



Creep properties and microstructure of a precipitation-strengthened ferritic Fe–Al–Ni–Cr alloy

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Abstract

The ferritic alloy Fe–10Cr–10Ni–5.5Al–3.4Mo–0.25Zr–0.005B (wt.%), strengthened by coherent B2-structured (Ni,Fe)Al precipitates with a volume fraction of 13 vol.% and average precipitate radius of 62 nm, was subjected to creep in the stress range 30–300 MPa and the temperature range 600–700 °C. The stress dependence of the steady-state strain rate can be represented by a power law with high apparent stress exponents of 6–13 and high apparent activation energies of 510–680 kJ mol^{−1}. Threshold stresses at all studied temperatures were observed, ranging from 69 to 156 MPa, from which a true stress exponent of ~4 and a true activation energy of 243 ± 37 kJ mol^{−1} were determined, which are equal to those for dislocation creep and lattice diffusion in the ferritic matrix, respectively. Based on these mechanical results and detailed electron microscopy observations, the creep mechanism was rationalized to be general dislocation climb with repulsive elastic interaction between coherent precipitates and the matrix dislocations.

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1. Introduction

Ferritic steels are an attractive alternative to austenitic steels for high-temperature applications (e.g., in thermal power plants) due to their lower cost and thermal expansion, and their higher thermal conductivity. However, the poor creep resistance of ferritic steels at temperatures above 600 °C limits their usage [1]. The existence of a miscibility gap between body-centered cubic (bcc) Fe and B2-NiAl in the Fe–Ni–Al system enables precipitation hardening of the bcc Fe matrix phase by the formation of B2-ordered NiAl precipitates over a wide range of

compositions and volume fractions [2–4]. Due to a small lattice parameter mismatch between the NiAl precipitates and the ferritic matrix (0.2887 and 0.2866, respectively [5]), a high volume fraction of coherent precipitates can be obtained, mirroring the γ/γ' structure found in Ni-based superalloys [6,7]. This characteristic two-phase microstructure endows these alloys with excellent coarsening and creep resistance at high temperature.

The addition of molybdenum to Fe–Ni–Al alloys (near Fe–10Ni–15Al and Fe–3Ni–10Al at.% composition) was found to improve coarsening resistance of the B2 (Ni,Fe)Al precipitates [8–10]. The partitioning of molybdenum to the Fe matrix leads to an increase in lattice parameter and thus a reduction of the lattice mismatch. This in turn reduces the elastic contribution to the precipitate

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energy [8]. In Mo-free alloys with high B2 precipitate volume fractions, the precipitates undergo morphological changes, from spherical to cuboidal, and to parallelepiped rectangular, during high temperature aging [8]. In contrast, B2 precipitates in Mo-containing alloys remain spherical and evidence of coalescence is less frequently observed, which was assigned to Mo segregating to the matrix/precipitate interfaces [7]. Molybdenum also improves grain boundary cohesion in ferritic steels [11], thus improving their ductility.

To improve oxidation resistance, Cr was added to these creep-resistant Fe–Ni–Al and Fe–Ni–Al–Mo alloys containing B2-ordered coherent NiAl precipitates [12–15]. Room-temperature mechanical properties of a Fe–10Cr–10Ni–3.4Mo wt.% alloy containing a range of Al concentrations (from 3 to 10 wt.%) were studied and it was found that the Fe–10Cr–10Ni–6.5Al–3.4Mo wt.% alloy (with additions of 0.25Zr–0.005B (wt.%), labeled FBB8) exhibits an optimized combination of superior strength and hardness, and reasonable ductility [13]. Extensive characterizations of this ferritic alloy, after homogenization at 1200 °C for 0.5 h and aging at 700 °C for 100 h (both terminated by air-cooling) were done by transmission electron microscopy (TEM) and atom-probe tomography (APT) [16]. They reveal the coherent B2-ordered (Ni, Fe)Al precipitate structure with an average radius of 65 nm and a volume fraction of 18% [16]. The high-temperature creep properties of this alloy, however, are unknown. To our knowledge, only one tensile creep experiment at 700 °C [14], was conducted for stresses ranging from 60 to 150 MPa, where creep strain rates vary from 10^{-8} to 10^{-5} s $^{-1}$.

