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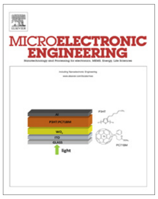

Residual stress gradients in electroplated nickel thin films

Yasin Kilinc [a], Ugur Unal [b,c], B. Erdem Alaca [a,c,*]

[a] Department of Mechanical Engineering, Koç University, Istanbul, Turkey
[b] Department of Chemistry, Koç University, Istanbul, Turkey
[c] Koç University Surface Science and Technology Center, Istanbul, Turkey

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ABSTRACT

Residual stress gradients in electroplated nickel films of 1 μm thickness are characterized for a wide range of current densities (1–20 mA/cm^2) and electroplating temperatures (30–60 °C) in a nickel sulfamate bath. Although a variety of stress measurements is available, exploration of stress gradients remain unstudied at the scale of 1 μm. Stress gradients – unlike uniform stresses – can cause significant bending even in monolayered released structures. Moreover, examples of misinterpretation of wafer curvature data as a measure of stress gradients exist in the literature. Based on these motivations, monolayered Ni microcantilevers are employed in this work as mechanical transducers for the characterization of stress gradients within the nickel film. Experiments are supported with finite element simulations. Residual stress gradient is found to vary in the range of about −130 to 70 MPa/μm with the sign change indicating a transition from downward to upward deflection of the microcantilever. Thus, a window of electroplating parameters is established yielding zero residual stress gradients, *i.e.* straight cantilevers, without the use of any additive agents.

1. Introduction

Nickel has a wide range of applications in microelectromechanical systems (MEMS) due to its attractive mechanical and magnetic properties. Advances in electroplating and extensive employment of thick nickel layers in MEMS applications make electroplating a method of choice for the deposition of nickel. Submicron deposition thicknesses are reported as well [1]. In MEMS literature, nickel sulfamate-based nickel electroplating is mostly carried out in a temperature and current density range of 30–60 °C and 1–20 mA/cm^2, respectively, while the pH of the electrolyte remains in the range of 3.5–5.0.

Among various MEMS applications one can mention Ni structures serving as micromechanical resonators with magnetic actuation in various biological and physical sensor studies [2–5]. Ni cantilevers fabricated by LIGA provide platforms for mechanical property measurements [6,7]. Ni layers are also utilized as micromechanical switches [1,8,9]. Especially in optical readout schemes, where large scatter of light reflected from severely bent microstructures results in lower signal amplitudes, initial bending of the cantilever is undesired. Hence, deposition processes need to be tightly controlled to eliminate bending upon release.

Unlike uniform residual stresses that lead to in-plane deformations, residual stress gradients are primarily responsible for out-of-plane bending [10]. In Fig. 1, one such cantilever is shown. Two scenarios for stress distribution along the cross-section A–B are possible. Depending on fabrication conditions that cause either increasing compressive or tensile stresses through film thickness from B to A, downward or upward deflection is obtained.

In the literature, uniform residual stresses in thin films are studied extensively with various techniques [8,11–13]. However, residual stress gradients in thin metallic films remain mostly unstudied. Among the limited number of accounts one can mention the recent study on strain-gradient-free 4.5-μm-thick Ni cantilevers obtained through multiple electroplating steps [14]. A few other studies [10,11,15] determined residual stress gradients for metallic materials under some specific deposition conditions with fairly limited fabrication parameter range. Therefore a systematic stress gradient characterization as a function of fabrication parameters supported by a microstructural analysis would provide a much needed input in the field of electroplated nickel films to understand out-of-plane deformations encountered in surface micromachining. This work comprises such a study on stress gradients in Ni thin films, where the effect of uniform stresses is omitted in measurements.

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* Corresponding author at: Department of Mechanical Engineering, Koç University, Istanbul, Turkey. Tel.: +90 212 338 17 27; fax: +90 212 338 1548.
E-mail address: ealaca@ku.edu.tr (B.E. Alaca).

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In the remainder of this work, a mechanical framework for the measurement of residual stress gradients will be discussed first followed by a description of employed experimental procedures using 1-μm-thick, 10-μm-wide and 100-μm-long cantilevers. Results are then presented leading to a useful set of deposition parameters for nickel sulfamate bath resulting in near-zero bending of Ni cantilevers.

2. Residual stress gradients and their characterization

Residual stresses in microstructures can be modeled with the combination of a uniform stress (σ) and a stress gradient across the thickness ($\nabla\sigma$). Uniform residual stresses are found in either tensile or compressive form leading to in-plane expansion or contraction of the microstructure after release. In contrast, the relief of a residual stress gradient induces a bending moment resulting in the formation of a curved microstructure with a strain gradient of opposite sign as shown in Fig. 2a.

