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Load-partitioning in an oxide dispersion-strengthened 310 steel at elevated temperatures

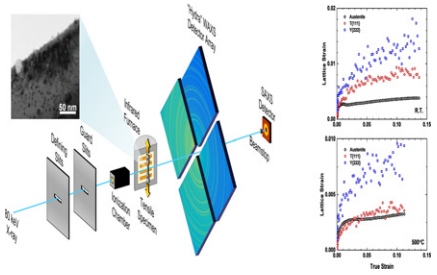
Yinbin Miao[a,b,*], Kun Mo[a], Zhangjian Zhou[c], Xiang Liu[b], Kuan-Che Lan[b], Guangming Zhang[b,c], Jun-Sang Park[a], Jonathan Almer[a], James F. Stubbins[b,d]

[a] Argonne National Laboratory, Lemont, Illinois 60439, United States
[b] University of Illinois at Urbana-Champaign, Urbana, Illinois 61801, United States
[c] University of Science and Technology Beijing, Beijing 100082, China
[d] International Institute for Carbon-Neutral Energy Research (I2CNER), Kyushu University, Fukuoka 819-0395, Japan

HIGHLIGHTS

- An austenitic oxide dispersion-strengthened stainless steel was developed and investigated for nuclear applications.
- Size distribution and orientation relationship of the ultra-fine oxygen-enriched nanoparticles were measured.
- *In situ* tensile tests were conducted at various temperatures to reveal the strengthening effect of precipitates.
- Only the ultra-fine oxygen-enriched nanoparticles are capable of retaining their strengthening effect at high temperatures.
- The austenitic oxygen dispersion-strengthened stainless steel showed advantageous mechanical strength.

GRAPHICAL ABSTRACT



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ABSTRACT

Here the high temperature tensile performance of an oxide dispersion-strengthened (ODS) 310 steel is reported upon. The microstructure of the steel was examined through both transmission electron microscopy (TEM) and synchrotron scattering. *In situ* synchrotron X-ray tensile investigation was performed at a variety of temperatures, from room temperature up to 800 °C. Pyrochlore structure yttrium titanate and sodium chloride structure titanium nitride phases were identified in the steel along with an austenite matrix and marginal residual α′-martensite. The inclusion phases strengthen the steel by taking extra load through particle-dislocation interaction during plastic deformation or dislocation creep procedures. As temperature rises, lattice strain measurement implies that the load partitioning effect of conventional precipitate phases starts to diminish, whereas those ultra-fine oxygen-enriched nanoparticles continue to maintain a considerable amount of extra lattice strain. Introduction of oxygen-enriched nanoparticles in austenitic steel is shown to improve the high temperature performance, making austenitic ODS steels promising for advanced nuclear applications.

* Corresponding author at: Argonne National Laboratory, Lemont, IL60439, United States.
E-mail address: ymiao@anl.gov (Y. Miao).

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1. Introduction

Advanced nuclear fission reactors and prospective fusion conceptions involve an extreme in-core environment and bring out tough material challenges for the nuclear industry [1]. Mechanical alloying is capable of introducing a dense and dispersed distribution of ultra-fine oxygen-enriched nanoparticles into steel matrices in order to produce oxide dispersion-strengthened (ODS) steels [2,3]. These nanoparticles can not only pin dislocations to inhibit plastic deformation and dislocation creep [4], but also trap irradiation-induced defects to suppress radiation damage evolution and consequent degradation of material performance [5,6]. Therefore, ODS steels are regarded as promising candidates for structural materials in future nuclear systems [7].

Due to their intrinsic advantages in mechanical strength and radiation resistance, ferritic/martensitic (F/M) ODS steels have been frequently studied for potential applications in fission reactor pressure vessels (RPVs) or fusion reactor first walls (FWs) [4,8,9]. Meanwhile, with high-temperature phase stability, austenitic steels also have exceptional corrosion and creep resistance. Combined with enhanced mechanical strength and radiation tolerance originating from the ultra-fine nanoparticles, austenitic ODS steels are expected to qualify for in-core structural materials of advanced reactors, such as fuel cladding for supercritical water reactors. Thus, austenitic ODS steels have been developed and investigated in order to explore their prospective use in nuclear industry [10–15].

As part of the long search for austenitic ODS steels that are practical for advanced nuclear designs, an ODS 310 stainless steel was developed and investigated in this study. As the origin of the outstanding properties of ODS steels, the microstructural characteristics of the ultra-fine oxygen-enriched nanoparticles determine the macroscopic performance. Hence, it is important to examine the microstructure of this ODS 310 steel, especially the properties of these nanoparticles and their responses to externally applied stress. This task calls for a combined utilization of advanced materials characterization techniques. The diffraction contrast imaging of transmission electron microscopy (TEM) is capable of revealing the number density and size distribution of nanoparticles, whereas the high-resolution transmission electron microscopy (HRTEM) can capture the crystal structure of nanoparticles along with their orientation relationship with the austenite matrix. Additionally, having extraordinary beam energy and intensity, synchrotron radiation is an ideal tool to characterize inclusion phases that have marginal volume fractions [16–18] such as oxygen-enriched nanoparticles. In fact, synchrotron-based techniques have already been successfully applied to the characterization of ODS steels in the past few years [9,19-21].

