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MICROSTRUCTURES AND MECHANICAL PROPERTIES OF ODS FERRITIC STAINLESS STEELS FOR HIGH TEMPERATURE SERVICE APPLICATIONS

In this study, ODS ferritic stainless steels were fabricated using a commercial alloy powder, and their microstructures and mechanical properties were studied to develop the advanced structural materials for high temperature service applications. Mechanical alloying and uniaxial hot pressing processes were employed to produce the ODS ferritic stainless steels. It was revealed that oxide particles in the ODS stainless steels were composed of Y-Si-O, Y-Ti-Si-O, and Y-Hf-Si-O complex oxides were observed depending on minor alloying elements, Ti and Hf. The ODS ferritic stainless steel with a Hf addition presented ultra-fine grains with uniform distributions of fine complex oxide particles which located in grains and on the grain boundaries. These favorable microstructures led to superior tensile properties than commercial stainless steel and ODS ferritic steel with Ti addition at elevated temperature.

Keywords: Stainless steel, oxide dispersion strengthening, oxide particles, tensile strength

1. Introduction

The core structural materials of next generation nuclear systems are expected to operate under extreme environments, i.e., higher temperatures and dose rates than commercial light water reactors. To realize these systems, it is necessary to develop advanced structural materials having high creep and irradiation resistance [1,2]. Beyond the nuclear system applications, recently, advanced structural materials have been required for high temperature service applications in fossil power, defense, aerospace industries and so on [3]. Oxide dispersion strengthening (ODS) is one of the promising ways to improve the mechanical property at high temperatures. This is mainly attributed to uniformly distributed nano-oxide particle with a high density, which is extremely stable at the high temperature and acts as effective obstacles when the dislocations are moving [2]. In this study, to develop the advanced structural materials for high temperature service applications, ODS ferritic stainless steels were fabricated using commercial alloy powders and their microstructural and mechanical properties were investigated.

2. Experimental

In alloy design prospective, ferritic stainless steel 430L with extremely low carbon was employed, because this commercial alloy has been widely used as structural materials due to high corrosion, oxidation resistance and good formability. Nominal composition of the stainless steel 430L powder is Fe(bal.)-16.5 Cr-0.7Mn-0.7Si-0.02C in wt%. To fabricate ODS steel based on the stainless steel 430L, some additional elements were incorporated in raw material preparation. Tungsten (W) is one of the strong ferrite formers and the solid solution elements to enhance the high temperature strength [4]. In this study, 2 wt.% of W was added in the ODS ferritic stainless steels. Dispersions of yttrium oxide (Y_2O_3) particles are not adequate for effective strengthening of ODS steels. Kim et. al. reported that extremely coarse Y-Fe-O complex oxides having a diameter between 20 nm and even 30 μm were precipitated in a Fe-Y_2O_3 model alloy during the heating process to 1150 ^{o}C [5]. Some minor alloying elements were known to reduce the diameter and increase the number density of oxide particles by the formation of specific complex oxides [6,7]. Titanium (Ti) is the most effective element to refine the strengthening particles forming Y-Ti-O type complex oxides with a high number density [7]. To investigate the effect of alloying elements, furthermore, hafnium (Hf) was also added in 0.6 wt%. The chemical compositions of the materials were summarized in Table 1.

TABLE 1

Chemical compositions of commercial stainless steel and ODS ferritic stainless steels (in wt%)

Mateirals	Elements								
	Fe	Cr	Si	Mn	C	W	Ti	Hf	Y2O3
STS 430L	bal.	16.5	0.7	0.7	<0.02	—	—	—	—
ODS1	bal.	16.5	0.7	0.7	<0.02	2	0.5	—	0.35
ODS2	bal.	16.5	0.7	0.7	<0.02	2	0.5	0.6	0.35

