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Tensile creep of directionally solidified NiAl–9Mo in situ composites

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Abstract

Well-aligned Mo fiber-reinforced NiAl in situ composites were produced by specially controlled directional solidification. The creep behavior parallel to the growth direction was studied in static tensile tests at temperatures between 900 °C and 1200 °C. A steady-state creep rate of $10^{-6}\,s^{-1}$ was measured at 1100 °C under an initial applied tensile stress of 150 MPa. Compared to binary NiAl and previously investigated NiAl–Mo eutectics with irregularly oriented Mo fibers, this value demonstrates a remarkably improved creep resistance in NiAl–Mo with well-aligned unidirectional Mo fibers. A high-resolution transmission electron microscope investigation of the NiAl/Mo interface revealed a clean semi-coherent boundary between NiAl and Mo, which enabled an effective load transfer from the NiAl matrix to the Mo fibers, and thus leads to the remarkably increased creep strength. The stress exponent, n, was found to be between 3.5 and 5, dependent on temperature. The activation energy for creep, Q_c, was measured to be 291 ± 19 kJ mol^{-1}, which is close to the value for self-diffusion in binary NiAl. Transmission electron microscopy observations substantiated that creep occurred by dislocation climb in the NiAl matrix. The Mo fiber was found to behave in a quasi-rigid manner during creep. A creep model for fiber-reinforced metal matrix composites was applied for an in-depth understanding of the mechanical behavior of the individual components and their contribution to the creep strength of the composite.

Keywords: Fiber reinforced composite; Creep resistance; Interface

1. Introduction

Ni-base superalloys have been used as high-temperature (HT) structural materials in gas turbine engines for more than half a century. Although a remarkable gain of performance has been achieved through alloy modifications and single crystal growth, further improvements of the HT properties of Ni-base superalloys have come to a limit since the operating temperatures (~1230 °C = $0.85T_m$) in modern gas turbine engines are approaching the melting point T_m of these alloys. However, even higher operating temperatures are still desired to enhance the efficiency of power plants.

With an attractive combination of a high melting point ($T_m = 1638$ °C) and low density ($\rho \sim 6\,g\,cm^{-3}$) [1] compared to those of Ni-base superalloys ($T_m = 1280$–1350 °C, $\rho = 8$–$8.5\,g\,cm^{-3}$), NiAl has been considered as a potential candidate for structural applications at HT to replace Ni-base superalloys. However, besides its low ductility at ambient temperature, in particular its intrinsic poor creep resistance at HT has prevented its structural application as load-bearing components such as turbine blades. Incorporating continuous reinforcements like refractory metallic fibers or lamella in NiAl to form a composite has been proven to be an effective way of strengthening at high temperatures. The mechanical properties of NiAl–refractory metal eutectic in situ composites like NiAl–9 at.% Mo have been extensively investigated by many researchers since the early 1990s [2–8]. Misra et al. [2] and Joslin [4] have demonstrated the remarkable improvement of mechanical properties of NiAl–9Mo alloys produced by directional solidification compared to binary NiAl. Joslin reported a steady creep rate of $10^{-6}\,s^{-1}$ at

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1300 K under a stress of 80 MPa compared to binary NiAl having the same creep rate at the same temperature only at a stress of 20 MPa, although the microstructure of the investigated alloys consisted mainly of cellular structures with irregularly aligned Mo fibers. Recently, with the development of directional solidification (DS) technology, a much steeper temperature gradient (>10 °C mm^{-1}) can be realized during the DS processing. This maintains a planar solidification front and thus generates a well-aligned fibrous eutectic microstructure of NiAl–9Mo with the Mo fibers aligned parallel to the growth direction. Although such NiAl composites possess anisotropic mechanical properties, they exhibit extraordinary strength in the fiber direction. Accordingly, Bei and George [6] and Hu et al. [8] have measured a remarkably enhanced HT tensile strength along the growth direction in DS NiAl–9Mo. Dudová et al. [7] also reported an extremely high compressive creep resistance of this material with a steady creep rate of 3×10^{-6} s^{-1} at 900 °C at a compressive stress of 200 MPa. This excellent HT strength makes DS NiAl–9Mo a promising candidate material for structural applications like turbine blades in the next generation of gas turbine engines. However, in spite of this significant progress, the reported creep investigations on NiAl–9Mo with well-aligned fibers, especially the tensile creep behavior at HT (>1000 °C), are still rare although such data are of vital importance not only for engineering design purposes but also in an academic sense for understanding the mechanisms and physics of the creep behavior to allow quantitative predictions of in-service material performance in these NiAl–9Mo composites.

