**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**

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Abstract: An application of high-energy wide angle synchrotron X-ray diffraction to investigate the tensile deformation of 9Cr ferritic/martensitic (F/M) ODS steel is presented. With tensile loading and in-situ X-ray exposure, the lattice strain development of matrix was determined. The lattice strain was found to decrease with increasing temperature, and the difference in Young's modulus of six different reflections at different temperatures reveals the temperature dependence of elastic anisotropy. The mean internal stress was calculated and compared with the applied stress, showing that the strengthening factor increased with increasing temperature, indicating that the oxide nanoparticles have a good strengthening impact at high temperature. The dislocation density and character were also measured during tensile deformation. The dislocation density decreased with increasing of temperature due to the greater mobility of dislocation at high temperature. The dislocation character was determined by best-fit methods for different dislocation average contrasts with various levels of uncertainty. The results shows edge type dislocations dominate the plastic strain at room temperature (RT) and 300 °C, while the screw type dislocations dominate at 600 °C. The dominance of edge character in 9Cr F/M ODS steels at RT and 300 °C is likely due to the pinning effect of nanoparticles for higher mobile edge dislocations when compared with screw dislocations, while the stronger screw type of dislocation structure at 600 °C maybe explained by the activated cross slip of screw segments.

Keywords: synchrotron XRD; mean internal stress; dislocation character; dislocation density

1. Introduction

Oxide dispersion strengthened (ODS) ferritic /martensitic steels are considered to be promising candidates for the breeding blanket material in a fusion reactor and fuel cladding tubes in Gen IV fission reactors [1, 2, 3]. The well-distributed nanoscale particles within the steels are critical to their excellent irradiation resistance, high-temperature strength, and superb creep properties [4, 5, 6]. These nanoscale particles are effective obstacles for dislocation movement which provide additional strengthening of the F/M matrix especially at high temperature, when compared with reduced activation Ferritic/Martensitic (RAFM) steels, such as Eurofer 97, F82H and ORNL 9Cr2W1V [7, 8].

Dislocations are important microstructure features, as their mobility greatly impacts the strength and plasticity of crystal materials. A knowledge of the dislocation density, dislocation character, and the interaction

between dislocations and dispersed particles during plastic deformation in a material is essential not only from a fundamental perspective, but also for engineering applications. The dislocation density can be measured by direct methods such as transmission electron microscope (TEM) and indirect methods such as X-ray diffraction (XRD). X-ray line profile analysis is a proven technique for studying the dislocation density in deformed metals [9, 10]. In addition, the modified Williamson-Hall (W-H) plot and the modified Warren-Averbach analysis have been applied to calculate dislocation density, twin-boundaries, and stacking-faults [11]. The W-H plot has also been applied to investigate the dislocation densities of 80% cold-rolled ODS-Eurofer steel annealed at different temperatures in ref. [12]. Some works have been done using the X-ray Bragg profile to determine the dislocation character (edge type and screw type) [13, 14, 15]. However the evolution of the dislocation character during the entirety of plastic deformation in ODS steel is still far from complete, especially the behaviour at high temperature.

In this study, an in-situ high-energy synchrotron radiation X-ray was used to monitor the microstructural evolution throughout the tensile deformation processes of a 9Cr F/M ODS steel from room temperature (RT) to 600 °C. The mean internal stress, dislocation density, and dislocation character were investigated. The dislocation density was also measured by using transmission electron microscope (TEM) at RT, 300 °C and 600 °C.

2. Experimental Procedure

9Cr F/M ODS steel with a nominal composition of Fe-9Cr-0.06C-1.5W-0.18Si-0.5Ti-0.35Y₂O₃ was fabricated by mechanical alloying (MA), hot isostatic pressing (HIP), and hot working (hot forging and hot rolling) [16, 17]. Then, the 9Cr F/M ODS steel was cut into tensile specimens by electrical discharge machining (EDM) with a gauge section of 1.49 mm × 0.76 mm × 7.60 mm. The in-situ synchrotron tensile tests were conducted at the 1-ID beamline at the Advanced Photon Source (APS) at Argonne National Laboratory (ANL), as illustrated in Figure 1. The tensile test with a strain rate of 10⁻⁴ s⁻¹ was held at each stress/strain state to allow X-ray diffraction scanning along the gauge part of the tensile specimen in order to catch its necking area using an MTS closed-loop servo-hydraulic test frame (model 858) [18, 19]. Experimental temperature conditions from RT up to 600 °C were examined. The diffraction analysis was performed by using a monochromatic 70 keV synchrotron beam with a 300 μm × 300 μm cross-section. The

