Microstructure and Mechanical Property of ODS Ferritic Steels Using Commercial Alloy Powders for High Temperature Service Applications

Sanghoon Noh*, Byoung-Kwon Choi, Suk Hoon Kang, Tae Kyu Kim
Nuclear Materials Division, Korea Atomic Energy Research Institute, Yuseong-gu, Daejeon, Republic of Korea
**Corresponding author: shnoh@kaeri.re.kr*

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]. Beyond the nuclear applications, recently, advanced structural materials have been required for high temperature service applications in fossil power, defense, aerospace industries and so on. 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. In this study, as a preliminary examination to develop the advanced structural materials for high temperature service applications, ODS ferritic steels were fabricated using commercial alloy powders and their microstructural and mechanical properties were investigated.

2. Methods and Results

2.1 Experimental procedure

In alloy design prospective, authors focused on low carbon ferritic stainless steel 430L, because this alloy has high corrosion, oxidation resistance and good formability. Nominal composition of the stainless steel 430L powder is Fe(bal.)-16.5Cr-0.7Mn-0.7Si-0.02C in wt%. To fabricate ODS steel based on stainless steel 430L, some additional elements were incorporated in raw material preparation. Tungsten (W) is strong ferrite former and one of the solid solution elements to enhance the high temperature strength [2]. In this study, 2 wt.% of W was added in the ODS ferritic steels. Yttrium oxide (Y_2O_3) particles are not adequate for effective strengthening of ODS steels. Titanium (Ti) is well known to refine the strengthening particles forming Y-Ti-O type complex oxides with a high number density [3]. To investigate the effect of alloying elements, furthermore, zirconium (Zr) and hafnium (Hf) were also added in 0.6 wt%, respectively. The chemical compositions of the materials were summarized in Table I.

The ODS ferritic stainless steels were fabricated by mechanical alloying (MA) and uniaxial hot pressing (UHP) processes. The MA is essential process that the continuous collision between grinding media and raw powders with a high revolving energy makes the repeated crushes and cold welding of powders, which eventually create the homogenous mixing and alloying in the constitution elements. The commercial alloy 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 2h at a heating rate of 10^{o}C/min. The process was carried out in a high vacuum (<5×10^{-4} Pa) under a hydrostatic pressure of 80 MPa in uni-axial compressive loading mode. After the process, the pressure was relieved and the samples were cooled in the furnace. For microstructural observations, ODS ferritic 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 18V with 0.5mA at -40 °C. The grain morphology and precipitate distributions were observed by a transmission electron microscope. To evaluate the mechanical property, Vickers hardness tests were carried out under 4.9N for 10s.

Table I: Chemical compositions of ferritic stainless steels and ODS steels (in wt%)

Elements	Materials				
	430L	430LW	ODS1	ODS2	ODS3
Fe	bal.	bal.	bal.	bal.	bal.
Cr	16.5	16.5	16.5	16.5	16.5
Si	0.7	0.7	0.7	0.7	0.7
Mn	0.7	0.7	0.7	0.7	0.7
C	<0.02	<0.02	<0.02	<0.02	<0.02
W	-	2	2	2	2
Ti	-	-	0.5	0.5	0.5
Zr	-	-	-	0.6	-
Hf	-	-	-	-	0.6
Y_2O_3	-	-	0.35	0.35	0.35

2.2 Microstructure of ODS ferritic steels

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 steel with Ti addition (ODS1) had inhomogeneous grain distribution, which showed the co-existed microstructure with fine (130nm) and coarse (1.7μm) grains. However, both ODS ferritic steels with Zr (ODS2) and Hf (ODS3) had quite homogeneous and ultra-fine grain distributions as shown in Fig. 1(b) and (c). Mean grain sizes of ODS steels with Zr and Hf were evaluated as 193 and 163nm, respectively. It is estimated that Zr and Hf additions in the ODS ferritic stainless steel leads to the significant decrease of the grain size and homogeneous grain distribution.

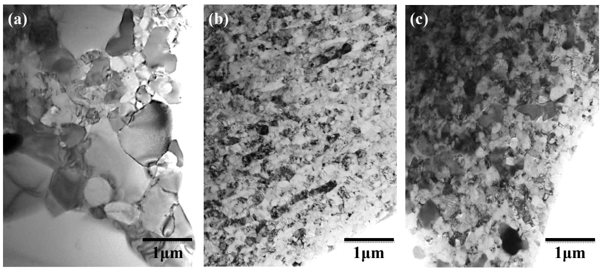

Fig. 1. Grain morphology of the ODS ferritic steels with (a)Ti, (b)Zr, and (c)Hf additions.

