Title: Synchrotron study on load partitioning between ferrite/martensite and nanoparticles of a 9Cr ODS steel

Authors: Kun Mo^{1,2*}, Zhangjian Zhou³, Yinbin Miao¹, Di Yun², Hsiao-Ming Tung⁴, Guangming Zhang³, Weiying Chen¹, Jonathon Almer², and James F. Stubbins¹

¹ Department of Nuclear, Plasma, and Radiological Engineering, University of Illinois at Urbana-Champaign, Urbana, IL 61801, USA

² Argonne National Laboratory, Argonne, IL 60439, USA

³ School of Materials Science and Engineering, University of Science and Technology Beijing, Beijing 10083, China

⁴ Institute of Nuclear Energy Research, Atomic Energy Council, Taoyuan 325, Taiwan

***Corresponding Author:** Kun Mo, Tel: +1-630-252-3455, Email: kunmo@anl.gov

Keywords: ODS, synchrotron radiation, nanoparticles, load partitioning

Abstract

Oxide dispersion strengthened (ODS) steels exhibit exceptional radiation resistance and high-temperature creep strength when compared to traditional ferritic and ferritic/martensitic (F/M) steels. Their excellent mechanical properties result from very fine nanoparticles dispersed within the matrix. In this work, we applied a high-energy synchrotron radiation X-ray to study the deformation process of a 9Cr ODS steel. The load partitioning between the ferrite/martensite and the nanoparticles was observed during sample yielding. During plastic deformation, the nanoparticles experienced a dramatic loading process, and the internal stress on the nanoparticles increased to a maximum value of 3.5 GPa, which was much higher than the maximum applied stress (~986 MPa). After necking, the loading capacity of the nanoparticles was significantly decreased due to a debonding of the particles from the matrix, as indicated by a decline in lattice strain/internal stress. Due to the load partitioning, the ferrite/martensite slightly relaxed during early yielding, and slowly strained until failure. This study develops a better understanding of loading behavior for various phases in the ODS F/M steel.

1. Introduction

With growing global interest in nuclear energy, the need for development of new metallic materials able to meet stringent requirements for use in advanced nuclear systems has become a more urgent goal. [1] Oxide dispersion strengthened (ODS) steel is an emerging class of nanostructured alloys that contain nanoparticles with very high number density. These nanoparticles are effective barriers for dislocation movement, and thus provide additional strengthening and creep resistance to materials. [2] The ODS steels also display exceptional irradiation resistance due to the “sink” effect of the nanoparticles. [2, 3] Thus, ODS steels are considered to be some of the most promising metallic materials for advanced nuclear systems, such as fusion systems or Gen IV nuclear plants, featuring high operating temperatures and high neutron fluence. The working temperature of ODS steels is expected to exceed 600°C, compared to 350-550°C for current ferritic/martensitic (F/M) steels. [3-5]

In order to develop suitable ODS steels and to optimize their chemical compositions, it is necessary to investigate the essential characteristics of the ODS steels (carbide particles, precipitates, nano-clusters, crystallites, nano-features, etc.). [6] Current analysis methods to characterize the microstructure of ODS alloys include small angle neutron scattering (SANS), transmission electron microscopy (TEM), and atom-probe-tomography (APT). [2, 6] However, none of these techniques can reveal the dynamic behavior of nanoparticles during deformation.

In this study, a high-energy X-ray diffraction technique was employed to investigate the nature of an ODS steel under applied stresses. This experimental technique was originally developed for neutron diffraction due to neutron's deep penetration in metallic materials. [7-10] Recently developed some of the largest third-generation synchrotron light sources, e.g. European Synchrotron Radiation Facility (FR), Advanced Photon Source (USA), and SPring-8 (JP) can provide high-energy X-ray diffraction with high flux, which can probe bulk metallic materials to

study their dynamic deformation behavior. [11-13] Based on this technique, the evolution of lattice strains for various phases (oxide and nitride particles, and host matrix) during *in-situ* tensile loading was directly and continuously measured. The dynamic behavior of the nanoscale particles and the corresponding response of the F/M matrix were clarified.

