Acta Materialia 155 (2018) 236–244

Contents lists available at ScienceDirect

Acta Materialia

journal homepage: www.elsevier.com/locate/actamat

Full length article

Experimental and modeling study of compressive creep in 3D-woven Ni-based superalloys

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ARTICLE INFO

Article history:
Received 3 January 2018
Received in revised form
29 May 2018
Accepted 31 May 2018
Available online 14 June 2018

Keywords:
Nickel alloys
3D weaving
Finite element analysis
High-temperature mechanical behavior
Creep buckling

ABSTRACT

Micro-architectured Ni-based superalloy structures, with Ni-20Cr-3Ti-2Al (wt.%) composition and γ/γ′-microstructure, are created by a multi-step process: (i) non-crimp orthogonal 3D-weaving of ductile, 202 μm diameter Ni-20%Cr wires, (ii) gas-phase alloying with Al and Ti, (iii) simultaneous transient-liquid phase (TLP) bonding between wires and homogenization within wires *via* interdiffusion, (iv) solutionizing to create a single-phase solid solution, and (v) aging to precipitate the γ′ phase. The creep behavior of these 3D-woven γ/γ′ nickel-based superalloys is studied under uniaxial compression *via* experiments at 825 °C and *via* finite element (FE) analysis, using a 3D model of the woven structures obtained through X-ray micro-tomography. The creep strain rate for the woven Ni-based superalloy is higher than that for the bulk superalloy due to the lower solid volume fraction of the woven structure, while the creep exponents are identical. The compressive creep behavior is sensitive to the geometry of the woven structures: fewer wires perpendicular to the load and fewer bonds between wires cause lower creep resistance of the woven structure, due to a reduction in load transfer from the longitudinal wires (which are primarily load-bearing) and the perpendicular wires. Creep buckling of longitudinal wires drastically reduces creep resistance of the woven structure, confirming the importance of maintaining longitudinal wires vertical and parallel to the uniaxial compression direction. Finally, reducing wire cross-section, *e.g., via* oxidation, reduces creep resistance. The oxidation kinetics of the wire structures at 750, 825, and 900 °C displayed parabolic rate constants comparable to commercial Ni-based superalloys, but indicates that up to 35% of the wire cross-section is oxidized after 7 days at 825 °C, such that oxidation-resistant coatings are needed for long-term use in oxidative environment.

1. Introduction

In recent years, artificially structured materials with stochastic architectures have received significant interest [1,2], not only because of their desirable properties such as low density, and high specific strength, stiffness, and surface area [3–5], but also for their broad range of non-structural applications, including heat/sound absorption, damping, filters, catalyst substrates, and fuel cell interconnects [6–9]. An important characteristic of these structured materials is their ability to optimize various properties in an isotropic or anisotropic manner by topologically tailoring nano/micro-architectures [10]. Some examples of the structured materials with regular micro-architectures are micro-lattice structures [11,12], woven structures [5,13], and topology-optimized lattices [14–16].

Nickel-based superalloys are well suited for structural applications at elevated temperatures up to approximately 1050 °C [17–19]. Fabrication of topologically tailored micro-architectures from Ni-based superalloys can achieve optimized combination of high strength/stiffness and high heat transfer properties for efficient cooling for thermo-structural applications [5,9,20]. Zhao et al. [9] fabricated topologically optimized 3D-woven architectures from Cu and Ni-20Cr wires and examined their permeability in three orthogonal directions by employing both computational and experimental approaches. They showed that the optimized structures achieved nearly four times higher permeability with minimal reduction in stiffness. Erdeniz et al. [5] reported a process, which is employed in the present work, for the fabrication of 3D-woven Ni-

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https://doi.org/10.1016/j.actamat.2018.05.067
1359-6454/

based superalloys with metallurgical bonding between wires by bypassing significant manufacturing hurdles such as limited ductility and difficulty in joining that comes with superalloy wires. They decoupled the weaving and alloying steps by using ductile Ni-20Cr precursor wires for 3D-weaving and post-textile alloying *via* pack cementation. Additionally, Erdeniz et al. [20] demonstrated that the bonding between wires could lead to higher strength at ambient temperature and they developed a technique to use transient-liquid-phase (TLP) bonding for this purpose. In particular, understanding the mechanical behavior of the woven structures at elevated temperatures is very important for thermo-structural applications of Ni-based superalloys with topologically optimized architectures. There are no reports on the high-temperature mechanical behavior, especially creep deformation behavior, of the woven structures consisting of Ni-based superalloy wires.

In the present work, we employ continuum-mechanics simulations to probe creep deformation of 3D-woven structures which allow for a systematic variation of topological and architectural features much more easily than *via* experiments. Based on 3D models of the woven structures previously obtained from X-ray micro-tomography [5] and using experimental creep data of the bulk alloy as input, the creep deformation behavior of the woven Ni-based superalloys is simulated under uniaxial compression *via* finite element (FE) analysis, which is then compared with experimental creep strain data. We also examine the creep deformation behavior by digitally modifying the geometry of the woven structures, such as the number of Z wires and the number of bonds between the wires. Additionally, we consider creep buckling as a potential deformation mechanism, which can cause lower creep resistance of the woven structure under uniaxial compression at elevated temperatures, by employing both computational and experimental approaches.

