Temperature Effect of Elastic Anisotropy and Internal Strain Development in Advanced Nanostructured Alloys: an *in-situ* Synchrotron X-ray Investigation

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ABSTRACT

Nanostructured ferritic alloys (NFAs) are promising structural materials for advanced nuclear systems due to their exceptional radiation tolerance and high-temperature mechanical properties. Their remarkable properties result from the ultrafine ultrahigh density Y-Ti-O nanoclusters dispersed within the ferritic matrix. In this work, we performed *in-situ* synchrotron X-ray diffraction tests to study the tensile deformation process of the three types of NFAs: 9YWTV, 14YWT-sm13, and 14YWT-sm170 at both room temperature and elevated temperatures. A technique was developed, combining Kroner's model and X-ray measurement, to determine the intrinsic monocrystal elastic-stiffness constants, and polycrystal Young's modulus and Poisson's ratio of the NFAs. Temperature dependence of elastic anisotropy was observed in the NFAs. An analysis of intergranular strain and strengthening factors determined that 14YWT-sm13 had a higher resistance to temperature softening compared to 9YWTV, attributed to the more effective nanoparticle strengthening during high-temperature mechanical loading.

1 INTRODUCTION

The excellent material properties of nanostructured ferritic alloys (NFAs) under conditions of high temperature, high pressure, and high irradiation make them a promising structural material for use in future nuclear engineering applications [1][2]. Unlike many traditional iron-based steels, the mechanical strength of NFAs is doubled at both room temperature and elevated temperatures [3] with a creep rate of NFAs that is 6-orders lower in magnitude [4]. NFAs are also highly resistant to radiation damage in that they have the capability of limiting radiation-induced helium bubbles within an ultrafine size (~1nm) without forming large voids which can cause the swelling of materials [5][6][7][8]. Through the mechanical alloying processes [9][10][11][12], NFAs such as 14YWT [9][13] and EUROFER97 [14][15] were developed as high performance oxide dispersion strengthened (ODS) alloys with a high-density ultra-fine oxygen-enriched nanoclusters both within the grains and along the grain boundaries

[16][17][18]. The Y-Ti-O-enriched nanoclusters within 14YWT were observed to be uniform in size (2-4nm), and structurally coherent with the ferritic matrix [13][19][20][21][22]. The induced strain energy from the solute-solute repulsion and the presence of oxygen in the form of oxygen-vacancy pair [16][17] makes these nanoclusters ultra-stable in size even at high temperatures (0.92 of the alloy melting temperature), under high pressure [23][24] and under high irradiation conditions [5][6][7]. Via Orowan strengthening, these nanoclusters effectively constrain the grain boundary activities and dislocation migrations. The nanoclusters within grain boundaries can also suppress the grain growth of NFAs during fabrication and thermal mechanical processing. Hence, the grain size of NFAs, usually in sub-micron range, is preserved after fabrication and provides additional strengthening (Hall-Petch strengthening) to the alloys [25].

In order to further enhance the material properties of NFAs, it is important to understand the mechanical behavior and material response when both external load and elevated temperature are applied. Different chemical compositions and different thermal-mechanical treatments can cause variations in the nanocluster size and grain size for different NFAs, leading to various responses to external tensile loadings [26][27][28]. In this study, we adopted a high-energy synchrotron X-ray diffraction technique to investigate the microstructural development of three types of NFAs during *in-situ* uniaxial tensile tests at the temperature from room temperature (RT) to 600°C. By applying Kroner's model [29] to interpret the experimental data, the basic mechanical properties including Young's modulus and Poisson's ratio were attained with elastic-stiffness constants (C_{11} , C_{12} , and C_{44}). The increase in elastic anisotropy with temperature was observed following the temperature dependence of elastic-stiffness constants. Previous studies have shown that the nanoclusters in both 14YWT and MA957 were invisible in the XRD diffraction measurement due to their non-stoichiometric nature. As the direct lattice strain measurements of nanoclusters are not applicable [30] the mean internal stresses of the ferritic matrix and strengthening factors (SFs) [31] were calculated to represent the strengthening effect of the nanoclusters during plastic deformation.