2. Experimental procedures

2.1. Material development

The base materials used for manufacturing the ODS steel include nitrogen-gas-atomized pre-alloyed steel powders and Ti and Y_2O_3 powders. The composition of mixed powders was designed to be Fe-9Cr-0.1C-1.5W-0.2V-0.5Ti-0.35 Y_2O_3 , in weight percent. The base materials were mechanically alloyed in an argon atmosphere using a planetary ball mill at 300 rpm with a ball-to-powder mass ratio of 5:1 for 30 hours. Milled powders were then degassed, sealed, and consolidated by hot isostatic pressing (HIP) under a pressure of 100 MPa at 1150°C for 3 hours. The consolidated sample was then forged at 1150°C with a forging ratio of 3:1. Additional hot rolling was performed three times at 1150°C with a reduction ratio of about 20% each time. In order to develop the F/M phase, the sample was heat-treated at 1050°C for 1 hour with water quenching and then heat-treated at 750°C for 2 hours with air-cooling.

2.2. Diffraction Measurements

The *in-situ* tensile tests measured with high-energy X-ray diffraction were carried out at the 1-ID beamline at the Advanced Photon Source at Argonne National Laboratory. The uniaxial tensile test was conducted using an MTS closed-loop servo-hydraulic test frame (model 858) with a maximum force of ± 15 kN at room temperature. [14, 15] The specimen, with a gauge section of $1.49 \text{ mm} \times 0.76 \text{ mm} \times 7.60 \text{ mm}$ (width $\times$ thickness $\times$ length), was loaded on the grips without applying external stress from the MTS test frame. After that, 0.1 N (or stress of ~ 88 MPa) load

was applied to fix the tensile specimen in position. Then the specimen was subjected to increasing uniaxial tensile stresses up to failure with *in-situ* X-ray exposure. The tensile test 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. Diffraction measurements were performed every 10 seconds with a monochromatic 70 keV ($\lambda = 0.0177$ nm) X-ray beam with a beam size of $300 \times 300 \mu\text{m}^2$. The experiment utilized the “Hydra” detector array, which consists of four area detectors (GE angio type), for X-ray diffraction measurements. The distance between the sample and the detector was ~ 2.08 m.

2.3. Diffraction analysis

Figure 1 shows a typical 2D diffraction pattern measured using the “Hydra” detector array (the panel tilting angle was 37°). Debye-Scherrer rings with various radii were reflected from the lattice planes, which had various interplanar spacings (or d -spacings) d_{hkl} . Under external applied loads, the diffraction rings were not perfectly circular, but were ellipsoidal due to the internal stress on the specimen. For the tensile tests, the vertical loading along the axial direction of the specimen, i.e. the Y-axis in Fig. 1, according the Poisson effect, caused internal lattice straining along the Y-axis and contraction along the X-axis. Due to the lattice strain development, the radii of the Debye rings shortened in the axial direction (Azimuth (η) = 90° and 270°) and lengthened in the transverse direction (Azimuth (η) = 0° and 180°). The d -spacing, d_{hkl} , can be converted to lattice strain, ε_{hkl} , by measuring the changes in the d -spacing with respect to the reference d -spacing, d_{hkl}^0 , using the following equation ([11, 16, 17]):

$$\varepsilon_{hkl} = \frac{d_{hkl} - d_{hkl}^0}{d_{hkl}^0} \quad (1)$$

The reference lattice spacing d_{hkl}^0 is determined by first interpolating the measured $d_{hkl}(\eta=0^\circ)$

and $d_{hkl}(\eta=90^\circ)$ during the elastic deformation, and then finding d_{hkl}^0 at their intersection.

