Microstructural characteristics and mechanical properties of a mechanically alloyed 15Cr ODS ferritic steel

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1. Introduction

For the application of cladding material of a sodium-cooled fast reactor (SFR), superior creep resistance at high temperature and irradiation resistance are required. As ferritic/martensitic and ferritic steels suffer from loss of creep- and tensile-strengths above 600 °C, oxide dispersion strengthened (ODS) ferritic/martensitic and ferritic steels are considered as promising structural materials of SFR cladding tube [1,2]. In this study, microstructural evolution and mechanical properties of a ODS ferritic steel, which was designed and fabricated in Korea Atomic Energy Research Institute (KAERI) by mechanical alloying (MA) and hot isostatic pressing (HIPing) were investigated [3].

2. Methods and Results

2.1 Experimental procedure

A 15Cr-ODS ferritic steel, designated as T500, was investigated in this study. Chemical compositions of the ferritic steel are shown in Table I. Excessive oxygen (Ex.O) in Table I defined as oxygen content subtracted oxygen coupled with Y_2O_3 from the total oxygen concentration in the ferritic steel.

The alloy was manufactured by mechanical alloying of metallic raw powders of Fe, Cr, Mo, Ti and Y_2O_3 by a Symoloyer horizontal ball-mill (CM20) with rotation speed of 240 rpm for 48 hours in Ar-4%H_2 mixed gas condition. After MA process, the powders are charged into a stainless steel capsule. Then, deoxidization and degassing for the capsule was performed at 400 °C for 1 hour in Ar-4%H_2 and below 10^{-4} Torr. Temperature for hot isostatic pressing was conducted at 1150 °C for 3 hours with a pressure of 100 MPa. After that, hot rolling was carried out at 1150 °C for total rolling ratio of 82 %. Finally, the alloy was heat-treated for recrystallization at the same temperature for 1 hour.

For a microstructural observation, focused ion beam (FIB) was applied to extract a specimen at several areas for scanning transmission electron microscope (STEM) to figure out the grain morphology and oxide particles dispersed in the alloy. In the case of mechanical properties, plate-type small size specimens were tested from room temperature to 800 °C for tensile test. Also,

Table I: Chemical composition of 15Cr-ODS ferritic steel (T500) in wt.%.

Fe	Cr	Mo	Ti	Y_2O_3	C	Ex.O
Bal.	14.85	0.99	0.31	0.34	0.007	0.18

on-going research on the creep-rupture test at 700 °C under the applied stress of 155 MPa is summarized.

2.2 Microstructural evolution

Fig. 1 and 2 show the STEM bright field micrographs of the grain morphology and oxide particles dispersed in the alloy. Fine spherical oxide particles 5-30 nm in diameter are embedded in the equiaxed grains, which have size of a few hundred of nanometers. To figure out the chemical compositions of the oxide particle, energy dispersive x-ray spectroscopy (EDS) analysis was conducted (Fig. 2). (Y,Ti)-rich oxide particle was confirmed and Fe and Cr were depleted in the oxide particle; this result indicates that the original Y_2O_3 powders are dissolved into the ferritic matrix during the MA process and (Y,Ti)-rich oxide particles were nucleated and coarsened during heat treatment [4].

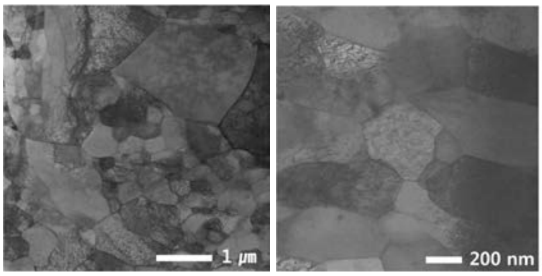

Fig. 1. STEM bright field micrographs of 15Cr-ODS ferritic steel.

