Journal of Nuclear Materials 467 (2015) 50–57

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

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

Guangming Zhang [a, b], Zhangjian Zhou [a, *], Kun Mo [c], Yinbin Miao [b], Xiang Liu [b], Jonathan Almer [d], James F. Stubbins [b]

[a] School of Materials Science and Engineering, University of Science and Technology, Beijing, Beijing 100083, China
[b] Department of Nuclear, Plasma and Radiological Engineering, University of Illinois at Urbana-Champaign, IL 61801, USA
[c] Nuclear Engineering Division, Argonne National Laboratory, Argonne, IL 60439, USA
[d] X-ray Science Division, Argonne National Laboratory, Argonne, IL 60439, USA

HIGHLIGHTS

- The tensile deformation of 9Cr ODS steel was studied by synchrotron irradiation.
- The evolution of internal mean stress was calculated.
- The evolution of dislocation character was determined by best-fit method.
- Edge type dominates plasticity at RT and 300 °C, while screw type dominates at 600 °C.

ARTICLE INFO

Article history:
Received 10 April 2015
Received in revised form
29 July 2015
Accepted 7 September 2015
Available online 11 September 2015

Keywords:
Synchrotron XRD
Mean internal stress
Dislocation character
Dislocation density

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 may be explained by the activated cross slip of screw segments.

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 [1–3]. The well-distributed nanoscale particles within the steels are critical to their excellent irradiation resistance, high-temperature strength, and superb creep properties [4–6]. 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

* Corresponding author.
E-mail address: zhouzhj@mater.ustb.edu.cn (Z. Zhou).

http://dx.doi.org/10.1016/j.jnucmat.2015.09.014
0022-3115/

activation ferritic/martensitic (RAFM) steels, such as Eurofer 97, F82H and ORNL 9Cr2WTaV [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) [13–15]. 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-0.35Y_2O_3 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 Fig. 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 mm 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 LaB_6 TEM.

3. Results and discussion

3.1. Macro-mechanical properties

Fig. 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 650 K in a reduced activation ferritic/martensitic (RAFM) steel [21]. The serrations were accompanied by an increase in work-

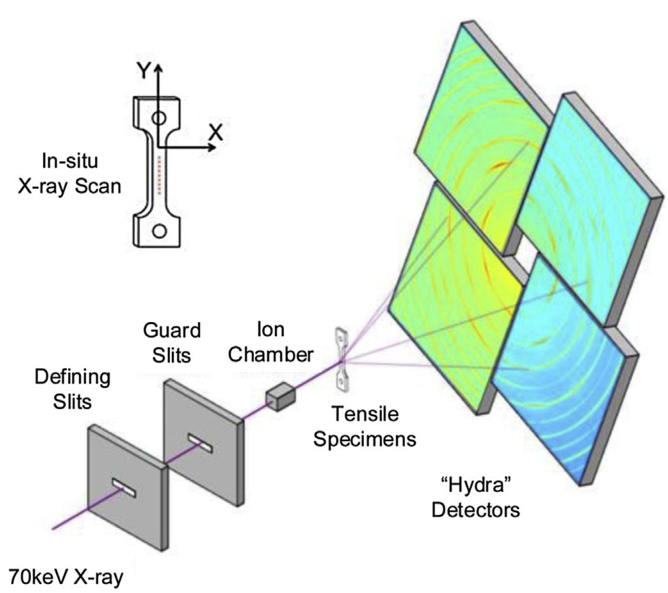

Fig. 1. Experimental set-up of the tensile test with in-situ synchrotron X-ray diffraction.

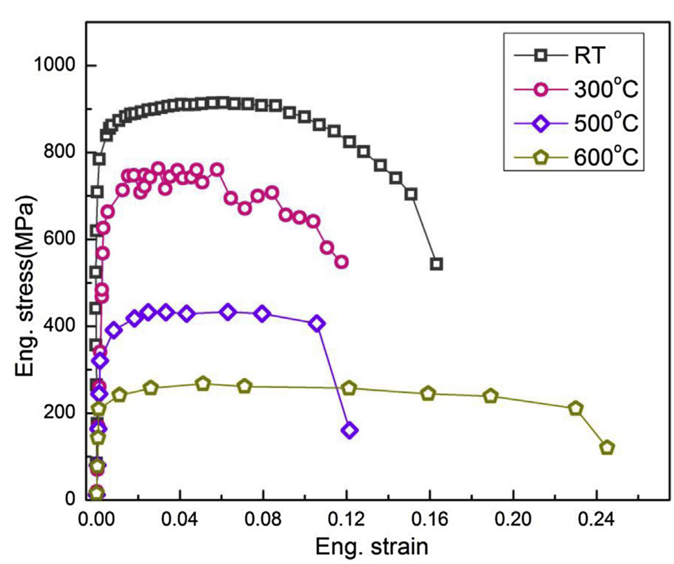

Fig. 2. Engineering stress vs. engineering strain from RT to 600 °C.