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# Improved very high cycle bending fatigue behavior of Ni microbeams with Au coatings

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ABSTRACT

This work investigated the effect of an 850-nm-thick electroplated Au coating on the very high cycle bending fatigue behavior of electroplated Ni microbeams tested under resonance in air at high frequencies (~9 kHz). The S–N curves show longer fatigue lives for the coated microbeams by at least a factor of 5 compared to the uncoated ones. This beneficial effect is demonstrated to be related to the delay in oxygen-assisted void formation, and therefore in void-assisted fatigue crack nucleation and growth in Ni. The improvement in fatigue life is limited by the fatigue degradation of the Au coating, which is also controlled by the formation of nanosized voids. Once a fatigue crack in the coating reaches the interface, delamination occurs, leading to exposure of the underlying Ni to air and faster, "uncoated-like", fatigue degradation thereafter. This study highlights that thin, noble metal coatings can significantly improve the fatigue lives of metallic microbeams whose very high cycle fatigue behavior is sensitive to the environment and controlled by void formation.



## 1. Introduction

Thin hard coatings are routinely employed in structural materials, particularly metals, to protect them from corrosion and wear [1,2]. Their use for improving fatigue life is less straightforward [3–10]. Any surface coating that could potentially delay fatigue crack nucleation at or near the surface would in principle improve the high cycle fatigue (HCF) behavior of a metal (whose life is mainly dominated by the nucleation of fatigue cracks) [8]. However, the coating may break at the location of the extrusions developing at the surface of the underlying metal due to persistent slip band (PSB) formation, thereby resulting in little benefit [9]. Alternatively, secondary cracking resulting from the presence/deposition of the coating (either due to cracking of the coating [5,11] or cracking of the metal underneath the coating due to surface modification resulting from the coating process [12]) can also decrease the fatigue life of the component [10]. Cracking of the coating is more likely if large residual tensile stresses are present [5,6]. Secondary cracking may not reduce fatigue life if the presence of the coating results in large compressive residual stresses that can effectively improve the fatigue life by decreasing the effective stress intensity factor range (i.e. decreasing crack growth rates) [13,14], with the opposite effect for residual tensile stresses. Stoudt et al. argued an effective coating should exhibit the following five properties to prevent fatigue crack nucleation: "(i) hardness — to suppress the development of the characteristic PSB-induced surface morphology, (ii) toughness — to resist cracking where subsurface PSBs intersect the film, (iii) cyclic work hardenability — to prevent slip localization, (iv) residual compressive stresses — to counter the effects of tensile stresses, and (v) adherence — to maintain surface film contact with the substrate" [10]. They demonstrated that a 5-$\mu$m-thick nanometer-scale multilayered coating, consisting of 125 40-nm-thick Cu/Ni bilayers and likely having the five aforementioned characteristics, significantly improved the fatigue life (by more than one order of magnitude) of bulk polycrystalline Cu.

In contrast to bulk metals, only a few studies have investigated the role of coatings on the fatigue properties of small scale materials, such as thin films and microbeams [15–18]. Additional coating characteristics may be required because of the possible size effects on fatigue mechanisms. For example, our recent studies highlighted strong size effects on the bending high cycle fatigue behavior of electroplated Ni microbeams [19]. We measured ultralow fatigue crack growth rates for these microbeams (average values down to ~$10^{-14}$ m/cycle), and revealed a discontinuous

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process for both fatigue crack nucleation and propagation that involved void formation, most likely based on vacancy condensation processes [19]. The environment plays a significant effect on fatigue lives that are three orders of magnitude longer *in vacuo* than in air. Studies on irradiation of metals (whereby large concentrations of vacancies are introduced) have shown that oxygen stabilizes void nucleation compared to the other vacancy cluster defects, by decreasing the void surface energy through a chemisorption process [20–24]. It is therefore possible that the formation of voids during cyclic loading of the Ni microbeams in air is facilitated by the presence of oxygen, resulting in faster nucleation and growth of small cracks. In this paper, we investigated the role of ~850-nm-thick electroplated Au coatings on the fatigue behavior of these Ni microbeams, particularly its effect on void-controlled fatigue crack nucleation.

