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ScienceDirect

Acta Materialia 83 (2015) 137–148

Acta MATERIALIA

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Deformation mechanisms in a precipitation-strengthened ferritic superalloy revealed by in situ neutron diffraction studies at elevated temperatures

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Received 18 April 2014; revised 23 September 2014; accepted 27 September 2014

Abstract—The ferritic superalloy Fe–10Ni–6.5Al–10Cr–3.4Mo strengthened by ordered (Ni,Fe)Al *B2*-type precipitates is a candidate material for ultra-supercritical steam turbine applications above 923 K. Despite earlier success in improving its room-temperature ductility, the creep resistance of this material at high temperatures needs to be further improved, which requires a fundamental understanding of the high-temperature deformation mechanisms at the scales of individual phases and grains. In situ neutron diffraction has been utilized to investigate the lattice strain evolution and the microscopic load-sharing mechanisms during tensile deformation of this ferritic superalloy at elevated temperatures. Finite-element simulations based on the crystal plasticity theory are employed and compared with the experimental results, both qualitatively and quantitatively. Based on these interphase and intergranular load-partitioning studies, it is found that the deformation mechanisms change from dislocation slip to those related to dislocation climb, diffusional flow and possibly grain boundary sliding, below and above 873 K, respectively. Insights into microstructural design for enhancing creep resistance are also discussed.
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Keywords: Neutron diffraction; Ferritic superalloy; High-temperature deformation behavior

1. Introduction

Ferritic steels currently used in power plants are limited to a temperature range of 723–873 K and a stress level of 15–100 MPa due to the limited creep resistance and poor corrosion resistance of these steels at high temperatures. To improve the thermal efficiency of steam turbines, there is a need to raise the steam temperature in ultra-supercritical steam turbines to 1033 K in the near future. To this end, a class of precipitation-strengthened ferritic superalloys has been developed and fabricated by vacuum arc melting and vacuum induction melting methods [1–9]. In analogy to the microstructure of Ni-based superalloys, the β/β′ ferritic superalloy has the *A2*-type (Strukturbericht notation) disordered body-centered-cubic (bcc) matrix (also called β phase here). The strengthening phase is the ordered *B2*-type (Strukturbericht notation of CsCl-type crystal structure) NiAl precipitates (also called β′ phase here). The β′ phase has a volume fraction below 20%, and is coherent with the β matrix. The alloy is designed as a candidate structural material for ultra-supercritical steam-turbine applications above 923 K. Two critical issues with this class of materials are the limited creep properties above 873 K and the poor room-temperature ductility. Thus, our previous research has focussed on the microstructural characterization of precipitates, the effect of chemistry on the microstructure and room-temperature mechanical properties, and the computational design of optimized compositions for ductility enhancement [1–6]. The creep properties were investigated by examining the role of microstructures and by searching for slow diffusing elements to reduce coarsening and therefore creep rates [7,8]. Further optimization of the high-temperature mechanical properties of these materials requires a fundamental understanding of the deformation mechanisms at the polycrystalline grain level and at the β/β′ phase level [7–9].

The underlying strengthening mechanisms in precipitation- or dispersion-strengthened materials can be generally divided into two categories: strengthening through the load transfer mechanism for large particles (e.g. micron-scale reinforcement in composites) and strengthening through elastic interactions between dislocations and precipitates, especially for nanosized precipitates [10]. Transmission electron microscopy (TEM) is the preferred experimental technique to characterize dislocation–particle interactions [9], while the diffraction techniques have become a popular way to identify the load transfer behavior in multiphase materials. Neutron diffraction (ND) takes advantage of the large beam size and deep penetration capability, and thus is able to provide a statistical measure of the lattice

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http://dx.doi.org/10.1016/j.actamat.2014.09.053
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spacing averaged over a large number of grains inside polycrystalline materials. With in situ loading and heating capabilities, the dynamic evolution of lattice strain, texture and peak width can be further investigated under different temperatures or various loading modes.

In recent works that utilize in situ ND measurements under tensile or creep deformation [11–13], the understanding of the deformation mechanisms in γ/γ′ Ni-based superalloys has been advanced by (i) the demonstration of a slip system change in the γ′ phase, (ii) investigation of load sharing between the γ and γ′ phases, (iii) an estimate of the critical resolved shear stress (CRSS) for γ′, and (iv) determination of the constrained thermal expansion coefficients. The accurate determination of lattice strains in the γ and γ′ phases is always complicated because of the low lattice misfit between γ and γ′ and the resulting overlapping of their fundamental reflection peaks. ND can easily detect the relatively high diffraction intensity from γ′ superlattice reflections, if the superalloy contains an intermediate to high volume percentage of γ′ (typically >40 vol.%). On the other hand, diffraction studies of superalloys with a low volume fraction of γ′ pose additional requirements on the instrument resolution and neutron beam if high data quality and sensitivity are desired.

