The microstructure and mechanical properties of Al-containing 9Cr ODS ferritic alloy

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Abstract

In this study, a 9Cr oxide-dispersion strengthened (ODS) alloy with additional corrosion resistant element Al was fabricated by mechanical alloying (MA) and hot pressing (HP) to explore the impact of Al on the microstructure and mechanical property of a 9Cr ODS alloy. It is found that the Al completely dissolved into the Fe-Cr matrix after milling for 30h. The minor phases in the Al-containing 9Cr ODS ferritic alloy were investigated by a high-energy X-ray, and were identified to be orthorhombic- $YAlO_3$ (YAP), bcc- $Y_3Al_5O_{12}$ (YAG), monoclinic- $Al_2Y_4O_9$ (YAM), and hexagonal- $YAlO_3$ (YAH). These phases were further confirmed by selected area diffraction pattern (SADP), energy dispersive spectroscopy (EDS), and high resolution transmission electron microscopy (HRTEM). In addition, their volume fractions were also calculated from the integrated intensities. According to the analysis of the particles and their formation sequences, the larger particles (greater than 100 nm) are identified as mainly YAG and Al_2O_3 particles, while the particles with small size (less than 30 nm) are likely primarily YAM, YAH, and YAP particles. The yielding strength (YS) and ultimate tensile strength (UTS) at RT are 563 MPa and 744 MPa, respectively, while the YS and UTS at 700 °C are 245 MPa and 276 MPa, respectively. Although the addition Al in ODS alloys decreases the strength at RT, the values at high temperature are similar to those obtained for 9Cr ODS alloys strengthened by fine Y-Ti-O particles.

Keywords: synchrotron, tensile deformation, nanoscale particles, microstructure

1. Introduction

The conflicts between the growing demand for energy and the goal of decreasing reliance on fossil fuels has motivated a worldwide interest in advanced nuclear energy [1, 2]. Advanced nuclear reactors require new structural materials with excellent properties including high temperature

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strength, irradiation resistance, and corrosion resistance [1, 3]. Ferritic/Martensitic oxide dispersion strengthened (ODS) alloys are one category of candidate structural materials for fuel cladding in next generation fission reactors [4, 5, 6] due to their high creep strength [7, 8, 9] and high irradiation resistance [10, 11, 12]. However, the poor corrosion resistance of F/M ODS alloys limits their development and applications in hostile environments such as the supercritical pressurized water reactor (SCPWR) [13, 14]. Recent study showed that the addition of Al in (12-19) Cr ferritic ODS alloys improved the corrosion resistance [15, 16, 17]. However, the $\alpha - \alpha'$ phase separation in the high Cr content ODS alloy actually results in deteriorated mechanical properties [18, 19]. Enenstam et al. [20] optimized the Fe-based alloy with a composition of 10Cr-4Al in a series of 10Cr alloys to balance the weldability, microstructural degradation, and corrosion resistance. So far, there is no report on the investigation of adding Al to a 9Cr ODS alloy.

The present study is an attempt to explore the possibility of fabrication of a 9Cr ODS alloy with the addition of 4.5wt% corrosion resistance element Al. The microstructure and mechanical properties of the Al-containing 9Cr ODS ferritic alloy were investigated.

