The comparison of microstructures and mechanical properties between 14Cr-Al and 14Cr-Ti ferritic ODS alloys

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The outstanding material performance stems from their unique microstructure: the highly stable nano-sized oxide particles. It was reported that high content of chromium (\(\sim 13\%\)) in the ODS alloys is effective to suppress corrosion [11,12], while the high chromium content (\(\sim 14\%\)) can result in a thermal aging embrittlement [13,14]. Some researchers have reported the effectiveness of Al addition in ODS ferritic alloys for the improvement of corrosion resistance in Lead-cooled fast reactor (LFR) and supercritical water reactor (SCWR) [15–17]. Therefore, it is expected that an adequate combination of the Cr and Al contents will benefit for ODS ferritic alloys in the application of high temperature corrosion environment.

However, there are also some results to prove that the added Al decreased the strength of ODS alloys [15], which may be caused by the coarsened grains and different types of particles (Y-Al-O) compared with the ODS alloys without Al [18,19]. In this present study, in order to figure out the different effect of Al and Ti on the microstructures and mechanical properties of 14Cr ODS alloys. The high-energy synchrotron X-ray technique was applied to study the different responses of both matrix and nanoscale particles to externally applied stresses for 14Cr-Ti ODS and 14Cr-Al ODS alloys, respectively. The microstructure and fracture surface for these two materials were also investigated.

1. Introduction

With the improvement in safety, energetic efficiency and sustainability for advanced nuclear systems leading to higher demanding for the structural materials [1–3], the oxide dispersion strengthening (ODS) ferritic alloys have attracted increasing attentions as one of candidate materials, because of their exceptional irradiation resistance [4–6], excellent high temperature strength, and creep resistance [7,8]. The outstanding material performance stems from their unique microstructure: the highly stable nano-sized oxide particles [9,10]. It was reported that high content of chromium (\(\sim 13\%\)) in the ODS alloys is effective to suppress corrosion [11,12], while the high chromium content (\(\sim 14\%\)) can result in a thermal aging embrittlement [13,14]. Some researchers have reported the effectiveness of Al addition in ODS ferritic alloys for the improvement of corrosion resistance in Lead-cooled fast reactor (LFR) and supercritical water reactor (SCWR) [15–17]. Therefore, it is expected that an adequate combination of the Cr and Al contents will benefit for ODS ferritic alloys in the application of high temperature corrosion environment.

2. Materials and methods

2.1. Investigated materials

Nitrogen-gas-atomized powders were mixed with 0.35 wt% nano sized \(Y_2O_3\) powders and 0.4 wt% titanium powders for 14Cr-Ti ODS alloys, while nitrogen-gas-atomized powders were mixed with 0.35 wt% nano sized \(Y_2O_3\) powders and 4.5 wt% aluminum powders for 14Cr-Al ODS alloys. Then, two kinds of mixed powders were mechanical alloyed in a high-energy planetary ball milling with same parameters.
to-powder weight ratio of 5:1, rotation speed of 300 rpm, and total milling time of 30 h) under pure argon atmosphere, respectively. Subsequently, the as-milled powders were consolidated by hot isostatic pressing (HIP) at 1150 °C under a pressure of 150 MPa for 2 h. The as-HIPed samples were forged at 1250 °C with a forging ratio of 3:1 and were then hot rolled at 1150 °C in two stages with total reduction ratio of 40%. Finally, the as-rolled samples were tempered at 650 °C for 2 h to relax the residual elastic stress. The chemical compositions of two kinds of as-rolled 14Cr ODS alloys were measured by destructive chemical analysis method. Table 1 shows the results.

2.2. Synchrotron WAXS investigation

The in-situ synchrotron tensile investigations were conducted at the 1-ID beamline at the Advanced Photon Source (APS), Argonne National Laboratory (ANL). The miniature tensile specimens with the gauge sections of 1.20 mm × 0.50 mm × 5.00 mm were cut from the two types of materials. The tensile tests were conducted at ambient temperature with a strain rate of 1 × 10⁻⁴ s⁻¹. The X-ray scattering was performed using a monochromatic 70 keV synchrotron beam with a 300 μm × 300 μm cross-section. The Hydra detector array including four identical GE-41RT 2D detectors was used to obtain the diffraction signals, as shown in Fig. 1.