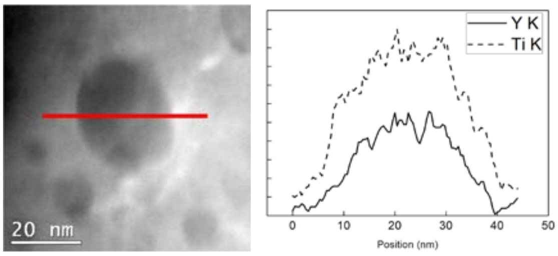

Fig. 2. STEM/EDS line scan analysis of Y and Ti for a (Y,Ti)-rich oxide particle in 15Cr-ODS ferritic steel.

Further studies to identify the (Y,Ti)-rich oxide particles were not performed in this study; however, the oxide particles were reported as Y_2O_3-TiO_2 (Y_2TiO_5) and Y_2O_3-$2TiO_2$ ($Y_2Ti_2O_7$) bi-oxides for MA/annealed powders [5] and Y_2O_3-$2TiO_2$ ($Y_2Ti_2O_7$) oxide for ODS ferritic steel [6].

2.3 Mechanical properties

Fig. 3 shows the tensile test results performed with the strain rate of 3.33×10^{-4} s^{-1} with the experimental ODS ferritic steel (14YWT) for comparison. The ultimate tensile strength was 1085 MPa at room temperature and gradually decreased to 253 MPa at 800 ^{o}C. On the other hand, the elongation was maintained to about 23 % from room temperature up to 600 ^{o}C and suddenly dropped to about 8 % at 700 ^{o}C. In particular, at room temperature, while the elongation is higher than other ODS steels, the ultimate tensile strength is lower; also, it is unusual that the elongation is decreased above 600 ^{o}C as the increase of elongation was usually reported above that temperature in other ODS steels [7,8]. Further studies on the scanning electron microscope (SEM) near the location of failure and for the fracture surface are in progress to understand such behaviors.

On-going creep-rupture test was conducted at 700 ^{o}C. Though the ultimate tensile strength at 700 ^{o}C is slightly lower than other ODS steels, the creep-rupture time is quite different compared to other ODS steels following the Larson-Miller parameters [7]. Creep-rupture test at low stress level is in progress to demonstrate the creep behavior along the grain boundary and near the oxide particles.

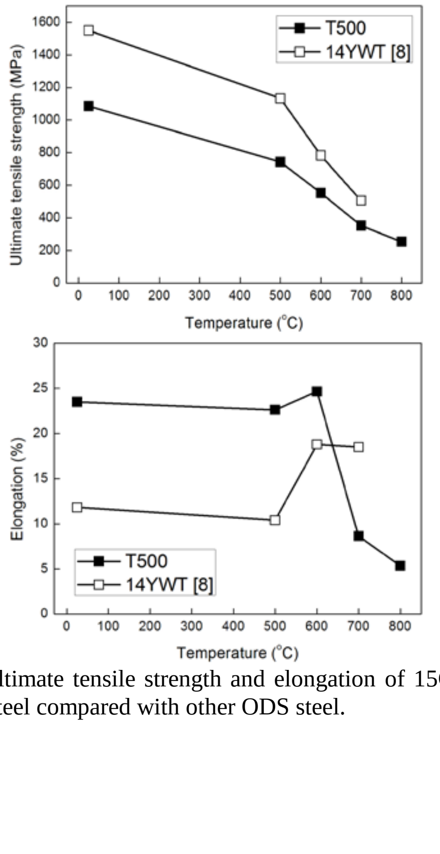

Fig.3. Ultimate tensile strength and elongation of 15Cr-ODS ferritic steel compared with other ODS steel.

3. Conclusions

15Cr-ODS ferritic steel designated as T500 was designed/fabricated in KAERI by mechanical alloying (MA) and hot isostatic pressing (HIPing) process. Fine spherical oxide particles are randomly distributed in the equiaxed grains. For the oxide particles, the presence of Y and Ti were confirmed while Fe and Cr were depleted. The ultimate tensile strength was about 1085 MPa and the elongation was about 23 % at room temperature, which is comparable with other ODS steels. However, further studies on the manufacturing and heat treatment conditions are required to enhance the creep-rupture strengths.

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