Therefore, the purpose of the current study was to explore the creep behavior of unidirectional Mo fiber-reinforced NiAl composites and to analyze the individual contributions of the Mo fiber and NiAl matrix to the composite creep behavior.

2. Experimental procedure

Ni–44.5Al–9.2Mo (in at.%) buttons were produced by inductive melting from high purity (>99.99%) Ni, Al and Mo. Thereafter, the NiAl–Mo buttons were melted in an alumina crucible and directionally solidified in a Bridgman furnace with a specially controlled liquid metal cooling under protective argon atmosphere. A detailed description about the directional solidification process can be found elsewhere [9]. The growth rate of the ingot was $R = 20$ mm h^{-1}. The local temperature gradient produced by metal cooling was ~11.7 °C mm^{-1}. The NiAl–9Mo rods after DS were 8 mm in diameter and 30 mm in length. Their composition Ni–43.8Al–9.5Mo (in at.%) was measured by X-ray energy dispersive spectroscopy (EDX) attached to a scanning electron microscope (SEM). For simplification, this composition will be designated as NiAl–9Mo in the following.

The creep behavior of DS NiAl–9Mo ($R = 20$ mm h^{-1}) composites was examined by tensile creep tests at 900–1200 °C at constant load with an initial tensile stress of 80–220 MPa in vacuum (2×10^{-2} Pa) with a mechanical testing machine (Schenk Hydropuls PSB250). Dog-bone-shaped tensile specimens with a gage volume of $1 \times 1.3 \times 18.2$ mm^3 were cut from the DS rods by electron discharge machining (EDM). The axis of the tensile specimens was parallel to the growth direction, and thus also to the fiber axis. Prior to the creep test, some dog-bone-shaped specimens were mechanically polished on one side. After the creep test this polished surface was examined by SEM (FEG LEO1530) using secondary electron imaging (SEI) and backscatter electron imaging (BSEI) for fractography of Mo fibers and the NiAl matrix. The local chemistry of the samples was analyzed by EDX/SEM (Oxford Link ISIS/JEOL JSM-7000F).

The microstructure after creep deformation was analyzed by transmission electron microscopy (TEM). Owing to the too small cross-section of the creep tensile specimens (1×1.3 mm^2), the TEM specimens were prepared from samples deformed by compression creep on DS NiAl–9Mo with a cylindrical shape of ∅4 mm × 6.25 mm. The compression direction was parallel to the cylinder axis and growth direction. The compression test was performed at 1100 °C under an initial compressive stress of 150 MPa in vacuum. Since thermal residual stresses in the NiAl and Mo phase [8] are already relieved at elevated temperatures above 1000 °C, in which the stress free temperature is about 577 °C for NiAl [10] and 871–982 °C for Mo [11], and moreover, at the temperatures above 1000 °C the running sliding systems for tension and compression are basically in the same; thus it is considered that there is no essential difference in dislocation structures between the specimens subjected to tensile and compressive deformations and therefore it will not influence the formed dislocation structure and the late discussion about the creep behavior and mechanisms of NiAl–9Mo composites based on the microstructures obtained by TEM observation with compressive samples. The TEM observation was carried out on samples cut parallel to the cylinder axis/growth direction. The TEM foils were prepared by ion milling (Duo Mill™ 600 of Gatan). The disk-shaped specimens (∅3 mm × 0.1 mm) were firstly dimpled to a thickness of ~20 μm in the disk center and then ion-milled. The acceleration voltage and incidence angle of the argon ion beam were 5 kV and 12°, respectively. The TEM foils were then imaged in an analytic TEM (JEM FX2000-II) for routine analysis and an FEG Tecnai F20 for high-resolution TEM (HRTEM) imaging. The HRTEM micrographs were treated by Gatan Digital-Micrograph® 3.9 to reveal interface dislocations at the NiAl/Mo interface. Fine precipitates and the local chemistry in the Mo fibers were analyzed by scanning transmission electron microscopy (STEM)/EDX and STEM/high angle annular dark field (HAADF) imaging.