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Letter

Properties of sputter deposited Ni-base superalloys for microelectromechanical systems

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ABSTRACT

Nickel-base superalloy 718 films have been sputter deposited to thicknesses of 20 μm. These as-deposited films were single-phase nanocrystalline solid solutions, which possessed tensile strengths of 1 GPa but negligible ductility. Standard 718 aging heat treatments resulted in modest grain growth, atypical precipitation behavior, significant ductility, and impressive strengths at both ambient (2 GPa at 25 °C) and elevated (750 MPa at 700 °C) temperatures. The density of grain boundaries was shown to have a dramatic effect on the precipitation pathways of this alloy.

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The vast majority of commercial microelectromechanical systems (MEMS) are fabricated out of silicon, predominantly vapor deposited polysilicon or deep reactive ion etched single-crystalline Si. The reliance on silicon as a structural material is at odds with traditional engineering structures. Expansion of the current MEMS materials suite for use in harsh environments, particularly elevated temperatures, would facilitate a greater variety of MEMS applications. Advanced metallic systems are especially attractive for use in MEMS-scale sensing and power applications because they offer higher density, electrical and thermal conductivity, strength, ductility, and toughness. Pure metals such as Al, Au, Ag, and Pt are routinely incorporated into MEMS devices as part of surface micromachining processes. Furthermore, the development of the LIGA (a German acronym for lithography, electroplating and molding) process has been used to produce high-aspect ratio Ni structures for load-bearing applications [1]. However, room temperature creep and loss of mechanical strength resulting from microstructural instabilities preclude the use of pure metal thin films or LIGA Ni structures at elevated temperatures [2–6]. By contrast, bulk Ni-base superalloys possess an attractive balance of elevated temperature properties but are not normally shaped and processed on the micron scale.

High temperature metal alloys, such as Ni-based superalloys, owe their superior properties to the fact that they are multi-element and multi-phase materials. Trying to replicate the chemistry and microstructure of these alloys using deposition processes, such as electrodeposition or chemical vapor deposition, would be extremely difficult. However, the physical nature of magnetron sputtering has shown to produce films with chemistries similar to the target material. In the current study, thick films of a commercial Ni-base superalloy, Haynes 718, were deposited using magnetron sputtering. The chemistry, microstructure, and mechanical properties of these films were analyzed, and heat treatments were performed to assess their influence on high temperature performance.

For the current study, films were magnetron sputtered from a commercial alloy 718 target (from Haynes International) using sputtering parameters (Table 1) that were determined from a design of experiment exercise focused on minimizing residual stress in the films [7]. Argon was used as the sputtering gas and, since Ar pressure was the key variable determined to drive residual stress development, depositions were performed at the lowest Ar pressures that sustained a stable plasma. Films were deposited on silicon and brass substrates in two different deposition chambers and, film thicknesses of approximately 20 μm were achieved. Films deposited on Si substrates adhered to the substrate without peeling. Freestanding films were obtained by cleaving the substrate with a diamond scribe. This led to delamination of the films and, lingering Si was removed by mechanical polishing and a XeF_2 gas etch. Films deposited on brass substrates peeled cleanly off the substrate with no apparent residue.

As-deposited films were prepared for transmission electron microscopy (TEM) using a Struers twin jet electropolisher and an electrolyte of

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Table 1
Sputtering parameters used for depositions performed on silicon and brass substrates.

Substrate	Target area (cm^2)	Power (W)	Ar pressure (Pa)	Target to substrate distance (cm)
Silicon	81	450	0.13	10
Brass	468	1500	0.06	10

15 vol.% perchloric acid and 85 vol.% ethanol. TEM observations revealed the as-deposited films to be a nanocrystalline solid solution with an average grain size of 45 ± 10 nm (Fig. 1(a)). The formation of a nanocrystalline solid solution was attributed to the high quench rates inherited from the sputtering process; the alloy was effectively quenched from the vapor phase. An as-deposited 718 film was etched with a waterless Kalling's reagent and analyzed in cross section with scanning electron microscopy (SEM). The SEM investigation showed equiaxed grains throughout the full thickness of the film (Fig. 1(b)) and indicated that mechanical properties are independent of film thickness. The chemistry of the as-deposited films was measured using energy-dispersive spectroscopy on a JEOL 8600 Superprobe with the bulk alloy 718 material serving as a standard. Results of this analysis (Table 2) indicated that the composition of the sputtered films was almost identical to the bulk alloy target. Lighter elements such as Al and Ti were represented in similar proportion to the heavier elements like Mo. The lightest elements in this alloy C, B, Si, and Mn were present in small amounts and were therefore unable to be measured with energy-dispersive spectroscopy.

