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MICROSTRUCTURES AND TENSILE PROPERTIES OF Fe-Cr-Al OXIDE DISPERSION STRENGTHENED FERRITIC ALLOYS FOR HIGH TEMPERATURE SERVICE COMPONENTS

In present study, Fe-22Cr-4.5Al oxide dispersion strengthened ferritic alloys were fabricated using a pre-alloyed powder with different minor alloying elements, and their microstructures and tensile properties were investigated to develop the advanced structural materials for high temperature service components. Planetary-typed mechanical alloying and uniaxial hot pressing processes were employed to fabricate the Fe-Cr-Al oxide dispersion strengthened ferritic alloys. Microstructural observation revealed that oxide dispersion strengthened ferritic alloys with Ti, Zr additions presented extremely fine micro-grains with a high number density of nano-scaled oxide particles which uniformly distributed in micro-grains and on the grain boundaries. These oxide particles were confirmed as a fine complex oxide, $Y_2Zr_2O_7$. These favorable microstructures led to superior tensile properties than commercial ferritic stainless steel and oxide dispersion strengthened ferritic alloy with only Ti addition at elevated temperature.

Keywords: Fe-Cr-Al; ferritic alloy; oxide dispersion strengthening; complex oxide; tensile strength

1. Introduction

Advanced structural materials have been essentially required for high temperature service components in nuclear/fusion, fossil power, defense, aerospace industries and so on [1-3]. Oxide dispersion strengthened (ODS) ferritic alloy is the most prospective structural material for high temperature service components, because of excellent creep, irradiation and corrosion resistance [1]. These superior properties come from microstructural combinations between fine ferritic matrix and nano-scaled oxide particles, which is extremely stable at the high temperature and acts as effective obstacles when the dislocations are moving in a ferritic matrix [2,3]. High Cr ferritic steels show excellent corrosion resistance due to the formation of rigid passive oxide layers on the surface such as Cr_2O_3. Al oxide could also be very sufficient passive layer to prevent corrosion and oxidation at elevated temperatures, by several weight percent of Al addition in some Fe alloys. Due to ideal combination of Cr and Al oxide films, therefore, Fe-Cr-Al ferritic alloys are considered to be suitable structural materials for high temperature service components [4,5]. Therefore, Fe-Cr-Al ODS alloys are considered to be ideal solution for superior high temperature strength and corrosion resistance. Pre-alloyed and commercial Fe-Cr-Al will be very useful to have uniform microstructures and reduce the fabrication costs. In this study, to develop the advanced structural materials for high temperature service components, Fe-22Cr-4.5Al ODS ferritic alloys were fabricated using a commercial pre-alloyed powder and their microstructures and tensile properties were investigated.

2. Experimental

To fabricate Fe-Cr-Al ferritic ODS alloys in this study, a commercial pre-alloyed powder was employed and its chemical composition was Fe(bal.)-22Cr-4.5Al-3Mo-0.5Si-0.02C in wt.% (manufactured by Sandvik Osprey Ltd., U.K.). This powder was fabricated by a high pressure Ar gas atomization process and has spherical shape with a particle size range of 38~150 μm. This ferritic alloy shows excellent corrosion, oxidation resistance with good phase stability at high temperatures due to the dense formation of passive film, consisted of chromium and aluminum oxides on the surface. Some minor elements, titanium (Ti) and zirconium (Zr) were also incorporated before a mechanical alloying process. The chemical compositions of the commercial ferritic stainless steel and Fe-Cr-Al ODS ferritic alloys were summarized in TABLE 1. The ODS ferritic alloys were fabricated by mechanical alloying (MA) and uniaxial hot pressing

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(UHP) processes. The commercial pre-alloyed powders and some raw powders were mechanically alloyed by a planetary typed ball-milling apparatus. The MA is essential process that the continuous collision of hardened steel balls and raw powders makes the repeated crushes and cold welding. This eventually create the uniform alloying or incorporation between metals and ceramics, Y_2O_3 in this study. MA atmospheres are thoroughly controlled in high purity argon (99.999%). The MA process was performed at a rotation speed of 200 rpm for 48 h with a ball-to-powder weight ratio of 15:1. The MA powder was then charged in a cylindrical carbon mold and consolidated using an UHP at 1150°C for 2 h at a heating rate of 10°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 process, the pressure was relieved and the samples were cooled in the furnace. To fabricate the thin foil specimens for TEM observations of micro-grain morphology and oxide particle distributions, ODS ferritic alloys were mechanically wet ground and a twin-jet polished using a solution of 5% $HClO_4$ + 95% methanol in vol. % at 18 V with 0.5 mA at –40°C. The oxide particles were also observed using extraction carbon replica samples by an FE-TEM. To evaluate tensile properties, 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.

