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

Guangming Zhang^{a,b}, Zhangjian Zhou^{a,*}, Kun Mo^c, Yinbin Miao^{b,c}, Shaofu Li^a, Xiang Liu^b,
Man Wang^a, Jun-Sang Park^d, Jonathan Almer^d, James F. Stubbins^b

^aSchool of Materials Science and Engineering, University of Science and Technology, Beijing, Beijing 100083, China

^bDepartment of Nuclear, Plasma and Radiological Engineering, University of Illinois at Urbana-Champaign, IL 61801, USA

^cNuclear Engineering Division, Argonne National Laboratory, Argonne, IL 60439, USA

^dX-ray Science Division, Argonne National Laboratory, Argonne, IL 60439, USA

Abstract

In this study, two kinds of 14Cr ODS alloys (14Cr-Al and 14Cr-Ti) were investigated to reveal the different effects between Al and Ti on the microstructures and mechanical properties of 14Cr ferritic ODS alloys. The microstructure information such as grains, minor phases of these two alloys has been investigated by high-energy X-ray diffraction and transmission electron microscopy (TEM). The in situ synchrotron X-ray diffraction tensile test was applied to investigate the mechanical properties of these two alloys. The lattice strains of different phases through the entire tensile deformation process in these two alloys were analyzed to calculate their elastic stresses. From the comparison of elastic stress, the strengthening capability of $Y_2Ti_2O_7$ is better than TiN in 14Cr-Ti, and the strengthening capability of YAH is much better than YAM and AlN in 14Cr-Al ODS. The dislocation densities of 14Cr-Ti and 14Cr-Al ODS alloys during tensile deformation were also examined by modified Williamson-Hall analyses of peak broadening, respectively. The different increasing speed of dislocation density with plastic deformation reveals 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.

Keywords: synchrotron X-ray diffraction, tensile deformation, nanoscale particles, microstructure

1. Introduction

With the improvement in safety, energetic efficiency and sustainability for advanced nuclear systems leading to higher demanding for the structural materials [1, 2, 3], the oxide dispersion strengthening (ODS) ferritic alloys have attracted increasing attentions as one of candidate materials, because of their exceptional irradiation resistance [4, 5, 6], excellent high temperature strength, and creep resistance [7, 8]. The outstanding material performance stems from their unique

*Address: Laboratory of Special Ceramics and Powder Metallurgy, School of Material Science and Engineering, University of Science and Technology Beijing, Beijing 100083, P.R.China Tel: +86-10-62334951 Email: zhouzhj@mater.ustb.edu.cn

microstructure: the highly stable nano-sized oxide particles [9, 10]. It was reported that high content of chromium (>13%) in the ODS alloys is effective to suppress corrosion [11, 12], while the high chromium content (>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, 16, 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 caused by the coarsed 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.

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 (the ball-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 2h. 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 2h 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.

Table 1: The chemical compositions (wt.%) of the ODS ferritic alloys investigated in the present study.

Materials	C	Cr	W	V	Si	Y	Ti	Al	O	N	S	P	Fe
14Cr-Ti-ODS	0.06	13.8	1.01	0.18	0.24	0.18	0.41	-	0.21	0.43	0.005	0.01	Bal.
14Cr-Al-ODS	0.06	13.8	1.02	0.18	0.23	0.18	-	4.4	0.18	0.46	0.004	0.01	Bal.

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

$1 \times 10^{-4} \text{ s}^{-1}$. The X-ray scattering was performed using a monochromatic 70 keV synchrotron beam with a $300 \mu\text{m} \times 300 \mu\text{m}$ 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.

Figure 1: Experimental set-up of the uniaxial tensile test with in-situ synchrotron X-ray diffraction [20].

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 and perpendicular to uniaxial tensile direction were integrated to give the lattice strains in two directions, $\varepsilon_{hkl} = (d_{hkl} - d_{hkl}^0) / d_{hkl}^0$, respectively. The d_0 is defined as the d-spacing when elastic stress is free, which is first determined by two linear fitting on the data of two directions during the elastic deformation, and finding d_0 at the intersection of two fitted line. In order to minimize the effect of elastic and plastic anisotropy, the average bulk lattice strains for the ferritic matrix were calculated from the lattice strains of $\{110\}$, $\{200\}$, $\{211\}$, $\{310\}$, and $\{321\}$ reflections using the weighted averaging single peaks developed by Daymond [21]. This method has been used to calculate the average bulk lattice strain for the matrix of Zircaloy-2 by averaging five Zircaloy-2 peaks [22].