In this paper, we present a systematic creep study of an alloy very close to FBB8, in the temperature range 600–700 °C and stress range 30–300 MPa. We find a temperature-dependent threshold stress, below which no measurable creep strain is observed. Using this threshold stress, we determine that the true stress exponent is ~ 4 , which is consistent with prior creep studies of ferritic Fe–Cr–Ni–Al steels [12,15], and a true creep activation energy of 243 ± 37 kJ mol $^{-1}$, which is equal to that for lattice diffusion of the ferritic matrix. Based on these results, possible creep mechanisms are discussed. The microstructure of the alloy in the as-heat-treated condition and after an interrupted creep test is analyzed by TEM. The presence of Zr-rich phases and nanoscale secondary precipitates is established and the precipitate size and inter-precipitate distance distribution of the primary B2-(Ni, Fe)Al precipitates is determined from superlattice dark-field TEM images. The precipitate–dislocation interaction after the interrupted creep test reveals that dislocation climb is most commonly observed where a strong interaction of dislocations and interfaces can be established.

2. Experimental procedures

The alloy chemical composition was measured by direct-current plasma mass spectroscopy performed by ATI Wah

Chang (Albany, OR) as Fe–10.4Cr–10.2Ni–5.5Al–3.4Mo–0.31Zr–<0.001B wt.%, or Fe–10.7Cr–9.3Ni–10.9Al–1.9Mo–0.18Zr–<0.005B at.% (close to the FBB8 composition of Fe–10Cr–10Ni–6.5Al–3.4Mo–0.25Zr–0.005B wt.% studied in previous work) [13,14,16]. These alloys were drop-cast by Sophisticated Alloys (Butler, PA). The cast billet was homogenized at 1200 °C for 0.5 h in evacuated and sealed quartz tubes, air-cooled, aged at 700 °C for 100 h in air and air-cooled onto a ceramic block. After the heat treatment, compression and tensile creep specimens were fabricated by electro-discharge machining. The compression specimens had dimensions of either $8 \times 8 \times 16$ mm or $10 \times 10 \times 20$ mm. The threaded dog-bone tensile specimens had a gage length and cross-section diameter of 35 and 6.6 mm, respectively.

Constant load compressive and tensile creep experiments were performed in air at 600, 650 and 700 °C. The samples were heated in a three-zone furnace, with the temperature controlled within 1 °C by a thermocouple placed within 10 mm of the sample. For compressive creep experiments, the specimen was placed between boron-nitride-lubricated platens, made of either sapphire or silicon carbide. Sample displacement was measured on the load train with a linear variable displacement transducer (LVDT) with a resolution of 10 μ m. The applied load was increased when a well-defined minimum strain rate was observed. For tensile creep experiments, the specimen was screwed on grips. An extensometer was attached to the two ends of the sample gage length. An LVDT was used to measure displacement with a resolution of 10 μ m. The cumulative creep strain per sample, in both compressive and tensile tests, was kept below 10%, well under the value where barreling or necking might occur. Strain rates at a given stress were calculated by measuring the slope of the strain vs. time in the steady-state creep regime.

The microstructure and dislocation–precipitate interactions were analyzed in the heat-treated condition before and after creep deformation. A tension creep test with a stress level of 107 MPa at 700 °C was interrupted after 2.3% tensile strain and the sample was air-cooled under load. Discs with a diameter of 6 mm and a thickness of 1 mm were cut from the gage length perpendicular to the loading axis. The discs were mechanically ground to a final thickness of 100 μ m using SiC paper. TEM samples with a diameter of 3 mm were cut by a rotary disc cutter. The samples were thinned to electron transparency by electro-polishing using an electrolyte of 20% perchloric acid in methanol at –30 °C. Superlattice dark-field TEM imaging and energy-filtered imaging was performed in a Zeiss Libra200MC microscope at an acceleration voltage of 200 kV. High angle annular dark-field (HAADF) and annular dark-field (ADF) or diffraction contrast (DC) scanning transmission electron microscopy (STEM) imaging was done in a FEI Titan 80-300 microscope equipped with a Fishione HAADF detector and a ChemiSTEM system for high resolution energy dispersive X-ray spectroscopy (EDS). For STEM imaging a semi-convergence

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