

Long term stability and mechanical properties of Al_2O_3 –NiAl composites reinforced with partially fragmented long fibers

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ABSTRACT

Long fiber reinforced NiAl composites, fabricated by high temperature processing, are designed for long term thermal exposure during service. This treatment causes partial fragmentation of the long fibers, which degrades the mechanical performance of the composite. We investigated the mechanical properties of Al_2O_3 –NiAl long fiber composites, fabricated by hot pressing and exposed to 2000 h annealing at 700 °C and 1100 °C. The composites showed basically good thermal stability during annealing except for grain coarsening of NiAl. Owing to fiber fragmentation during cooling after composite fabrication, the mechanical performance of the composites is determined by the load partitioning in a mixture of long and broken fibers. We propose a method to calculate the strength of such composites, based on microstructural information and composite fracture mechanisms. The theoretical predictions show good agreement with experimental results.

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1. Introduction

In the past several decades considerable research has been carried out on the intermetallic phase NiAl with respect to its potential application in advanced gas turbine engine components [1,2]. In spite of its high melting point (1638 °C), low density (5.88 g/cm³), good thermal conductivity, and excellent oxidation resistance, the limited strength and creep resistance at high temperatures as well as poor toughness at room temperature remain as the main obstacles for structural applications of NiAl. A method to overcome these drawbacks is to reinforce NiAl with ceramic particles, whiskers, or fibers. Among the numerous possible reinforcements, a single-crystalline Al_2O_3 continuous fiber (sapphire fiber) stands out by its excellent thermal stability and creep resistance at high temperatures.

Bowman et al. [3] examined the mechanical properties of NiAl composites with 25% (volume fraction) sapphire fibers over the temperature range of 25 °C to ~1000 °C, and Hu et al. [4–6] investigated the chemistry, microstructure as well as the mechanical properties up to 1100 °C for composites with 50% fiber content. In all of these investigations, fiber fragmentation occurred, owing to the mismatch of thermal expansion of fiber and matrix. Primarily because of fiber fragmentation during fabrication, the degraded

mechanical properties of the composites, especially the poor composite strength, became the main problem of such composites.

Considering the mechanics of composites reinforced with fragmented fibers, the shear-lag model has been used to calculate the effective length, stress distribution and transfer, as well as stress concentrations, following the initial shear-lag analysis by Cox [7]. Zeng [8] and Landis [9] studied the fiber length under effective loading and the stress concentration in the composites with short fibers, assuming the matrix to support the tensile stress; Xia [10] concentrated on the analysis of the stress transfer. All of these approaches proved the shear-lag model to be accurate for analyzing the composite constituent properties, only if a more detailed micromechanical analyses and a clear understanding of their implications for the mechanical behavior of the composites were supplied in advance. With an understanding of the fracture behavior and the micromechanics of NiAl composites by correlating microstructure, interface structure, and composite mechanical properties, it is feasible to apply the shear-lag model for predicting the tensile strength of such composites.

Due to the in-service requirements of NiAl composites, the purpose of the current investigation was to examine the long term thermal stability with respect to the microstructure, interface structure, and mechanical properties of NiAl composites. It was the objective to clarify the involved micromechanics and fracture mechanisms under a tensile stress, so that the strength of NiAl composites reinforced by a mixture of long and fragmented sapphire fibers could be quantitatively predicted based on a modified Rule of Mixtures (ROM) and a shear-lag analysis.

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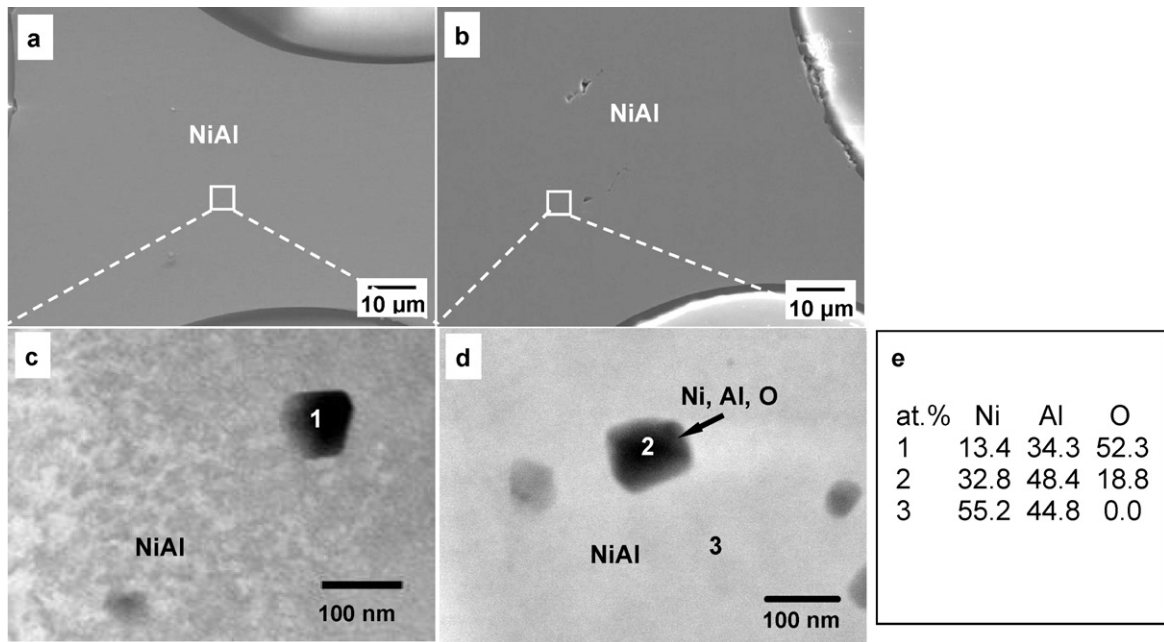