## 2. Methods

### 2.1. Specimen geometry and material properties

The specimens were fabricated with the MetalMUMPs® process from MEMSCAP. MetalMUMPs is an electroplated nickel micromachining process, which includes the patterning of a thick layer of photoresist that forms a patterned stencil for the electroplated Ni. The electroplating process is at 30 °C, with a current density of 20 mA/cm$^2$ and a pH level of 4 [25]. The Ni bath consists of nickel sulfamate, nickel bromide, nickel, and boric acid, with a concentration of Ni in the bath of 99.9%. A 20-$\mu$m-thick structural layer of Ni is electroplated on top of a 0.55-$\mu$m-thick Cu base layer, and the top of the Ni layer is covered with a 0.5-$\mu$m-thick Au layer. The fabrication process allows an optional coating of the metallic layer with a 0.85 $\mu$m-thick electroplated layer of Au deposited on the top surface and side walls in selected regions [26]. Fig. 1 shows the geometry of the Au-coated fatigue specimens which are on-chip microresonators (shown in Fig. 1(a)), consisting of a large fan-shaped mass connected to a Au-coated Ni microbeam (see Fig. 1(b) for the location of the Au coating, covering the entire microbeam). The microbeam is ~60 $\mu$m long and the width of the narrowest Ni section is ~11.5 $\mu$m which produces a normalized stress gradient under bending: $\eta = \frac{1}{\sigma_{max}}\frac{d\sigma}{dx} = 17\%\,\mu m^{-1}$. The cross-section SEM image (see Fig. 1(c)) shows the 0.85-$\mu$m-thick Au coating along the sidewalls, and the 1.35-$\mu$m-thick Au coating at the top. The fan shaped mass has two sets of interdigitated fingers (two "combs"), one for electrostatic actuation and one for capacitive sensing.

The Ni layer has a strong ⟨001⟩ crystallographic out-of-plane texture and a columnar microstructure, with columnar grains that are ~5–10 $\mu$m in height [27]. The average grain size (diameter) is 1.2 $\mu$m, based on SEM images of FIB cross-sections. The tensile properties were measured using microtensile testing of dog-bone shaped specimens [27]: yield stress of 656 ± 70 MPa, tensile strength of 873 ± 26 MPa, and plastic strain at failure of 7.4 ± 2.8%. The high strength of this electroplated Ni is consistent with previous reports, knowing that the strength is affected by grain size (Hall-Petch effect) and the amount of impurities (likely resulting in solid solution strengthening), which both depend on the Ni bath composition and deposition conditions [28,29]. The stress-strain curve was fitted with the following Ramberg-Osgood equation:

$$\varepsilon = \frac{\sigma}{E_{Ni}} + \left(\frac{\sigma}{K}\right)^{\frac{1}{n}} \quad (1)$$

with $E_{Ni}$ = 172 GPa (a value consistent with the texture [27]), $K$ = 1451 MPa, and $n$ = 0.136.

The average grain size of the Au coating is 650 nm based on SEM images of the FIB cross-sections. The tensile properties of the Au coating could not be measured directly. Instead, the elastic modulus of Au, $E_{Au}$, was calculated using finite element modelling to match the measured effect of the Au coating on the microresonator's resonance frequency ($f_0$); see sections 2.3. A value of 71 GPa was obtained for $E_{Au}$ (see section 3.1). Nanoindentation was also performed on the top surface of the Au coating to determine its hardness. The top surface was first polished using FIB milling and then indentation was performed using a Cube corner 3-sided pyramid tip made of Diamond and having a tip radius of ~100 nm (half angle = 35.26°, included angle = 90°). The resulting hardness was 3.02±1.1 GPa obtained from 7 measurements taken at locations 2 $\mu$m apart with maximum indentation depth of 120 nm. The large hardness value and therefore high strength seems consistent with the small grain size and possible strengthening due to impurities.

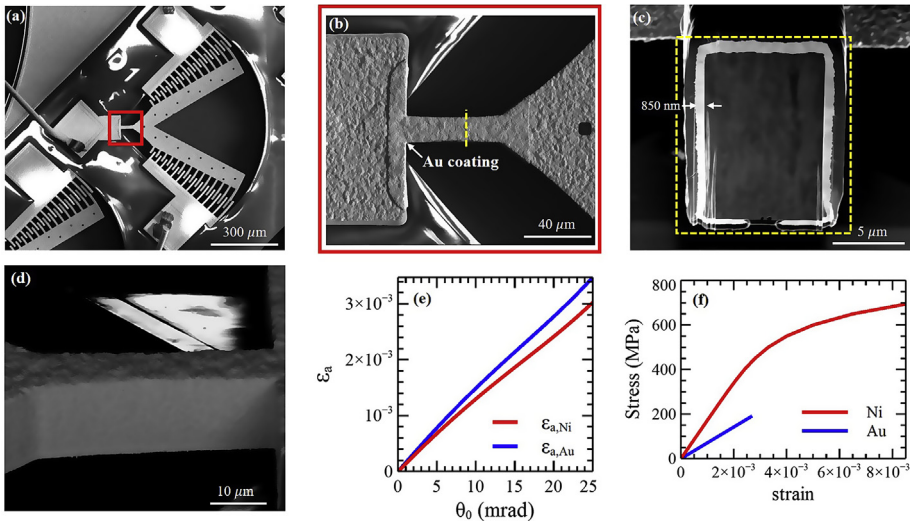


**Fig. 1.** (a) & (b) SEM images of the microresonators with Au coated microbeams; (c) Cross section of a coated microbeam; (d) SEM image of the Au side wall prior to fatigue testing, (e) $\varepsilon_a$ and $\theta_0$ relationship; (f) stress-strain curves for Ni and Au.