2. Material and methods

The starting materials with a nominal composition of Fe-9Cr-4.5Al-0.06C-1.5W-0.18Si-0.35Y₂O₃ were mechanically alloyed (MA) using a planetary ball milling in a pure argon atmosphere at 300 rpm with a ball-to-powder mass ratio of 5:1 for different intervals. The as-milled powders for both 15 h and 30 h of milling and the 30 h milling powders with subsequently annealing at 1200 °C were investigated by scanning electron microscope (SEM, LEO-1450), X-ray diffraction (XRD, D/MAX-RB), and back scattered electron beam (BSEB). The as-milled powders after milling for 30 h were then consolidated by hot pressing (HP) using a graphite die under a pressure of 35 MPa at 1200°C for 2 h. The BN ceramic chips were put between the powders and the graphite die to avoid their reaction. The synchrotron irradiation of the as-HPed sample (9CrAl ODS alloy) was conducted at the 1-ID beamline at the Advanced Photon Source (APS) at Argonne National Laboratory (ANL). The diffraction analysis was performed by using a monochromatic 65keV synchrotron beam. The "Hydra" detector array was utilized to collect the diffraction information. Transmission electron microscopy (TEM) thin foil specimens were prepared by ion milling. The microstructure and chemical composition were characterized by TEM (JEM-2010) with an energy dispersive spectroscope (EDS). The nanoscale particles were investigated by high resolution transmission electron microscope (HRTEM). The as-HPed sample with a relative density of 98.6% was cut into tensile specimens with a gauge length of 5 mm. Tensile tests were carried out at room temperature (RT) and 700°C with a strain rate of 10⁻³ s⁻¹. The fracture surfaces of post-tensile specimens were also studied by SEM.

3. Results and Discussion

3.1. Mechanical alloying

The XRD patterns of the as-milled powders, after milling for 15 h and 30 h respectively, were shown in Figure 1. It can be seen that the Al peak still exists after milling for 15h, but disappears after 30 h, which indicates that pure Al cannot completely dissolve into the Fe-Cr matrix until 15h of milling. In addition, the previous study showed the complete dissolution of Y₂O₃ in the MA process after milling for 30 h [21]. Therefore, it can be confirmed that it is sufficient for the starting powders to form a supersaturated solid solution by MA for 30 h. Therefore only the

Figure 1: XRD patterns of the as-milled powders and the annealed powders.

Figure 2: The as-milled powders after 30 h: (a) the morphology of powders by SEM; (b) the morphology of powder at cross-section by BSEB.

as-milled powders after milling for 30 h were annealed at temperature of 1200 °C for 2 h. The XRD pattern of the as-annealed powders was also shown in Figure 1, no new phase (obvious precipitate) was distinguished after annealing at 1200 °C. One should note that the minor phases were not identified due to the limited precision of XRD. Therefore, the results simply reveal that no major phase occurs after annealing at 1200 °C. The minor phases will be discussed later by high energy X-ray and HRTEM investigations.

Figure 2 shows the morphology of the as-milled powders after 30 h of milling. The size of the powders shown in Figure 2 (a) ranges from 100 to 250 μm with an average size of about 170 μm , Sakasegawa [22] concluded that larger powders (greater than 90 μm) have lower oxygen concentrations than smaller powders (less than 45 μm), which provides a great benefit to the creep resistance. The cross section morphology by BSEB confirms the complete dissolution of Al into the matrix after milling for 30h, which can be seen in Figure 2 (b).

3.2. Microstructure characterization

Figure 3: Synchrotron X-ray diffraction line profile of the 9CrAl ODS alloy; (Ferrite matrix phases, along with minor phases, orthorhombic $YAlO_3$ (yttrium aluminum perovskite, YAP), bcc $Y_3Al_5O_{12}$ (yttrium aluminum garnet, YAG), monoclinic $Al_2Y_4O_9$ (yttrium aluminum monoclinic, YAM), and hexagonal $YAlO_3$ (yttrium aluminum hexagonal, YAH) were recognized.)

The 9CrAl ODS alloy was examined by high-energy X-ray technique. Figure 3 shows the 90° integrated intensity as a function of the d-spacing. Multiple types of Y-Al-O are expected to precipitate in the 9CrAl ODS alloy during the process of consolidation [23, 24]. The large peaks were identified as ferrite, and four types of minor precipitates (Y-Al-O) were identified as: orthorhombic- $YAlO_3$ (YAP), bcc- $Y_3Al_5O_{12}$ (YAG), monoclinic- $Al_2Y_4O_9$ (YAM), and hexagonal- $YAlO_3$ (YAH). This result agrees with the ref. [25] that all these four types mixed Al-Y oxides were identified in INCONEL alloy MA6000. The integrated intensities were used to calculate the

volume fractions of the corresponding phases based on Equation 1 [26].

