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EFFECT OF RHENIUM ADDITION ON MICROSTRUCTURES AND MECHANICAL PROPERTIES OF OXIDE DISPERSION STRENGTHENED FERRTIC STEELS

In this study, to investigate effects of rhenium addition on the microstructures and mechanical properties, 15Cr-1Mo ODS ferritic steels with rhenium additions were fabricated by the mechanical alloying, hot isostatic pressing, and hot rolling processes. Unremarkable differences on grain morphologies and nano-oxide distributions were estimated in the microstructure observations. However, the ODS ferritic steels with 0.5 wt.% rhenium showed higher tensile and creep strengths at elevated temperature than that without rhenium. It was found that rhenium is very effective to improve the mechanical properties.

Keywords: oxide dispersion strengthened steel, rhenium, oxide particles, tensile, creep

1. Introduction

ODS ferritic steel is one of the most suitable structural materials for advanced nuclear components, because of superior creep and irradiation resistances by a presence of homogeneously distributed nano-oxide particles with a high density [1,2]. This unique microstructure of ODS ferritic steels usually depends on the alloy composition, which mainly consists of Cr, W, Ti and Y_2O_3 [3,4]. Chromium concentration over the 12 wt.% gives excellent compatibility and corrosion resistance in the various coolants by forming a thin passive oxide film on the surface [2,5]. Titanium is an essential alloying element in ODS steels because this forms extremely fine Y-Ti-O enriched particles by a combination with Y_2O_3 with high density in the matrix during hot annealing process. The interaction between fine Y-Ti-O particles and highly dense dislocation is primary strengthening mechanism and this leads superior tensile and creep strength at elevated temperatures [2,3]. Tungsten and molybdenum are also the most effective elements for solid-solution strengthening to improve the mechanical properties at elevated temperatures. However, excessive addition leads to the deterioration of mechanical properties due to the formation of brittle Fe_2(W, Mo), called as Lavas phase during long-term operations [6,7].

In this study, effects of rhenium addition on microstructure and mechanical properties of ODS ferritic steel were investigated. 15Cr-1Mo ODS Mechanical alloying, hot isostactic pressing and hot rolling processes were employed to fabricate the ODS ferritic steels, and tensile test as well as creep rupture test were examined to evaluate the properties at elevated temperatures.

2. Experimental

A Chemical composition of ODS ferritic steel, Fe(bal.)-15Cr-1Mo in wt.% with some minor alloying elements including Ti, Zr, Y, and O was employed in this study. Because of high Cr contents over 12 wt.% with C contents below 0.05wt%, this results fully stable ferritic phase even up to a melting point, so that any additional strengthening effects could not be expected during fabrication process and heat treatment process. Rhenium (Re) was also added in a variety of 0, 0.1 and 0.5 wt.%, so that ODS ferritic steels with the different Re contents were designated as R0-, R1- and R2-ODS steels, respectively. The ODS ferritic steels were fabricated by mechanical alloying (MA), hot isostatic pressing (HIP) and hot rolling (HR) processes. Metallic raw powders and Y_2O_3 were mechanically alloyed by a horizontal ball-mill apparatus for 40 h in high purity argon gas. The MA was performed with a ball-to-powder weight ratio of 10:1. After MA process, alloy powders were then charged in a stainless steel capsule. The capsules were degassed and sealed at 400°C below a vacuum level of 5×10^{-3} torr for 3h. The HIP was carried out at 1150°C for 3h at a heating rate of 5°C/min and followed by furnace cooling. HIPed ODS steels were hot-rolled at 1150°C in a fixed rolling direction for a plate, which resulted in a final

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TABLE 1

Chemical compositions of the ODS ferritic steels (in wt.%)

	Fe	Cr	Mo	Ti	Zr	Re	Y	O	Y_2O_3
R0	bal.	14.88	0.90	0.12	0.24	—	0.26	0.19	0.33
R1	bal.	14.70	0.93	0.09	0.26	0.11	0.27	0.19	0.34
R2	bal.	15.10	0.91	0.10	0.25	0.48	0.26	0.20	0.33

reduction rate of 70% in a thickness. Finally, ODS ferritic steel plates were annealed at 1150°C for 1h and cooled down in the air to relive the residual stress. The chemical compositions of ODS ferritic steels were given in Table 1. The chemical compositions and oxygen concentration of the ODS ferritic steels were analyzed by ICP-AES and inert gas fusion methods.

