image analysis and are given in table II. The mean grain sizes were 1.09μm and 4.36μm, and the aspect ratio of each material was 1: 2.1 and 1:1.5, respectively. The mean grain size of ODS steel was 25% of ferritic steel, and the mean aspect ratio of ODS steel reached 143% against ferritic steel. The bright field TEM images of ODS steel steel and ferritic steel are shown in fig. 2. First, when compared with fig. $2(a)$ and $2(b)$, fig. $2(a)$ shows fine dispersoids. Fig. 2(c) and (d) are carbon

(a) (b)
Fig. 1. SEM images of (a) ODS steel steel, (b) ferritic steel (b)

Table II: Mean grain sizes and mean aspect ratio of materials measured by SEM materials measured by SEM

		Mean grain size Mean aspect ratio	
ODS steel	1.09um	1:2.09	
Ferritic steel	$4.36 \mu m$	1:1.46	

extraction replica sample images showing precipitate extraction replica sample images showing precipitate distributions. Fig. 2(c) indicates an image of complex oxide particles, which consist of Y-Ti-O or Y-Ti-Cr-O with a mean diameter of 9.7nm in 15Cr ferritic ODS steel. Fig. 2(d) shows an image of coarse Cr precipitation with a mean diameter of 67nm in ferritic steel. Despite the same fabrication processes, it is considered that the reason for the finer grain size of considered that the reason for the finer grain size of ODS steel than ferritic steel is owing to the presence of fine nano-oxide particles in the grains or at the grain boundary. This can be a nucleation site when the recrystallization occurs during the hot consolidation process. Furthermore, oxide particles suppress boundary migration by a pinning effect when the grain growth occurs nano-oxide particles in the grains or at the grain
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2.3 Effect of Y2O³ on the mechanical properties properties of the 15Cr ferritic steels

Fig. 3 exhibits the stress-strain curve of the materials Fig. 3 exhibits the stress-strain curve of the materials at room temperature and 700° C. The strain-stress curve at RT in fig. 3(a) shows that the ultimate tensile strength (UTS) of ODS steel is significantly higher than strength (UTS) of ODS steel is significantly higher than ferritic steel, but the elongation is somewhat less. The stress-strain curve in fig. 3(b) was tested at 700℃. Ferritic steel without Y_2O_3 showed a low tensile strength and high elongation, whereas ODS steel has a higher UTS than ferritic steel. However, it was shown that ODS steel has a decreased elongation against ferritic steel.

3. Summary

The effects of Y_2O_3 addition on the microstructure The effects of Y_2O_3 addition on the microstructure and tensile properties of 15Cr ferritic steels were investigated. Fine complex oxide particles were observed in 15Cr ferritic ODS steel. They were $\frac{dP}{dP}$ homogeneously dispersed in the Fe-Cr matrix and regarded to cause grain growth suppression, despite fact that the ferritic steel was fabricated using the same pow $221-22$ Le particles were app
steel. They were pp.
Fe-Cr matrix and [4]

(c) Fig. 2. TEM images of ODS and ferritic steels: Thin foil images of (a) ODS steel, and (b) ferritic steel, and carbon extraction replica sample image of (c) ODS steel and, (d) ferritic steel

Fig 3. Stress-strain curves of ODS steel and ferritic steel at (a) room temperature, and (b) 700° C (a) (b)
strain curves of ODS steel and ferritic
mperature, and (b) 700° C

-der metallurgy process. In addition, the results of the tensile test showed that ODS steel has a significantly high tensile strength at high temperature, which is considered to be the strengthening effect of the nano oxide particle distribution.

Acknowledgements

This work was supported by the National Research Foundation of Korea(NRF) grant funded by the Korea government(MEST) (No. 2012M2A8A1027872). ing effect of the nano-
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REFERENCES

[1]T. OKUDA, M. FUJIWARA, Dispersion behaviour of oxide particles in mechanically alloyed ODS steel, JOURNAL OF MATERIALS SCIENCE LETTERS LETTERS, 14 pp.1600-1603, 1995.

[2] R. Lindau, A. Moeslang, Mechanical and microstructural properties of a hipped RAFM ODS-steelv, J. Nucl. Mater. 307–311, pp. 769–772 , 2002.

[3] S. Ukai, M. Fujiwara, Perspective of ODS alloys application in nuclear environments, J. Nucl. Mater. 307-311, pp. 749–754, 2002. application in nuclear environments, J. Nucl. Mater. 307–311,
pp. 749–754, 2002.
[4] C.H. Zhang, Characterization of the oxide particles in Al-

added high-Cr ODS ferritic steelsm, J. Nucl. Mater. 417,pp. 221–224, 2011.