$$V_i = \frac{\frac{1}{n_i} \sum_{j=1}^{n_i} \frac{I_i^j}{R_i^j}}{\frac{1}{n_a} \sum_{j=1}^{n_a} \frac{I_a^j}{R_a^j} + \frac{1}{n_{p1}} \sum_{j=1}^{n_{p1}} \frac{I_{p1}^j}{R_{p1}^j} + \frac{1}{n_{p2}} \sum_{j=1}^{n_{p2}} \frac{I_{p2}^j}{R_{p2}^j} + \dots} \quad (1)$$

where, V_i is the volume fraction of phase i ; i represents Ferrite (subscript a) and various precipitates phases (subscripts $p1, p2, \dots$); I_i^j is the integrated intensity of the reflection of j (for a specific hkl) for phase i ; n_i is the number of the reflections analysed for a certain phase; and R_i^j is the material scattering factor for a specific phase (i) and reflection (j), which has an expression in equation 2 as follows:

$$R_{hkl} = \frac{1}{V^2} [|F|^2 p (\frac{1 + \cos^2 2\theta}{\sin^2 \theta \cos \theta})] e^{-2M} \quad (2)$$

where V is the volume of the unit cell; F is the structure factor; p is the multiplicity of the reflection; $\frac{1 + \cos^2 2\theta}{\sin^2 \theta \cos \theta}$ is the Lorentz-polarization factor as a function of diffraction angle θ ; and e^{-2M} is the temperature factor. The volume fraction of each distinguishable precipitate phase was calculated and is listed in Table 1. These values were averaged from synchrotron wide-angle X-ray scattering (WAXS) data collected at three different X-ray exposures. YAG has the largest volume fraction, up to 0.51%, while the YAM has the smallest at 0.16%. Hsiung et al. reported a primarily YAM distribution in the K3-ODS alloy, which was fabricated by hot extrusion at 1150 °C [27]. Zhang et al. reported that the nano-sized oxides in the MA956 consist mainly of $YAlO_3$ in a perovskite structure (YAP) [28]. YAH was found to be the main species among oxides in the Ni-based ODS alloy MA6000 [25]. Dou et al. [29] confirmed that the hot extrusion temperature has a clear effect on both the particles size and number density, and reported a primarily YAH phase after 1050 °C, and the occurrence of YAP after 1150 °C [30]. According to the internal oxidation and polymorphic transition reactions: $2Y_2O_3 + Al_2O_3 (\gamma) = Y_4Al_2O_9$ (YAM), $Al_2O_3 (\gamma) + Y_4Al_2O_9$ (YAM) = $4 YAlO_3$ (YAH), YAH transforms to YAP due to the affinity of oxygen for different solute atoms, and finally, $Al_2O_3 (\gamma) + 3 YAlO_3$ (YAP) = $Y_3Al_5O_{12}$ (YAG) [31, 32]. The volume fraction relationship for the four particle types (YAG > YAP > YAH > YAM) may be explained by this formation sequence, which may due to the sintering process at 1200 °C and excess oxygen.

Table 1: Volume fractions of all the synchrotron-distinguishable precipitate phases in the 9CrAl ODS alloy.

Formula	Structure	Volume Fraction
YAM	orthorhombic	0.16±0.026%
YAH	hexagonal	0.21±0.086%
YAP	perovskite	0.38±0.034%
YAG	garnet	0.51±0.023%

The typical morphology of the 9CrAl ODS alloy is shown in Figure 4. Figure 4 (a) shows a TEM bright field image of the 9CrAl alloy. The grain size is as large as several microns. The multiscale precipitates with sizes ranging from several to hundreds of nanometers distribute both in the grains and on the grain boundaries. Figure 4 (b) shows a view of the nanoscale particles in a grain of the 9CrAl ODS alloy. It can be seen that the smaller sized particles with a high number

Figure 4: The bright field image of TEM for the 9CrAl ODS alloy in: (a) low magnification; (b) high magnification.

