



Regular Article

Deposition-based synthesis of nickel-based superalloy microlattices

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ABSTRACT

Nickel-based superalloy microlattices with periodic hollow struts were fabricated through a combination of two deposition-based methods - electrodeposition and pack cementation - via the following steps: (i) creating a template by self-propagating photopolymer waveguide prototyping, (ii) coating the template with nickel by electroplating, (iii) etching away the template, (iv) coating the Ni lattice with Cr, Al, and Ti by pack cementation, (v) diffusing the Cr, Al, and Ti rich coatings into the nickel to achieve a homogenized alloy and (vi) optionally, aging the alloy to create γ' -precipitates. The resulting nickel-based superalloy microlattices are 2–4 times stronger than pure Ni counterparts.

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Microlattice structures [1–7] are a subgroup of micro-architected materials [8–12] that provide alternatives for stochastic foams. Due to their regular structures, unlike foams, they offer good energy absorption, high specific strength and stiffness, which can be tailored for specific applications by modifying the micro-architecture [13–15]. Microlattices have been fabricated from various metals and alloys using a variety of techniques [16], such as investment casting [17], deformation forming [18], textile techniques [9–11,19], additive manufacturing [5], and the self-propagating photopolymer waveguide technique [2]. In particular, the last method has been used recently to manufacture pure nickel microlattices with a range of relative densities (controlled via wall thickness) resulting in tunable properties [2]. Fabricating such materials from high-temperature alloys, such as Ni-based superalloys, is desirable to create creep- and oxidation-resistant structures that can be actively cooled, using the space between and/or within the hollow struts. Superalloy microlattices are also of interest as cores in lightweight, high-temperature sandwich structures. However, electrodeposition is practically achievable only for pure Ni, and cannot be employed for the complex compositions typical of Ni-based superalloys. One potential solution is to decouple the electro-deposition step, which creates the structure, and a later alloying step. This approach was recently demonstrated for Ni-Cr wires woven into 3D architected structures, to which Al and Ti were added via pack cementation technique, a chemical vapor deposition process, to achieve, after heat treatment, the γ/γ' microstructure and the associated high strength displayed by Ni-based superalloys [9,19–22].

In this study, the pack cementation technique is used to transform pure Ni microlattice structures, fabricated via self-propagating photopolymer waveguide prototyping, into oxidation-resistant and γ' -strengthened Ni-based superalloys. For this purpose, we coat pure Ni microlattices with Cr, Al and Ti via pack cementation, and achieve, after homogenization and aging, a uniform Ni-Cr-Al-Ti composition with γ/γ' superalloy microstructure. We describe here the microstructural evolution during this process and the high-temperature mechanical properties of the resulting structures.

Polymer microlattice templates were fabricated by a self-propagating, photopolymer waveguide process described in detail elsewhere [23]. The polymer structures were then electroplated with $\sim 40\ \mu\text{m}$ nickel using a standard nickel sulfamate plating solution at a low current density of $20\ \text{mA}/\text{cm}^2$. To render the polymer surface electrically conductive for electroplating, a 500 nm nickel layer was deposited by electron beam evaporation using a rotary planetary fixture, achieving complete coverage of the lattice. Electron beam evaporation was selected over electroless nickel deposition to avoid addition of phosphorous, which is detrimental to superalloy properties. After electroplating, the top and bottom surface of the microlattice was sanded to expose the polymer, which was subsequently etched out by immersion in 1.5 M NaOH solution.