$$\bar{\varepsilon} = \frac{\sum_{hkl} T_{hkl}(\theta, \psi) m_{hkl} E_{hkl} \varepsilon_{hkl}}{\sum_{hkl} T_{hkl}(\theta, \psi) m_{hkl} E_{hkl}} \quad (1)$$

where, m_{hkl} is the multiplicity of the (hkl) reflection, E_{hkl} is the Young's modulus of the (hkl) orientation, ε_{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:

$$T(h_i) = \frac{I(h_i)/R(h_i)}{\frac{1}{n} \sum_{j=1}^n I(h_j)/R(h_j)} \quad (2)$$

, where, $I(h_j)$ is the integrated intensity of reflection h_j , and $R(h_j)$ 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:

$$R_{hkl} = \frac{1}{V^2} [|F|^2 p (\frac{1 + \cos^2 2\theta}{\sin^2 \theta \cos \theta})] e^{-2M} \quad (3)$$

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, 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].

$$\Delta K = \frac{0.9}{D} + (\frac{\pi M^2 b^2}{2})^{\frac{1}{2}} \rho^{\frac{1}{2}} (KC^{\frac{1}{2}}) \quad (4)$$

where $K = 2 \sin \theta / \lambda$; $\Delta K = 2 \cos \theta \Delta \theta / \lambda$; θ is the diffraction angle, and $\Delta \theta$ is the half of 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 ρ 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 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

Figure 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. Beside the main peaks, the line profiles were zoomed in to find the minor phases. In 14Cr-Ti ODS alloy, the typical NaCl structure TiN and pyrochlore structure $Y_2Ti_2O_7$ were identified, while in the 14Cr-Al alloy, the hexagonal $YAlO_3$ (yttrium aluminum hexagonal, YAH), monoclinic $Al_2Y_4O_9$ (yttrium aluminum monoclinic, YAM), and AlN were distinguished.

Figure 2: Synchrotron X-ray diffraction line profiles for the 14Cr-Ti ODS and 14Cr-Al ODS alloys.

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 Figure 3. The average grain size is about 300 nm as shown in Figure 3(a), the TiN with size of hundreds nanometers are shown in Figure 3(b). In order to give the statistic size distribution of $Y_2Ti_2O_7$ nanoscale particles, the TEM BF image of carbon extraction replica was shown in Figure 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 Figure 3(d).

The TEM results of 14Cr-Al ODS alloy are shown in Figure 4. There are no complete grains in the view field of Figure 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 Figure 4 (b). Figures 4 (d) and (e) shows the selected area diffraction patterns (SADP) of the labeled particles (#1 and #2) in Figure 4 (a), respectively. The particle #1 was identified as the YAH particle at zone axis $[3\bar{3}1]$ and the particle #2 was identified as the YAM particle at zone axis $[7\bar{2}2]$. 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- $Y_3Al_5O_{12}$ (YAG) particles were not distinguished using both TEM and high-energy X-ray, the inexistence of YAG and YAP cannot be concluded, which maybe 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 Figure 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 Figure 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.

Figure 3: The TEM results of 14Cr-Ti ODS alloy: (a) the BF image in low magnification, (b) the BF image in high magnification, (c) the distribution of nano scale particles $Y_2Ti_2O_7$ by the carbon extraction replica, (d) the statistic size distribution of nanoscale particles in (c).

Figure 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 Figure 4 (a), (e) the selected area diffraction pattern of particle # 2 in Figure 4 (a), (f) the statistic size distribution of nanoscale particles in (c).

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].

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

Figure 6 shows the true stress-lattice strain curves of five different reflections in both directions of perpendicular and parallel to the tensile loading direction for the 14Cr-Ti ODS and the 14Cr-Al ODS alloy, respectively. The lattice strains of five reflections in two directions for two materials increase with the increasing applied stress. Young's modulus E is the slope in the direction of parallel to the tensile loading direction in the elastic regime, which is calculated by assuming a linear fit between stress and strain, as shown in Table 2. It can be seen that the Young's modulus of 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 ODS alloy. The lattice constants for these two materials were also calculated according to d_{hkl}^0 . The lattice constants for 14Cr-Ti and 14Cr-Al ODS alloys are (2.883 ± 0.00101) angstrom and (2.874 ± 0.00098) angstrom, 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 $\sim$ 1.27, Fe $\sim$ 1.27, Ti $\sim$ 1.45, Al $\sim$ 1.43 angstrom), 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 and the TiN precipitates in 14Cr-Ti

ODS. Figure 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 equation 1.

Figure 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.

Table 2: Young's modulus of the matrix at five different reflections for 14Cr-Ti ODS and 14Cr-Al ODS, respectively.