TABLE 1

Chemical compositions of commercial stainless steel and Fe-Cr-Al ODS ferritic alloys (in wt.%)

Mateirals	**Elements**									
	Fe	**Cr**	**Si**	**Mn**	**C**	**Mo**	**Al**	**Ti**	**Zr**	**Y_2O_3**
STS 430L	bal.	16.5	0.7	0.7	<0.02	—	—	—	—	—
ODS1	bal.	22.0	0.5	—	0.02	3.0	4.5	0.5	—	0.35
ODS2	bal.	22.2	0.5	—	0.02	3.0	4.5	0.5	0.6	0.35

3. Results and discussion

Micro-grain and nano-scaled oxide particle morphologies in Fe-Cr-Al ODS ferritic alloys are presented in Fig. 1. The ODS ferritic alloys with Ti (ODS1) and Ti, Zr (ODS2) additions had typically equiaxed and quite homogeneous micro-grain distributions as shown in Fig. 1(a) and (b). An ODS ferritic alloy with Ti, Zr additions exhibited much more fine micro-grains than ODS ferritic alloy with only Ti addition. Mean grain sizes of ODS ferritic alloys with Ti and Ti, Zr were measured as 480 and 160 nm, respectively. The ODS ferritic alloy with Ti addition (ODS1) had somewhat inhomogeneous particle distribution, which consisted of the co-existence with fine (below 10 nm) and coarse (over 23 nm) particles. In contrast, the ODS ferritic alloy with Ti, Zr additions (ODS2) had quite uniform oxide particle distributions in the micro-grains. There were also more oxide particles on grain boundaries in the ODS2 than those in the ODS1. The number density of the both ODS ferritic alloys were so different, they were estimated to 4.02×10^{22} m^{-3} for ODS1 and 6.44×10^{22} m^{-3} for ODS2, respectively. Bright field TEM images with extracted carbon replica specimens showing oxide particle morphology and size distribution were exhibited in Fig. 2. Mean particle size were similar in both ODS ferritic alloys, which were estimated as 9.96 nm in ODS1 and 11.3 nm in ODS2. Based on TEM observations, results of image analysis on oxide particle size distribution, mean particle size, and number density were summarized in Fig. 3.

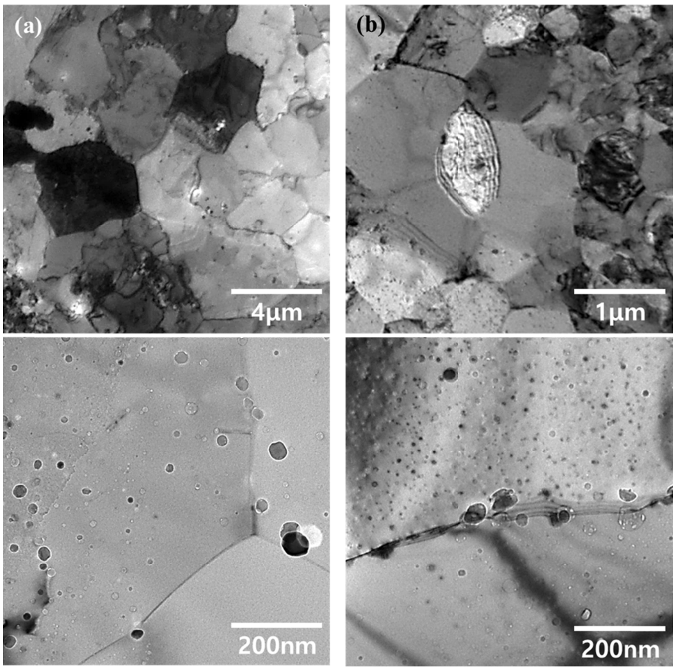

Fig. 1. Micro-grain and oxide particle distribution of the Fe-Cr-Al ODS ferritic alloys with (a)Ti, (b)Ti, Zr additions