2 MATERIALS AND EXPERIMENTAL PROCEDURE

Three different NFAs were employed in the present study: 9YWT [32][33], 14YWT-sm13 [34], and 14YWT-sm170 [10][35][36]. Both 14YWT-sm13 and 14YWT-sm170 have the same nominal composition: Fe-14Cr-3W-0.4Ti-0.3Y₂O₃ (wt.%), while the average nanoparticle size is slightly different: ~3nm and ~4nm, respectively. The 9YWT has a nominal composition of Fe-9Cr-2W-0.4Ti-0.2V-0.05C-0.3Y₂O₃ with the nanoparticle size of ~5nm.

The *in-situ* uniaxial tensile tests with high-energy synchrotron X-ray diffraction characterization were conducted at the 1-ID-E hutch beamline at the Advanced Photon Source at Argonne National Laboratory. The experimental setup and the specimen dimensions are shown in Fig. 1. Uniaxial tensile tests were conducted on an MTS closed-loop servo-hydraulic test frame (model 858) with a maximum force of ± 15 kN [37][38]. All the NFAs were machined into the SS-J3 type tensile specimen, as shown in Fig.1. During tensile tests, each specimen was subjected to increasing uniaxial tensile stresses up to failure with a strain rate of $2 \times 10^{-4} \text{ s}^{-1}$. A monochromatic 72keV X-ray beam, with beam-size of $100 \times 100 \mu\text{m}^2$ was used to perform the diffraction measurements. The experiment utilized the "Hydra" detector array, which consists of four area detectors (G1-41RT), for X-ray diffraction measurements [38]. The sample to the detector distance was ~1.7m. The NFAs were tested at various temperatures: RT,

300°C, 500°C, and 600°C using an infra-red furnace at ambient air.

3 RESULTS AND DISCUSSION

3.1 Macro-Scale Tensile Deformation

The engineering stress-strain diagrams of the three NFA materials tested at different temperatures are shown in Fig. 2. The corresponding ultimate tensile strengths (UTS) and the yield strengths (YS) are listed in Table 1. Both UTS and YS were observed to decrease with increasing temperature, which is in accordance with previous studies on different NFAs [38][39][40][41]. Among the tested NFAs, 14YWT-sm170 exhibited the best ductility at all studied temperatures; 9YWTV shows the highest strength at all studied temperatures except at 600°C, and 14YWT-sm13 shows the highest UTS value at 600°C. From 500°C to 600°C, a decrease in UTS is 13.2% for 14YWT-sm13, and a decrease in UTS is 11.6% for 14YWT-sm170. However, the reduction in UTS is 49.7% for 9YWTV. Obviously, at a higher temperature regime, 14YWT materials show higher resistance to temperature softening than 9YWTV.

The Debye-Scherrer rings of stress-free samples (before tensile loading) at room temperature are shown in Fig. 3. All the samples in this study show strong texture as indicated by the intensity variation around the azimuth of the Debye-Scherrer rings. Note that the Debye-Scherrer ring of a reflection, i.e. {222} in 14YWT-sm170, is incomplete and thus the Bragg condition at the ϕ of 90° (or 270°) is not met. Therefore, this reflection is inappropriate for studying the lattice strain development in tensile direction. During the uniaxial tensile test, the radii of Debye-Scherrer rings decreased along the loading direction and increased perpendicular to the loading direction due to the Poisson's effect. Therefore, the lattice strain (ϵ_{hkl}) can be determined with the corresponding d -spacing by the Eq. (1) [42]:

$$\epsilon_{hkl} = \frac{d_{\sigma}^{\phi} - d_0}{d_0}, \quad (1)$$

where d_0 is the reference d -spacing of an $\{hkl\}$ reflection before loading is applied [38], and d_{σ}^{ϕ} is the d -spacing of the same reflection measured along ϕ when the sample is subject to the applied stress σ . At $\phi=90^\circ$ and 270° , the lattice strains close to the loading direction are measured. The lattice strain development of the representative reflections of the NFAs at both room temperature and 600°C is shown in Fig. 4. The elastic modulus of each reflection can be attained from the slope of lattice strain vs. applied stress curve in the elastic regime. Here, the elastic modulus of each reflection clearly shows a smaller value at 600°C than that at room temperature. In all the cases, {310} reflection has the lowest elastic modulus. The hook-shaped loading curve was observed in the test of 14YWT-sm170 at low temperatures (Fig. 4(c1)). Similar behavior of internal stress development has been observed in many of the materials with multiple phases: the lattice strain of the metallic matrix slightly decreases during early yielding, but increases afterwards until necking [30][38][43][44][45]. At 600°C, temperature softening eliminates this behavior in 14YWT-sm170.

3.2 Elasticity Properties and Elastic Anisotropy Evolution

In order to derive important mechanical properties of the NFAs (i.e. Young's modulus and Poisson's ratio) from the results of the *in-situ* experiments, a reliable model is required to correlate the single grain behavior and the material bulk response. The Voigt, Reuss and Kroner models have been

extensively used to predict the overall properties (e.g. bulk, shear and Young's moduli and Poisson's ratio) of polycrystalline materials based on their elastic-stiffness constants for monocrystalline. Unlike the Voigt and Reuss models, however, Kroner's model can be used to estimate more accurate material elastic properties due to a more reasonable assumption in the orientation dependence of elastic parameters [46][47]. Therefore, we used this model [48] to evaluate basic mechanical properties of all three NFAs. Here each grain is defined as an inclusion in a homogeneous matrix with the strain-stress relation of a grain denoted as

$$\sigma_{ij} = (C_{ijkl} + r_{ijkl}(\Omega))\varepsilon_{kl} \text{ or } \varepsilon_{ij} = (S_{ijkl} + t_{ijkl}(\Omega))\sigma_{kl}, \quad (2)$$

where σ_{ij} (or σ_{kl}) and ε_{ij} (or ε_{kl}) are the stress and strain of a grain, respectively; C_{ijkl} and S_{ijkl} are the stiffness and compliance tensors; $r_{ijkl}(\Omega)$ and $t_{ijkl}(\Omega)$ represent the interaction among grains, and Ω is the single grain volume. When we evaluate the overall constitutive relation of the bulk material, the interactions among grains will not manifest themselves by satisfying the following equations

$$\int_D r_{ijkl} dD = \int_D t_{ijkl} dD = 0, \quad (3)$$

where D is the bulk volume. Therefore, the strain-stress relation in bulk materials can be defined as [49]

$$\bar{\sigma}_{ij} = (C_{ijkl})_B \bar{\varepsilon}_{ij} \text{ or } \bar{\varepsilon}_{ij} = (S_{ijkl})_B \bar{\sigma}_{ij}, \quad (4)$$

where $(C_{ijkl})_B$ and $(S_{ijkl})_B$ are bulk stiffness and compliance tensors, respectively. With the elastic-stiffness constants C_{11} , C_{12} , and C_{44} , the shear stiffness $c' = (C_{11} - C_{12})/2$ and anisotropy parameter $A = 2C_{44} / (C_{11} - C_{12})$ can then be evaluated respectively. According to the definition, the parameter A represents the shear resistance ratio between the elastically hardest orientation and softest orientation, making it the most appropriate measurement for determining the material's elasticity anisotropy.

With respect to the NFAs at two temperature conditions (RT and 600°C), the elastic modulus for each reflection plane was obtained by a linear fitting of the slope of the lattice strain-applied stress curves for each reflection, listed in Table 2. At both RT and 600°C, the {222} crystallographic planes are the strongest and the {200} crystallographic planes are the weakest. The only exception is 14YWT-sm13 at RT, where elastic modulus of {110} crystallographic planes are slightly larger than that of {222} crystallographic planes, most likely due to measurement errors. The elastic moduli of all the crystallographic planes except {222} are considerably smaller when measured at 600°C than those at RT, whereas {222} crystallographic planes develop a larger modulus at 600°C. Such phenomenon suggests a temperature dependence of the anisotropic behavior of NFAs [38][50].