sample to detector distance was set as 2082.5 mm. The ‘Hydra’ detector array containing four identical GE angio type 2D detectors with a pixel size of 0.2 was utilized to collect the wide angle diffraction information. The 15° region of the Debye-Scherrer rings near the tensile direction was integrated to obtain the lattice strain of the matrix, $\varepsilon_{11} = (d - d_0)/d_0$, peak broadening and peak intensity. The TEM samples were lifted out from the gauge part of the tensile specimen near the fracture surface by an FEI HELIOS 600i FIB. Diffraction contrast images were used to characterize the dislocation density after tensile deformation. All TEM images were taken on a JEOL 2010 LaB₆ TEM.

3. Results and discussion

3.1. Macro-mechanical properties

Figure 2 shows the engineering stress-engineering strain curves for the 9Cr F/M ODS steel tested at RT, 300 °C, 500 °C, and 600 °C. The material’s yield strengths (YS) are 845 MPa, 665 MPa, 343 MPa and 216 MPa, respectively, and ultimate tensile strengths (UTS) are 915 MPa, 760 MPa, 432 MPa and 268 MPa, respectively. The UTS dropped from 915 MPa to 268 MPa from RT to 600 °C, while the total elongation dropped from 0.16 at RT to 0.122 at 500 °C, and then increased to 0.245 at 600 °C, which agrees well with the results of the ODS EUROFER97 alloy reported in ref. [20]. Although the tensile tests were stopped periodically in order to capture the necking area, the stress–strain curves were smooth with the exception of the curve of 300 °C. The serrated points at 300 °C may be ascribed to dynamic strain aging (DSA) according results from Mariappan et al. stating DSA occurred at intermediate temperature around 650K in a reduced activation ferritic/martensitic (RAFM) steel [21]. The serrations were accompanied by an increase in work-hardening and a loss of ductility.

Figure 3 shows the true stress-lattice strain curves for six different $\langle hkl \rangle$ // tensile direction families of grains. The lattice strains of six reflections are hook-shaped at RT, while those of 600 °C are not visible. The hook-shaped region corresponds to the period that matrix phase starts to transfer load to the precipitate phases. This phenomenon indicates that the load partitioning between matrix and nanosclae particles became weaker with increasing temperature [22]. The lattice strains of six reflections increase with the increasing of stress, and decrease with increasing temperature. The lattice strain at RT is almost twice that at 600 °C for each lattice plane. Young’s modulus E is the slope calculated by assuming a linear fit between stress and strain in the elastic regime, as shown in Table 1. At two different temperatures, the (200) plane was the softest plane of all

six planes, while the (321) reflection remained at intermediate range at RT, and became the hardest plane at 600 °C, indicating that elastic anisotropy of lattice planes behave individually with temperature. This temperature dependence has been discussed in 14YWT by Stoica et al.[23]. The larger difference of E among six reflections at 600 °C than that at RT reveals the increasing anisotropy with increasing temperature. In previous works [24, 25, 26], the lattice strain of the specific reflection (211) was selected to represent the lattice strain of the Fe-Cr matrix, but the texture index [27] should be considered due to the temperature dependence of elastic anisotropy.