Figure 5: Size distribution of the oxide particles in the matrix of the 9CrAl ODS alloy.

density distribute uniformly in the grain. The oxides particles size distribution was detected by the careful statistical image processing method using TEM pictures with different magnifications. Figure 5 shows the result, it can be seen that the effective diameter with the highest frequency is about 8 nm. Most of the particles are smaller than 15 nm. The large precipitates (greater than 100 nm) seen in Figure 4 (a) are also included in this result, where their fraction is less than 1 %.

Figure 6: (a) selected area diffraction pattern of particle # 1 in Figure 4, (b) the EDS of particle # 1, (c) the EDS of particle # 2 in Figure 4.

In order to identify the different precipitates in the 9CrAl ODS alloy, the precipitates were further investigated by EDS, selected area diffraction pattern (SADP), and HRTEM. The labeled particle (# 1) in Figure 4 (a) with a size of 240 nm was examined by SADP as shown in Figure 6 (a) and EDS as shown in Figure 6 (b). This particle was identified as $Y_3Al_5O_{12}$ (YAG) at the zone axis $[\bar{1}11]$, while the atomic ratio of Al/Y ≈ 1.68 for this particle confirms the YAG (Al/Y ≈ 1.67). Another large precipitates (# 2) in Figure 4 (a) was also examined by EDS, the so large atomic ratio of Al/Y (≈ 37) indicates the very less-yttrium in this large particle, indicating the existence of Al_2O_3 , which agrees with ref. [28]. However, the Al_2O_3 is not distinguishable in Figure 3, which may be ascribed to the very low volume fraction of Al_2O_3 or the nonuniform distribution of Al_2O_3 in the specimen.

Table 2: Measured and calculated inter-planar distances (d) and angles (α) of the nanoscale particle in Figure 7 and possible indexing.

d(Å), α	d_1	d_2	d_3	α_{12} (°)	α_{23} (°)	α_{13} (°)
YAP	{1-2-1} 2.61	{201} 2.37	{122} 1.97	78.14	47.43	125.57
Measured	2.6	2.4	2.0	76.25	48.86	125.11
YAM	{13-1} 3.14	{202} 3.51	{1-3-1} 3.14	63.44	63.43	126.87
Measured	3.2	3.4	3.2	63.48	62.94	126.42

The nanoscale particles in Figure 4 (b) were investigated by HRTEM. Figure 7 (a) and (b) show a YAP particle with a diameter of about 36 nm, and Figure 7 (c) and (d) show a YAM

Figure 7: (a) HRTEM micrograph of an YAP particle with diameter of about 36 nm, (b) FFT diagram of the micrograph in (a); (c) HRTEM micrograph of an YAM particle with diameter of about 10 nm, (d) FFT diagram of the micrograph in (c).

particle with a diameter of about 10 nm. Table 2 lists the measured inter-planar spacing and angles of these particles. The measured values for these particles are consistent with those of $YAlO_3$ (YAP) oxide particles at the zone axis $[23\bar{4}]$ according to PDF # 701677 and $Y_4Al_2O_9$ (YAM) at the zone axis $[101]$ according to PDF # 830935, respectively. However, no YAH particle was distinguished by HRTEM, the absence of YAH cannot be concluded due to the YAH peaks in Figure 3. According to the above analysis for particles and their formation sequences, the larger particles (greater than 100 nm) are assumed to be mainly YAG and Al_2O_3 , while the smaller sized particles (less than 30 nm) may be primarily YAM, YAH, and YAP particles.

3.3. Mechanical properties

Figure 8: Strian-stress curves of the 9CrAl ODS alloy at RT and 700 °C.