The least squares estimation was used to obtain the optimized combination of elastic-stiffness constants (C_{11} , C_{12} , and C_{44}) that minimize the following expression:

$$\sum_{hkl} \left(\frac{E_{hkl}^{\text{exp}} - E_{hkl}^{\text{cal}}}{E_{hkl}^{\text{exp}}} \right)^2 + \sum_{hkl} \left(\frac{\nu_{hkl}^{\text{exp}} - \nu_{hkl}^{\text{cal}}}{\nu_{hkl}^{\text{exp}}} \right)^2, \quad (5)$$

where E and ν represent the Young's modulus and Poisson's ratio. The superscript “exp” and “cal” represent the values obtained from experimental measurement and computation based on Kroner's model, respectively. The summation was done over all the measurable $\{hkl\}$ reflections. In this study, an increment of 0.5 GPa was adopted with all elastic-stiffness constants for the optimized combination. Note that the $\{110\}$ family reflection was not included in the calculations due to its high texture that conflicts with the homogeneous matrix assumption of the Kroner model. A typical fitting result for 14YWT-sm13 at 600°C with the optimized elastic-stiffness constants is shown in Fig. 5. The orientation parameter $3a$ in Fig. 5 takes the form: $3a=3(h^2k^2+k^2l^2+l^2h^2)/(h^2+k^2+l^2)^2$.

As the incomplete diffraction ring prevented an analysis of the elastic modulus of $\{222\}$ for 14YWT-sm170, only 9YWTV and 14YWT-sm13 were analyzed to attain their elastic-stiffness constants. It was then possible to calculate the corresponding values of the shear stiffness and the anisotropy parameter. The elastic-stiffness constants obtained with Eq. (5) are listed in Table 3, and the basic properties of materials are presented in Fig. 6. As the testing temperature increases, materials' softening reflected by the decrease in Young's modulus and the increase in Poisson's ratio was observed in both NFAs (Fig. 6(a, b)). As shown in Fig. 6(c), the shear modulus decreases with the increasing temperature. The temperature dependence of bulk elastic anisotropy was studied through the development of Poisson's ratio as the function of temperature, and the materials' intrinsic monocrystal elastic anisotropy was determined through the anisotropy parameter A . As seen in Fig. 6(d), the anisotropy parameter exhibited very little change in the low-temperature regime ($T < 400^\circ\text{C}$), but dramatically increased in the high-temperature regime ($T > 400^\circ\text{C}$). This increase indicates that the elastic strain response of crystalline in the NFAs develops a higher anisotropy when loaded at high temperatures.

The orientation dependence of Young's modulus of 9YWTV and 14YWT-sm13 was then plotted in 3D to visually compare their elastic anisotropy (see Fig. 7). For modulus at RT and 600°C, 9YWTV apparently displays stronger anisotropy, meaning that the elastic modulus in 9YWTV shows the larger difference for various orientations than 14YWT-sm13.

To further investigate the anisotropic behavior of grains orienting in different directions at various temperatures, we calculated the intergranular strains $\Delta\varepsilon^{\text{II}}$ to examine the difference between the real lattice strain $\varepsilon_{\{hkl\}}$ and the projected linear elastic strain accordingly, which is defined as

$$\Delta\varepsilon^{\text{II}} = \varepsilon_{\{hkl\}} - \sigma/E_{\{hkl\}}. \quad (6)$$

Here, plastic deformation begins within those grains that have orientations that activate their dislocation slip systems upon reaching the point of critical shear stress. Other grains at different orientations, however, sustain more loads in that they continue to respond to the load elastically, which causes a redistribution of stress among these grains. Compressive intergranular strain accumulates for the grains that first reach the yielding point, while the grains in other orientations that are plastically hard still experience a tensile intergranular strain. Therefore, the onset of the accumulating of compressive intergranular strains of grain aligned in softer orientations is an indication of the elastic anisotropy