The internal stress, σ , of each of the phases can be calculated following the von Mises criterion (assuming isotropic polycrystalline) [12, 17-19]:

$$\sigma = \sqrt{\frac{(\sigma_{11} - \sigma_{22})^2 + (\sigma_{22} - \sigma_{33})^2 + (\sigma_{33} - \sigma_{11})^2}{2}} \quad (2)$$

In the above equation, σ_{11} is the axial principal stress, and σ_{22} and σ_{33} are the transverse principal stresses. σ_{22} and σ_{33} are equal to each other for the flat-plate specimen in this study. The values of σ_{11} , σ_{22} , and σ_{33} are calculated by [11, 17]:

$$\sigma_{11} = \frac{E}{1+\nu} \epsilon_{11} + \frac{\nu E}{(1+\nu)(1-2\nu)} (\epsilon_{11} + \epsilon_{22} + \epsilon_{33})$$

$$\sigma_{22} = \sigma_{33} = \frac{E}{1+\nu} \epsilon_{22} + \frac{\nu E}{(1+\nu)(1-2\nu)} (\epsilon_{11} + \epsilon_{22} + \epsilon_{33})$$

In the above equations, E is the elastic constant and ν is Poisson's ratio. Both E and ν for each phase can be measured from the lattice strain development in the elastic regime.

3. Results and discussion

Figure 2 shows the stress-strain diagram of the 9Cr ODS steel. The engineering stress, σ_e , was calculated by $\sigma_e = F/A$, where F is the load on the sample and A is the area of the cross-section. F was measured by the MTS loading frame, while A was measured before the tensile test. The applied stress on the specimen was approximated by the true stress, σ_t , which was calculated by engineering stress (σ_e) and strain (ϵ) by: $\sigma_t = \sigma_e(1+\epsilon)$. Young's modulus for the 9Cr ODS sample was measured to be ~203 GPa using ultrasonic techniques. The 0.2% yield strength was

measured to be ~845 MPa. The ultimate tensile strength (UTS) was measured to be ~ 915 MPa, and developed at the strain of ~8.6%. Sample necking began after the sample reached the UTS, and continued until sample failure. The total elongation of the 9Cr ODS sample was ~16.2%.

Three major phases were observed from synchrotron X-ray diffraction: the F/M matrix, and titanium nitride (TiN) and $\text{Y}_2\text{Ti}_2\text{O}_7$ particles. From electronic microscopic study, it was found that the size of the TiN particles ranged from tens of nanometers to 1 micrometer (Fig. 3), while the size of the $\text{Y}_2\text{Ti}_2\text{O}_7$ particles was found to be much smaller, and in the range of several to tens of nanometers (Fig. 4). Despite a very small fraction (~0.35 weight percentage) of these $\text{Y}_2\text{Ti}_2\text{O}_7$ nanoparticles, their major reflections can be recognized with high-energy X-ray diffraction. The most significant reflections of $\text{Y}_2\text{Ti}_2\text{O}_7$ are (222) and (440) reflections with d -spacings of ~2.91 and 1.78Å, respectively. The corresponding value of the lattice constant is ~10.09 Å, which is in good agreement with the literature value [20].

In order to compare the internal stress developments of all phases, F/M matrix (211) reflection, $\text{Y}_2\text{Ti}_2\text{O}_7$ (222) reflection, and TiN (111) reflection were selected to represent their bulk response during deformation. One should note that a single reflection for each phase only represents its distinct families of grain orientations. Here, the lattice strains interpreted from a single reflection that represents a specific grain set are assumed to be representative of the bulk properties. Figure 5 shows the lattice strain development for F/M matrix before necking. Five reflections including (110), (200), (321), (211), and (310) were measured. All reflections show similar loading behavior during tensile test. Figure 6 shows the modeled orientation dependent elastic constants for body-centered cubic (BCC) Fe using Reuss's, Voigt's and Kröner's models. [21] Although the orientation dependent Young's modulus/elastic constant for the F/M matrix is slightly different from the pure BCC Fe, these two metals' tendency of elastic constants in the function

of orientation parameter (defined as $3A$, $A=(h^2k^2+h^2l^2+l^2k^2)/(h^2+k^2+l^2)^2$, [22]) should be similar due to their identical base material and similar crystal structure. (Detailed descriptions for the models of the orientation dependent elastic constants can be found in ref. [22-26]) The elastic constants of (110), (321), and (211) reflections are found to be close to the bulk elastic constant. Among these reflections, (211) reflection was selected to represent the bulk material because the interpreted lattice strains, and the calculated internal von Mises stress of this reflection approach the mean values of all reflections. For the dispersed particles including TiN and $Y_2Ti_2O_7$, the most intense diffraction peak of each phase ((222) reflection of $Y_2Ti_2O_7$, and (111) reflection of TiN) was selected to represent their overall properties. The diversity of lattice strain development for different crystal orientations is minor due to the negligible intergranular interaction between the dispersed particles.

