Phase-specific deformation behavior of a NiAl–Cr(Mo) lamellar composite under thermal and mechanical loads

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ABSTRACT

Phase-specific thermal expansion and mechanical deformation behaviors of a directionally solidified NiAl –Cr(Mo) lamellar *in situ* composite were investigated by using real-time *in situ* neutron diffraction during compression at elevated temperatures up to 800 °C. Tensile and compressive thermal residual stresses were found to exist in the NiAl phase and Cr_{ss} (solid solution) phase, respectively. Based on the evolution of lattice spacings and phase stresses, the phase-specific deformation behavior was analyzed qualitatively and quantitatively. Estimates of phase stresses were derived by Hooke's law on the basis of a simple method for the determination of stress-free lattice spacing in *in situ* composites. During compressive loading, the NiAl phase yields earlier than the Cr_{ss} phase. The Cr_{ss} phase carries much higher stress than the NiAl phase, and displays consistent strain hardening at all temperatures. The NiAl phase exhibits strain hardening at relatively low temperatures and softening at high temperatures. During unloading, the NiAl phase yields in tension whereas the Cr_{ss} phase unloads elastically. In addition, post-test microstructural observations show phase-through cracks at room temperature, micro cracks along phase interfaces at 600 °C and intact lamellae kinks at 800 °C, which is due to the increasing deformability of both phases as temperature rises.

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1. Introduction

As promising candidates for high temperature structural applications in energy conversion systems [1,2], NiAl-based eutectic composites by directional solidification were actively investigated in the early 1970s [3,4], and have drawn renewed interest since 1990s [5–10]. The composites can provide significant enhancement in room temperature toughness and elevated temperature creep strength over monolithic NiAl. The combination of intermetallic compound NiAl and a refractory metal phase, such as Cr, is a eutectic system that has been well characterized in terms of processing microstructures and macroscopic mechanical properties. By directional solidification, *in situ* composites are derived where the NiAl matrix phase and the fibrous refractory metal phase are well aligned parallel to the growth direction of [100] for both phases [3–6,8]. With small additions of impurity elements such as

Mo and V, the fiber-matrix morphology changes to lamellar one with the two alternating phases, and the [100] growth direction to [111] [3,5,6]. In either case, the room temperature toughness and high temperature creep strength of the composite are significantly improved compared to those of single-phase NiAl. For example, the fracture toughness of fibrous NiAl–34Cr and lamellar NiAl–28Cr–6Mo (all compositions in this paper are in atomic percent) can reach ~ 20 MPa$\sqrt{m}$ and ~ 24 MPa$\sqrt{m}$ at room temperature, respectively, as opposed to ~ 6 MPa$\sqrt{m}$ for polycrystalline NiAl or the conventionally NiAl–34Cr cast alloy [5]. The creep strength of the fibrous NiAl–34Cr and lamellar NiAl–28Cr–6Mo at a strain rate of 1×10^{-6} s^{-1} at 1017 °C can achieve ~140 MPa and ~160 MPa, respectively, compared to ~20 MPa for a [001] single crystal NiAl [5].

The reinforcing mechanisms are generally associated with the well-aligned microstructure and superior mechanical properties of the refractory metal phase. Heredia et al. [11] summarized three toughening mechanisms, namely, crack trapping, crack renucleation and ductile-phase bridging, for the NiAl-based fibrous/ lamellar composites. Yang et al. [6] regarded the improved room temperature fracture toughness in a layered NiAl–Cr(Mo)

composite as the result of plastic rupture of the Cr solid solution (Cr_{ss}) layers and the domain-to-domain variations in the lamellar fracture mode. In our previous study [12], we found in a NiAl−Cr(Mo) nano-layered composite that the Cr_{ss} layers with thickness of ~400 nm can bear very high stresses and deform plastically before fracture, unlike in bulk form, where it fractures in a relatively brittle fashion at significantly lower stresses. While the above findings can help understand the mechanisms for room temperature toughness improvement, published literature concerning the mechanisms for high temperature creep strength enhancement are sparse. Although it is expected that the refractory metal phase should play a key role in such enhancement at elevated temperatures, the knowledge of phase-specific deformation behavior are still needed to elucidate the detailed mechanisms.

The development of *in situ* X-ray/neutron diffraction techniques allow investigation of the stress−strain response of the composite and its constituent phases simultaneously [13−20]. By measuring the elastic strain in each phase, the stress partitioning between the constituent phases can be determined using Hooke's law and their contributions to the overall mechanical behavior of the bulk can be evaluated. In our prior study [12], real-time *in situ* neutron diffraction was successfully employed to estimate the phase-specific stress increments upon the applied stresses in a nano-layered NiAl−Cr(Mo) lamellar composite. Note that the thermal residual stresses are expected during directional solidification due to differences in the coefficients of thermal expansion (CTE) of constituent lamellae, which will be further discussed in this study. The total phase-specific stresses, i.e. the sum of stress increments and thermal residual stresses, were not derived, though there are two typical methods to determine the thermal residual stress. One direct method is to determine the residual elastic strain with knowledge of a reference lattice parameter of stress-free NiAl and Cr_{ss} powders. The stress-free powders are hard to obtain from the bulk composite since the powders should be nano-scale to avoid involving any nano-layers. The other indirect route is analytical solution based on total stress balance with knowledge of CTE of individual phase as well as the joining temperature at which the two phases begin to bond in solid state. The joining temperature is actually difficult to define and measure. In order to precisely examine the phase-specific deformation behavior of the composite, it requires quantitative understanding of the thermal residual stresses in individual phases in the first place.

