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Young's modulus of electroplated Ni thin film for MEMS applications

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Abstract

The Young's modulus of an electroplated nickel (Ni) thin film suitable for microelectromechanical applications has been investigated as a function of process variables: the plating temperature and current density. It was found that the Young's modulus is approximately 205 GPa at plating temperatures less than 60 °C, close to that of bulk Ni, but drastically drops to approximately 100 GPa at 80 °C. The inclusion of ammonium and sulphate ions by hydrolysis is believed to be responsible for the sharp drop. The Young's modulus of 205 GPa is for a Ni film plated at J=2 mA/cm^2 and it decreases to 85 GPa as the plating current density is increased to 30 mA/cm^2. The results imply that at low current density, the plating speed is slow and there is sufficient time for the as-plated Ni atoms to rearrange to form a dense coating. At high currents, the plating speed is high, and the limited mass transport of Ni ions leads to a less dense coating.

Keywords: Young's modulus; Electroplated Ni thin film; MEMS

Electroplated metal thin films comprising nickel (Ni) and nickel–iron (NiFe) are commonly used for microelectromechanical systems (MEMS) as they provide a simple and cheap technology with superior material properties and device performances. Various MEMS devices such as pressure sensors, thermal actuators, microcoils, micromotors and microstructures based on plated metals have been fabricated and studied [1–10].

From both the design and fabrication points of view, material properties such as thermal conductivity and Young's modulus are of particular importance. Commonly, MEMS devices are first designed, the structures are then simulated and optimised by finite element analysis, and finally they are fabricated. The bulk properties are often used for MEMS design and modelling. However, the resulting device performance after fabrication can be very different from the predicted performance due to a dependence of the properties of plated thin metals upon processing. This leads to the redesign and re-fabrication of MEMS devices, increasing the development time and cost.

It is known that the microstructure and mechanical properties of a plated thin film depend upon the plating conditions such as temperature, concentration and current density [2,11–14]. Surprisingly, little effort has been made to clarify the relationship between processing conditions and mechanical properties of plated thin metal films, particularly of plated Ni. In this letter, we report the results of an experimental investigation into the effects of plating temperature and current density upon the Young's Modulus of electroplated Ni thin films. The measurement of Young's modulus in thin films is problematic and has been the subject of much research around the world [15–17].

Nickel thin films were electroplated from a solution of sulphamate nickel plating solution,[1] consisting of nickel sulphamate (300 g/l), nickel chloride (10 g/l) and boric acid (40 g/l). The purity of the plating solution is >99.99%, with metal impurity densities of Na<10 ppm, Ca <2 ppm and others at ≤1 ppm. The plating bath comprises a hot water bath, a tank for the plating solution with a lid to prevent vaporization (with two electrodes inserted through it), a control circuit, and a Ni plate anode with an area of ∼3 cm^2. A magnetic pellet was used to stir the solution, and

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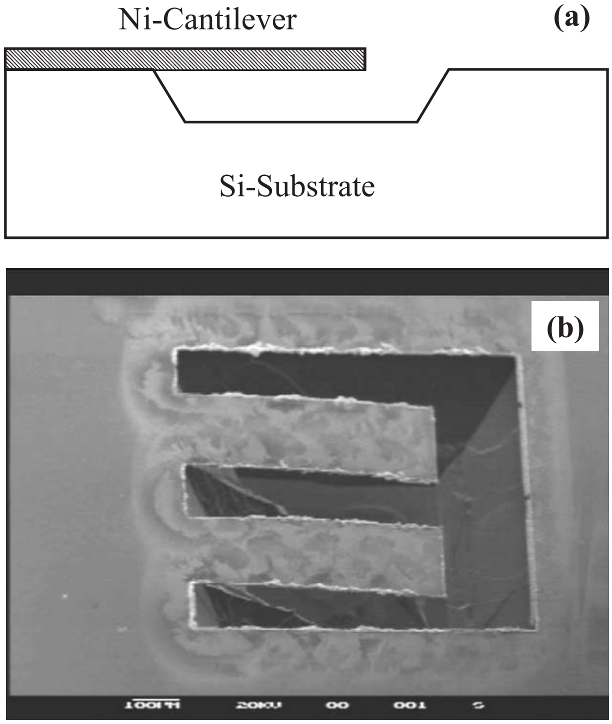

Fig. 1. The cross-sectional diagram (a) and SEM picture (b) of a Ni cantilever cut from an electroplated Ni film plate, and released by etching in KOH solution.

thereby keep the solution concentration uniform and prevent the formation of hydrogen bubbles on the sample surface. The plating temperature was adjusted in the range of 20–80 °C to an accuracy of ±2 °C, and the current density was in the range of 2–30 mA/cm^2.

