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Fatigue-induced thick oxide formation and its role on fatigue crack initiation in Ni thin films at low temperatures

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Abstract

This study highlights an oxidation-assisted fatigue crack initiation mechanism in 20 μm thick electroplated Ni films under loading conditions relevant for a wide range of microelectromechanical systems, such as extreme stress gradients at the surface (50% decrease over the first micrometer from the surface). Microresonators subjected to in-plane bending at ~8 kHz were fatigued for billions of cycles in humid air, at 30 °C, 50% relative humidity (RH), and 80 °C, 90% RH, for maximum stress amplitudes up to ~500 MPa (~55% of the ultimate tensile strength). Transmission electron microscopy (TEM) revealed highly localized thick oxides (~1 μm) on specimens fatigued for several billions of cycles. These oxides are two to three orders of magnitude thicker than the regular native oxides at these low temperatures, and only form at the location of cyclic slip bands. These oxides appear to be thicker for higher partial pressures of water, based on the TEM comparison of one specimen fatigued at 30 °C, 50% RH to one fatigued at 80 °C, 90% RH. Fatigue microcracks were observed within these highly localized thick oxides. Finite element models were also employed to confirm these results based on the interpretation of the evolution of the devices' resonance frequency. This oxidation-assisted fatigue crack initiation mechanism at low temperatures constitutes a significant departure from the established mechanisms for bulk metals and their environmental effects. A possible explanation for the different governing mechanism is the presence of extreme stress gradients in these microscale components. Under these loading conditions, the classical fatigue crack initiation mechanisms are not operational, allowing this alternative mechanism to become dominant. This study highlights the need to further understand the coupled size and environmental effects on the fatigue of metallic thin films.

Keywords: Fatigue crack initiation; Electroplated nickel; Thin films; Environmental effects; Oxidation

1. Introduction

The environmental effects on the fatigue behavior of bulk metals are well documented, with fatigue lives that can be orders of magnitude longer in vacuum compared to air for reactive metals [1]. It is commonly accepted that the formation of persistent slip bands (PSBs) and accompanying surface morphology changes (persistent slip markings, PSMs) that result in fatigue crack initiation is not affected by the environment [1]. Instead, the significant increase in fatigue life is thought to be the result of slower growth rates of stage I cracks in inert environments [2]. Specifically, one mechanism assumes that chemisorbed oxygen may prevent the "rewelding" of small, crystallographic fatigue cracks [3]. As such, metals fatigued in vacuum have a large density of PSBs associated with small (i.e. less than 10–20 μm) cracks whereas they have much fewer PSBs in air from which longer cracks (>100 μm) grow and reach stage II propagation mode until critical fracture [1].

While these environmental effects are fairly well understood for bulk metals, much fewer studies have focused on the environmental effects on the fatigue of microscopic metallic components [4–6]. Several factors are distinctly different at the nano-/microscales, which could affect the

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role of the environment on the fatigue of small-scale metallic components. First, the critical cracks leading to fatigue failure are much shorter, often limited by the component's size (micrometer range). Hence the fatigue life is mainly dictated by the nucleation and propagation of short, stage I fatigue cracks. These short fatigue cracks can also experience extreme stress gradients (e.g. in the case of microscopic notches or bending of narrow components), further reducing their propagation rates. The environmental effects could therefore be much more pronounced given the close proximity of the cracks to the surfaces for most of the fatigue life. In addition, the fatigue crack nucleation process (and therefore possibly the environmental effects) can differ from the aforementioned PSB/PSM formation for small grains and/or film thicknesses (below ~1 μm), transitioning to individual dislocations and interface-mediated damage behavior such as cracking and voiding along grain boundaries or twin boundaries [7]. Last but not least, some microelectromechanical systems (MEMS) applications, such as microresonators for accelerometers or gyroscopes, require reliability studies, including environmental effects, in the very high cycle fatigue regime in which billions of cycles may accumulate [8,9].

