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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 (~1 nm) without forming large voids which can cause the swelling of materials [5–8]. Through the mechanical alloying processes [9–12], 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 [16–18]. The Y-Ti-O-enriched nanoclusters within 14YWT were observed to be uniform in size (2–4 nm), and structurally coherent with the underline ferritic matrix [13,19–22]. 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 [5–7]. 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 [26–28]. In this study, we adopted a high-energy synchrotron X-ray diffraction technique to investigate the microstructural develop-

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ment 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: 9YWTV [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-0.3Y_2O_3 (wt%), while the average nanoparticle size is slightly different: ~3 nm and ~4 nm, respectively. The 9YWT has a nominal composition of Fe-9Cr-2W-0.4Ti-0.2V-0.05C-0.3Y_2O_3 with the nanoparticle size of ~5 nm.

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 2×10^{-4} s^{-1}. A monochromatic 72 keV X-ray beam, with beam-size of 100×100 μm^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 ~1.7 m. 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 [38–41]. 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

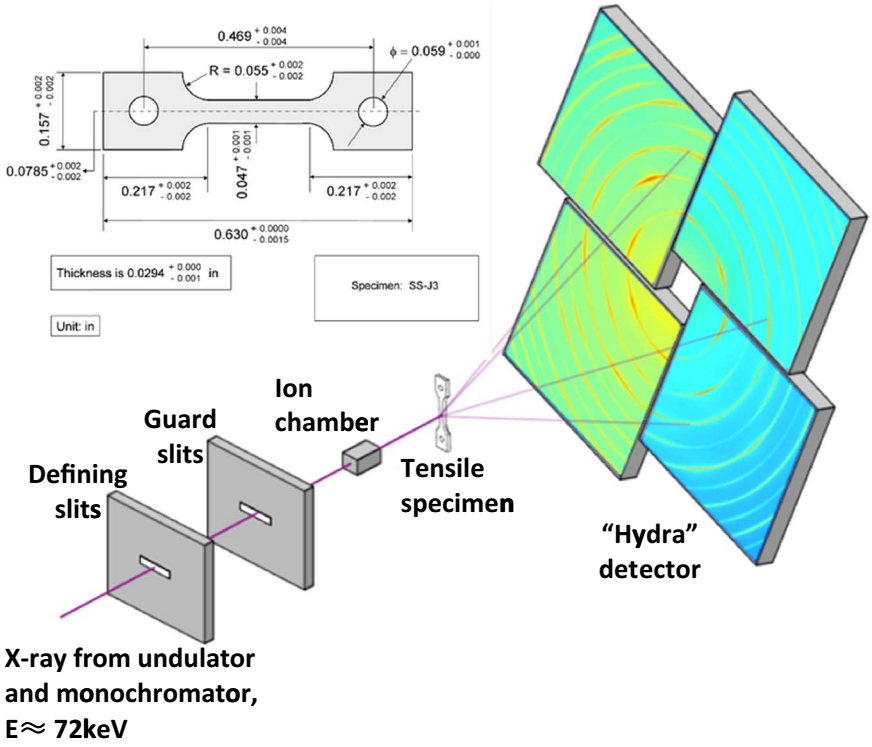

Fig. 1. *In-situ* synchrotron X-ray diffraction test setup.