Young's modulus E (GPa)	{110}	{200}	{211}	{310}	{321}
14Cr-Ti-ODS	208	170	209	184	213
14Cr-Al-ODS	203	153	197	174	206

Figure 7: The true stress-average bulk lattice strain curves in tensile loading direction for 14Cr-Ti ODS alloy and 14Cr-Al ODS alloy.

Figure 7 shows the average bulk lattice strain as a function of true stress before necking. It can be seen that there is an 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 alloy is due to the bad ductility, which makes the Y-Ti-O nanoscale particles in 14Cr-Ti ODS have no chance to express their full potential to strengthen matrix. In order to further understand this phenomenon, the lattice responses of various precipitates phases and the ferritic matrix to the uniaxial tensile stress for both 14Cr-Ti ODS and 14Cr-Al ODS alloy are illustrated in Figure 8 (a).

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 $Y_2Ti_2O_7$ 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: $E_t(111)=337$ GPa [30], $E_{y2}(222)=286$ GPa [32], $E_a=309$ GPa [33], $E_h(102)=242$ MPa [34], $E_m(22-1)=199$ MPa [31], $E_{f1}=193$ GPa and $E_{f2}=205$ GPa [35], (subscripts t for TiN, $y2$ for $Y_2Ti_2O_7$, a for AlN, h for YAH, and m for YAM, $f1$ for matrix of 14Cr-Al, $f2$ for the matrix of 14Cr-Ti).

Figure 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 $Y_2Ti_2O_7$ 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 can not 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 YAM and AlN in 14Cr-Al ODS alloy.

Figure 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

Figure 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.

Figure 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.

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 Figure 9 (b). The good linearity shown in this figure 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

Figure 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.

The fracture morphologies of two strained specimens are shown in Figure 10. Figure 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 Figure 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 Figure 10 (c) (d). It 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% ($7.53 \text{ g/cm}^3 / 7.77 \text{ g/cm}^3$) and 99% ($7.12 \text{ g/cm}^3 / 7.19 \text{ g/cm}^3$), respectively. Therefore, the cracks on the fracture surface may be induced by the stress

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 Equation 5.

$$YS = (\sigma_p^2 + (\sigma_m + \sigma_k + \sigma_{dbh})^2)^{\frac{1}{2}} \quad (5)$$

where, σ_p , σ_m , σ_k , σ_{dbh} are dislocation forest hardening, solid solution hardening, Hall-Petch strengthening, and the dispersed barrier 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 σ_m , 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 σ_k , the Al caused a larger size of grains, which decreased the strength of 14Cr-Al ODS. Third, the dispersed barrier hardening, σ_{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 $Y_2Ti_2O_7$ particles in 14Cr-Ti ODS alloy. Fourth, dislocation forest hardening, σ_p . 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 $Y_2Ti_2O_7$, while in the 14Cr-Al alloy, the hexagonal $YAlO_3$ (yttrium aluminum hexagonal, YAH), monoclinic $Al_2Y_4O_9$ (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 lower strength, but much larger elongation than 14Cr-Ti ODS. From the comparison of elastic stress, $Y_2Ti_2O_7$ is better strengthening phase than TiN in 14Cr-Ti, and YAH is much better than YAM 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.

5. Acknowledgement

This work was supported by National Magnetic Confinement Fusion Program of China under Grant No. 2015GB121006 and International S&T Cooperation Program of China (No.2013DFG62090). Argonne National Laboratory's work was supported under U.S. Department of Energy Contract No. DE-AC02-06CH11357.