A copper seed layer of thickness 60 nm was deposited on a Si substrate with a 5-nm-thick interlayer of Cr using a sputtering system. (The thin Cr layer promotes adhesion between Cu and the Si-substrate). The Ni layer was plated onto the Cu seed layer to a deposition thickness of 1.5–3 μm. A laser machining system[2] was used to cut Ni cantilevers of length 500 μm and width 150 μm out of the electroplated Ni thin film using UV light. The cantilevers were then released using a KOH etchant of concentration 20% in wt at a temperature of 85 °C to remove the Si from beneath the cantilevers. The typical etch time was 1.5 h. A typical cantilever is shown in the SEM micrograph of Fig. 1b with a cross-sectional diagram in Fig. 1a. The edges of metal beams cut by laser are typically very rough, and there is re-deposition of metal debris. However, the scale of the roughness and the size of debris is very small compared to the dimensions of the beam and their effects on the extraction of Young's modulus is negligible.

A DekTak 8 machine was used to conduct a beam bending experiment with a force in the range of 1–50 μN. In brief, the Dektak machine was used as an instrumented microhardness tester and was used to measure the transverse tip deflection u of the beams as a function of increasing tip load F. Linear beam theory for a beam of uniform rectangular section, of length L, width w and thickness t predicts a linear relation between u and F according to

$$u = \frac{4}{Ew}\left(\frac{L}{t}\right)^3 F + \kappa L \qquad (1)$$

where E is the Young's modulus, and κ the curvature of the beam. Thus the slope of a plot of u versus F can be used to deduce a value for E. The presence of residual stress within the cantilever leads to an initial curvature of the beam but does not affect the linear relation Eq. (1). It was found that the curvature of the beam strongly depends on the surface preparation and initiating plating conditions, and varies significantly from specimen to specimen without exhibiting a clear correlation with the process parameters, therefore we will not try to correlate the curvature or the gradient stress with the Young's modulus here.

A typical plot of transverse tip displacement versus transverse force for a Ni beam is shown in Fig. 2. Initially, the displacement u increases linearly with increasing force F, but departs from the linear relationship in the large force regime; the linear approximation is only applicable for small deflections of a beam. At large deflections, a different formula is needed [15]. The slope of the plot in the linear regime is used to extract a value for E via Eq. (1). It should be noted that the beams are sufficiently compliant that the contribution to the overall deflection due to compliance of the supports is negligible and can be ignored in the calculation of E.

The effect of plating temperature upon Young's modulus is shown in Fig. 3, for a constant plating current density of J=2 mA/cm^2. The Young's modulus is approximately 205 GPa at a temperature of less than 60 °C, and this is close to the value of 210 GPa for bulk Ni. However, the modulus drops drastically to approximately 100 GPa at 80 °C. The inclusion of ammonium and sulphate ions by hydrolysis is believed to be responsible for the sharp drop. The Ni layer plated at 40 °C has a value of 228±15 GPa that is higher

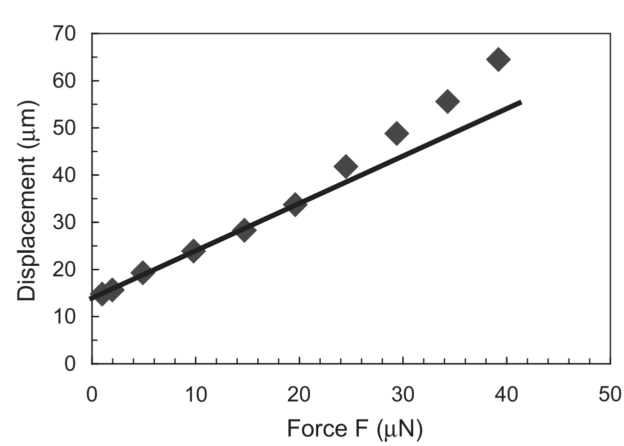

Fig. 2. The compliance of a representative cantilever Ni beam, electroplated at T=60 °C and J=8 mA/cm^2.

[2] NewWave Research, laser machining system mode PXC.

than that of bulk value, but the difference is within the experimental scatter. At 80 °C, however, the Young's modulus drops to approximately 100 GPa, about half of the bulk value. The dramatic reduction of Young's modulus has been reproduced in repeated experiments, confirming that it is not due to experimental scatter.

The effect of plating current density in the range of 2–30 mA/cm^2 upon Young's modulus is plotted in Fig. 4. The results were obtained at a plating temperature of 60 °C. As the plating current density increases, the Young's modulus decreases linearly from a near bulk value of 205 to 85 GPa.