The synchrotron diffraction signals can be used to monitor the lattice strain evolution of different phases during the tensile deformation. The 15° region of the Debye-Scherrer rings near the uniaxial tensile direction were integrated to study the evolution of dislocation density in the process of tensile deformation from the synchrotron diffraction signals [24,25,20].

![Fig. 1. Experimental set-up of the uniaxial tensile test with in-situ synchrotron X-ray diffraction [20].](image)

where $V$ is the volume of the unit cell; $F$ is the structure factor; $p$ is the multiplicity of the reflection; $L_{\text{av}}$ is the Lorentz-polarization factor as a function of diffraction angle $\theta$; and $e^{-2M}$ is the temperature factor, which was calculated according to the Debye temperature and experimental temperature.

The modified Williamson-Hall (W-H) method has been applied to investigate the evolution of dislocation density in the process of tensile deformation from the synchrotron diffraction signals [24,25,20].

$$\Delta K = \frac{0.9}{D} + \left( \frac{n M^2 b^4}{2} \right) e^{2M} (K \lambda^2)$$

where $K = 2 \sin \theta / \lambda$; $\Delta K = 2 \cos \theta / \lambda$; $\theta$ is the diffraction angle, and $\Delta \theta$ is the full width at half maximum (FWHM); $D$ is the average grain size; $M$ is an adjustable parameter depending on the effective outer cut-off radius of dislocations, where $M = 2$ was used for all deformed samples [26]. $b$ is the Burgers vector of dislocations and $p$ is dislocation density; and $C$ is the dislocation contrast factor, which depends on different possible combinations of Burgers vectors, line vectors of dislocations, the diffraction vector and anisotropic elastic constants [27].

2.3. Electron microscopy investigation

The microstructures of two materials were investigated from both thin foil samples and carbon extraction replicas using transmission electron microscopy (TEM, JEOL JEM-2100) equipped with EDS. The thin foil samples were mechanically thinned to 100 μm and punched into 3 mm discs, and then electropolished with 5% perchloric acid and 95% methanol at −20 °C using a Struer Tenupol-5 twin-jet polisher. The carbon extraction replicas were prepared from the mechanically polished surfaces. The polished surface was pre-etched by aqua regia solution (75% hydrochloric acid + 25% nitric acid) for 20 s, and then coated by a carbon film. The carbon film covering almost all of the nano-sized oxide particles were detached from matrix by electro-etching in a dilute acid solution (5% perchloric acid + 95% methanol) with a voltage of 30 V at 20 °C and then mounted on a Cu grid. Thus, the conventional TEM

Table 1

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<th>Materials</th>
<th>C</th>
<th>Cr</th>
<th>W</th>
<th>V</th>
<th>Si</th>
<th>Y</th>
<th>Ti</th>
<th>Al</th>
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<td>Cr-Ti-ODS</td>
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<td>13.8</td>
<td>1.01</td>
<td>0.18</td>
<td>0.24</td>
<td>0.18</td>
<td>0.41</td>
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<td>0.21</td>
<td>0.43</td>
<td>0.005</td>
<td>0.01</td>
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<tr>
<td>Cr-Al-ODS</td>
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<td>13.8</td>
<td>1.02</td>
<td>0.18</td>
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<td>–</td>
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<td>0.46</td>
<td>0.004</td>
<td>0.01</td>
<td>Bal.</td>
</tr>
</tbody>
</table>

where $\varepsilon_{\text{bulk}}$ is the lattice strain for the matrix of Zircaloy-2 by averaging the lattice strains of {110}, {200}, {211}, {310}, and {321} respectively. The $dhkl$ is defined as the d-spacing when elastic stress is free, $E_{hkl}$ is the Young’s modulus of the (hkl) orientation, $\rho_{hkl}$ is the lattice strain of the (hkl) reflection, and $T_{hkl}$ is the Harris texture index [23], which is defined by the following equations:

$$T_{hkl} = \frac{1}{n} \sum_{j=1}^{n} \left( \frac{T_{hj}/R(hj)}{R(hj)} \right)$$

where, $I(hj)$ is the integrated intensity of reflection $hj$, and $R(hj)$ is the integrated intensity calculated from the structure factor and other parameters for a samples with a random orientation of crystallites, which has the following expression:

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

is the multiplicity of the (hkl) reflection, $\rho_{hkl}$ is the Young’s modulus of the (hkl) orientation, $\rho_{hkl}$ is the lattice strain of the (hkl) reflection, and $T_{hkl}$ is the Harris texture index [23], which is defined by the following equations:

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Table 1

The chemical compositions (wt%) of the ODS ferritic alloys investigated in the present study.
bright field (BF) images were taken from the thin foil samples and carbon extraction replicas. In addition, the fracture surfaces of the post-strained specimens and the microstructure morphologies of unstrained specimens were investigated by scanning electron microscope (SEM, LEO-1450).

3. Results and discussion

3.1. Phase identification

Fig. 2 shows the synchrotron X-ray diffraction line profiles for both 14Cr-Ti ODS alloy and 14Cr-Al ODS alloy. The main peaks for both materials are phases of the ferritic matrix. The line profiles were zoomed in to find the minor phases. In 14Cr-Ti ODS alloy, the typical NaCl structure TiN and pyrochlore structure Y$_2$Ti$_2$O$_7$ were identified, while in the 14Cr-Al alloy, the hexagonal YAlO$_3$ (yttrium aluminum hexagonal, YAH), monoclinic Al$_2$Y$_4$O$_9$ (yttrium aluminum monoclinic, YAM), and AlN were distinguished.

In order to confirm the phases, the microstructures of these two materials were investigated by TEM and EDS. The TEM results of 14Cr-Ti ODS alloy were shown in Fig. 3. The average grain size is about 300 nm as shown in Fig. 3(a), the TiN with size of hundreds nanometers are shown in Fig. 3(b). In order to give the statistic size distribution of Y$_2$Ti$_2$O$_7$ nanoscale particles, the TEM BF image of carbon extraction replica was shown in Fig. 3(c), which the statistic distribution results with average size of about 9.6 nm ranging from 4.1 nm to 31.5 nm can be seen in Fig. 3(d).

The TEM results of 14Cr-Al ODS alloy are shown in Fig. 4. There are no complete grains in the view field of Fig. 4(a) due to their large grain size. The large size precipitate AlN was found in the matrix and confirmed by EDS, as shown in Fig. 4(b). Fig. 4(d) and (e) shows the selected area diffraction patterns (SADP) of the labeled particles (#1 and #2) in Fig. 4(a), respectively. The particle #1 was identified as the YAH particle at zone axis [331] and the particle #2 was identified as the YAM particle at zone axis [722]. Hsiung et al. [28] reported a primarily YAM distribution in the K3-ODS alloy and the YAH was found to be the main species among oxides in the Ni-based ODS alloy MA6000 [29]. Therefore, from the results of TEM and high-energy X-ray, the YAM and YAH particles can be assumed as the main species oxide particles in 14Cr-Al ODS alloy. Though orthorhombic-YAlO$_3$ (YAP) and bcc-Y$_3$Al$_5$O$_{12}$ (YAG) particles were not distinguished using both TEM and high-energy X-ray, the inexistence of YAG and YAP cannot be concluded, which may be ascribed as the small volume fraction. Due to different crystal structures of Y-Al-O particles (YAM, YAH, YAP, and YAG) in ODS alloys, the multiple types of nanoscale particles Y-Al-O shown...
in Fig. 4(c) cannot be distinguished one by one. The size distribution of multiple types Y-Al-O particles with an average size of about 15 nm ranging from 5.03 nm to 80.25 nm was shown in Fig. 4(d). From the results of two materials, the 14Cr-Al ODS alloy has much larger grain and slightly larger particles than 14Cr-Ti ODS alloy, which may have a significant influence on mechanical properties.

3.2. Macroscopic stress-strain and load partitioning

Fig. 5 shows the engineering stress-engineering strain curves of the 14Cr-Ti ODS alloy and 14Cr-Al ODS alloy tested at ambient temperature. The yield strength (YS) and ultimate tensile strength (UTS) for 14Cr-Ti ODS alloy are 937 MPa and 977 MPa, respectively, while those values of 14Cr-Al ODS alloy drop to 530 MPa and 681 MPa, respectively. The total elongation of the 14Cr-Ti ODS and 14Cr-Al ODS are 0.021 and 0.16, respectively. It is obvious that the 14Cr-Al ODS alloy has lower strength, but much larger elongation compared to 14Cr-Ti ODS, which agrees with the result in ref. [15].