3.2. Mean internal stress vs. true stress

In order to investigate the strengthening effect of nanoscale particles, i.e. the strength increase in ODS steel compared with the strength of non-ODS steel. The mean internal stress of the matrix was calculated for comparison with the applied stress loaded by the tensile machine. The mean internal stress relies on the mean lattice strain of matrix, which was calculated from the lattice strains of (110), (200), (211), (310), (321) and (411) reflections using the weighted averaging algorithm developed by Daymond [28]. This method has been used to calculate the mean lattice strain for the matrix of Zircaloy-2 by averaging five Zircaloy-2 peaks [29, 30]. First, the texture index T as one of the necessary parameters for this method was calculated. The texture index T of the reflection $h_i = h_i k_i l_i$ is defined by the following equations:

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

, 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 sample with a random orientation of crystallites, which has the following expression:

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

where V is the volume of the unit cell; F is the structure factor; p is the multiplicity of the reflection; $\frac{1 + \cos^2 2\theta}{\sin^2 2\theta \cos \theta}$ 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. Thus, the mean internal stress $\bar{\sigma}$ was defined as equation 3.

$$\bar{\sigma} = \bar{E} \frac{\sum_{hkl} T_{hkl}(\Phi, \Psi) m_{hkl} E_{hkl} \varepsilon_{hkl}}{\sum_{hkl} T_{hkl}(\Phi, \Psi) m_{hkl} E_{hkl}} \quad (3)$$

where, $\bar{E}$ is the average Young's modulus of the bulk matrix, values which were taken from [31] for F82H (given in Table 2) due to its similarity to the bulk matrix of 9Cr ODS; and m is the multiplicity of the reflection. Thus, the mean internal stress and true stress are shown in Figure 4. It can be seen that the difference begins after yielding, and reaches a peak at the onset of necking. As is known, the exceptional mechanical properties of ODS steels are ascribed to the dispersed nanoscale particles and the fine grains according to the Orowan strengthening and Hall-Petch strengthening. The difference between applied stress and the mean internal stress was mainly induced by the Orowan strengthening. Then, the difference between the internal stress of matrix and the true stress at the necking point were used to analyze the strengthening effect of nanoscale particles. A given strengthening factor (SF) was defined as the percentage increase of strength of ODS steel comparing with matrix of ODS steel, which is shown in equation 4,

$$SF = \frac{\sigma_{Applied} - \sigma_{Matrix}}{\sigma_{Matrix}} \quad (4)$$

Where, the SF was calculated using the applied stress $\sigma_{Applied}$ and the mean internal stress σ_{Matrix} at necking point in Figure 5. This shows that the SF has a strong temperature dependence, which increases with increasing temperature from RT to 600 °C. However, the unexpected results of the SF at 300 °C being much higher than that at 500 °C, may be ascribed DSA. The solution atoms diffuse around dislocations, further strengthening the obstacles, which may provide an additional strengthening effect at 300 °C. Though the load partitioning became weaker with increasing temperature due to the higher mobility of dislocations, the increasing SF with increasing temperature indicates the increasing percentage of stress strengthened by nanoscale particles. Therefore, the nanoscale particles plays a more important role for strengthening at high temperatures comparing with other strengthening mechanisms.

3.3. Dislocation evolution

The full width at half-maximum (FWHM) of Fe-Cr matrix peaks were measured during the process of tensile deformation at RT, 300 °C, 500 °C, and 600 °C. The CeO₂ sample was used as the reference material for obtaining instrumental broadening profile. The FWHM of Fe-Cr matrix was calibrated corresponding to instrumental broadening profile using the relation as follows:

$$FWHM = [(FWHM_{measured})^2 - (FWHM_{instrumental})^2]^{\frac{1}{2}} \quad (5)$$

Figure 6 shows the FWHM evolutions of eight bcc Fe-Cr peaks of (200), (211), (220), (310), (222), (321),

(400) and (330) during tensile straining in the 9Cr F/M ODS steel at RT and 600 °C respectively. The FWHM of all Fe-Cr peaks increased with the increasing strain during the macroscopic deformation. As mentioned in [32], the FWHM Fe(400) > the FWHM Fe(330) and FWHM Fe(310) > the FWHM Fe(222) indicates anisotropic strain broadening. The relationship between the FWHM of the eight peaks also changes with temperature, since FWHM Fe(222) > the FWHM Fe(211) at RT while FWHM Fe(222) < FWHM Fe(211) at 600 °C. This indicates a temperature dependence of the anisotropic strain broadening. The modified W-H plot was applied to calculate the dislocation density by the following equation:

$$\Delta K = \frac{0.9}{D} + \left(\frac{\pi M^2 b^2}{2} \right)^{\frac{1}{2}} \rho^{\frac{1}{2}} (KC^{\frac{1}{2}}) \quad (6)$$