[50][51][52][53]. As shown in the variation of the compressive intergranular strains with applied stress for {110} grains in the NFAs (see Fig. 8) the onset of compressive intergranular strain for the 14YWT-sm170 and 14YWT-sm13 alloys is quite similar at 500°C and 600°C, denoting an insignificant softening at these temperatures. Note that the elastic anisotropy is also restrained in 14YWT while 9YWTV at 500°C obviously shows a higher yielding point than at 600°C. With the rise in temperature, the compressive intergranular strain starts to accumulate at a much lower external loading value in 9YWTV, indicating an obvious “softening” of the grains.

3.3 Mean Internal Stress

In order to understand the mechanically strengthening mechanism during the plastic deformation and the loading status for both ferritic matrix and nanoparticles, we calculated the mean internal stress of the matrix and compared it with the externally applied stress. The difference between the internal stress and the externally applied stress refers to the load status of various phases within the NFAs. It can help explain the nanoparticles strengthening effects. The mean internal stress is defined as [54][55]:

$$\sigma_{matrix} = \bar{E} \frac{\sum_{hkl} T_{hkl} m_{hkl} E_{hkl} \varepsilon_{hkl}}{\sum_{hkl} T_{hkl} m_{hkl} E_{hkl}}, \quad (7)$$

where $\bar{E}$ is the average Young's modulus of the ferritic matrix (with the values of $\bar{E}$ derived from previous study [45], and the fraction term is the mean internal strain defined as a weighted average lattice strain ε_{hkl} for various {hkl} reflections. The weighting parameters T_{hkl} , m_{hkl} and E_{hkl} are the texture index, the multiplicity and the stiffness for reflection {hkl}, respectively [56]. Here, six reflections were included in the calculation: {110}, {200}, {211}, {310}, {321} and {222}. The 9YWTV and 14YWT-sm13 were selected for detailed analysis, and a comparison of mean internal stress developed during the tensile and the externally applied stress are provided in Fig. 9. In all the tests at various temperatures, the mean internal stress was smaller than the actually applied stress after yielding, the difference is caused by the load re-distribution and transfer between phases and grains during plastic deformations. As the main strengthener in these alloys, nanoparticles play an important role in deformation particularly in the load re-distribution in plasticity. For 9YWTV, the difference between mean internal stress and externally applied stress is significant at room temperature, which decreased with an increase in temperature. At 600°C, this difference is almost negligible (Fig. 9(a)). For 14YWT-sm13, the difference between mean internal stress and externally applied stress is significant at room temperature. The difference remains and cannot be neglected at 600°C (Fig. 9(b)). To better quantify the difference between mean internal stress and external applied stress ($\sigma_{applied}$), the strengthening factors $SF = (\sigma_{applied} - \sigma_{matrix}) / \sigma_{matrix}$ [54] for both alloys, which are summarized in Table 4, were calculated. The larger SF of 14YWT-sm13 at 600°C shows that the strengthening of ferritic matrix mainly derives from the function of nanoparticles at room temperature and elevated temperatures. These ultra-fine uniformly distributed nanoparticles pin the dislocation movement, causing the loading transfer between the nanoparticles and the grains, therefore enhancing the strength of the material. In comparison, a significant decrease in SF from 500°C to 600°C was found in 9YWTV. This decrease indicates a higher temperature dependence of the strengthening for 9YWTV due to the lack of ultra-fine uniformly distributed nanoparticles, compared to 14YWT-sm13, particularly at the high-temperature regime.