Fig. 6 shows the true stress-lattice strain curves of five different reflections in both directions of perpendicular and parallel to the tensile loading direction, respectively for: (a) the 14Cr-Ti ODS alloy; (b) the 14Cr-Al ODS alloy.

**Fig. 4.** The TEM results of 14Cr-Al ODS alloy: (a) the BF image in low magnification, (b) the EDS result for AlN, (c) the distribution of nanoscale particles Y-Al-O by the carbon extraction replica, (d) the selected area diffraction pattern of particle # 1 in Fig. 4(a), (e) the selected area diffraction pattern of particle # 2 in Fig. 4(a), (f) the statistic size distribution of nanoscale particles in (c).

**Fig. 5.** Strain-stress curves of the 14Cr-Ti and 14Cr-Al ODS alloys at ambient temperature.

**Fig. 6.** The true stress-lattice strain curves of five different reflections in both directions of perpendicular and parallel to the tensile loading direction, respectively for: (a) the 14Cr-Ti ODS alloy; (b) the 14Cr-Al ODS alloy.
all five reflections for the 14Cr-Al ODS alloy are smaller than those values of 14Cr-Ti ODS alloy. This indicates that the Al softened the matrix of 14Cr-Al ODS alloy. The lattice constants for these two materials were also calculated according to Eq. (1). The lattice constants for 14Cr-Ti and 14Cr-Al ODS alloys are (2.883 ± 0.00101) Ångström and (2.874 ± 0.00098) Ångström, respectively. The lattice constant of 14Cr-Al is about 0.3% larger than that of 14Cr-Ti ODS alloy. According to the atomic radius for the matrix elements (Cr ~ 1.27 Å, Fe ~ 1.27 Å, Ti ~ 1.45 Å, Al ~ 1.43 Å), the atomic radius of Al is 12.6% larger than those of the matrix elements (Fe and Cr). Therefore, the larger lattice constant of 14Cr-Al may be ascribed to the solid solution effect of Al into Fe-Cr matrix. Though, the atomic constant of Ti is also larger than those of matrix elements, most of Ti was used to form the Y-Ti-O particles in 14Cr-Ti ODS alloys. Fig. 6 also shows that the lattice strain begins slightly compressive shifts with increasing applied stress after samples’ yielding, indicating the onset of load partitioning.[30,31] In order to compare the load partitioning phenomenon in these two materials, the average bulk lattice strains were calculated based on Eq. (1).

Fig. 7 shows the average bulk lattice strain as a function of true stress before necking. It can be seen that there is a prominent compressive shift in the curve of 14Cr-Al ODS, while the curve of 14Cr-Ti ODS just shifts a little. This illustrates the load transfer between the matrix and nanoscale particles occurred in 14Cr-Al ODS alloy are more significant than that in 14Cr-Ti ODS. However, this phenomenon does not prove the worse strengthening effect of Y-Ti-O particles in 14Cr-Ti ODS than the Y-Al-O particles in 14Cr-Al. The less load transfer in 14Cr-Ti ODS may be induced by the stress from YS to UTS, which the enhanced dislocation pile-up led to the increase in lattice strain of different precipitates during this period. The different increasing speed of dislocation density between these two materials revealed the better strengthening effect of Y-Ti-O particles in 14Cr-Ti ODS than that of Y-Al-O particles in 14Cr-Al ODS alloys.

Due to the limited analyzable reflections of the precipitates phases, only the specific lattice strains of the precipitates were measured and used as the representatives of the phase. Generally for 14Cr-Al ODS and 14Cr-Ti ODS alloys, during the elastic deformation region, the lattice strains of the analyzable precipitate phases in these two materials are all close to the lattice strains of their ferritic matrix, respectively. After the samples yield, the lattice strains of precipitate phases become much larger than those of their ferritic matrix. This difference in lattice strains for two materials implies that the significant amount of the stress transfers to the precipitates from matrix by the particle-dislocation interactions. In addition, the lattice strain of Y2Ti2O7 is much larger than that of TiN in 14Cr-Ti ODS alloy, and the lattice strains of YAH and YAM are much larger than that of AlN in 14Cr-Al ODS alloy, which revealing a strong dependence of partitioning phenomenon on the particle size.