Where $K = 2\sin\theta/\lambda$; $\Delta K = 2\cos\theta\Delta\theta/\lambda$; θ is the diffraction angle, and $\Delta\theta$ is half of the FWHM of the diffraction peak; D is the average grain size; M is an adjustable parameter, which should be fitted together with the rest of the parameters of the model. In this experimental, $M = 2$ was used for all samples to give the level of accuracy expected by the W - H approach [12]. 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 [33]. The dislocation contrast factors C for pure edge and pure screw dislocations at different temperatures were calculated which have concerned anisotropic elastic constants caused by temperature.

Shintani et al. obtained q from the coefficients of H^2 in the linear function and compared the measured and theoretical q values of pure edge type and pure screw type dislocations to determine the fraction of each [13]. Figure 7 shows the W-H plots at two plastic strain levels (0, 3.4%) at RT by using the dislocation contrast factor of pure edge type, dislocation contrast of pure screw type and best-fit method, respectively.

In each plot, a least-square fitting line is drawn with a R^2 coefficient. It can be seen that the R^2 coefficient changed with increasing strain. At a plastic strain of 0%, the dislocation contrast factor of screw type dislocations exhibited a better linear fit ($R^2 = 0.944$) than that of edge type dislocations, while the dislocation contrast factor of edge type dislocations exhibited a better linear fit ($R^2 = 0.963$) at a plastic strain of 4.3%. Li et al. [34] investigated the dislocation character by selecting a best fit from the modified W-H plot using five different corresponding dislocation contrast factors according to different ratios of pure edge and pure screw dislocations. Schafler et al. [14] have proven that the specific fraction of each dislocation type can be determined by comparing experimental and theoretical C -values. In order to obtain in-situ results of dislocation

character, this method was refined such that the dislocation average contrast factor was divided into 100 steps from 100% edge type with 0% screw type to 0% edge type with 100% screw type, one couple of examples were shown in Figure 7(c). Then the best-fit result was selected to calculate the dislocation density and determine the dislocation character during process of tensile deformation at RT, 300 °C, 500 °C and 600 °C.

Figure 8 shows the evolution of dislocation densities with plastic deformation at RT, 300 °C, 500 °C, and 600 °C. Although the results of dislocation density obtained by the evaluation method are only semi-quantitative, the results still can be used to investigate the influence of deformation and temperature on the dislocation density and characters. It can be seen that the dislocation density increased quickly to a maximum in the very beginning region and then remains stable with increasing plastic strain at these four temperatures. The maximum dislocation density at the four tested temperatures is about $(6.05 \pm 0.37) \times 10^{14} \text{ m}^{-2}$ (RT), $(5.13 \pm 0.45) \times 10^{14} \text{ m}^{-2}$ (300 °C), $(2.91 \pm 0.27) \times 10^{14} \text{ m}^{-2}$ (600 °C), respectively. The increase speed of dislocation density decreased with increasing temperature due to the higher mobility of dislocations at high temperature. The dislocation density also decreased with increasing temperature with an exception at 300 °C. As mentioned in the description of macro-mechanical properties of 9Cr F/M ODS steel, DSA occurs at 300 °C. The solution atoms diffuse around dislocations further strengthening the obstacles. Thus, the dislocation density at 300 °C is slightly higher than that at RT.