In this work, we present high-temperature thermomechanical loading and in situ ND studies on the above-mentioned β/β′ ferritic superalloy. The microstructure-based understanding of the deformation mechanisms will be derived from information gained on the phase-level and grain-level, e.g. how the applied load is partitioned among different grain families and between the two phases. The above understanding relies critically on high-quality experiments to resolve the low-intensity superlattice diffraction peaks of the β′ phase. Recently, development of state-of-the-art thermomechanical loading capabilities as well as high neutron flux at the VULCAN Engineering Materials Diffractometer of the Spallation Neutron Source (SNS), Oak Ridge National Laboratory (ORNL), has opened unique opportunities for advanced structural materials research and has improved the capability of quantitatively characterizing the secondary phases with low volume fractions and low intensity peaks [14,15]. Therefore, exploiting this capability in this work, lattice strain measurements were conducted under quasi-static tensile deformation in the temperature range 623–973 K. Here, the quasi-static tension refers to a stepwise load-and-hold at different stress levels for the collection of diffraction data, as opposed to a continuous loading with a constant strain rate. Evolution of load-sharing mechanisms at grain and phase levels is investigated in plastic deformation regimes. The interpretation of load transfer is supported by a crystal plasticity finite-element simulation that includes the effect of creep on the temporal lattice strain evolution. The results demonstrate that a change in deformation mechanism as a function of stress and temperature can be identified through different diffraction signatures, as reflected by lattice strain, peak width or peak intensity.

2. Methods

2.1. Material

The nominal composition of the alloy studied here is Fe–12.7Al–10.2Cr–9Ni–1.9Mo–0.14Zr–0.024B (at.%), or Fe–6.5Al–10Cr–10Ni–3.4Mo–0.25Zr–0.005B (wt.%). The ingot was fabricated by induction melting at Sophisticated Alloys, Inc. Rods cut from the ingot were sealed in quartz tubes under vacuum for the purpose of subsequent heat treatment, which included homogenization at 1473 K for 30 min, followed by air cooling, aging at 973 K for 100 h, and then air cooling. The average grain size was ~100 μm, and the polycrystalline material was slightly textured. The slight texture was indicated by a value of 2 for the spherical harmonics preferred orientation correction factor in GSAS software when fitting the measured diffraction pattern. Inside each grain, there was a homogeneous distribution of β′ precipitates with an average diameter of 130 nm and a volume fraction of ~18%. The volume fraction was determined in our previous works by atom probe tomography (APT), analytical electron microscopy (AEM) and ultra-small-angle X-ray scattering [1,7]. The corresponding TEM images have been shown previously in Ref. [1].

2.2. Neutron diffraction experiments

Screw-threaded cylindrical samples with a gauge length of 40 mm and a gauge diameter of 6.75 mm were prepared for in situ tensile experiments. The ND experiments were conducted at the VULCAN instrument [14,15]. The measurable diffraction d-spacing range of 0.5–3.5 Å was obtained by using a chopper setting of 20 Hz spinning frequency. The diffraction geometry provided lattice strain measurements parallel and perpendicular to the loading directions (i.e. both axial and transverse directions with respect to the sample geometry). The incident beam size was defined by a 6 mm horizontal and 6 mm vertical incident slit and a set of 5 mm collimators. Because of the lack of a high-temperature extensometer, the cross-head displacement was used to estimate macroscopic strains at elevated temperatures. The sample was loaded with high-temperature water-cooled grips on the VULCAN-MTS load frame and heated by the copper induction coils. The sample was maintained in the middle of the induction coils, as shown in Fig. 1a. The open configuration of the heating coil allows the neutron beam to diffract only from the sample without shadowing from the copper tube. The temperature gradient in the sample gauge length was measured by an infrared camera, which was verified to be within 10 K in the neutron gauge length at various testing temperatures up to 1273 K. An example of the infrared camera image is exhibited in Fig. 1b. One K-type thermocouple was spot welded at the center of the sample gauge length and another ~4 mm away from the center. The central thermocouple was for the sample temperature control, while the off-center one was used to monitor the temperature difference with respect to the central one and also to set a maximum temperature limit for protection against overheating. In situ tension experiments were performed under a constant load control mode with stepwise-loading schedules at isothermal conditions of 623, 773, 873 and 973 K. The ND data were collected for 40 min at each stress level. Three stress levels were chosen for unloading, if the sample did not fracture at the maximum applied load. Peak fitting was done using the VDRIVE software [16].

A typical ND pattern measured at the VULCAN instrument is displayed in Fig. 2a, with the normalized intensity on a logarithmic scale. The coherency between the precipi-

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