Figure 8 shows the tensile properties of the 9CrAl ODS alloy at RT and 700 °C. The yield strength (YS) and ultimate tensile strength (UTS) at RT are 563 MPa and 744 MPa, respectively, while the YS and UTS at 700 °C drop to 245 MPa and 276 MPa, respectively. The YS and UTS at RT are lower than those of 9Cr ODS alloys, while the values for the 9CrAl ODS alloy at 700 °C are similar to those for 9Cr ODS alloys [33, 34, 35, 36] and much higher than those for non-ODS alloys such as CLAM and Eurofer 97 [37, 38]. As is well known, Al can influence the ductility and strength of the ODS alloys. The strength decreases with the increasing of the Al fraction in the level of lower than 0.1 wt% at 700 °C [23], the UTS of 14Cr-Al ODS alloy is much lower than that of 14Cr-Ti ODS alloy at both RT and 700 °C [39]. From these results, both the elongation and strength of the 9CrAl ODS alloy at 700 °C are acceptable compared to 9Cr ODS alloys. Figure 9 shows the fracture surface of specimens tested at RT and 700 °C. It can be seen that the fracture surface of the specimen tested at RT contains a cleavage type feature, which is surrounded by a dimple type feature. In spite that the fracture surface at 700 °C is covered by a thin oxidation layer, the large dimple can still be seen clearly, which reveals the good ductility of the 9CrAl ODS alloy at 700 °C.

Figure 9: SEM images of fracture surfaces of the specimens strained at different temperatures: (a) RT; and (b) 700 °C.

3.4. Possible mechanism

The thermal stability of multiple types Y-Al-O in INCONEL alloy MA6000 has been investigated at high temperature up to 850 °C [40]. In this work, they reported that there are no detectable coarsening of particles and no energy for transformation of particles at this temperature. Therefore, the particles should be very stable in the process of tensile test at 700 °C. The excellent mechanical properties of ODS alloys at high temperature can be ascribed to the dispersed nano sized particles (Orowan strengthening and the cutting mechanism) [41]. The strengthening effect of the Orowan mechanism depends on the particle size and number density while the cutting mechanism is related to the coherency between the particles and the matrix. The smaller sized particles (YAM, YAH, and YAP) have been verified to tend to be coherent or semi-coherent with the matrix [27, 30], which may yield good strengthening phases. In the process of tensile deformation at 700 °C, the nanoscale particles in the grains impede the movement of dislocations and the large particles on the grain boundaries hinder the sliding of grain boundaries, as shown in Figure 10. In addition, the 9CrAl ODS alloy has a larger grain size compared with 9Cr ODS alloys without Al addition, the large grain sized materials may be stronger at high temperature due to less tendency for grain boundary sliding [42]. Therefore, although Al softened the matrix of 9CrAl ODS alloy, the 9CrAl ODS alloy still possess good mechanical property at 700 °C.

4. Conclusion

The 9CrAl ODS alloy was fabricated by MA and HP. It is found that the Al completely dissolved in the Fe-Cr matrix after milling for 30h. The precipitates in the as-HPed sample were identified as orthorhombic- $YAlO_3$ (YAP), bcc- $Y_3Al_5O_{12}$ (YAG), monoclinic- $Al_2Y_4O_9$ (YAM), and hexagonal- $YAlO_3$ (YAH) by high-energy X-ray techniques. In addition, the volume fractions of the four types of precipitates were calculated from the integrated intensities. The microstructure was investigated by TEM and HRTEM. The size distribution of particles ranges from several to hundreds of nanometers, and most of the particles are smaller than 15 nm. The large particles (greater than 100 nm) are concluded to be YAG and Al_2O_3 by SADP and EDS, while the small size particles primarily belong to YAM, YAH, and YAP. The YS and UTS at RT are 563 MPa and 744 MPa, respectively, while the YS and UTS at 700 °C remain at 245 MPa and 276 MPa, respectively. The elongation increases from 16% to 19% with the temperature increasing from RT to 700 °C.

Figure 10: TEM bright field image of the gauge areas of the post strained specimens at 700 °C.

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