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The ODS ferritic stainless steels were fabricated by mechanical alloying (MA) and uniaxial hot pressing (UHP) processes. The MA is inevitable process that the continuous collision between grinding media (high hardness steel balls in this study) and raw powders with a high revolving energy makes the repeated crushes and cold welding of raw powders, which eventually create the uniform mixing and alloying in the constitution elements. The commercial stainless steel powders and some raw powders were mechanically alloyed by a planetary ball-mill apparatus. The atmosphere was thoroughly controlled in ultra-high purity argon (99.9999%) gas. The MA was performed with a ball-to-powder weight ratio of 10:1. The MA powder was then consolidated using UHP at 1150^{o}C for 2 h at a heating rate of 10^{o}C/min. The process was carried out in a high vacuum ($<5\times10^{-4}$ Pa) under a hydrostatic pressure of 80 MPa in uni-axial compressive loading mode. After the consolidation process, the hydrostatic pressure was relieved and the ODS steels were cooled in the furnace. For microstructural observations, ODS ferritic stainless steels were mechanically wet ground and a twin-jet polished to fabricate the thin foil specimens using a solution of 5% $HClO_4$ + 95% methanol in vol.% at 18 V with 0.5 mA at –40°C. The grain morphology and precipitate distributions were observed by a transmission electron microscope. To evaluate the mechanical property, tensile tests were carried out at room temperature and elevated temperatures. The miniaturized and sheet-typed tensile specimens were machined with 5 mm of a gauge length, 1.2 mm of a width and 0.5 mm of a thickness. Tensile tests were performed at room temperature and 700°C at a strain rate of 6.7×10^{-3} s^{-1} in the air.

3. Results and discussion

Microstructural images of grain morphology on ODS ferritic stainless steels are shown in Fig. 1. All ODS ferritic steels showed typically equiaxed ferrite grains because of uniaxial hot pressing process. The ODS ferritic stainless steel with Ti addition (ODS1) had quite inhomogeneous grain distribution, which consisted of the co-existed microstructure with fine (130 nm) and coarse (1.7 μm) grains. However, both ODS ferritic stainless steels with Hf (ODS2) had quite uniform and ultra-fine grain distributions as shown in Fig. 1(b). Mean grain sizes of ODS steels with Hf were evaluated as 163 nm. It is estimated that Hf additions in the ODS ferritic stainless steel leads to the significant decrease of the grain size and homogeneous grain distribution.

Bright field TEM images showing the nano-oxide particle distributions in a micro-grain of the ODS ferritic stainless steels were presented in Fig. 2. The ODS1 showed inhomogeneous particle distribution consisted of very fine (<15 nm) and coarse (>30 nm) oxide particles with bimodal diameters. In contrast, ODS2 had fairly fine and homogeneous oxide particle distribution than ODS1. Mean diameter was evaluated as 6.3 nm. Analysis results of the chemical elements by the TEM-EDS revealed that fine oxide particles in the micro-grains of ODS1 were composed of Y-Si-O and Y-Ti-Si-O complex oxides as shown in Fig 3. The oxide particles in ODS steels with Ti and Y_2O_3 additions are precipitated as specific oxides, namely $Y_2Ti_2O_7$ and Y_2TiO_5, which are formed by a combination of Y, Ti, and O during the consolidation process such as hot isostatic pressing and extrusion processes [7]. Si is also one of the very high affinity elements with O. Kim et al. reported a formation of $Y_2Si_2O_7$ complex oxides in the AISI 316L ODS austenitic steel which included 0.81wt.% of Si [8]. ODS2 showed quite different oxide particles in the micro-grains. Y-Hf-Si-O complex oxides were observed. Interestingly, more oxide particles in the ODS2 were existed on the grain boundaries than those in ODS1 as shown in Fig. 4. Precipitates on the grain boundary can be favorable nucleation site for recrystallization stage and play an important role as an obstacle when the grain boundary migrates, so called ‘pinning effect’. It was identified that this led to ultra-fine and uniform grains in the ODS ferritic stainless steel with a Hf addition.