References

- [1] T. Abram, S. Ion, Generation-IV nuclear power: A review of the state of the science, *Energy Policy* 36 (12) (2008) 4323–4330.
- [2] P. Yvon, F. Carré, Structural materials challenges for advanced reactor systems, *Journal of Nuclear Materials* 385 (2) (2009) 217–222.
- [3] S. J. Zinkle, G. Was, Materials challenges in nuclear energy, *Acta Materialia* 61 (3) (2013) 735–758.
- [4] G. Odette, M. Alinger, B. Wirth, Recent developments in irradiation-resistant steels, *Annu. Rev. Mater. Res.* 38 (2008) 471–503.
- [5] J. Brodrick, D. Hepburn, G. Ackland, Mechanism for radiation damage resistance in yttrium oxide dispersion strengthened steels, *Journal of Nuclear Materials* 445 (1) (2014) 291–297.
- [6] A. Certain, S. Kuchibhatla, V. Shutthanandan, D. Hoelzer, T. Allen, Radiation stability of nanoclusters in nano-structured oxide dispersion strengthened (ODS) steels, *Journal of Nuclear Materials* 434 (1) (2013) 311–321.
- [7] R. Klueh, P. Maziasz, I. Kim, L. Heatherly, D. Hoelzer, N. Hashimoto, E. Kenik, K. Miyahara, Tensile and creep properties of an oxide dispersion-strengthened ferritic steel, *Journal of Nuclear Materials* 307 (2002) 773–777.
- [8] S. Ohtsuka, S. Ukai, H. Sakasegawa, M. Fujiwara, T. Kaito, T. Narita, Nano-mesoscopic structural characterization of 9Cr-ODS martensitic steel for improving creep strength, *Journal of Nuclear Materials* 367 (2007) 160–165.
- [9] N. Cunningham, Y. Wu, D. Klingensmith, G. Odette, On the remarkable thermal stability of nanostructured ferritic alloys, *Materials Science and Engineering: A* 613 (2014) 296–305.
- [10] S. Li, Z. Zhou, P. Wang, H. Sun, M. Wang, G. Zhang, Long-term thermal-aging stability of a 16Cr-oxide dispersion strengthened ferritic steel at 973K, *Materials & Design* 90 (2016) 318–329.
- [11] H. S. Cho, H. Ohkubo, N. Iwata, A. Kimura, S. Ukai, M. Fujiwara, Improvement of compatibility of advanced ferritic steels with super critical pressurized water toward a higher thermally efficient water-cooled blanket system, *Fusion engineering and design* 81 (8) (2006) 1071–1076.
- [12] H. Cho, A. Kimura, Corrosion resistance of high-Cr oxide dispersion strengthened ferritic steels in super-critical pressurized water, *Journal of Nuclear Materials* 367 (2007) 1180–1184.
- [13] A. L. Rouffé, J. Crépin, M. Sennour, B. Tanguy, A. Pineau, D. Hamon, P. Wident, S. Vincent, V. Garat, B. Fournier, Effect of the thermal ageing on the tensile and impact properties of a 18% Cr ODS ferritic steel, *Journal of Nuclear Materials* 445 (1) (2014) 37–42.
- [14] J. Chao, C. Capdevila, M. Serrano, A. Garcia-Junceda, J. A. Jiménez, M. K. Miller, Effect of α - α' phase separation on notch impact behavior of oxide dispersion strengthened (ODS) Fe20Cr5Al alloy, *Materials & Design* 53 (2014) 1037–1046.
- [15] A. Kimura, R. Kasada, N. Iwata, H. Kishimoto, C. Zhang, J. Isselin, P. Dou, J. Lee, N. Muthukumar, T. Okuda, et al., Development of Al added high-Cr ODS steels for fuel cladding of next generation nuclear systems, *Journal of Nuclear Materials* 417 (1) (2011) 176–179.
- [16] S. Takaya, T. Furukawa, M. Inoue, T. Fujisawa, T. Okuda, F. Abe, S. Ohnuki, A. Kimura, Corrosion resistance of Al-alloying high Cr-ODS steels in stagnant lead-bismuth, *Journal of Nuclear Materials* 398 (1) (2010) 132–138.
- [17] T. Liu, L. Wang, C. Wang, H. Shen, Effect of Al content on the oxidation behavior of Y2Ti2O7-dispersed Fe-14Cr ferritic alloys, *Corrosion Science*.
- [18] C. Zhang, A. Kimura, R. Kasada, J. Jang, H. Kishimoto, Y. Yang, Characterization of the oxide particles in Al-added high-Cr ODS ferritic steels, *Journal of Nuclear Materials* 417 (1) (2011) 221–224.
- [19] T. Liu, L. Wang, C. Wang, H. Shen, H. Zhang, Feasibility of using Y2Ti2O7 nanoparticles to fabricate high strength oxide dispersion strengthened Fe–Cr–Al steels, *Materials & Design* 88 (2015) 862–870.
- [20] Y. Miao, K. Mo, Z. Zhou, X. Liu, K.-C. Lan, G. Zhang, M. K. Miller, K. A. Powers, Z.-G. Mei, J.-S. Park, et al., On the microstructure and strengthening mechanism in oxide dispersion-strengthened 316 steel: A coordinated electron microscopy, atom probe tomography and in situ synchrotron tensile investigation, *Materials Science and Engineering: A* 639 (2015) 585–596.
- [21] M. R. Daymond, The determination of a continuum mechanics equivalent elastic strain from the analysis of multiple diffraction peaks, *Journal of applied physics* 96 (8) (2004) 4263–4272.
- [22] F. Xu, R. Holt, M. Daymond, R. Rogge, E. Oliver, Development of internal strains in textured Zircaloy-2 during uni-axial deformation, *Materials Science and Engineering: A* 488 (1) (2008) 172–185.
- [23] V. Valvoda, M. Järvinen, On the harris texture index, *Powder Diffraction* 5 (04) (1990) 200–203.
- [24] G. Zhang, Z. Zhou, K. Mo, Y. Miao, X. Liu, J. Almer, J. F. Stubbins, The evolution of internal stress and dislocation during tensile deformation in a 9Cr ferritic/martensitic (F/M) ODS steel investigated by high-energy X-rays, *Journal of Nuclear Materials* 467 (2015) 50–57.
- [25] L. Wang, M. Li, J. Almer, Investigation of deformation and microstructural evolution in Grade 91 ferritic–martensitic steel by in situ high-energy X-rays, *Acta Materialia* 62 (2014) 239–249.
- [26] R. Renzetti, H. Sandim, R. Bolmaro, P. Suzuki, A. Möslang, X-ray evaluation of dislocation density in ODS-Eurofer steel, *Materials Science and Engineering: A* 534 (2012) 142–146.