The diffraction peaks for various phases were obtained by integrating the diffraction rings (Fig. 1) along azimuth (from 0 to 360°), as depicted in Fig. 7. Figure 8 shows the lattice strain development before necking for the F/M matrix (211) reflection, $Y_2Ti_2O_7$ (222) reflection, and TiN (111) reflection. In the elastic regime, the slopes of the lattice strain development to the applied stress for the F/M matrix and $Y_2Ti_2O_7$ and TiN particles were measured to be 200 (± 8), 200 (± 18), and 228 (± 20) GPa in the axial/loading direction (along ϵ_{11} development), and -676 (± 20), -480 (± 64), and -841 (± 82) GPa in the transverse direction (along ϵ_{22} development). The slopes of the ϵ_{11} development are close to the elastic constants when tested independently. The bulk elastic constants are ~ 203 [27], ~ 253 [28], and ~ 251 [29] GPa for the F/M matrix and $Y_2Ti_2O_7$ and TiN particles, respectively. The difference in values measured by X-ray diffraction and by independent tests is partly due to the anisotropic crystallographic stiffness of each of the phases. More importantly, the difference in values is caused by their interactions and load

partitioning. [7-9] The corresponding Poisson's ratios for the F/M matrix, $\text{Y}_2\text{Ti}_2\text{O}_7$, and TiN were measured to be 0.29, 0.41, and 0.27, respectively.

For the entire process of plastic deformation, the lattice strain of the F/M matrix slightly decreased during early yielding, but increased afterwards (Fig. 6&8). This indicates that the F/M matrix was moderately relaxed during early yielding, and strained as the applied stress was increased. The relaxation with the minimum internal von Mises stress of 696 MPa (when the applied stress is 912 MPa) is due to load transfer to the particles. In contrast, the lattice strains for both $\text{Y}_2\text{Ti}_2\text{O}_7$ and TiN increased uninterruptedly from yielding to sample necking. The lattice strain for $\text{Y}_2\text{Ti}_2\text{O}_7$ increased at a nearly linear rate (i.e. the slope of curve in Fig. 8: $(d\sigma_t/d\varepsilon_{11}) \approx \text{constant}$; σ_t is the applied stress, and ε_{11} is lattice strain) in the plastic regime up to the start of necking. The slope of curve for TiN increased as the applied stress increased, indicating a weak capacity for taking load as a function of applied stress/macrosopic strain. Following different trends of lattice strain development, the lattice strain for all three phases extends in various ranges during plastic deformation. During early yielding, the lattice strains for the F/M matrix (211) reflection, $\text{Y}_2\text{Ti}_2\text{O}_7$ (222) reflection, and TiN (111) reflection were 3.5×10^{-3} , 5.6×10^{-3} , and 4.6×10^{-3} , corresponding to the internal von Mises stresses (Eq. 2) of 696, 1121, and 1049 MPa, respectively. The applied stress measured by the loading frame was 843 MPa, which is about 150 MPa higher than the internal stress of the F/M matrix, and about 300 and 200 MPa lower than the internal stresses of the $\text{Y}_2\text{Ti}_2\text{O}_7$ and TiN particles, respectively. This is a typical load partitioning that often starts during early yielding. [11, 18, 19] The load partitioning became more significant with increasing applied stress, and achieved a maximum value before necking, when the internal von Mises stress for the F/M matrix and $\text{Y}_2\text{Ti}_2\text{O}_7$ and TiN particles was measured to be 796, 3504, and 1747 MPa (lattice strains of 4.0×10^{-3} , 17.5×10^{-3} , and 7.6×10^{-3}), respectively. When necking occurred, the applied stress was measured to be ~986 MPa, which is

about 190 MPa greater than the internal stress of the F/M matrix, ~800 MPa lower than the internal stress of the TiN particles, and less than 1/3 of the internal stress of the $\text{Y}_2\text{Ti}_2\text{O}_7$ particles.