In Fig. 3, tensile properties of the ODS ferritic stainless steels was summarized. The tensile strengths of ODS ferritic stainless steel were exceptionally higher than commercial stainless steel 430L at both room and elevated temperatures. Ultimate tensile strengths (UTS) of ODS1, 2 exhibited 1060,

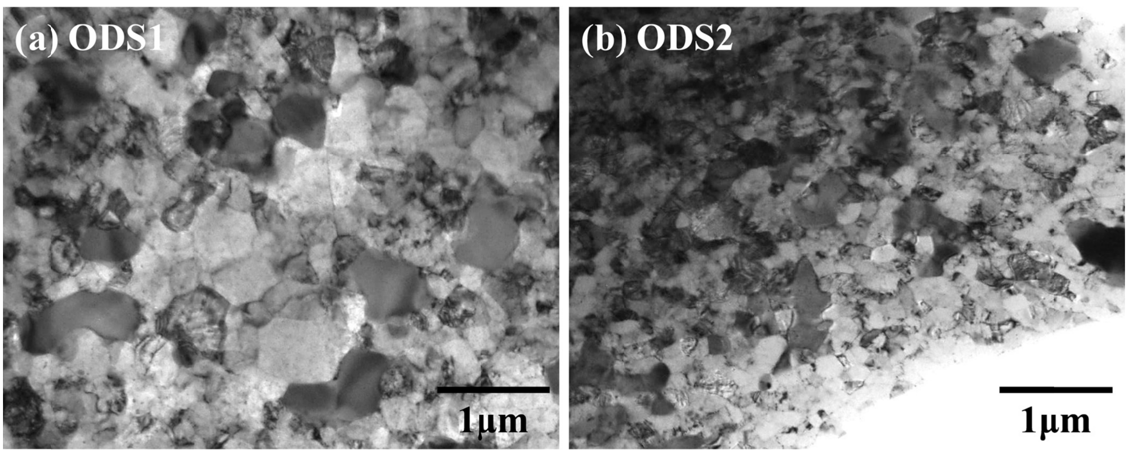

Fig. 1. Grain morphology of the ODS ferritic stainless steels with (a)Ti, (c)Hf additions

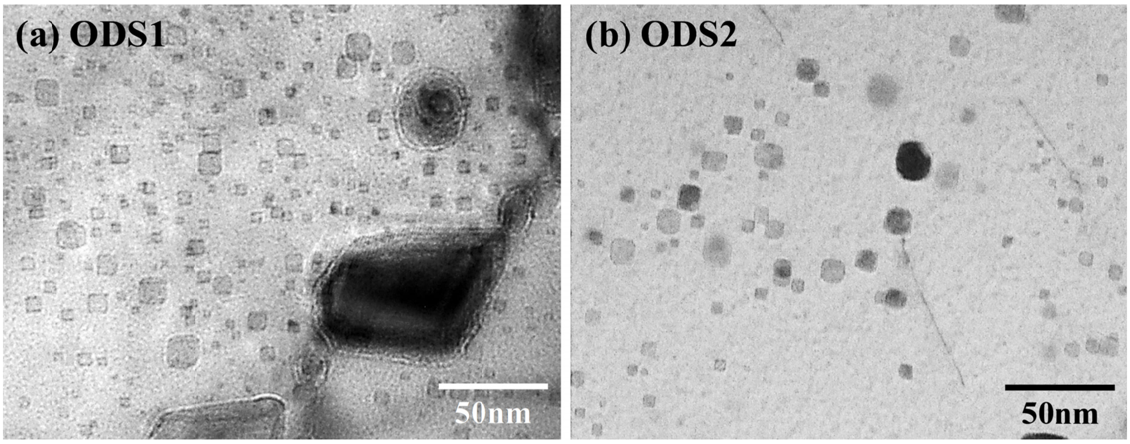

Fig. 2. Oxide particles in a micro-grain of the ODS ferritic stainless steels with (a)Ti, (b)Hf additions