For pack chromization, the Ni microlattice was embedded in a pack consisting of 65 wt% alumina (filler), 30 wt% Cr (source), and 5 wt% NH_4Cl (activator) powders contained within an alumina crucible. The loaded crucible was placed at the water-cooled end of a tube furnace flushed with Ar and pushed into the hot zone of the furnace. The deposition process was conducted at $1000\ ^\circ\text{C}$ for times up to 8 h or until an average of 14 wt% Cr content was achieved, based on weight measurements before and after the treatment. The Cr-rich coatings on the

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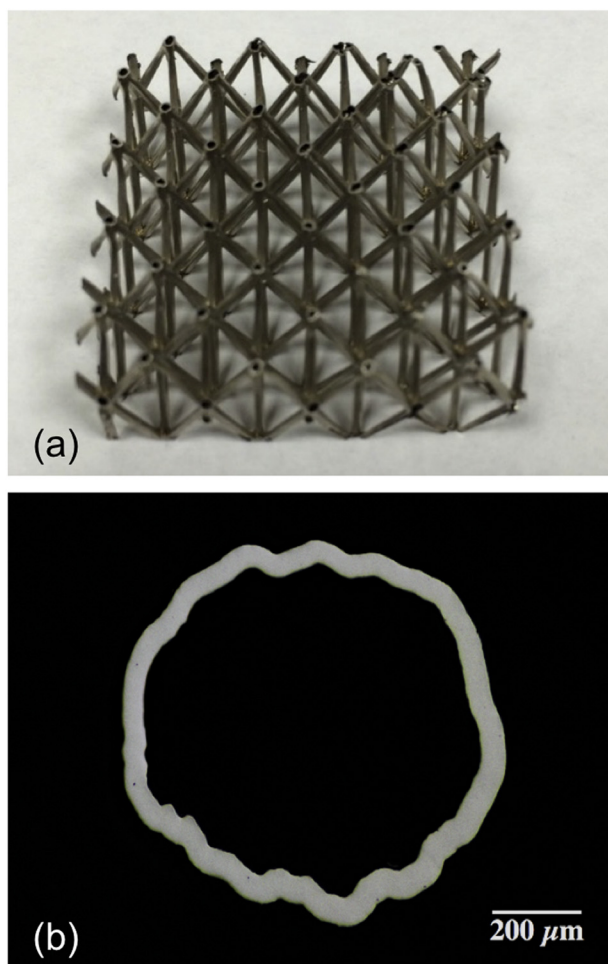


Fig. 1. (a) Photograph of an unalloyed Ni microlattice structure, after electrodeposition and polymer removal, with $35 \times 35 \times 14$ mm dimensions, consisting of $4 \times 4 \times 1$ unit cells (each $\sim 9 \times 9 \times 14$ mm) and (b) optical micrograph of polished radial cross-section of one of the interconnected hollow Ni struts forming the microlattice structure.

inner and outer surfaces of the Ni hollow struts were then diffused into the walls in a homogenization step performed at 1100°C for 24 h under vacuum. Al and Ti were then deposited on the surfaces of the Ni-Cr hollow struts using a similar pack cementation method. The pack consisted of 57 wt% alumina (filler), 30 wt% Ti (source), 10 wt% Raney Ni (Ni-50 wt% Al, source) and 3 wt% NH_4Cl (activator) powders. The sample and the pack were once again placed in an alumina crucible, loaded in the tube furnace at 1000°C and held for 20 min. Alumino-titanized samples were then subjected to a second homogenization treatment ($1100^\circ\text{C}/24$ h) to achieve walls with uniform composition, followed by a solutionizing treatment at 1200°C for 2 h and a subsequent aging treatment at 900°C for 16 h, all under vacuum, followed by cooling in the water-chilled furnace. To prevent loss of Al due to evaporation during homogenization and aging, a ~ 10 g piece of Ni-50 wt% Al alloy was placed in the same crucible next to the samples.

Following the heat treatment, the samples were prepared using standard metallographic techniques for optical and scanning electron microscopy (SEM). To reveal γ' precipitates, aged samples were etched for 30 s with a solution consisting of 33 vol% acetic acid, 33 vol% nitric acid, 33 vol% deionized water, and 1 vol% hydrofluoric acid. Compositional data were obtained using an SEM equipped with an energy dispersive X-ray spectrometer (EDS). Hardness data were obtained using a Vickers microhardness tester with an applied load of 10 g and dwell time of 10 s. For each data point, at least five measurements were taken and the average is reported. Hot compression tests were conducted with a strain rate of $5 \times 10^{-4} \text{ s}^{-1}$ at 788°C in air on specimens with

dimensions of 4 unit cells in length (~ 35 mm), 4 unit cells in width (~ 35 mm) and 1 unit cell in height (13–14 mm).