CONCLUSIONS

In this work, *in-situ* high-energy synchrotron X-ray diffraction tests were performed to study the basic mechanical properties and internal stress development of three types of NFAs: 9YWTV, 14YWT-sm13, and 14YWT-sm170 at both room temperature and elevated temperatures. A combination of Kroner's model and X-ray measurements were used to derive the intrinsic monocrystal elastic-stiffness constants and polycrystal elastic parameters of the NFAs. An increase was observed in the elasticity anisotropy in all of the materials studied with an increase in temperature. An analysis of the intergranular strain of 14YWT-sm13 indicated a higher resistance to temperature softening compared to 9YWTV. A further analysis of the strengthening factors indicated differences in the applied and mean internal stress. This discrepancy was due to the nanoparticle strengthening mechanism. 14YWT-sm13 displays stronger strengthening compared to 9YWTV, particularly at the higher temperatures.

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	T (°C)	9YWTV	14YWT -sm13	14YWT -sm170
UTS	RT	1.78	1.38	1.10
	300	1.55	1.12	1.00
	500	1.34	0.90	0.59
	600	0.67	0.78	0.52
YS	RT	1.48	1.11	0.87
	300	1.19	0.84	0.73
	500	0.54	0.46	0.50
	600	0.47	0.44	0.37

Table 1: Ultimate tensile strengths (UTS) and yield strengths (YS) of the NFAs at various temperatures: (units: GPa).

T	Sample	Fitted Elastic modulus E_{hkl} for each peak (GPa)					
		{1 1 0}	{2 0 0}	{2 1 1}	{2 2 0}	{3 1 0}	{2 2 2}
RT	9YWTV	215	181	219	218	196	233
	14YWT-sm13	236	175	214	233	193	231
	14YWT-sm170	231	178	205	236	198	NaN
600 °C	9YWTV	160	129	189	177	153	261
	14YWT-sm13	198	140	201	198	159	244
	14YWT-sm170	172	153	179	179	156	NaN

Table. 2. Elastic modulus for various $\{hkl\}$ reflection; the uncertainty of the values is around ± 5 GPa.

	9YWTV			14YWT-sm13		
T (°C)	C₁₁	C₁₂	C₄₄	C₁₁	C₁₂	C₄₄
RT	213.0	109.0	119.0	199.0	77.0	111.0
300	202.0	97.0	125.0	172.0	65.5	99.5
500	194.0	119.0	125.0	186.0	122.0	111.0
600	186.0	135.0	131.0	187.0	125.0	129.0

Table 3. The best fit of elastic-stiffness constants using Eq. (5). The unit is GPa.

Sample	RT	300°C	500°C	600°C
9YWTV	0.1612	0.1660	0.1937	0.0818
14YWT-sm13	0.2114	0.4636	0.1819	0.2304

Table. 4. The strengthening factors (SFs) for 9YWTV and 14YWT-sm13 at various temperatures.

Figure 1. *In-situ* synchrotron X-ray diffraction test setup.

Figure 2. Stress-strain diagrams of the tested NFAs: (a) 9YWTV; (b) 14YWT-sm13; and (c) 14YWT-sm170 at various temperatures.

Figure 3. Diffraction patterns for (a) 9YWTV, (b) 14YWT-sm13, and (c) 14YWT-sm170. “90°” denotes the tensile loading direction.

Figure 4. Lattice strain - applied stress relations of three NFAs at room temperature: (a1) 9YWTV; (b1) 14YWT-sm13; (c1) 14YWT-sm170; and at 600 °C: (a2) 9YWTV; (b2) 14YWT-sm13; (c2) 14YWT-sm170.

Figure 7. Orientation dependence of Young's modulus (unit: GPa) at RT and 600°C for (a) 9YWT and (b) 14YWT-sm13.

Figure 6. Temperature dependence of elastic mechanical properties for the NFAs: (a) The bulk elastic modulus; (b) Poisson's ratio (c) shear modulus; and (d) anisotropy parameter evolution.

Figure 8. Intergranular strain evolution for the NFAs (a) 9YWTV, (b) 14YWT-sm13, and (c) 14YWT-sm170.

Fig. 9: Comparison of mean internal stress and applied stress for (a) 9YWTV and (b) 14YWT-sm13 at various temperatures (Red: RT; Blue: 300°C; Magenta: 500°C; and Cyan: 600°C). The solid symbols and hollow symbols are applied stresses and internal stresses, respectively.