To illustrate the above considerations, a previous study on electroplated nickel films with MEMS-relevant dimensions highlighted an unexpected fatigue crack nucleation process [4]. Fatigue tests performed in laboratory air revealed localized fatigue-induced oxide thickening, with surface oxides up to 400 nm in thickness, which are orders of magnitude thicker than native oxides. The observed oxidation was strictly limited to areas in which extrusions and intrusions were found, contrary to other fatigue-enhanced oxidation mechanisms [10]. Propagating fatigue cracks were observed at the location of these thick oxides, making this "PSB oxide thickening" mechanism a possible fatigue crack initiation mechanism. Whether this particular mechanism is relevant for a larger range of materials or component sizes has not yet been established. However, it appears unlikely that the numerous fatigue studies on PSB/PSM formations at the macroscale, including detailed transmission electron microscopy (TEM) and focused ion beam (FIB)/scanning electron microscopy (SEM) investigations (e.g. see Refs. [1,11–13]), would all have missed the formation of thick oxides. Therefore, a better understanding of the underlying oxidation process and its role in fatigue crack nucleation is required to further predict its range of applicability. The present study not only confirms the occurrence of highly localized thick surface oxides in electroplated Ni films but also provides critical information regarding the role of temperature (30 °C vs. 80 °C) and water vapor (partial pressures of ~2 vs. ~42 kPa) on these localized surface oxides. Specifically, electroplated Ni MEMS microresonators were cycled for billions of cycles in three different environments [14,15]. Here, the previously published fatigue results, including resonance frequency (f_0) evolution and SEM observations [14,15], are further analyzed using a combination of TEM and finite element modeling (FEM), in order to understand the fatigue crack initiation process in these electroplated Ni microspecimens.

2. Methods

2.1. Fatigue specimens and material

The fatigue microresonators are depicted in Fig. 1. They consist of a notched cantilever beam at the base of a fan-shaped mass and two comb drives (interdigitated fingers on each side of the mass), of which one allows for electrostatic actuation and the other allows for capacitive motion sensing. A fatigue test consists of driving a microresonator at resonance by periodically keeping track of f_0 (~ 8 kHz), which leads to fully reversed sinusoidal in-plane bending of the notched beam. The fatigue damage occurs at the notch root, and the evolution of f_0 is an indication of the extent of fatigue damage [14]. It was demonstrated that no external cooling is necessary during a fatigue test for the investigated range of applied plastic strain amplitudes (up to ~0.05%), given that only small volumes at the notch root of the beam experience cyclic plasticity [15] (see discussion in Section 2.2). The microresonators were fabricated with the 15th run of the MetalMUMPs process (MEMSCAP). The structural layer is a 20 μm thick Ni layer that was electroplated at 30 °C, with a current density of 20 mA cm^{-2} and a pH level of 4 [16]. The microstructure is mainly columnar with 5–10 μm long elongated grains that are 1–2 μm wide, and smaller (<1 μm) equiaxed grains in between the columnar grains [14]. The tensile properties (and the associated standard deviations) were measured on five microtensile uniaxial specimens fabricated with the same run as follows: elastic modulus, $E_{Ni} = 166 \pm 19$ GPa, 0.2% yield strength, $\sigma_y = 656 \pm 70$ MPa and tensile strength, $\sigma_{uts} = 873 \pm 26$ MPa [15]. The following Ramberg–Osgood type constitutive equation was used to fit the data:

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

with $K' = 1451$ MPa, $n' = 0.136$ and elastic modulus, $E_{Ni} = 172$ GPa at 30 °C (value based on a previous modal analysis matching the experimental f_0 [15]) and 166 GPa at 80 °C [15].

The notched cantilever beam (see Fig. 1b and c) is ~40 μm long and 15 μm wide, with a ~4 μm root-radius notch that is located ~10 μm from the base. The bending of this microscopic notched beam induces extreme stress gradients (under nominally elastic conditions, the normalized stress gradient is –50% μm^{-1} over the first micrometer from the surface, or an average of –36% μm^{-1} over the first 2 μm [15]). As a result of these extreme stress gradients compared to notched, macroscale components (that are at most ~–1% μm^{-1}) [17], the fatigue cracks that nucleated were arrested (i.e. did not propagate to failure) over the investigated range of maximum applied stress amplitudes [14,15].