2. Experimental procedures

2.1. Fabrication of bulk and 3D woven superalloys

A bulk Ni-based superalloy button with a composition of Ni-20Cr-3Ti-2Al was arc melted from Ni-20Cr pellets (99.9% purity), Ti sponges (99.9% purity), and Al pellets (99.9% purity). The button was flipped over five times, after each arc melting sequence that lasted 30 s. The resulting alloy was then solutionized at 1200 °C for 2 h and aged at 900 °C for 16 h. Creep samples were cut out of this button *via* electro-discharge machining (EDM).

The other type of materials used in this study are non-crimp 3D orthogonal woven structures fabricated from soft-annealed Ni−20Cr wires, with a diameter of 202 μm. A detailed description of the weaving process is given in Ref. [5]. Woven samples, cut to a length of 15 mm, a width of 25 mm, and a thickness of 3.5 mm (corresponding to 11 wire layers, with an approximate mass of 7 g), were buried in a pack mixture consisting of 57 wt% Al_2O_3 powders (20−50 μm particle size) as filler, 30 wt% Ti powders (99.5% purity, −325 mesh) and 10 wt% Raney Ni precursor powders (Ni−50 wt% Al, 150 μm particle size) as Al and Ti sources, and 3 wt% NH_4Cl powders (100 μm particle size) as activator. Approximately 40 g of pack was poured in a steel retort, where the internal pressure rises at elevated temperatures, with the cut specimen placed at the center of the retort and fully buried in the pack. The inner wall of the steel retort was spray-coated with boron nitride to minimize contamination. The retort was placed at the water-cooled end of a tube furnace that was heated to 1000 °C. After flushing the furnace tube with Ar for 15 min, the retort was pushed into the hot zone of the furnace, where it was held for 60 min. The retort was then pulled back to the water-cooled end of the tube and cooled there for 15 min. The specimens were removed from the pack and ultrasonicated in acetone for 2 h to remove all pack remnants.

Four layers of the Al- and Ti-coated woven structures were stacked, sandwiched between two superalloy (Inconel X750, 1 mm thickness) sheets, and placed in a jig to hold the sandwich structure together. The jig was then placed in a vacuum furnace and the following heat treatment was carried out: (i) transient liquid phase bonding at 1140 °C for 30 min, (ii) homogenization at 1100 °C for 48 h, (iii) solutionizing at 1200 °C for 2 h, and (iv) aging at 900 °C for 16 h. The bonded sandwich structure was then removed from the furnace and cut with a diamond blade into smaller samples for creep test. The creep samples were intended to have final dimensions of 8 mm × 8 mm x 16 mm. However, due to insufficient bonding between the layers, the sandwich structure fell apart upon cutting and we were only able to obtain creep specimens with dimensions of 8 mm × 8 mm x 7 mm (equivalent of two woven layers).

2.2. Creep testing of bulk and 3D woven superalloys

Compressive creep experiments were performed at 825 ± 2 °C on bulk and woven samples with dimensions of 5 mm × 5 mm x 11 mm and 8 mm × 8 mm x 7 mm, respectively. The samples were placed between silicon carbide platens lubricated with boron nitride and heated in air in a three-zone furnace with the temperature measured using a K-type thermocouple placed within 1 cm of the specimen. They were subjected to uniaxial compression by Ni-based superalloy rams in a compression cage using dead loads. Sample displacement was monitored with a linear variable displacement transducer with a resolution of 6 μm, resulting in a minimum measurable strain increment of 3×10^{-4}. A strain rate was obtained by measuring the slope of the strain vs. time plot in the steady-state (secondary) creep regime. After a measurable steady-state strain rate was achieved, the applied load was increased to obtain another steady-state strain rate. Thus, a single specimen provided minimum creep rates for a series of increasing stress levels, at the end of which the total strain did not exceed 10%.

2.3. Static oxidation tests

Woven samples were tested for their oxidation behavior at three different temperatures (750, 825, 900 °C) up to 1 week (168 h) in static air. Samples, placed in alumina crucibles, were weighed before the tests with an analytical balance with an accuracy of 0.0001 g. Two samples per temperature were held in the furnace for a pre-specified duration (3, 6, 12, 24, 48, 96, 168 h) and weighed again at the end of each step after cooling to room temperature in air. The mass gain data were then used to assess the oxidation kinetics.

2.4. Metallography and microscopy

Samples were polished down to 0.05 μm using standard metallographic techniques. Select specimens were etched with a 33 vol% water +33 vol% acetic acid +33 vol% nitric acid +1 vol% hydrofluoric acid solution to reveal the γ'-Ni_3(Al,Ti) precipitates. An optical microscope and a scanning electron microscope equipped with an energy dispersive X-ray spectrometer were used for microscopic and chemical analyses.

3. Computational model and methodology

3.1. Governing equations

The relationship describing the creep behavior of metallic foams has originally been developed by Gibson and Ashby [21]