One should note that the load partitioning between the Fe matrix and second-phases occurred at a lower stress (around the yield point). (Fig. 8) In the elastic deformation regime, only the lattice spacing alters to compensate the external applied stress. Therefore, the lattice strain is linear to the stress. As this stress exceeds critical resolve shear stress (CRSS), dislocations are activated so that plasticity starts to account for a part of deformation, resulting in the retardation of the increase of lattice strain. In ODS steels, dense dispersive nanoscale oxygen-enriched particles help suppress the gliding of dislocations, majorly through the Orowan looping mechanism [30, 31]. In the beginning of plastic deformation, the CRSS of Orowan looping is relatively low. Thus, the nanoparticles only take a marginal fraction of the stress. However, accumulation of Orowan loop formation around the nanoparticles enlarges the equivalent size of these nanoparticles while the nanoparticle density remains the same. This significantly raises the CRSS of the nanoparticles and finally eliminates the probability of dislocation bypassing them. This source shortening phenomenon [32] forces successive dislocation to form pile-up structures behind the nanoparticles that they are incapable of bypassing, transferring huge amounts of stress towards these nanoparticles. Consequently, the lattice strain of matrix decreases in the later stage of plastic deformation. (Fig. 8)

The $\text{Y}_2\text{Ti}_2\text{O}_7$ nanoparticles exhibit a superior capacity for taking load. The final achieved internal stress of ~3.5 GPa is much higher than many strengthening phases in metallic materials, including carbides [17, 19] and cementite [11] in iron-based steels, and carbides in Ni-based alloys [33]. Compared to these particles, the loading capacity of $\text{Y}_2\text{Ti}_2\text{O}_7$ nanoparticles is considerably higher, despite a much lower volume fraction of nanoparticles within the ODS steel.

Similar to $\text{Y}_2\text{Ti}_2\text{O}_7$ particles, TiN particles withstand a relatively high internal stress during plastic deformation, although the values of the internal stress are much smaller than those of the $\text{Y}_2\text{Ti}_2\text{O}_7$ particles. Because the TiN particles are so much larger in size, and sparse in space (Fig. 3), the particles are not as effective as oxide dispersions in impeding dislocation movements [23, 30, 34].

During sample necking, the load partitioning among the matrix and particles reverses from the manner before necking. Figure 9 shows lattice strain vs. macroscopic strain for all phases in the 9Cr ODS steel. This analysis is based on the diffraction measurements on the fixed points on the necking part of the specimen. The lattice strain of the F/M matrix slightly increased during necking until close to failure. On the contrary, the lattice strain of both the $\text{Y}_2\text{Ti}_2\text{O}_7$ and TiN particles decreased to different extents with increasing applied strain during necking. Evidently, the nanoparticles experience a much more significant decrease in internal stress than the TiN particles. Either fracture or debonding can cause the particles to lose their capability for taking load, which leads to the drop in internal stress. Figure 10 shows the fracture surface of the 9Cr ODS sample, where nanoscale $\text{Y}_2\text{Ti}_2\text{O}_7$ particles were found in almost all of the dimples. The integrity of $\text{Y}_2\text{Ti}_2\text{O}_7$ particles indicates that the particles are not fractured, but are debonded from the matrix. Due to debonding, the nanoparticles lost the capability of taking load, and consequently maintained a relatively low level of lattice strain/internal stress until sample failure. Therefore, although $\text{Y}_2\text{Ti}_2\text{O}_7$ particles provide significant strengthening for the 9Cr ODS steel, their impact is limited in elastic and plastic deformations to before necking, with a maximum internal stress of ~ 3.5 GPa. When necking occurs, further macroscopic straining cannot put more load on the nanoscale particles, and can only cause a decrease in internal stress.