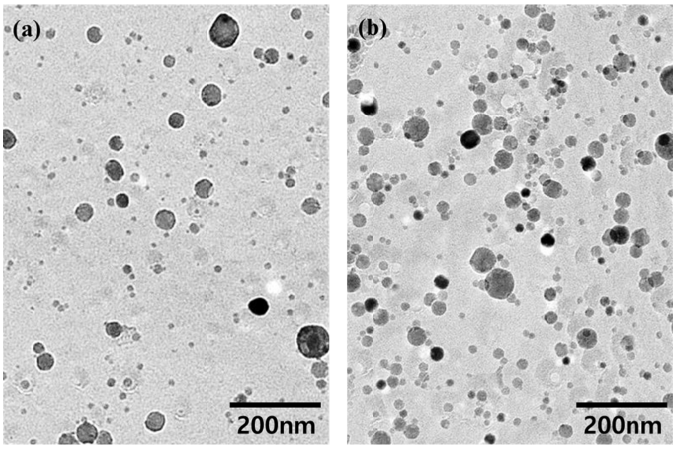

Fig. 2. Oxide particles in the Fe-Cr-Al ODS ferritic alloys with (a)Ti, (b)Ti, Zr additions

Analysis results of the constitution elements for nano-scaled oxide particles by the TEM-EDS in Fig. 4. It is conformed that oxide particles in ODS1 were consisted of Y-Si-O complex oxides. Although 0.5 wt.% Ti was added in ODS1, chemical composition of complex oxide in ODS1 was Y 12.1

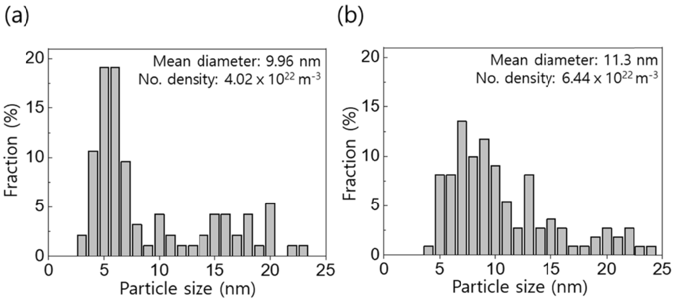

Fig. 3. Oxide particle distributions in the Fe-Cr-Al ODS ferritic alloys with (a) Ti, (b) Ti, Zr additions

at.%, Si 20.1 at.%, and O 67.6 at.%. There were many reports that oxide particles in ODS steels with Ti and Y_2O_3 additions are normally precipitated as specific oxides, namely $Y_2Ti_2O_7$ and Y_2TiO_5, which are formed by a combination of Y, Ti, and O [6]. However, $Y_2Si_2O_7$ complex oxides were formed in AISI 316L ODS austenitic steel included 0.8 wt.% of Si, because Si is also one of the very high affinity elements with oxygen [7]. ODS2 showed quite different oxide particles which confirmed as Y-Zr-O complex oxides. The chemical composition of complex oxide in ODS2 was Y 12.8 at.%, Zr 10.7 at.%, and O 48.5 at.%, which was well corresponded with $Y_2Zr_2O_7$.

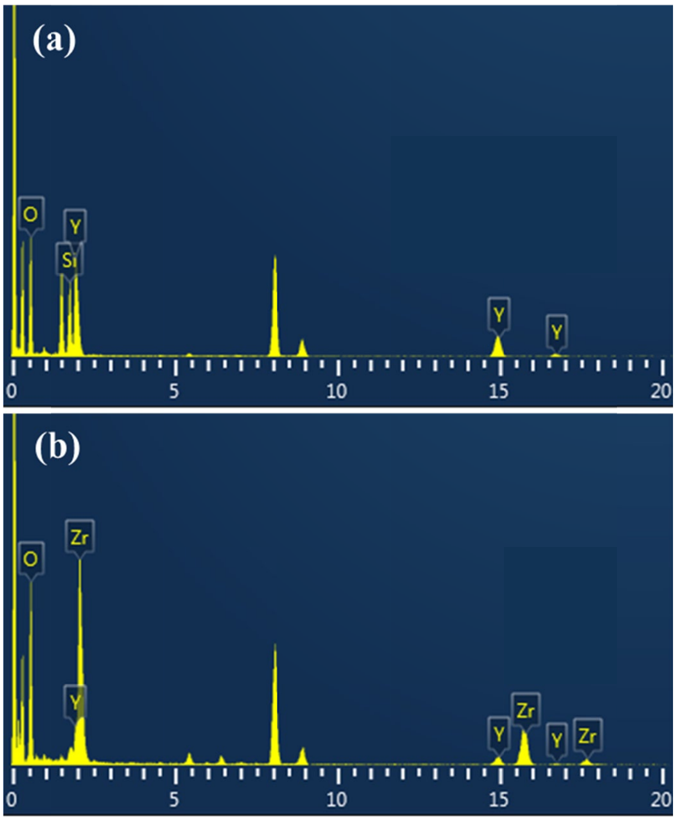

Fig. 4. TEM-EDS analysis of the oxide particles in the Fe-Cr-Al ODS ferritic alloys with (a) Ti, (b) Ti, Zr additions

