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MICROBRIDGE TESTING OF YOUNG'S MODULUS AND RESIDUAL STRESS OF NICKEL FILM ELECTROPLATED ON SILICON WAFER

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Microbridge testing is used to measure the Young's modulus and residual stresses of metallic films. Nickel film microbridges with widths of several hundred microns are fabricated by Microelectromechanical Systems. In order to measure the mechanical properties of nickel film microbridges, special shaft structure is designed to solve the problem of getting the load-deflection curves of metal film microbridge by Nanoindenter XP system with normal Berkovich probe. Theoretical analysis of the microbridge load-deflection curve is proposed to evaluate the Young's modulus and residual stress of the films simultaneously. The calculated results based on the experimental measurements show that the average Young's modulus and residual stress are around 190GPa and 175MPa respectively, while the Young's modulus measured by Nanohardness method on nickel film with silicon substrate is 186.8±7.34GPa.

KEY WORDS *nickel film microbridge, MEMS, mechanical property, load-deflection measurement*

1. Introduction

Microelectromechanical systems (MEMS) is a new technology to manufacture microsystem, microdevices and microstructure whose dimensions are only a few hundred microns. The materials used in MEMS are always in thin film form, based on certain substrates or composited with other thin films, which have an important role on the performance of MEMS devices and microstructures. The deposition processes of thin films and different thermal expansion coefficients always lead to residual stresses in thin films, which may change the performance of the devices. Characterizing, understanding and controlling the mechanical properties of MEMS materials have been an active research area during the recent years[1−5]. The mechanical behavior of material with thickness of a few microns might differ from those of the bulk material due to its size effect, micromachining method or specific microstructure *etc.* However, it's difficult to establish a suitable technique and standard with high degree of accuracy for the measurements of mechanical properties of MEMS materials. Early work on the measurements of mechanical properties of thin films involved in indentation methods, wafer curvature testing, bulge testing, microtensile testing, resonant frequency testing and beam bending method.

The submicron indentation of thin films on substrates is a common method to measure hardness and Young's modulus, while thin film on different substrate and large pressure of the indenter may have influence on thin film[6]. Wafer curvature methods can be used to measure the average stress and strain of thin film, but the stresses are affected by the thermal expansion or growth mismatch between the substrate and thin film, and also the measured stress is an average value of large part of film with substrate[7]. The bulge test uses square or rectangular membrane to determine the residual stress and elastic modulus from the stress-strain curves, but the stress concentration occurring at the four corners made it difficult to measure the yield strength and fracture strength of thin film[8], and the surface flaws presented in thin films may bring some errors. The sample holding problem is also occurred in microtensile testing[9], because the fragility of thin films and the flaws in sample are difficult to avoid in tensile testing. The resonant frequency method is used to measure the elastic modulus of a cantilever beam, but the experimental error may be large[10]. In order to avoid some of these difficulties, a new method based on the deflection of a freestanding cantilever microbeam has been developed[11]. This method eliminates all the substrate effects, and can measure both elastic and plastic properties, namely Young's modulus and yield strength; also the experimental error is low by appropriate selecting the size of the microbeam. However, this method may also bring some errors in determining the Young's modulus due to the spring of Nanoindenter, undercutting or insufficient etching at the beam support, slippage between the load applicator and the beam[12]. In order to obtain more accurate data, Espinosa *et al.*[1] propose a 3-D computational modeling for testing thin films in RF (radio frequency) MEMS switches, that is, membrane deflection experiment (MDE) and numerical simulation were used to obtain the Young's modulus and residual stress of freestanding thin membranes in RF MEMS switches. In this method, Nanoindenter was used to measure the membrane deflection, and finite element modeling was conducted using ABAQUS Implicit, version 5.7 in order to obtain the accurate values. Its main advantage is that measurement can be done accurately on wafer level. Later, Espinosa and his co-workers[3–5] modified this MDE to measure the mechanical properties of freestanding thin film, and the Young's modulus was obtained using a very simple equation. However, this method is very complicated and expensive, and also critical for measuring conditions. Recently Zhang *et al.*[13] has set up a novel analysis method to evaluate Young's modulus as well as residual stress and bending strength simultaneously for thin film in the form of microbridges. This method uses MEMS to fabricate samples and the sample holding problem and substrate effect can be avoided. In the same time, many samples having different sizes can be fabricated on the same wafer. Nanoindenter is used to measure the load-deflection curves of thin film microbridges, and by combining the theoretical analysis model, the Young's modulus and residual stress can be obtained. It's really an effective way to evaluate two basic parameters for materials in MEMS: Young's modulus and residual stress.