4. Conclusions

An application of high-energy synchrotron X-ray diffraction for 9Cr ODS steel was presented. With tensile loading and *in-situ* X-ray exposure, time-resolved lattice strain development was clarified. The load partitioning between the F/M matrix and the nanoparticles was found to occur during sample yielding. The nanoparticles experienced a dramatic loading process, and the internal von Mises stress on the nanoparticles increased to 3.5 GPa (the maximum applied stress is ~986 MPa) before sample necking. In contrast, the F/M matrix relaxed slightly during early yielding, and slowly strained until necking. However, the load partitioning process reversed during sample necking: the internal stress placed on the nanoparticles decreased rapidly, indicating a debonding of the particles from the matrix. This debonding led to a decrease in the capacity of nanoparticles to carry load.

5. Acknowledgement

This work was supported by the U.S. Department of Energy under grants 973 DOE INL 120293 and NEUP 09-516. Argonne National Laboratory's work was supported under U.S. Department of Energy contract DE-AC02-06CH11357. The authors would like to thank Dr. Meimei Li from the Argonne National Laboratory for technical assistance. The microstructural analysis was carried out in part in the Frederick Seitz Materials Research Laboratory Central Facilities, University of Illinois, which are partially supported by the U.S. Department of Energy under grants: DE-FG02-07ER46453 and DE-FG02-07ER46471.

6. References

- [1] Guérin, Y., Was, G. S., and Zinkle, S. J., 2009, "Materials Challenges for Advanced Nuclear Energy Systems," *MRS BULLETIN*, 34(1), pp. 10-14.
- [2] Odette, G. R., Alinger, M. J., and Wirth, B. D., 2008, "Recent developments in irradiation-resistant steels," *Annu Rev Mater Res*, 38, pp. 471-503.
- [3] Ukai, S., and Fujiwara, M., 2002, "Perspective of ODS alloys application in nuclear environments," *J Nucl Mater*, 307, pp. 749-757.
- [4] Alamo, A., Lambard, V., Averty, X., and Mathon, M. H., 2004, "Assessment of ODS-14%Cr ferritic alloy for high temperature applications," *J Nucl Mater*, 329, pp. 333-337.
- [5] Baluc, N., Gelles, D. S., Jitsukawa, S., Kimura, A., Klueh, R. L., Odette, G. R., van der Schaaf, B., and Yu, J. N., 2007, "Status of reduced activation ferritic/martensitic steel development," *J Nucl Mater*, 367, pp. 33-41.
- [6] Marquis, E. A., Hyde, J. M., Saxey, D. W., Lozano-Perez, S., de Castro, V., Hudson, D., Williams, C. A., Humphry-Baker, S., and Smith, G. D. W., 2009, "Nuclear reactor materials at the atomic scale," *Mater Today*, 12(11), pp. 30-37.
- [7] Daymond, M. R., Bourke, M. A. M., VonDreele, R. B., Clausen, B., and Lorentzen, T., 1997, "Use of Rietveld refinement for elastic macrostrain determination and for evaluation of plastic strain history from diffraction spectra," *J Appl Phys*, 82(4), pp. 1554-1562.
- [8] Clausen, B., Lorentzen, T., Bourke, M. A. M., and Daymond, M. R., 1999, "Lattice strain evolution during uniaxial tensile loading of stainless steel," *Mat Sci Eng a-Struct*, 259(1), pp. 17-24.
- [9] Clausen, B., Bourke, M. A. M., Brown, D. W., and Ustundag, E., 2006, "Load sharing in tungsten fiber reinforced Kanthal composites," *Mat Sci Eng a-Struct*, 421(1-2), pp. 9-14.
- [10] Daymond, M. R., 2004, "The determination of a continuum mechanics equivalent elastic strain from the analysis of multiple diffraction peaks," *J Appl Phys*, 96(8), pp. 4263-4272.
- [11] Young, M. L., Almer, J. D., Daymond, M. R., Haefner, D. R., and Dunand, D. C., 2007, "Load partitioning between ferrite and cementite during elasto-plastic deformation of an ultrahigh-carbon steel," *Acta Materialia*, 55(6), pp. 1999-2011.
- [12] Young, M. L., DeFouw, J., Almer, J. D., and Dunand, D. C., 2007, "Load partitioning during compressive loading of a Mg/MgB₂ composite," *Acta Materialia*, 55(10), pp. 3467-3478.
- [13] Young, M. L., Rao, R., Almer, J. D., Haefner, D. R., Lewis, J. A., and Dunand, D. C., 2009, "Load partitioning in Al₂O₃-Al composites with three-dimensional periodic architecture," *Acta Materialia*, 57(8), pp. 2362-2375.
- [14] Liss, K. D., and Yan, K., 2010, "Thermo-mechanical processing in a synchrotron beam," *Mat Sci Eng a-Struct*, 528(1), pp. 11-27.
- [15] Qu, D. D., Liss, K. D., Sun, Y. J., Reid, M., Almer, J. D., Yan, K., Wang, Y. B., Liao, X. Z., and Shen, J., 2013, "Structural origins for the high plasticity of a Zr-Cu-Ni-Al bulk metallic glass," *Acta Materialia*, 61(1), pp. 321-330.
- [16] Cheng, S., Wang, Y. D., Choo, H., Wang, X. L., Almer, J. D., Liaw, P. K., and Lee, Y. K., 2010, "An assessment of the contributing factors to the superior properties of a nanostructured steel using in situ high-energy X-ray diffraction," *Acta Materialia*, 58(7), pp. 2419-2429.
- [17] Wang, L., Li, M., and Almer, J., 2013, "In situ characterization of Grade 92 steel during tensile deformation using concurrent high energy X-ray diffraction and small angle X-ray scattering," *J Nucl Mater*, 440, pp. 81-90.
- [18] Pan, X., Wu, X. L., Mo, K., Chen, X., Almer, J., Ilavsky, J., Haefner, D. R., and Stubbins, J. F., 2010, "Lattice strain and damage evolution of 9–12%Cr ferritic/martensitic steel during in situ tensile test by X-ray diffraction and small angle scattering," *J Nucl Mater*, 407(1), pp. 10-15.