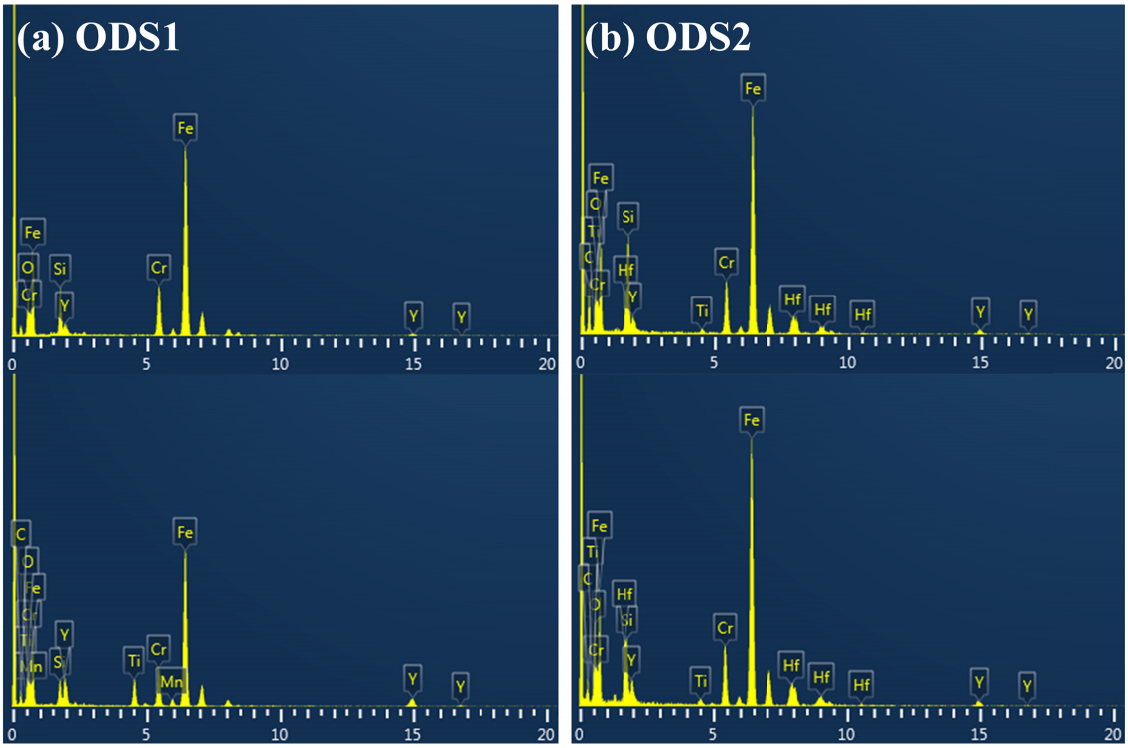

Fig. 3. TEM-EDS analysis of the oxide particles in ODS ferritic stainless steels with (a)Ti, (b)Hf

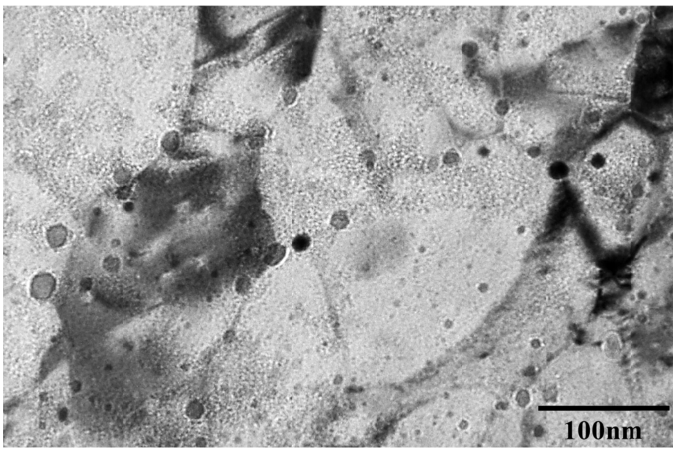

Fig. 4. Oxide particles on the grain boundaries in ODS ferritic stainless steel with Hf addition