In this study, the oxygen-enriched nanoparticles in a recently developed ODS 310 stainless steel were first *ex situ* investigated by both TEM and synchrotron X-ray scattering. Then *in situ* synchrotron tensile tests were conducted within a wide range of temperatures from room temperature, through the operation temperature of fuel cladding materials in light water reactors (350 °C) and supercritical water reactors (500 °C), up to those extreme temperatures usually found in accidental scenarios (650 °C and 800 °C), so that the responses of those nanoparticles to externally applied stress could be comprehensively studied to evaluate the qualification of austenitic ODS steels for advanced nuclear applications.

2. Experiments

The ODS 310 steel investigated in this study has the nominal composition as listed in Table 1. The base material powders were first mechanically alloyed in an inert atmosphere using a planetary ball mill at 300 rpm with a ball-to-powder ratio of 5:1 for 30 h. The milled powders were then degassed, sealed, and consolidated through hot isostatic pressing (HIP) under a pressure of 100 MPa at 1150 °C for 3 h. A similar manufacturing procedure has been used to produce a series of F/M and austenitic ODS steels with excellent performance.

Table 1
Nominal compositions of investigated ODS steels (wt%).

Material	Fe	Cr	Ni	Mo	Ti	Y_2O_3
ODS310	bal.	24.0	18.3	1.9	0.3	0.35

In order to prepare specimens suitable for TEM observation, 3 mm discs with a thickness of approximately 250 μm were cut from the bulk material using electric discharge machining (EDM). These discs were first mechanically thinned and polished to a 100 μm thickness and a 0.05 μm surface finish by sandpapers and diamond suspensions. The specimens were then electropolished to electron transparency for TEM observation using a solution containing 5 vol.% perchloric acid and 95 vol.% methanol at −14 °C. Both diffraction contrast imaging and HRTEM investigations were performed on a JEOL 2010 LaB_6 TEM with a 200 kV electron beam.

Synchrotron involved investigations were performed at Sector 1-ID, Advanced Photon Source (APS), Argonne National Laboratory (ANL). An 80.7 keV ($\lambda = 0.154$ Å) monochromatic synchrotron beam was used to illuminate the specimens to generate scattering signals. Four GE-RT41 detectors, each of which contains 2048 × 2048 pixels, were combined to form the HYDRA detector array to collect wide angle X-ray scattering (WAXS) signals. Meanwhile, a Scint-X detector was used to capture small-angle X-ray scattering (SAXS) information. The ODS 310 steel was also cut into miniature tensile specimens with gauge sections of 1.20 mm (width)×0.5 mm (thickness)×5.00 mm (length) by EDM. The tensile specimens were mounted on an MTS tensile stage with an infrared clamshell furnace for *in situ* synchrotron tensile investigation (see Fig. 1). The tensile tests were performed using a 2×10^{-4} s^{-1} strain rate at room and elevated temperatures up to 800 °C. Each synchrotron WAXS exposure took 4 s. The fracture surface of each strained specimen was investigated by a JEOL IT-100 scanning electron microscope (SEM).

The 10° regions of the Debye-Scherrer diffraction rings near the uniaxial tensile direction were integrated to provide the lattice spacings evolution during straining. The lattice strains were also calculated accordingly, $\varepsilon_{22}^{hkl} = (d^{hkl} - d_0^{hkl})/d_0^{hkl}$, where, *hkl* is the Miller index of the measured diffraction ring, d^{hkl} is the measured lattice spacing between two neighboring {hkl} planes, d_0^{hkl} is the measured lattice spacing of the unloaded specimen at the testing temperature. Likewise, the lattice strain at the horizontal direction, ε_{11}, can also be deduced. ε_{33} was assumed to be equal to ε_{11}. For the austenite matrix, the average bulk lattice strain was derived from the lattice strains of {111}, {200}, {220}, {311}, {222}, {400}, and {331} reflections using the weighted averaging algorithm developed by Daymond [22] and successfully applied by Miao et al. to austenitic ODS steels [14]. The temperature-dependent elastic stiffness tensor of 316 stainless steel [23] was adopted in this study due to the similarity of elastic moduli among austenitic steels and the lack of related data for 310 stainless steels.

Aside from the lattice strain, diffraction peaks also contain information about dislocation density, stacking fault portion (for FCC materials), and grain size of the samples. The modified Williamson-Hall (W-H) analysis was used to interpret this information from the broadening of diffraction peaks. In the modified W-H analysis, the peak broadening is governed by the following equation:

$$\Delta K = \left(\frac{1.5\alpha+\beta}{a}\right) W(g) + \frac{0.9}{D} + \left(\frac{\pi A^2 b^2}{2}\right)^{\frac{1}{2}} \rho^{\frac{1}{2}} \left(K\bar{C}^{\frac{1}{2}}\right), \quad (1)$$