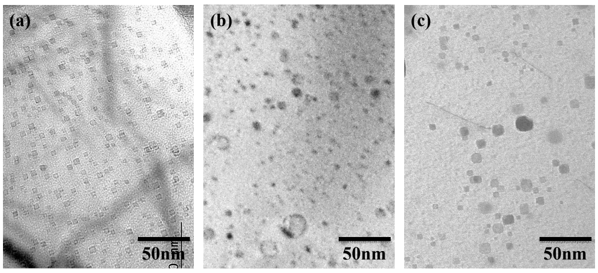

Fig. 2. Oxide particles in a micro-grain of the ODS ferritic steels with (a)Ti, (b)Zr, and (c)Hf addition.

In Fig.2, bright field TEM images showing the nano-oxide particle distributions in a micro-grain of the ODS ferritic steels were presented. The ODS1 showed very fine and uniform distribution of oxide particles with a mean diameter of 5.2 nm. In contrast, ODS2 and 3 had slightly coarser oxide particles than ODS1. Mean diameters were evaluated as 6.5 and 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-Ti-O and Y-Si-O complex oxides. The oxide particles in Ti-added ODS steels 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 hot consolidation process [3]. Si is also very high affinity elements with O. Kim et al. found $Y_2Si_2O_7$ complex oxides in the AISI 316L ODS austenitic steel which included 0.81wt.% of Si [4]. ODS2 and 3 showed different oxide particles in the micro-grains. Y-Zr-O and Y-Hf-O complex oxides were observed. Interestingly, more complex oxide particles in the ODS2 and 3 were precipitated on the grain boundaries than those in ODS1 as shown in Fig. 3. 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 430L with Zr and Hf additions.

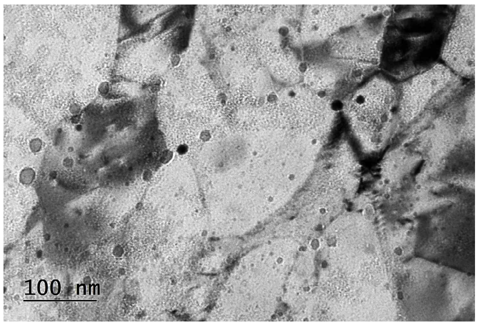

Fig. 3. Oxide particles on the grain boundaries in ODS ferritic stainless steel 430L with Hf addition.

2.3 Mechanical property of ODS ferritic steels

In Fig. 4, Vickers hardness of the stainless steels was summarized. Based on this result, a contribution of solid solution hardening by W addition could be evaluated to Δ55Hv. ODS1 showed high hardness, 340Hv. Combined contributions of the oxide particle and fine grain can be evaluated to Δ123Hv in ODS1. While oxide particle sizes and distributions of ODS2, 3 were similar with ODS1, they had higher hardness than ODS1. This is due to ultra-fine grains by oxide particle in the grain boundary in ODS2, 3. This result was well corresponded to results of microstructural observation. More detailed estimation will be performed to elucidate the strengthening mechanism in the ODS ferritic stainless steel 430L.

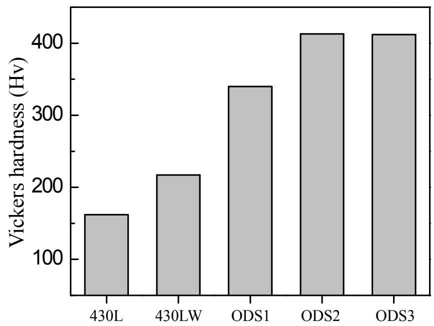

Fig. 4. Vickers hardness of the ferritic stainless steels and ODS steels.

3. Conclusions

In this study, ODS ferritic 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, Zr, and Hf. The ODS ferritic steel with Zr and Hf additions showed ultra-fine grains with fine complex oxide particles. The oxide particles were uniformly located in grains and on the grain boundaries. This led to higher hardness than ODS ferritic steel with Ti addition.

Acknowledgements

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