Fig. 1(a) shows a pure Ni microlattice structure prior to alloying, which consists of interconnected hollow struts. A polished cross-section of one of these hollow struts is given in Fig. 1(b) that shows the Ni wall with a nearly uniform, ~ 50 μm thickness. The strut does not have a perfectly circular cross-section, which is due to ridges in the surface of the sacrificial polymer scaffold, created via self-propagating photopolymer waveguide prototyping, on the surface of which Ni was deposited. As-deposited Ni has an ultrafine grain structure, hence, a relatively high hardness value of 2.89 GPa (295 HV), which drops to 1.37 GPa (140 HV) upon annealing at 1000°C for 30 min. Grain growth occurred during annealing, as revealed by etched cross-section (not shown here) in which 1–2 grains are typically spanning the entire wall thickness.

The EDS map shown in Fig. 2(a) reveals the distribution of Cr in an as-chromized Ni microlattice structure. The image shows a small portion of the radial cross-section of a hollow strut, from a specimen that was chromized at 1000°C for 8 h, which reached an overall Cr content of ~ 14 wt%. The as-coated sample shows high Cr concentration at the outer surface of the strut, as expected, as well as at its inner surface, indicating that the chromium halide gas penetrated into the hollow struts from their open ends, increasing the rate of Cr deposition during pack cementation. The cross-sectional images obtained via optical microscopy (OM) and SEM (in secondary electron mode) revealed little contrast. This indicates that the coating was formed as a solid solution without intermetallic phases, which is not surprising since Ni has a high solubility for Cr (>40 wt% at 1000°C). Upon annealing at 1100°C for 24 h, the

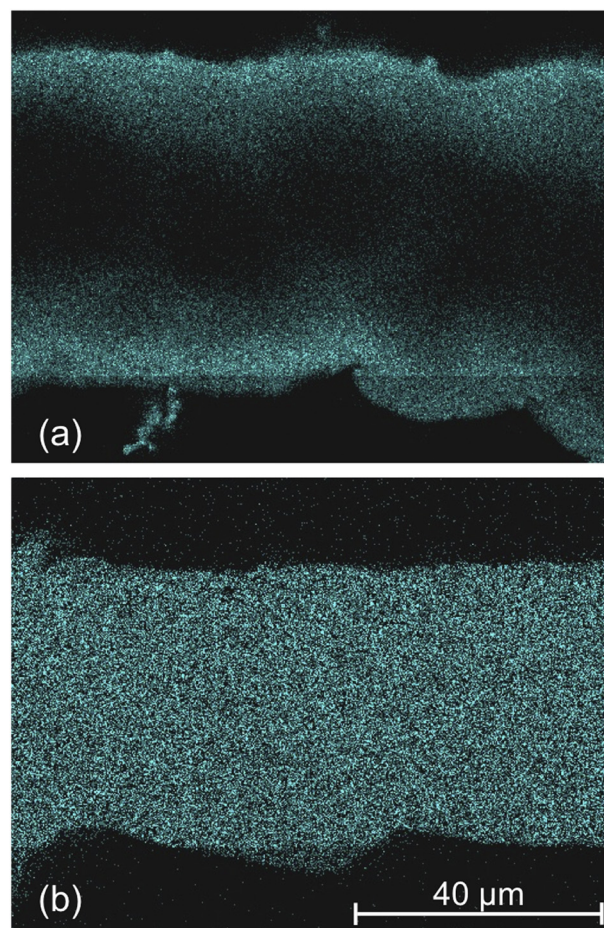


Fig. 2. EDS maps of polished cross-sections, showing the Cr distribution in (a) Cr-coated Ni wall chromized at 1000°C for 8 h showing a Cr gradient from wall surfaces to wall interior and (b) the same sample homogenized at 1100°C for 24 h with a uniform composition of Ni-14 Cr (wt%).

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