Due to the higher elastic constants of nanoscale particles than those of metallic phases, the relations in elastic stress between the ferritic matrix and the precipitates may vary with the relations in lattice strains between different phases. Therefore, the actual elastic stress of each precipitates phase was determined and then compared with that of the matrix to provide more useful information on the load partitioning phenomenon than the elastic strain. The Young’s modulus of the precipitates and ferritic matrix were derived from the elastic constants and were found to be: $E_{(111)} = 337$ GPa[30], $E_{(222)} = 286$ GPa[32], $E_{(102)} = 309$ GPa[33], $E_{(102)} = 242$ MPa[34], $E_{(221)} = 199$ MPa[31], $E_{(111)} = 193$ GPa and $E_{(200)} = 205$ GPa[35], (subscripts t for TiN, y2 for Y2Ti2O7, a for AlN, h for YAH, and m for YAM) for matrix of 14Cr-Al, $f_2$ for the matrix of 14Cr-Ti).

Fig. 8(b) shows the elastic stresses of different precipitates and ferritic matrix for 14Cr-Ti ODS and 14Cr-Al ODS alloys. Generally for these two samples, the difference of elastic stress between the ferritic matrix and the precipitates is significant. In the 14Cr-Ti ODS, the Y2Ti2O7 bears the much higher stress than TiN and matrix, which is agreed with the results of 9Cr ODS and ODS 304 steel[36,31], as explained as the size effect. In the 14Cr-Al ODS alloy, YAH particles bear the highest stress in three different precipitate phases (YAH, YAM, and AlN). As it is difficult to distinguish the YAH and YAM one by one, the size distribution of each phases cannot be figured out. In addition, the YAM has the same level stress with AlN. Therefore, the load partitioning not only has size effect but also matters with the elastic constants. Thus, the YAH phase is a better strengthening phase than YAH and AlN in 14Cr-Al ODS alloy.

Fig. 9 shows the dislocation density evolution in the entire process of tensile deformation for 14Cr-Ti ODS and 14Cr-Al ODS alloys. It can be seen that when plastic deformation was initiated in materials, the dislocation density increased faster in 14Cr-Ti ODS than that in 14Cr-Al ODS alloys. Wang et al.[25] reported two deformation stages in the tensile deformation of G91 at ambient temperature. The flow stress increased almost linearly with the W-H slope firstly and subsequently increased almost linearly with the W-H y-intercept up to the onset of necking. As is known, the W-H slope and W-H y-intercept represent dislocation density and grain size, respectively. Thus, the profiles of dislocation density vs. true stress from YS to UTS for 14Cr-Ti ODS and 14Cr-Al ODS alloys were shown in Fig. 9 (b). The good linearity shown in this Fig. 9 (b) validates the dominance of dislocation glide in the period of true stress from YS to UTS, which the enhanced dislocation pile-up led to the increase in lattice strain of different precipitates during this period. The different increasing speed of dislocation density between these two materials revealed the better strengthening effect of Y-Ti-O particles in 14Cr-Ti ODS than that of Y-Al-O particles in 14Cr-Al ODS alloys.

### 3.3. Electron microscopy investigations

The fracture morphologies of two strained specimens are shown in Fig. 10. Fig. 10 (a) shows a brittle type cleavage feature in the fracture surface of 14Cr-Ti ODS alloy, while most of surface is ductile dimpling fracture in 14Cr-Al ODS alloy, shown in Fig. 10 (b). It is noted that several cracks can be seen in the fracture surface of 14Cr-Ti ODS alloy. In order to confirm if the cracks formed before tensile deformation. The SEM images of microstructure morphologies of unstrained specimens of these two kinds of alloys were shown in Fig. 10(c). There can be seen that there are no large cracks in both materials. The relative density of 14Cr-Ti and 14Cr-Al ODS alloys were measured as 97% (7.53 g/cm³) and 99% (7.12 g/cm³/7.19 g/cm³), respectively. Therefore, the cracks on the fracture surface may be induced by the stress.