The residual stress gradient for a monolayer cantilever is given in Eq. (1) in terms of the modulus of elasticity (E) and tip deflection (δ) of a cantilever of length L [10]. The assumption of linear stress distribution, *i.e.* constant stress gradient, is frequently employed in the literature [10,14,16–18], which is especially proper for very thin films such as the 1-μm-thick nickel film of this study:

$$\nabla\sigma = E\frac{2\delta}{L^2} \tag{1}$$

Hence, the residual stress gradient can be determined by measuring process-dependent parameters of tip deflection and modulus of elasticity. While tip deflection can be measured by optical means, modulus of elasticity is determined through measuring the first-mode resonance frequency, f_1, of a cantilever. In case of a monolayer, homogeneous cantilever with a rectangular uniform cross-section, the relation between E and f_1 is given by Eq. (2). This relation remains unaltered by the presence of residual stress gradients:

$$f_1 = 0.162\frac{h}{L^2}\sqrt{\frac{E}{\rho}} \tag{2}$$

In Eq. (2) two new parameters, ρ and h, density and thickness of a cantilever, respectively, are introduced. Being a relatively process-independent parameter, ρ is taken as the bulk mass density of 8908 kg/m^3 throughout this work [19].

Fig. 2b provides a case study for the verification of Eq. (1), where a finite element analysis (FEA) of a cantilever is carried out. COMSOL Multiphysics 4.3 FEA software with 3d free-tetrahedral mesh elements is employed to study the release of a cantilever with a stress gradient of 1 MPa/μm. The thickness, width and length of the cantilever are chosen as 100 × 100 × 1000 μm, respectively. Fig. 2b depicts the linear variation of strain along the midline of a cross-section within the cantilever. Strain gradient from the analytical treatment deviates from the slope of the strain plot in Fig. 2b by less than 0.5%.

It is not uncommon to encounter wafer curvature measurements employed in the literature for the characterization of residual stress gradients. There are reports claiming wafer curvature measurements encompass the effect of stress gradients as well as uniform stresses [20]. However, use of wafer curvature data might be misleading, as there are cases where infinite radius of curvature, *i.e.* straight substrate, does not guarantee straight cantilevers upon release. Similarly, finite wafer curvature associated with thin film stresses might lead – in a seemingly counterintuitive fashion – to a straight cantilever.

To elucidate the misinterpretation associated with wafer curvature measurements, let us consider a <100> Si substrate of a diameter (D) of 10 mm and a thickness (T) of 500 μm. Let us also assume that a Ni film with a thickness (h) of 10 μm is coated on the top surface of this substrate. If there is a stress gradient of 20 MPa/μm in the Ni film (Fig. 3a), it leads to a negligible wafer curvature ($1/\rho$) of 8.864×10^{-4} m^{-1}. This can be computed by incorporating the resulting moment due to stress gradients into the moment equilibrium in a similar fashion to Stoney formulation. The associated formulation is given in Fig. 3a as well, and invariant biaxial modulus of <100> Si ($E/(1-\nu)$) is taken as 180.51 GPa [21]. If this curvature measurement is misinterpreted as an indication of uniform stress only, it would correspond to a stress level of less than 1 MPa. However, if one fabricates and releases a Ni cantilever of a length (L) of 100 μm, this seemingly harmless effect leads to a considerable tip deflection of 0.5 μm.

On the contrary, one can consider the same substrate/thin film system, where the thin film is under a uniform tensile stress of 100 MPa (Fig. 3b). Using Stoney formulation given in Fig. 3b, the associated wafer curvature is predicted to be 0.133 m^{-1}. This measurement is 150 times larger than the curvature in the presence of the aforementioned stress gradient case. More interestingly, as all associated stresses are uniform, they do not lead to any permanent bending of the cantilever upon release. Hence, a larger curvature does not necessarily mean a bending and stiction risk for surface micromachining.

Aforementioned loading scenarios are verified using COMSOL as described in connection with Fig. 2 as well. They support the claim that one has to release microstructures for a reliable characterization of stress gradients, and Stoney formulation alone is not an efficient way for the characterization of surface micromachining.

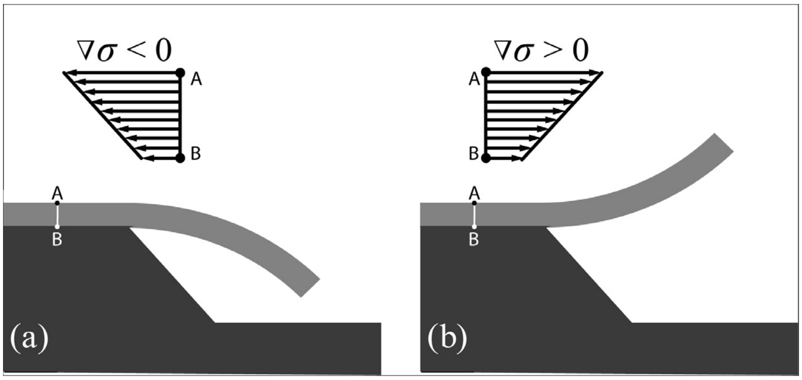

Fig. 1. The effect of residual stress gradient, $\nabla\sigma$, on bending of a monolayer cantilever. (a) With compressive stresses increasing with increasing distance from thin film-substrate interface, the cantilever bends towards the substrate. (b) If residual stresses become more tensile away from the interface, the cantilever bends upwards.