The grain and precipitate morphologies were observed by field emission scanning electron microscope (FE-SEM) and field emission transmission electron microscope (FE-TEM). ODS ferritic steels were buff-polished and electronically polished in 5% $HClO_4$ + 95% methanol solution in vol.% at 18 V with 0.5 mA at –50°C to remove the work hardened surface induced by mechanical buff-polishing. In order to observe the precipitate distributions, twin-jet polishing methods were employed for thin sample fabrication to observe the FE-TEM. Specimens for mechanical property evaluations were taken out by electro-discharge machining in the rolling direction. A sheet type tensile specimen with a gauge length of 25.4 mm, width of 3.7 mm, and thickness of 1 mm was used. The tensile tests were carried out at room temperature and 700°C in air at a strain rate of 1×10^{-3} s^{-1}. Creep rupture tests were carried out under 150, 200 MPa at 700°C in air.

3. Results and discussion

The SEM images of grain morphology observation on ODS ferritic steels with different Re concentration are shown in Fig. 1. Grain morphology of ODS ferritic steels were clearly distinguished by an observation of the back-scattered secondary electron image mode in the FE-SEM. Recrystallized and equiaxed micrograins might be formed at a stage of HIP process, and it seems to be occurred secondary recrystallization during hot rolling process at 1150°C because micrograins are elongated toward a hot rolling direction which is parallel to horizontal direction, however their aspect ratio was not so high as shown in Fig. 1(below). All alloys showed quite homogeneous grain distribution with 1~2 μm of a grain size. In spite of various Re concentration from 0 to 0.5 wt.%, it is hard to observe significant differences on grain morphologies of 15Cr-1Mo ODS ferritic steels.

To investigate the effects of Re addition on nano-oxide particle distributions of the ODS ferritic steels, a TEM observation was carried out. Bright field images of the ODS steels with different Re concentrations, (a) 0 wt.% Re, (b) 0.5 wt.% Re, are shown in Fig. 2. The mean intercept grain diameters of R0- and R2-ODS steels measured in a plane perpendicular to the rolling direction are 1.13 um and 1.06 um, respectively. Fine nano-oxide particles are quite homogeneously distributed in all ODS ferritic steels. Based on TEM observations, results of image analysis on nano-oxide particle size distribution, mean particle size, and number density were summarized in Fig. 3. Oxide particles over 10 nm in a diameter existed with a small fraction in the R0-ODS steels without Re addition, while very fine oxide particles under 10 nm were extensively distributed in the R2-ODS steel. Nevertheless, mean particle size and number density were not so different, they analyzed as 6.07 nm, 3.46×10^{22} m^{-3} for R0-ODS steel and 5.36 nm, 2.96×10^{22} m^{-3} for R2-ODS steel, respectively. Some researchers reported that oxide particles in ODS steels with minor alloying elements, such as Ti, Zr with Y_2O_3 are precipitated as specific oxides, namely $Y_2Zr_2O_7$ and Y_2TiO_5, which are formed by a combination of Y, O and Ti or Zr during the consolidation process [8]. In spite of 0.5 wt.% Re addition in R2-ODS steel, there were no traces of