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**Table 2**

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<th>Young’s modulus E (GPa)</th>
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<th>(310)</th>
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<td>170</td>
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<td>184</td>
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<td>197</td>
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**Fig. 7.** The true stress-average bulk lattice strain curves in tensile loading direction for 14Cr-Ti ODS alloy and 14Cr-Al ODS alloy.
concentration around voids in 14Cr-Ti ODS alloy. The voids may take partial responsibility for the bad ductility of 14Cr-Ti ODS alloy.

3.4. Possible mechanism

The microstructures and mechanical properties of two materials have been discussed above. Though the voids in 14Cr-Ti ODS alloy may influence its tensile property. The YS still can be used to compare with that of 14Cr-Al ODS alloy. The YS of 14YWT has been predicted successfully by a method developed by Kim et al. [37]. This method includes four strengthening mechanisms as shown in Eq. (5).

\[
YS = \left( \sigma_p^2 + (\sigma_m + \sigma_k + \sigma_{dbh})^2 \right)^{\frac{1}{2}}
\]

where, \(\sigma_p\), \(\sigma_m\), \(\sigma_k\), \(\sigma_{dbh}\) are dislocation forest hardening, solid solution hardening, Hall-Petch strengthening, and the dispersed barrier hardening, respectively.

![Fig. 8. Lattice strain/stress evolution of different phases in 14Cr-Al ODS and 14Cr-Ti ODS alloy: (a) lattice strain vs. true strain; (b) elastic stress vs. true strain.](image)

![Fig. 9. The dislocation density evolution in the 14Cr-Ti ODS and 14Cr-Al ODS alloys: (a) dislocation density vs. engineering strain; (b) dislocation evolution compared with true stress.](image)

![Fig. 10. The SEM images of fracture surfaces of the specimens strained and microstructure morphologies of unstrained specimens for: (a) (c) 14Cr-Ti ODS alloy; (b) (d) 14Cr-Al ODS alloy.](image)
hardening, respectively. As the mechanical property is related to the microstructure, thus the difference of mechanical properties between two materials can be explained as follows: First, solid solution hardening $\sigma_s$, Al softened the matrix of 14Cr ODS (shown in Table 2) because of the solid solution of Al into matrix. Second, the Hall-Petch strengthening $\sigma_p$, the Al caused a larger size of grains, which decreased the strength of 14Cr-Al ODS. Third, the dispersed barrier hardening, $\sigma_{dbh}$. The larger Y-Al-O particles (YAH and YAM) with low number density in 14Cr-Al ODS alloy has worse strengthening effect than the high density Y$_2$Ti$_2$O$_7$ particles in 14Cr-Ti ODS alloy. Fourth, dislocation forest hardening, $\sigma_f$. The dislocation density in 14Cr-Al ODS alloy increased slowly with the increasing deformation. These four aspects make the difference of these two materials.

4. Conclusion

The different effect of Al and Ti on the microstructure and mechanical properties of 14Cr ODS alloys were studied. In 14Cr-Ti ODS alloy, the minor phases were identified as the typical NaCl structure TiN and pyrochlore structure Y$_2$Ti$_2$O$_7$, while in the 14Cr-Al alloy, the hexagonal YAlO$_3$ (yttrium aluminum hexagonal, YAH), monoclinic Al$_2$Y$_2$O$_5$ (yttrium aluminum monoclinic, YAM), and AlN were distinguished. The 14Cr-Al ODS alloy has much larger grains and slightly larger particles compared to 14Cr-Ti ODS alloy. The 14Cr-Al ODS alloy has much larger elongation than 14Cr-Ti ODS. From the comparison of elastic stress, Y$_2$Ti$_2$O$_7$ is better strengthening phase than TiN in 14Cr-Ti and YAH is much better than YAH and AlN in 14Cr-Al ODS. The different increasing speed of dislocation density with plastic deformation revealed the better strengthening effect of Y-Ti-O particles in 14Cr-Ti ODS than that of Y-Al-O particles in 14Cr-Al ODS alloy.

Acknowledgement

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