- [27] T. Ungár, I. Dragomir, A. Revesz, A. Borbély, The contrast factors of dislocations in cubic crystals: the dislocation model of strain anisotropy in practice, *Journal of applied crystallography* 32 (5) (1999) 992–1002.
- [28] L. Hsiung, M. Fluss, S. Tumey, J. Kuntz, B. El-Dasher, M. Wall, B. Choi, A. Kimura, F. Willaime, Y. Serruys, HRTEM study of oxide nanoparticles in K3-ODS ferritic steel developed for radiation tolerance, *Journal of Nuclear Materials* 409 (2) (2011) 72–79.
- [29] G. Schaffer, M. Loreto, R. Smallman, J. Brooks, The nature of the dispersoids in INCONEL alloy MA6000, *Journal of materials science* 24 (9) (1989) 3261–3266.
- [30] G. Zhang, K. Mo, Y. Miao, X. Liu, J. Almer, Z. Zhou, J. F. Stubbins, Load partitioning between ferrite/martensite and dispersed nanoparticles of a 9Cr ferritic/martensitic (F/M) ODS steel at high temperatures, *Materials Science and Engineering: A* 637 (2015) 75–81.
- [31] Y. Miao, K. Mo, Z. Zhou, X. Liu, K.-C. Lan, G. Zhang, M. K. Miller, K. A. Powers, J. Almer, J. F. Stubbins, In situ synchrotron tensile investigations on the phase responses within an oxide dispersion-strengthened (ODS) 304 steel, *Materials Science and Engineering: A* 625 (2015) 146–152.
- [32] Y. Luan, Elastic properties of complex transition metal oxides studied by Resonant Ultrasound Spectroscopy, Ph.D. thesis, University of Tennessee, Knoxville, 2011.
- [33] R. Bruls, H. Hintzen, G. De With, R. Metselaar, The temperature dependence of the Young's modulus of MgSiN₂, AlN and Si₃N₄, *Journal of the European Ceramic Society* 21 (3) (2001) 263–268.
- [34] X. Zhan, Z. Li, B. Liu, J. Wang, Y. Zhou, Z. Hu, Theoretical prediction of elastic stiffness and minimum lattice thermal conductivity of Y₃Al₅O₁₂, YAlO₃ and Y₄Al₂O₉, *Journal of the American Ceramic Society* 95 (4) (2012) 1429–1434.
- [35] G. M. Stoica, A. D. Stoica, M. K. Miller, D. Ma, Temperature-dependent elastic anisotropy and mesoscale deformation in a nanostructured ferritic alloy, *Nature communications* 5.
- [36] K. Mo, Z. Zhou, Y. Miao, D. Yun, H.-M. Tung, G. Zhang, W. Chen, J. Almer, J. F. Stubbins, Synchrotron study on load partitioning between ferrite/martensite and nanoparticles of a 9Cr ODS steel, *Journal of Nuclear Materials* 455 (1) (2014) 376–381.
- [37] J. H. Kim, T. S. Byun, D. T. Hoelzer, C. H. Park, J. T. Yeom, J. K. Hong, Temperature dependence of strengthening mechanisms in the nanostructured ferritic alloy 14YWT: Part II Mechanistic models and predictions, *Materials Science and Engineering: A* 559 (2013) 111–118.

Internal stress evolution of different phases in the tensile deformation in 14Cr-Al ODS and 14Cr-Ti ODS alloy ($Y_2Ti_2O_7$ is better strengthening phase than TiN in 14Cr-Ti, and YAH is much better than YAM and AlN in 14Cr-Al ODS).