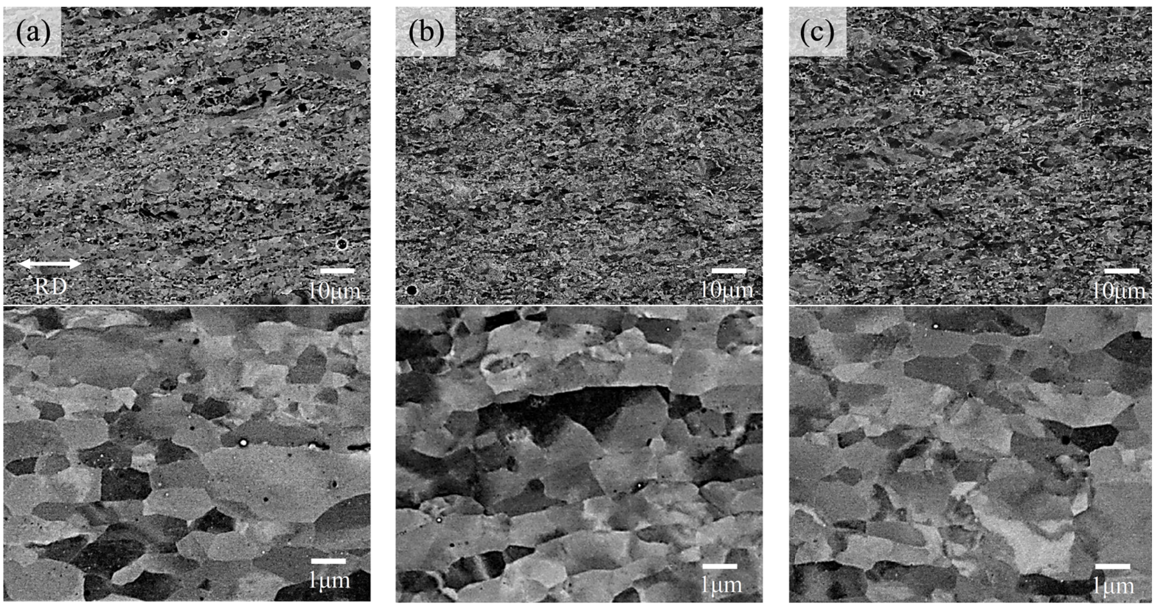

Fig. 1. Grain morphology of ODS ferritic steels with different Re concentration, (a) 0, (b) 0.1, (c) 0.5 wt.% Re

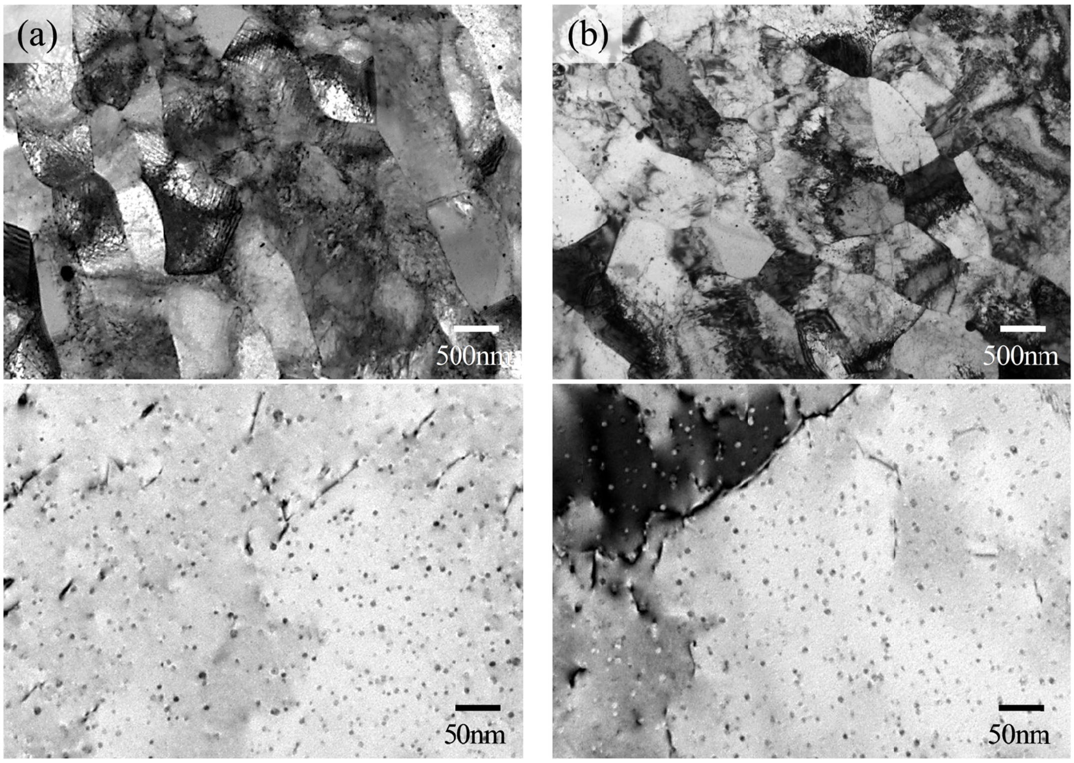

Fig. 2. Bright field images of the ODS steels with different Re concentrations, (a) 0, (b) 0.5 wt.% Re