- [19] Pan, X., Wu, X. L., Chen, X., Mo, K., Almer, J., Haefner, D. R., and Stubbins, J. F., 2010, "Temperature and particle size effects on flow localization of 9-12%Cr ferritic/martensitic steel by in situ X-ray diffraction and small angle scattering," *J Nucl Mater*, 398(1-3), pp. 220-226.
- [20] Tamura, M., Sakasegawa, H., Shiba, K., Tanigawa, H., Shinozuka, K., and Esaka, H., 2011, "Decomposition of Y₂Ti₂O₇ Particles in 8 Pct Cr Oxide-Dispersion-Strengthened Martensitic Steel during Tempering," *Metall Mater Trans A*, 42A(8), pp. 2176-2188.
- [21] "<http://materials.npre.illinois.edu/calculator/website.html>."
- [22] Hauk, V., 1997, *Structural and Residual Stress Analysis by Nondestructive Methods*, ELSEVIER.
- [23] Mo, K., 2011, "Microstructural Evolution and Mechanical Behavior in Nickel Based Alloys for Very High Temperature Reactor," PhD Thesis, University of Illinois at Urbana-Champaign, Urbana.
- [24] Hutchings, M. T., Withers, P. J., Holden, T. M., and Lorentzen, T., 2005, *Introduction to the Characterization of Residual Stress by Neutron Diffraction*, Taylor & Francis Group, LLC.
- [25] Fitzpatrick, M. E., and Lodini, A., 2003, *Analysis of Residual Stress by Diffraction using Neutron and Synchrotron Radiation*, Taylor & Francis Inc.
- [26] Noyan, I. C., and Cohen, J. B., 1987, *Residual Stress, Measurement by Diffraction and Interpretation*, Springer-Verlag, New York.
- [27] Kim, S. A., and Johnson, W. L., 2007, "Elastic constants and internal friction of martensitic steel, ferritic-pearlitic steel, and alpha-iron," *Mat Sci Eng a-Struct*, 452, pp. 633-639.
- [28] He, L. F., Shirahata, J., Nakayama, T., Suzuki, T., Suematsu, H., Ihara, I., Bao, Y. W., Komatsu, T., and Niihara, K., 2011, "Mechanical properties of Y₂Ti₂O₇," *Scripta Mater*, 64(6), pp. 548-551.
- [29] Pierson, H. O., 1996, *Handbook of Refractory Carbides and Nitrides: Properties, Characteristics, Processing, and Applications*, William Andrew Publishing.
- [30] Bacon, D. J., Kocks, U. F., and Scattergood, R. O., 1973, "The effect of dislocation self-interaction on the orowan stress," *Philos Mag*, 28(6), pp. 1241-1263.
- [31] Proville, L., and Bako, B., 2010, "Dislocation depinning from ordered nanophases in a model fcc crystal: From cutting mechanism to Orowan looping," *Acta Materialia*, 58(17), pp. 5565-5571.
- [32] Atkinson, J. D., Brown, L. M., and Stobbs, W. M., 1974, "Work-Hardening of Copper-Silica .4. Bauschinger Effect and Plastic Relaxation," *Philos Mag*, 30(6), pp. 1247-1280.
- [33] Mo, K., Tung, H.-M., Li, M., Almer, J., Chen, X., Chen, W., Hansen, J. B., and Stubbins, J., 2013, "Synchrotron radiation study on Alloy 617 and Alloy 230 for VHTR application," *J Press Vess-T Asme*, 135, p. paper No. 021502.
- [34] Chrzan, D. C., Morris, J. W., Osetsky, Y. N., Stoller, R. E., and Zinkle, S. J., 2009, "What is the Limit of Nanoparticle Strengthening?," *MRS BULLETIN*, 34(3), pp. 173-177.