1124 MPa with a sufficient total elongation (TE) of 15.2, 18.5% at room temperature, respectively. However, commercial stainless steel was relatively poor, which corresponded to less than 40% of the ODS ferritic stainless steels. High temperature tensile properties were also showed similar behaviors with the results at room temperature. The commercial stainless steel showed a post yield softening with significantly poor tensile strength at 700°C, whereas the ODS ferritic stainless steels had a favorable tensile strength and elongation. The UTS of ODS1, 2 were respectively evaluated as 314, 440 MPa with a TE of 18.6, 23.8% at 700°C. This is due to the effects of dispersion strengthening by nano-oxide particles in the grain and on the grain boundaries at high temperatures. The ODS2 with a Hf addition showed more superior tensile strength than the ODS1 at both room and 700°C. The ODS2 has ultra-fine grains with

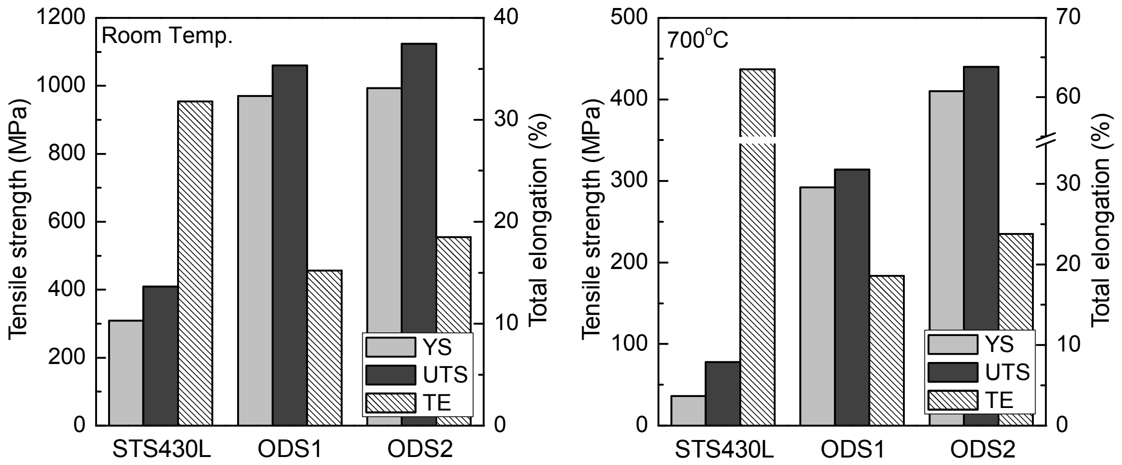

Fig. 5. Results of tensile tests on commercial stainless steel and ODS ferritic stainless steels

uniform nano-sized complex oxides, as coincided with the results of the microstructure observations. Extremely fine grains of the ODS steel contributed to an improvement in the tensile strength by the grain boundary strengthening. Additionally, fine nano-oxide particles are very stable even at a high temperature of up to 1300°C, and usually acts as an obstacle for the dislocation gliding when the material deforms, which is called 'Orowan strengthening' [9]. It is estimated that ODS ferrtic stainless steel with Hf additions showed favorable microstructures and tensile properties at elevated temperature for high temperature service applications.

4. Conclusions

In this study, ODS ferritic stainless steels were fabricated using commercial stainless steel 430L powder and their microstructures and mechanical properties were investigated. Morphology of micro-grains and oxide particles were significantly changed by the addition of minor alloying elements such as Ti, Hf. The ODS ferritic stainless steel with a Hf addition showed ultra-fine grains with uniform distributions of fine complex oxide particles which located in grains and on the grain boundaries. This led to more favorable tensile properties than ODS ferritic stainless steel with a Ti addition.

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