But their work was focused on non-metal films, such as silicon nitrides and oxides. For metal films, it is more difficult to get microbridge structure. One reason is that metal films are almost impossible to be patterned by dry etching, such as reactive ion etching (RIE), or by chemical wet etching. During the wet etching processes, it's difficult to control the micro size exactly. The other reason is that during the bulk silicon etching, the metal film can hardly withstand the chemical etching solution for a long time. Boutry *et al.* has ever tried to fabricate metal thin film microbridges by RIE to get rid of the silicon substrate, but the particle bombardment during RIE has some alternation of film characteristic[14].

In the present work, MEMS was used to overcome these difficulties, and succeeded in fabricating the nickel film microbridges. The residual stress and Young's modulus of nickel film microbridges were evaluated based on the measured load-deflection curves.

2. Fabrication of Nickel Film Microbridges and Experimental Method

Nickel film is one of the most important MEMS materials and many nickel film microstructures are electroplated with photoresist mask[15–17]. In this paper, microbridge samples are electroplated Ni films on single crystal silicon substrate. The major microfabrication steps for the microbridges are shown in Fig.1. (a) Three inches, p-type (100) silicon wafer was thermally oxidized. The thickness of SiO_2 was around 1.8μm. One side of the SiO_2 layers was etched off in buffered HF solution, while the other side of the SiO_2 layer was patterned to determine the rectangular silicon etching window. (b) On the exposed silicon side, Cr layer with a thickness of 15nm was sputter-deposited as adhesion layer, and then a layer of Ni was sputter-deposited as seed layer for electroplating. This seed layer of Ni was about 50nm. (c) Bulk Ni film was electroplated to the desired thickness, then patterned by diluted $FeCl_3$ solution. The very thin layer of Cr film was also removed by $Ce(SO_4)_2 \cdot 2(NH_4)_2SO_4$ solution to expose silicon substrate. (d) Silicon substrate was bulk micromachined by KOH anisotropic etching with the etch mask of SiO_2. The etching conditions are: T=80℃, H_2O : KOH=100 : 44 (weight ratio). To prevent Ni film from long time immersing in the hot KOH solution, the silicon substrate was put into a boot clamp, leaving only the side of Si to contact KOH. After the silicon substrate was etched through, the Cr layer under the Ni bridge was removed again by $Ce(SO_4)_2 \cdot 2(NH_4)_2SO_4$ solution. Thus, a freestanding nickel film microbridge was fabricated. The length of the microbridge ranges from 1000 to 2000μm, and the width ranges from 200 to 1000μm. The thickness of the microbridges for all samples was 3.7μm. The distances between each microbridges were larger than 500μm. The structure of the fabricated Ni film microbridge is shown in Fig.2.

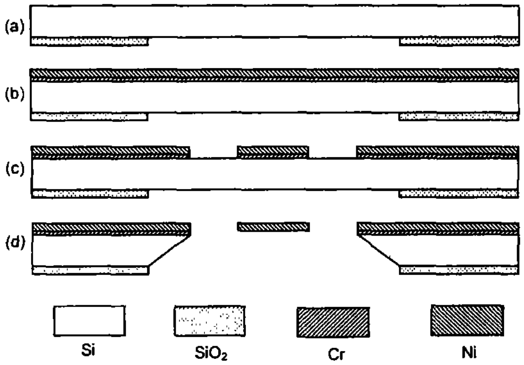

Fig.1 Fabrication processes of nickel microbridges:
(a) pattern the SiO_2 layer;
(b) electroplate the Ni film;
(c) pattern the Ni film;
(d) bulk-machined the silicon substrate.