Fig. 1. Representative X-ray diffraction pattern of the 9 Cr ODS sample

Fig. 2. The stress-strain diagram of the 9 Cr ODS sample

Fig. 3. Scanning electron micrograph in secondary electron mode of the 9 Cr ODS sample

Fig. 4. Transmission electron micrograph of nanoscale oxide particles embedded in the F/M
matrix

Fig. 5. Applied stress vs. lattice strain for the F/M matrix. ϵ_{22} and ϵ_{11} are the lattice strains perpendicular and parallel to the applied stress, respectively.

Fig. 6. Elastic anisotropy of Young's modulus for BCC Fe

Fig. 7. The X-ray diffraction peaks of the 9 Cr ODS sample converted from the Debye-Scherrer rings (Fig. 1). All of the diffraction peaks were identified. For clarity, only some of the peaks belonging to the F/M matrix and TiN and Y₂Ti₂O₇ phases are indicated here. The *d*-spacing regime ranging from ~2.1 to ~3.2 is enlarged in order to show the peaks of Y₂Ti₂O₇ with relatively weak intensity.

Fig. 8. Applied stress vs. lattice strain for all phases (F/M matrix (211) reflection, $Y_2Ti_2O_7$ (222) reflection, and TiN (111) reflection) in the 9Cr ODS steel. ϵ_{22} and ϵ_{11} are the lattice strains perpendicular and parallel to the applied stress, respectively.

Fig. 9. Lattice strain vs. macroscopic strain for all phases (F/M matrix (211) reflection, $Y_2Ti_2O_7$ (222) reflection, and TiN (111) reflection) in the 9Cr ODS steel. ϵ_{22} and ϵ_{11} are the lattice strains perpendicular and parallel to the applied stress, respectively.

Fig. 10. Scanning electron micrograph of the fracture surface of the 9 Cr ODS sample. The white particles were identified to be $\text{Y}_2\text{Ti}_2\text{O}_7$ by energy dispersive spectroscopy (EDS).