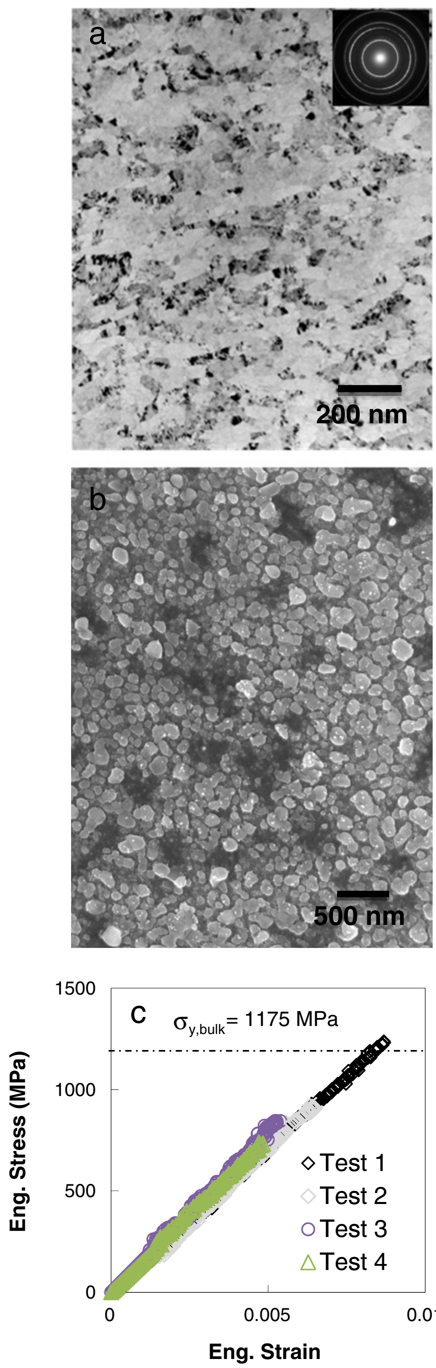

Fig. 1. (a) Bright field TEM micrograph of an as-deposited 718 film revealing the nanocrystalline grain size. The selected area diffraction pattern of the film (inset) corresponds to a single-phase Ni solid solution alloy. (b) SEM image of an etched as-deposited 718 cross section showing equiaxed grains through the foil's thickness. (c) Stress–strain curves from microtensile experiments performed on as-deposited 718 films show very high strengths but very limited plasticity.

Mechanical properties of the as-deposited films were investigated using microtensile testing techniques described elsewhere [8]. The films were sectioned into tensile geometries using wire electrical discharge machining by sandwiching the films between two aluminum sheets with nickel print to ensure good electrical conductivity and to restrict movement of the films during machining. Room temperature microtensile tests were conducted at a strain rate of 2×10^{-4}. The results of these experiments (Fig. 1(b)) demonstrated that the films exhibited linear elastic behavior in their as-deposited state. Very high strengths on the order of 1 GPa were observed, but scatter in the measured fracture strengths was several hundred MPa as a result of brittleness. Side-wall roughness associated with machining was the primary initiation site for brittle failure. This is similar to the scatter seen in the fracture strengths of Si specimens controlled by side wall, etch-induced defects [9].

The high energy and quench rates associated with the sputtering process resulted in a far-from-equilibrium supersaturated nanocrystalline single-phase microstructure. In contrast, the homogenized state for bulk alloy 718 is normally achieved by solutionizing and quenching from 1000 °C. The alloy is then typically aged at 720 °C for 8 h followed by a furnace cool to 620 °C, where it is held for an additional 8 h. This standard aging procedure results in a two-phase ($\gamma + \gamma''$) microstructure that provides a beneficial balance of properties. For comparison, the standard aging procedure was applied to the as-sputtered 718 films by sandwiching them between two alumina plates (to prevent curling) and heat treating in a vacuum of 10^{-4} Pa. The following microstructural changes were observed (Fig. 2(a)) following the standard aging procedure. The grains coarsened to an average size of 200 nm with a small fraction of larger grains in excess of 500 nm. The grain boundaries appeared more sharply defined indicating a more relaxed state. The second phase precipitates were also observed. The plate morphology and size of these precipitates was indicative of the orthorhombic ($D0_a$) δ-Ni_3Nb phase, whose presence was confirmed with X-ray diffraction [7].

Precipitation of δ-Ni_3Nb was unexpected. In bulk 718, metastable body centered tetragonal ($D0_{22}$) γ''-Ni_3Nb precipitates form first and only transform to the stable δ-Ni_3Nb phase after much longer aging times [10–12]. In bulk 718, nucleation of the δ-phase has been reported to occur at grain boundaries [13]. This observation suggests that the preferred formation of the δ-phase in nanocrystalline alloy 718 sputtered films may be associated with their significantly higher grain boundary densities.

Recognizing the microstructural differences that arise in aged bulk 718 and sputtered 718 films, room temperature microtensile tests