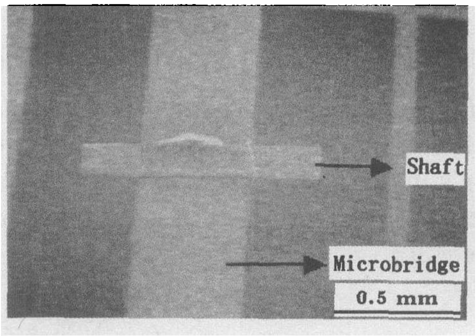

Fig.2 Structure of Ni film microbridge.

The Ni film microbridge testing was conducted on a Nanoindenter

XP system with normal Berkovich probe because long wedge tip is not available at present. In order to distribute the indentation force uniformly on the center of the bridge, a stiff shaft was fabricated by precision machining and glued at the center of the bridges. When the Berkovich probe pressed on the ceramic shaft, a similar linear load behavior was realized as that with a wedge tip. The material for the shaft was a kind of stiff ceramics, and its size is $600\mu\text{m}\times 80\mu\text{m}\times 50\mu\text{m}$.

3. Analysis

3.1 Effects of the shaft on the center deflection

Since the shaft fixed on the bridge center has certain size, the load distribution at the bridge center must not be the same as that with an ideal wedge tip. To evaluate its influence, FEM (finite element method) analysis is conducted to get the variance. The analysis is performed in the software package ANSYS 6.0 University High. Fig.3 shows the dependence of the microbridge deflection on the length ratio of the shaft and the microbridge. When the length ratio is within 10%, the deflection variance is within 3% (shown in Fig.3). For the tested samples, the length of the shaft is $80\mu\text{m}$, while the length of the microbridge is more than $1000\mu\text{m}$, so the influence of the shaft length on the deflection testing should be within 3%. On the other hand, the shaft is fixed on the microbridge center by precision machining method, there must be some excursion of its position from the central place. Fig.4 shows the deflection variance as a function of the shaft position. It can be noticed that when the deflection of the shaft position is within 12.5% from the center position of the bridge, the deflection variance is just within 5%, which is also acceptable for this measurements.

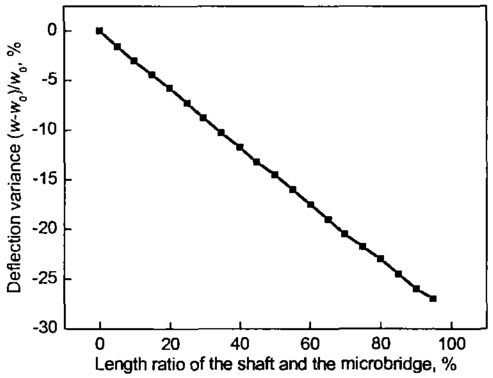

Fig.3 The dependence of microbridge deflection on length ratio of the shaft and the microbridge.

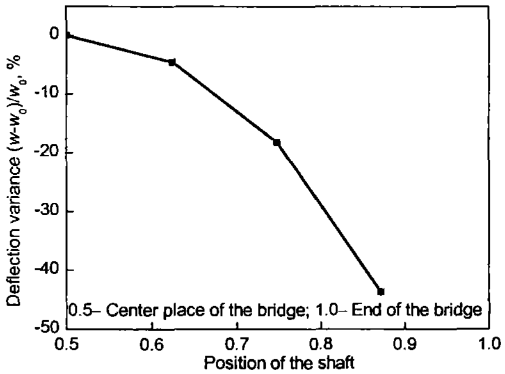

Fig.4 The dependence of microbridge deflection on the positions of the shaft.