The dramatic reduction of E at 80 °C is repeatable and may be attributed to the formation of sulphate and ammonium ions at T>70 °C due to hydrolysis [11]. It is expected that the inclusion of these nonmetallic species will change the microstructure and atomic arrangement of plated Ni, leading to a weaker and more compliant Ni film. The plating rate for the 80 °C samples was about 50% greater than those at $T \leq 60$ °C despite the same current density, indicating some inclusion of other species in the Ni film, and this is under further investigation.

The decrease of Young's modulus with increasing plating current density can be explained as follows. At low plating current density, the plating is limited by the reaction rate at the interface between solution and sample. There is a sufficient supply of Ni ions to the cathode, and the reduced Ni atoms have sufficient time to migrate to a relaxed position and to fill any remaining pores and gaps. The deposited Ni film is hence dense and possesses the bulk value of Young's modulus. As the plating current density increases, the plating rate rises, and the limited supply of Ni ions to the cathode leads to the formation of a depletion layer of Ni ions near the cathode surface: the so-called mass-transport limitation. Any incoming Ni ions will be captured by outgrowing microstructures, leaving behind pores and gaps in the film. The deposited layer is porous, rough and possesses a tensile residual stress [11]. Our AFM measurement on Ni samples showed the RMS value of roughness of the Ni film increases from 11 to 25 nm as J increased from 2 to 20 mA/cm^2.

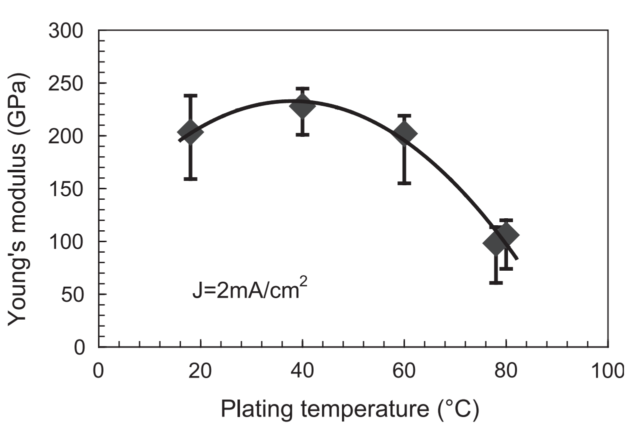

Fig. 3. Young's modulus of Ni thin film as a function of the plating temperature, with J=2 mA/cm^2.

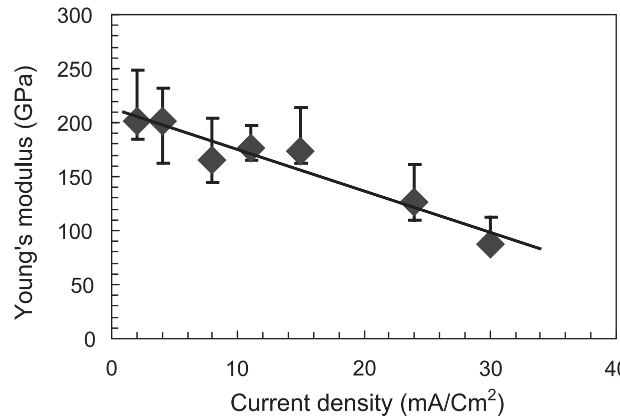

Fig. 4. Young's modulus as a function of plating current density plated at T=60 °C.

The mechanical properties of electroplated Ni strongly depend on the plating bath and process conditions. A Young's modulus of 182 GPa was obtained from thick Ni structures using a Watts bath, while a value of 93 GPa was obtained from those plated using a Sulphamate bath at a current density of 30 mA/cm^2 at 55 °C [15], which agrees with the value of 85 GPa obtained from our sample plated at 30 mA/cm^2.

Electroplated metal films typically have a columnar structure when plated at high temperatures and low rates. This becomes fine-grained with increasing the current density [13,14,18]. The fine-grained structure has been attributed to the low Young's modulus of 93 GPa, while the columnar structure of Ni corresponded to a high Young's modulus of 182 GPa [18]. This microstructural change is believed to be responsible for the change in Young's modulus observed here. The surface of electroplated Ni films have been investigated by SEM, and no cracks and pores were found under a magnification of 75 K, indicating that the pores in the film are in the nanometer range or even smaller.

In conclusion, the Young's modulus of thin film Ni formed by electroplating has been investigated as a function of temperature and plating current density. A plating temperature of 20 °C and a low current density (J=2 mA/cm^2) produces a film with a Young's modulus of 205 GPa close to that of bulk Ni. An elevation in plating temperature to 80 °C or of current density to J=30 mA/cm^2 leads to a drop in modulus by a factor of more than 2. It is postulated that the drop in modulus is associated with an increase in porosity of the coating. The sensitivity of modulus to the plating conditions is significant for the design and fabrication of MEMS devices using plating technologies such as LIGA.

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