In the present study, real-time *in situ* neutron diffraction measurements were performed during compression of a NiAl−Cr(Mo) lamellar composite to elucidate the temperature-dependent phase-specific deformation behavior, shedding light on the reinforcing mechanisms of the composite over monolithic NiAl. Post-test microstructures were also examined by a scanning electronic microscope (SEM) to acquire the temperature-dependent deformation modes.

2. Materials and experimental procedures

The NiAl−Cr(Mo) composite with the composition of 33Ni−33Al−31Cr−3Mo was produced by directional solidification in a high-temperature optical floating-zone furnace at a growth rate of 40 mm h^{-1}. Two other materials, monolithic NiAl with Cr solid solution [referred to as NiAl(Cr) henceforth] and a Cr solid solution alloy [referred to as Cr(NiAl, Mo) henceforth], were also produced using the same method to compare their thermal expansion behaviors in monolithic form with those of constituent phases in lamellar form, thus shedding light on the thermal residual stress state inside the composite. The compositions of NiAl(Cr) and Cr(NiAl, Mo) are 48Ni−44Al−8Cr and 78Cr−7Mo−8Ni−7Al, respectively, which are similar to those of the

constituent phases (lamellae) in the composite [21]. Details of the alloy fabrication and directional solidification processes can be found elsewhere [8,22], where similar procedures were used to produce other eutectic composites. The microstructures of the composite as examined in our prior study [12] show well-aligned lamellar morphologies both parallel and perpendicular to the growth direction (axial and transverse direction, respectively). The thicknesses of the NiAl and Cr_{ss} layers are ~800 nm and ~400 nm, respectively. Columnar grains were observed in the single-phase NiAl(Cr) and Cr(NiAl, Mo), indicating their polycrystalline morphology.

Cylindrical samples with the dimension of 6 mm in diameter and 12 mm in length were cut from the directionally solidified rods along the growth direction for *in situ* compression and heating experiments. The *in situ* compression tests were conducted on the VULCAN Engineering Materials Diffractometer at the Spallation Neutron Source, Oak Ridge National Laboratory (SNS, ORNL), as schematically illustrated in Fig. 1, where the loading direction is parallel to the growth direction <111> of both phases in the composite. The VULCAN diffractometer was described in detail elsewhere [23], but in brief, the loading axis is horizontal and at 45° to the incident neutron beam, allowing simultaneous measurement of lattice spacing both parallel and perpendicular to the loading direction, i.e. the longitudinal and transverse directions. Displacement control mode was used for the loading part at a rate of 2 mm h^{-1}, corresponding to an engineering strain rate of about 5×10^{-5} s^{-1} (the engineering strains were calculated as the ratio of the crossheads displacement to the initial sample length), while force control mode was employed for the unloading part with a period of 30 min. These rates were chosen to ensure adequate neutron diffraction data in a quasi-continuous manner as will be discussed later. For the high-temperature tests, the sample was heated by copper induction coils at a rate of 10 °C min^{-1}, and then held at the target temperature for 10 min before compression. During the heating and holding process, a small force of −50 N was applied on the sample to allow expanding under nearly constraint-free condition. A total of 7 samples were tested, including 6 *in situ* compression tests and an *ex situ* compression tests. The *in situ* compression tests were conducted at five different temperatures: 20, 200, 400, 600, and 800 °C. Two *in situ* tests were conducted at 20 °C for the purpose of repeatability validation, one with a large stain of ~3.5% and the other with a small strain of 0.5%. The neutron diffraction measurements of the two tests show reasonable consistency, which will be given later. Thus only one *in situ* test was conducted at each of the other four temperatures. One *ex situ*
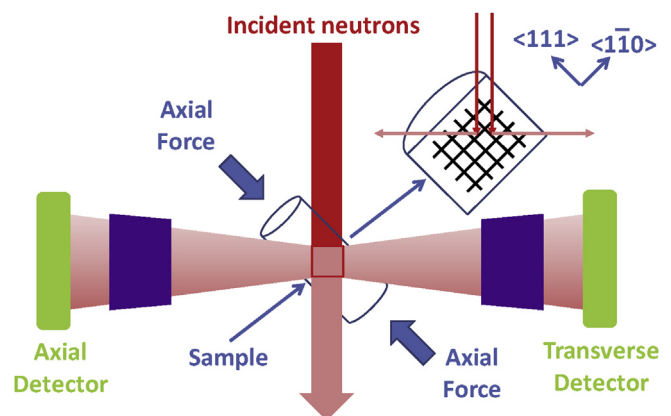

Fig. 1. Schematic of *in situ* neutron diffraction during compression of the NiAl−Cr(Mo) composite.