Figure 9 shows the density evolution of edge type and screw type dislocations during plastic deformation at RT, 300 °C and 600 °C. In general, the dislocation structure changes with increasing plastic strain and increasing temperature. At RT and 300 °C, the dislocation density of edge type dislocations is much higher than that of screw type dislocations, while the screw dislocation density is much higher than the edge dislocation density at 600 °C. The edge type dislocations dominate the plastic deformation at RT and 300 °C, while the proportion of edge type dislocations decreased with increasing temperature. As temperature increased up to 600 °C, the screw type dislocations began to dominate the plastic deformation. The temperature dependence of dislocation character during plastic deformation is an interesting issue, and may play an important role in the strengthening mechanisms in ODS steel. The edge type dislocations are assumed to play a minor role in the hardening of bcc metals due to their higher mobility compared with screw dislocations [35], and because the Orowan stress for edge dislocations is known to be much smaller than that for screw dislocations in MA956 [36]. Therefore, the edge dislocations bypasses nanoparticles more easily than screw dislocation at low

temperature (RT and 300 °C), which results in the stronger edge character [33]. At elevated temperature (600 °C), although the Orowan stress of screw dislocations is still larger than that of edge dislocations, the slip of screw dislocation can occur on {110}, {112}, and {123} planes including cross slip between these planes, which encourages the dominance of screw dislocations at 600 °C.

3.4. Dislocation densities of post-tensile specimens measured by TEM

TEM micrographs of post-tensile specimens near the fracture region at RT, 300 °C, and 600 °C are shown in Figure 10. The bright field TEM images were taken at around 3g conditions of either {110} or {200} reflections to provide the dislocation density information. Three images were used to measure the dislocation density for each condition. And the uncertainties were estimated by using the error of linear fitting, which is based on linear regression techniques. The high dislocation density and multiple sub-grains can be seen in the grains of specimen tested at RT. As the temperature increased to 600 °C, the sub-grains disappeared and the dislocation density decreased from $(6.6 \pm 2.4) \times 10^{14} \text{ m}^{-2}$ at RT to $(5.4 \pm 1.7) \times 10^{14} \text{ m}^{-2}$ at 300 °C to $(3.5 \pm 1.3) \times 10^{14} \text{ m}^{-2}$ at 600 °C, matching with the maximum dislocation density of $(6.05 \pm 0.37) \times 10^{14} \text{ m}^{-2}$ at RT, $(5.13 \pm 0.45) \times 10^{14} \text{ m}^{-2}$ at 300 °C, and $(2.91 \pm 0.27) \times 10^{14} \text{ m}^{-2}$ at 600 °C respectively as calculated from the X-ray profile line.

4. Conclusions

A high-energy wide angle synchrotron X-ray diffraction technique was applied to study the tensile deformation of a 9Cr F/M ODS steel. The lattice strain of six planes decreased with the increasing tensile test temperature, and the elastic anisotropy revealed the important temperature dependence. The mean internal stress was calculated and compared with the applied stress. The strengthening factor increased with the increasing temperature, indicating that oxide nanoscale particles provide an enhanced strengthening role at high temperatures. The dislocation density and character were also measured by the modified W-H plot during tensile deformation. The dislocation density decreased with increasing temperature due to the greater mobility of dislocations at high temperature. The TEM observation of post-tensile specimens confirmed the dislocation density change. The dislocation character was determined by a best-fit method for different dislocation average

contrasts with various levels of uncertainty. The results show that edge type dislocations dominate the plastic strain at RT and 300 °C, while the screw type dislocations dominate at 600 °C. The stronger edge character in 9Cr F/M ODS steel at RT and 300 °C is likely due to the pinning effect of nanoparticles for higher mobile edge dislocation when compared with screw dislocation, while the stronger screw type of dislocation structure at 600 °C maybe explained by activated cross slip of screw segments.

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Figure captions

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

Figure 2: Engineering stress vs. engineering strain from RT to 600 °C.

Figure 3: The true stress vs. lattice strain for six different $\langle hkl \rangle$ // tensile direction families of grains for 9Cr F/M ODS steel at (a) RT; and (b) 600 °C.

Figure 4: The true stress and mean internal stress of 9Cr F/M ODS steel during tensile test at RT, 300 °C, 500 °C, 600 °C.

Figure 5: The strengthening factor of 9Cr F/M ODS steel

Figure 6: Evolution of the FWHM of the Fe-Cr matrix as a function of strain during tensile deformation for 9Cr F/M ODS steel at (a) RT; and (b) 600 °C.