3.2 Evaluation of the Young's modulus and residual stress

After the load-deflection curves are gotten by the Nanoindenter measurements, the Young's modulus and residual stress can be determined by fitting the experimental load-deflection curve with the theoretical solution by the least square technique, as described in Ref.[13]

$$S=\sum_{i=1}^{n}[w_i^{\mathrm{e}}(Q_i)w_i^{\mathrm{t}}(Q_i,N_{\mathrm{r}},E_{\mathrm{f}})]^2 \qquad (1)$$

Where n is the number of data, w_i^{e} is the experimentally observed deflection, and $w_i^{\mathrm{t}}(Q_i, N_{\mathrm{r}}, E_{\mathrm{f}})$ is the theoretical deflection obtained by the following equations

$$w = -\frac{Q\tanh(kl/2)}{2N_{\mathrm{r}}k} + \frac{Ql}{4N_{\mathrm{r}}} - \frac{M_0}{N_{\mathrm{r}}}\left[\frac{1}{\cosh(kl/2)} - 1\right] \tag{2}$$

with $k = \sqrt{N_{\mathrm{r}}/D}$, $D = E_{\mathrm{f}}t^3/12$, where Q is the load force per unit width of the microbridge, l and t are the length and thickness of the microbridge, respectively. M_0 is a generalized force connecting the film and the substrate and expressed as follows

$$M_0 = \frac{Q\left[\dfrac{1}{\cosh(kl/2)} - 1\right]}{2k\tanh(kl/2)} \tag{3}$$

The iteration technique is used to regress the Young's modulus E_{f} and residual force N_{r}, which gives the residual stress as $\sigma_{\mathrm{r}} = N_{\mathrm{r}}/t$. In this model, the substrate deformation is neglected because the load-deflection curves are linear, and the metal film microbridge is easy to deflect than the silicon nitride thin film due to the length of Ni film microbridge is longer than its width.

4. Results and Discussion

The sizes of Ni microbridge and the evaluated Young's modulus and residual stress are listed in Table 1, and the typical experimental load-deflection curves of these samples are shown in Fig.5.

From the calculated results, one can find that the average value of Young's modulus of Ni film microbridges is around 190GPa, which is lower than the value of 207GPa of the bulk polycrystalline nickel[18]. While, Sharpe *et al.*[19–21] and Christenson *et al.*[22] have reported significantly lower modulus. Hemker *et al.*[16] measured the dog-bone shaped LIGA (lithographie graphik abformung) nickel microsample by tensile testing, and the measured Young's modulus was 180±24GPa, which is also lower than the value for bulk polycrystalline nickel[18], but in good agreement with that of LIGA Ni microsamples[19,20]. Our results are comparable to the results in references[16,19,20], but higher than that of LIGA Ni microsamples as reported by Stephens *et al.*[15] and Cho *et al.*[17]. This difference may be due to the different electroplating parameters and the testing methods. Normally, the Nanoindenter XP system can measure the Young's modulus of thin films on substrates directly, which is called the Nano-hardness method. The Young's modulus gotten by the nano-hardness method for the Ni sample with silicon substrate is 186.8±7.34GPa, as shown in Fig.6.

The testing results prove that the mathematical equations and the iteration processing to regress the mechanical parameters are somehow accurate to get the Young's modulus values, and the attached shaft structure on the microbridge does little effect on the results.

During the iteration processes, the residual stresses with Young's modulus can be also get in the microbridges. For the electroplating nickel samples, the average value is 174.7MPa for the Ni film microbridges. The large residual stress in the Ni microbridges corresponds to the phenomenon that the nickel film peels up easily at the edge of the three-inch silicon wafer because of the weak adhesion strength in the Ni/Si interface. As

Table 1 Calculated results of the nickel film microbridges

Sample	Size, μm			Young's modulus	Residual stress
	Length	Width	Thickness	GPa	MPa
1	1541	940	3.7	194.3	96.5
2	1541	195	3.7	177.1	188.4
3	1045	445	3.7	193.8	197.8
4	1038	352	3.7	211.1	177.1
5	1045	248	3.7	174.1	213.7
Average value				190.1	174.7