Tensile stress-strain curves of commercial ferritic stainless steel and Fe-Cr-Al ODS ferritic alloys were shown in Fig. 5. Yield strengths and ultimate tensile strengths of ODS ferritic alloys were substantially higher than commercial ferritic stainless steel 430L at both room temperature and 700°C. Ultimate tensile strengths (UTS) of ODS1, 2 evaluated as 1064, 1123 MPa with a sufficient total elongation (TE) of 18.9, 14.6% at room temperature, respectively. Commercial ferritic stainless steel 430L was significantly poor, which corresponded to less than 30% of the Fe-Cr-Al ODS ferritic alloys. However, total elongations of ODS ferritic alloys were lower than ferritic stainless steel 430L as shown in Fig. 5(a). This is too poor cold workability of ODS ferritic alloys to fabricate structural components like a sheet, wire, and tube, even though Al addition of 4.5 wt.% in this study. Tensile properties at an elevated temperature were also showed similar behaviors with the results at room temperature. The commercial ferritic stainless steel showed a post yield softening with extremely poor tensile strength at 700°C, whereas the Fe-Cr-Al ODS ferritic alloys had a superior tensile strength and favorable elongation. The UTS of ODS1, 2 were respectively evaluated as 362, 383 MPa with a TE of 49.1, 50.2% at 700°C. The ODS2 with Ti, Zr additions showed higher YS and UTS than the ODS1 at 700°C. Tensile properties of ODS ferritic alloys were well coincided with the results of the microstructural observations. This is due to uniformly distributed nano-scaled complex oxides with a high number density in the extremely fine micro-grains of ODS2. Especially, complex oxide particles on the grain boundaries was very effective to improve the strength and elongation by suppression of the grain boundary sliding at elevated temperatures. Moreover, nano-scaled complex oxides are very stable even at a high temperature of up to 1000°C [8]. These usually plays an important role as efficient obstacle to prevent dislocation gliding and grain boundary sliding when the material deforms, which is well known as 'Orowan strengthening' [9]. Therefore, it is estimated that Fe-Cr-Al ODS ferritic alloy with Ti, Zr additions showed favorable microstructures and tensile properties at elevated temperature for high temperature service components.

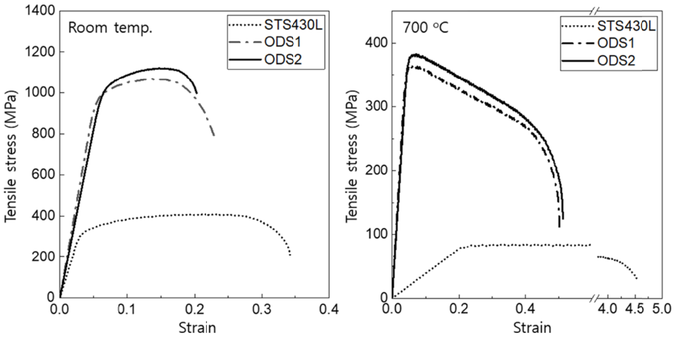

Fig. 5. Stress-strain curves in tensile tests on commercial stainless steel and Fe-Cr-Al ODS ferritic alloys

4. Conclusions

In this study, Fe-22Cr-4.5Al ODS ferritic alloys were fabricated using a commercial pre-alloyed 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. Microstructural observation and tensile property evaluation revealed that Zr addition in the Fe-Cr-Al ODS ferritic alloy was very effective to improve tensile properties at an elevated temperature. The Fe-Cr-Al ODS ferritic alloy with Ti, Zr additions showed ultra-fine grains with uniform distributions of complex oxide particles which located in micro-grains and on the grain boundaries. This led to higher tensile strength and elongation than ODS ferritic alloy with only Ti addition.

Acknowledgments

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