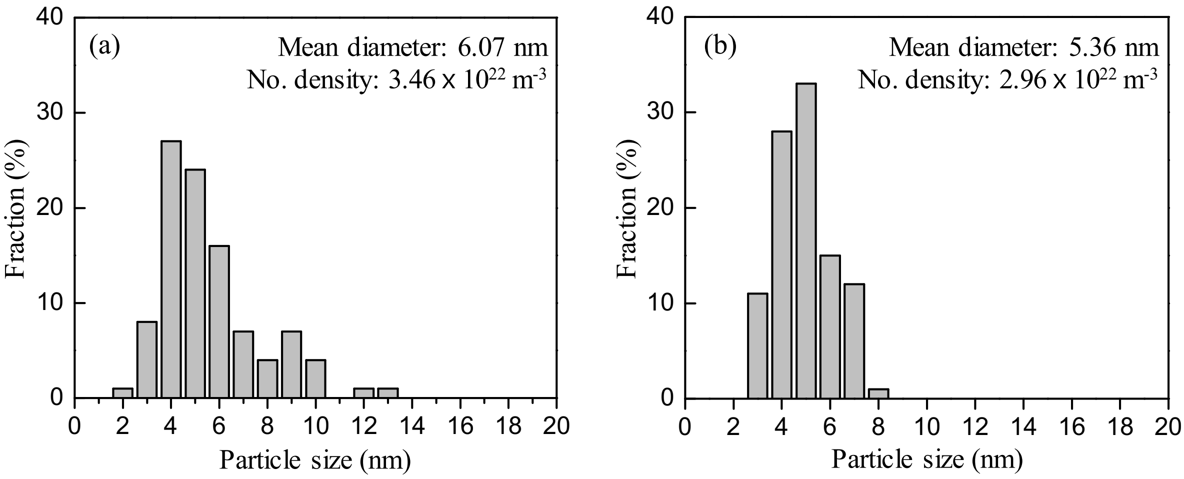

Fig. 3. Nano-oxide particle distributions of the ODS steels with different Re concentrations, (a) 0, (b) 0.5 wt.% Re

Re additions on precipitation and nano-oxide particles. Similar to a result of FE-SEM observation, there are no difference in micro-grain morphology and nano-oxide particle distribution even in different Re concentrations. Therefore, it is inferred that Re is completely solid-soluted in Fe-Cr alloy matrix and does not affect the microstructure of the 15Cr-1Mo ODS ferritic steels.

Tensile tests of the ODS ferritic steels with different Re concentrations were carried out at a room temperature and 700°C. Their results were presented in Fig. 4. The R0-ODS steel without Re addition showed higher tensile strength than R1- and R2-ODS steel with Re additions. As increase of Re concentrations, the total elongation was not dramatically changed as shown in Fig. 4(a). Lower yield strength with sufficient elongation at room temperature is considered to be very favorable, because it is more advantageous in the plastic working process such as cold rolling, drawing, and pilgering process to fabricate structural components. At 700°C, however, Re concentrations of ODS ferritic steels significantly affect the tensile strength. As increase of Re concentrations, tensile strengths of the ODS ferritic steels were increased. ODS steels with Re additions evaluated the high yield strength, 302 MPa for 0.1 wt% Re, 331 MPa for 0.5 wt.% Re, respectively. Compared to this, ODS ferritic steel without Re addition showed significantly low yield strength, 263 MPa. A change on total elongations of ODS ferritic steel with Re concentrations was quite dissimilar with tensile strength at the elevated temperature, however, the difference of