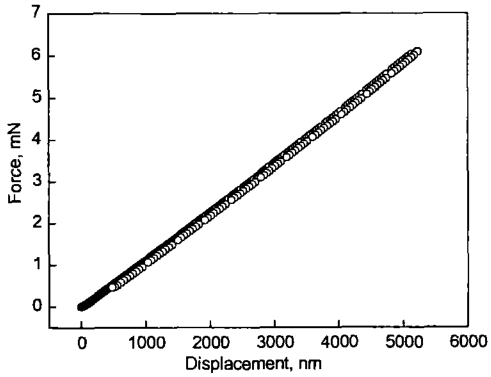

Fig.5 Measured load-deflection curves of the Ni film microbridges for sample 4.

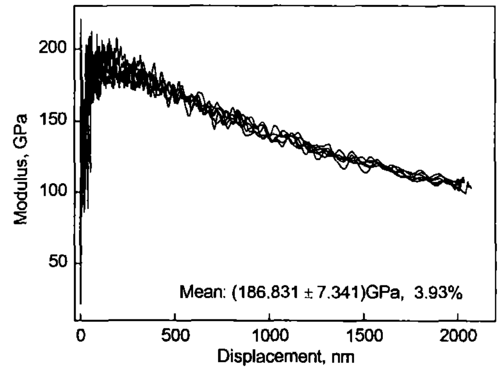

Fig.6 Nanoindentation measurement of nickel film on silicon substrate.

reported in the literature[23–27], Sotirova-Chakarova *et al.*[23] have shown that the residual stress in electroplated Ni film with thickness of several microns is in the range of 60–350MPa and the residual stress is dependent on the deposition conditions. Bastour *et al.*[24] studied the mechanical behavior of microgrippers realized by LIGA technique and found that the residual stress of electroplated Ni film is in the range of 30–160MPa. Manceau *et al.*[25] measured the residual stress of electroplated Ni film using a vibrational technique and it is about 150–300MPa for Ni films with thickness of 10μm. Recently Yi *et al.*[26] and Jeon *et al.*[27] both investigated the mechanical properties of electroless nickel films, and the residual stress is 50–250 and 160MPa respectively for the electroless nickel films. Thus, the residual stress in our electroplated Ni film is reasonable as compared with the results of references[23–27].

In addition, one can find from table 1 that there is a variation in Young's modulus and residual stress for nickel film microbridges. One possible reason is due to the current distribution in the fabrication process of the Ni film, this may result in ununiformity in film thickness. It has been shown that the change in film thickness of 0.05μm will result in a large change in Young's modulus and residual stress, so it is critical for the measurement of film thickness. Further, some flaws in the nickel microbridges may have effect on the measurements. The other possible reason is that the position where the Nanoindenter tip pressed on the microbridge may deviate from the central position of the film microbridge, this may lead to some errors in measuring the load-unload curves. On the other hand, when the Nanoindenter is used to measure the load-deflection curve, at the beginning, the measured data in the load-deflection curve can not be used because the contact state between the tip and the nickel film microbridge is not very stable, this may bring errors in evaluating the Young's modulus and residual stress. Thus, the evaluated data based on the load-deflection curves has some scatter.

5. Conclusions

Microbridge testing is used to measure the Young's modulus and residual stresses of metallic films. Nickel film microbridges are fabricated by Microelectromechanical Systems. In order to measure the load-deflection curve of nickel film microbridges, special shaft structure is designed to solve the problem of getting the load-deflection curves by Nanoindenter XP system with normal Berkovich probe. Theoretical analysis of the load-deflection curve of metal film microbridge is proposed to evaluate the Young's modulus and residual stress of the films simultaneously. The calculated results show that the average Young's modulus and the residual stress are around 190GPa and 175MPa respectively, while the Young's modulus measured by Nano-hardness method on nickel film with silicon substrate is 186.8±7.34GPa.

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