Fig. 1. Matrix chemistry in the cross-section of a Al_2O_3 –NiAl composite: (a) secondary electron image (SEI) of the as-fabricated state; (b) SEI of the 700 °C-annealed state; (c) and (d) corresponding STEM micrographs of (a) and (b); (e) results of semi-quantitative STEM/EDX analysis of (c) and (d).

2. Experimental procedure

The composites used in the current study were fabricated from matrix coated fibers by diffusion bonding. The procedure was as follows: continuous single crystalline α - Al_2O_3 (sapphire) fibers with a diameter of ~ 130 μm supplied by Saphikon were firstly coated with NiAl by a physical vapour deposition (PVD) process. A fiber volume fraction (V_f) of $\sim 50\%$ can be obtained by adjusting the thickness of the NiAl-coating to ~ 27 μm. The coated fibers were then placed into a channel-die and hot pressed at 1300 °C for 1 h with a pressure of 40 MPa in vacuum (5×10^{-3} Pa). Under these experimental conditions, composites with fully densified NiAl matrix and uniformly distributed Al_2O_3 fibers were produced. The as-fabricated sample size was 2 mm $\times$ 7 mm $\times$ 40 mm. For an examination of the long term thermal stability, some of the as-fabricated NiAl composites were further annealed at 700 °C and 1100 °C, respectively, for 2000 h. In the following, composite samples, only subjected to diffusion bonding, will be referred to as-fabricated samples; samples, which were annealed after diffusion bonding, will be termed annealed samples.

Both the as-fabricated and annealed composites were microcharacterized by electron microscopy, like scanning electron microscopy (SEM), scanning transmission electron microscopy (STEM) and energy dispersive X-ray spectroscopy (EDX). After a routine metallographic preparation of the sample surface, the composite samples were observed both along the transverse and the longitudinal direction in a SEM. The specimens for TEM studies were prepared by focused ion beam (FIB) milling. The detailed preparation procedure for TEM foils can be found elsewhere [4].

The interfacial shear strength of the composites was measured by fiber push-out tests at room temperature. For these tests, a slice with a thickness of ~ 1 mm was firstly cut out perpendicular to the fiber axial direction. The slice was then ground and polished to a final thickness of 0.3 mm and eventually glued onto a special specimen stage. The fiber push-out tests were carried out with a microhardness testing machine (Hahn & Kolb), equipped with a special diamond indenter. The load and fiber displacement during the fiber push-out tests were recorded by a PC based data acquisition system (HBM Hotting Balwin Messtechnik

MGC plus). The interfacial shear strength of the composites was calculated as:

$$\tau_{\text{debond}} = \frac{F_{\text{debond}}}{2\pi rh} \quad (1)$$

where τ_{debond} is the interfacial shear stress for complete debonding, also referred to as interfacial shear strength, F_{debond} is the maximum axial load for interface debonding, r is the radius of the tested fiber, and h is the thickness of the specimen.

The tensile tests were carried out on both as-fabricated and annealed composites at 700 °C and 1100 °C, using a servohydraulic mechanical testing machine (Schenk Hydropuls PSB250) in vacuum (5×10^{-3} Pa). The tensile testing temperatures were adjusted to the prior annealing temperatures, e.g. a sample annealed at 700 °C was subsequently tensile deformed at 700 °C as well. An axial extensometer was located on the sample holder but not directly on the sample surface owing to the small sample gauge length (28 mm). Tensile deformation proceeded with a strain rate of $1 \times 10^{-4} \text{ s}^{-1}$ until sample failure. For each composite state, at least three tests were conducted to ensure good statistics of the tensile testing results. After the tensile tests the fracture surfaces were examined by means of SEM/EDX.

3. Results

3.1. Microcharacterization

3.1.1. Matrix

The microstructures perpendicular to the axial fiber direction of the as-fabricated as well as the annealed composites at 700 °C are shown in Fig. 1a and b, respectively. Some particles with dark or grey contrast and a size of several ten nanometers can be observed in the NiAl matrix, as revealed by STEM/HAADF micrographs in Fig. 1c and d. EDX analysis identified these particles as (Ni,Al)-oxides (see Fig. 1e), which were generated as reaction products of NiAl and residual oxygen in the matrix during hot pressing and annealing [4]. The amount of oxide particles in the NiAl matrix increased with rising annealing temperature. No other chemical reactions or phase transformations were detected in the

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