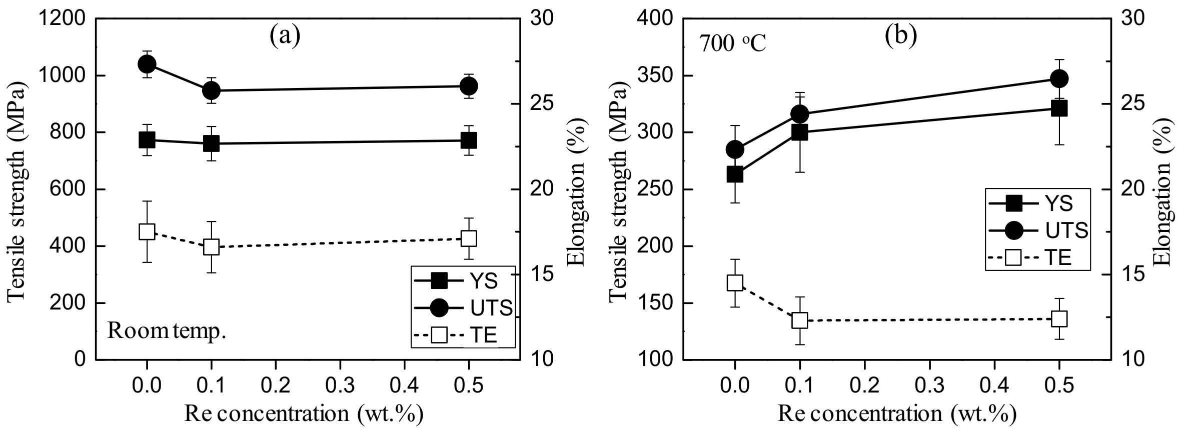

Fig. 4. Results of tensile tests on the ODS ferritic steels with different Re concentrations

total elongations with Re concentration was very slight. Creep rupture tests were also performed at 700°C and the results are plotted on log-log scale in Fig. 5. Although there was unremarkable difference in the microstructural analysis, the creep resistances of Re added ODS ferritic steels was higher than that without Re. This also coincides with the results of the tensile strength described above. It is well known that Re is inevitable alloying elements to improve the elevated temperature properties in many super alloys. This generally plays an important role to suppress the diffusivity of solid solution elements, such as W, Mo, and Co. Ni alloys with 3~6 wt.% Re additions presented exceptionally higher creep resistances than conventional super alloys at elevated temperatures [9-11]. In this study, it could be also assumed that Re additions in the ODS ferritic steel showed similar effects with a case of super alloys.

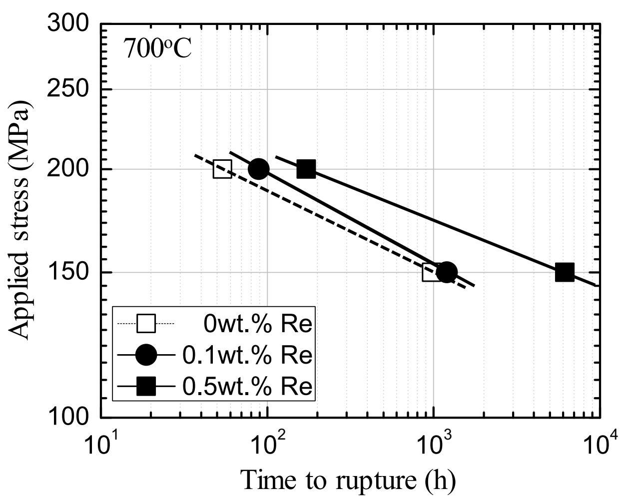

Fig. 5. Results of creep rupture tests on the ODS ferritic steels with different Re concentrations

4. Conclusions

15Cr-1Mo ODS ferritic steels with Re additions of 0, 0.1, and 0.5 wt.% were fabricated by the MA, HIP, and HR processes to investigate the microstructures and mechanical properties. There were no significant differences on grain morphologies and nano-oxide particle distribution. However, ODS ferritic steels with Re additions showed higher tensile and creep strengths at elevated temperature than that without Re. Therefore, it is considered that the Re is very effective alloying element of the ODS ferritic steel to improve the mechanical properties at elevated temperatures.

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