Figure 7: The modified Williamson-Hall plots for Fe-Cr peaks at different strains at RT using the dislocation contrast factor of (a) pure edge type dislocations; (b) pure screw type dislocations; and (c) best-fit method.

Figure 8: The evolution of dislocation density as a function of plastic strain for 9Cr F/M ODS steel at RT, 300 °C, and 600 °C.

Figure 9: The density evolution of edge type and screw type dislocations with increasing plastic strain of 9Cr F/M ODS steel at (a) RT; (b) 300 °C; and (c) 600 °C.

Figure 10: The bright field of TEM images of post-tensile testing specimens of 9Cr F/M ODS steel at (a) RT;

(b) 300 °C; and (c) 600 °C.

Table captions

Table 1: Young's modulus of the Fe-Cr matrix at six different reflections for 9Cr F/M ODS steel

Table 2: The Young's modulus of F82H from RT to 600 °C

Table 1 Young’s modulus of the Fe-Cr matrix at six different reflections for 9Cr F/M ODS steel.

Young's modulus E (GPa)	(110)	(200)	(211)	(310)	(321)	(411)
RT	213±2	175±2	204±3	191±1	211±3	212±1
300 °C	201±5	158±4	196±5	176±4	191±5	193±5
500 °C	196±5	116±6	192±7	147±6	205±7	186±5
600 °C	179±4	103±6	194±2	137±4	213±4	183±3

Table 2 The Young’s modulus of F82H from RT to 600 °C [30].

Young's modulus (GPa)	RT	300 °C	500 °C	600 °C
E	216	201	186	173

Figure

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Figure 2: Engineering stress vs. engineering strain from RT to 600 °C.

Figure (a)

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Figure (b)

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Figure (a)

Figure 6: Evolution of the FWHM of the Fe-Cr matrix as a function of strain during tensile deformation for 9Cr F/M ODS steel at (a) RT; and (b) 600 °C.

Figure (b)

Figure 6: Evolution of the FWHM of the Fe-Cr matrix as a function of strain during tensile deformation for 9Cr F/M ODS steel at (a) RT; and (b) 600 °C.

Figure (a)

Figure 7: The modified Williamson-Hall plots for Fe-Cr peaks at different strains at RT using the dislocation contrast factor of (a) pure edge type dislocations; (b) pure screw type dislocations; and (c) best-fit method

Figure (b)

Figure 7: The modified Williamson-Hall plots for Fe-Cr peaks at different strains at RT using the dislocation contrast factor of (a) pure edge type dislocations; (b) pure screw type dislocations; and (c) best-fit method

Figure (c)

Figure 7: The modified Williamson-Hall plots for Fe-Cr peaks at different strains at RT using the dislocation contrast factor of (a) pure edge type dislocations; (b) pure screw type dislocations; and (c) best-fit method

Figure 8: The evolution of dislocation density as a function of plastic strain for 9Cr F/M ODS steel at RT, 300 °C, and 600 °C.

Figure (a)

Figure 9: The density evolution of edge type and screw type dislocations with increasing plastic strain of 9Cr F/M ODS steel at (a) RT; (b) 300 °C; and (c) 600 °C.

Figure (b)

Figure 9: The density evolution of edge type and screw type dislocations with increasing plastic strain of 9Cr F/M ODS steel at (a) RT; (b) 300 °C; and (c) 600 °C.

Figure (c)

Figure 9: The density evolution of edge type and screw type dislocations with increasing plastic strain of 9Cr F/M ODS steel at (a) RT; (b) 300 °C; and (c) 600 °C.

Figure (a)

Figure 10: The bright field of TEM images of post-tensile testing specimens of 9Cr F/M ODS steel at (a) RT; (b) 300 °C; and (c) 600 °C.

Figure (b)

Figure 10: The bright field of TEM images of post-tensile testing specimens of 9Cr F/M ODS steel at (a) RT; (b) 300 °C; and (c) 600 °C

Figure (c)

Figure 10: The bright field of TEM images of post-tensile testing specimens of 9Cr F/M ODS steel at (a) RT; (b) 300 °C; and (c) 600 °C.