DETERMINATION OF YOUNG'S MODULUS OF PZT AND $Co_{80}Ni_{20}$ THIN FILMS BY MEANS OF MICROMACHINED CANTILEVERS

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Abstract: *This paper presents a technique to determine the Young's modulus and residual stress of thin films using a simple micromachined silicon cantilever as the test structure. An analytical relation was developed based on the shift in resonance frequency caused by the addition of a thin film on the cantilever. FEM simulations were performed which confirmed the validity of assumptions made in our analytical model. Resonance frequency measurements both before and after the deposition of the thin film improve the accuracy of the results. Experiments were performed on PZT deposited by pulsed laser deposition and on evaporated $Co_{80}Ni_{20}$ thin films.*

Keywords : *Young's modulus, cantilever, resonance frequency, PZT, CoNi.*

I- INTRODUCTION

Design of MEMS devices requires detailed information about material parameters such as the Young's modulus. As industry is increasingly focusing on micro devices, we need information on the mechanical properties of materials in the thin film domain, these properties can differ significantly from those of bulk materials. PZT is widely used for both piezo-electric actuation and sensing purposes [1,2]. Printer manufacturers are, for instance, trying to incorporate PZT as an active device layer in inkjet printheads [3]. Recently, excellent ferroelectric properties have been obtained by using PZT deposited by Pulsed Laser Deposition. However, accurate determination of mechanical properties of PZT is being hampered by the fact that up to now only mm-square areas can be deposited uniformly. Micrometer sized measurement devices provide a solution to this problem.

We devised a method to accurately determine the Young's modulus of PZT by using the shift in resonance frequency of micro cantilevers. Bending of the cantilever was used to determine the residual stress in a thin film. Experiments were conducted to measure both the resonance frequency before and after deposition of thin film PZT and $Co_{80}Ni_{20}$.

II- THEORY

II-1 YOUNG'S MODULUS

The relation between cantilever resonance and Young's modulus can be approximated by an analytical model, or calculated accurately using finite element software. In the following section both methods are compared. Subsequently, an analysis of the sensitivity of the calculated Young's modulus to uncertainties in measurement values is made.

II-1.1 ANALYTICAL MODELLING

A dynamic approach, based on the Euler-Bernoulli beam equation and the dependence of the resonance frequency on the flexural rigidity of composite cantilever, was used to develop the analytical model for determination of Young's modulus. The resonance frequency of a single material cantilever is [4]:

$$f_n = \frac{C^2}{2\pi}\frac{t}{L^2}\sqrt{\frac{E}{12\rho}} \qquad (1)$$

where f_n is the resonance frequency (Hz), C is 1.875 for fundamental frequency (f_0), E is the young's modulus, ρ is density, t is the thickness and l is the length of the cantilever.

Due to the addition of a thin film on the cantilever, its neutral axis shifts to a new position and this affects the flexural rigidity of composite cantilever. In addition there is an added mass effect, and both results in a change in the resonance frequency. By measuring this change in the resonance frequency before and after the deposition of thin film, one can compute the Young's modulus of an additional thin film deposited on the cantilever by using equation (2). A relation was developed on the basis of the new position of the neutral axis and on the assumptions that the material is linearly elastic, has uniform cross-section and that there is small cantilever deflection [4,5].

Symbols used in the equations are defined in Table 1. The Young's modulus introduced in equation (2) is the biaxial Young's modulus [6]:

$$E_f = \frac{1}{t_f^{\,3}} \left[\begin{array}{l} -2E_s t_s^{\,3} - 3t_f E_s t_s^{\,2} - 2E_s t_s t_f^{\,2} + 6(t_s\rho_s + t_f\rho_f)B \\ +2\sqrt{\begin{array}{l} E_s^2 t_s^{\,2} t_f^{\,4} + 3t_s^{\,3} E_s^2 t_f^{\,3} + \left(4E_s^2 t_s^{\,4} - 3AB\right) t_f^{\,2} + \\ \left(3E_s^2 t_s^{\,5} - 9ABt_s\right) t_f + E_s^2 t_s^{\,6} - 6ABt_s^{\,2} + \\ 9(t_s\rho_s + t_f\rho_f)^2 B^2 \end{array}} \end{array} \right] \tag{2}$$

where, $A = E_s t_s (t_s\rho_s + t_f\rho_f)$ *and*

$$B = \left(\sqrt{\frac{E_s t_s^{\,3}}{12 t_s \rho_s}} - 0.568\pi\Delta f_0 L^2 \right)^2$$

One of the most uncertain parameters is the thickness of the cantilever (t_s). By measuring the resonance frequency before deposition, the error caused by this uncertainty can be reduced [7].

Table-1: List of symbols

Symbol	Meaning
E_f	Biaxial Young's modulus of additional film
E_s	Biaxial Young's modulus of base layer (silicon)
t_f	Thickness of additional film
t_s	Thickness of base layer (silicon)
ρ_f	Density of additional film
ρ_s	Density of base layer (silicon)
L	Length of cantilever
Δf_0	Change in resonance frequency before and after deposition of thin film

II-1.2 FINITE ELEMENT SIMULATION

To check the validity of the assumption made in the analytical model, a finite element simulation was carried out using the COMSOL software package.

A full 3D Eigenfrequency analysis was performed for the silicon cantilever and composite cantilever separately, using the structural mechanics section of the MEMS package. Standard anisotropic elastic properties of single crystal silicon were used. In the model, the length axis of the cantilever was aligned parallel to the <110> direction of single crystal silicon. The additional film was treated as a fully isotropic material [8]. Different mesh sizes were used to observe the mesh dependency of the results. A 0.02 % deviation was observed when the number of elements increased fourfold, so we assumed that our mesh size was sufficient.

Table-2: Parameters used for comparison between FEM and analytical model

Parameter	Value
E_f	70 GPa
ρ_f	7700 kg/m^3
L	220 μm
W	33 μm
t_s	3 μm
t_f	100 nm

The FEM simulation was performed by assuming realistic values of the cantilever's geometrical dimensions and the Young's modulus of thin film as shown in Table 2. The shift in resonance frequency with and without the additional thin film obtained from eigenfrequency analysis of FEM simulation was used as an input in the analytical model described by equation (2) to calculate the Young's modulus of thin film. As a result, it was found that analytically calculated value of Young's modulus is about 14 % higher than the value assumed for FEM simulations.

II-2 RESIDUAL STRESS

Residual stress is the tension or compression in a thin film which can be calculated from the curvature of the cantilever that is caused by the addition of the thin film. The thin film was deposited after the release of the cantilever from the substrate. Deflection of the free end of the cantilever was measured both before and after deposition. The residual stress in the thin film was determined using Stoney's equation, which is valid when the thickness of film is very small as compared to the thickness of substrate [7]:

$$\sigma_f = \frac{1}{6}\frac{E_s t_s^{\,2}}{R t_f} \tag{3}$$

where,
σ_f= residual stress in thin film
R = radius of curvature of the beam = $L^2 / 2\xi$
ξ = deflection at the free end of beam.

The deflection of the cantilever due to the residual stress in the thin film was modelled in the COMSOL finite element software using the same assumptions as those listed in Table 2 and assuming 600 MPa of initial stress in the thin film. The stress in the base layer of the cantilever was set to zero. The bending of the cantilever is shown in Figure 1. The radius of curvature related to that deflection was then used in equation (3) to calculate the residual stress of the thin film, yielding a value of 621 MPa, which is about 3 % higher than the assumed value.

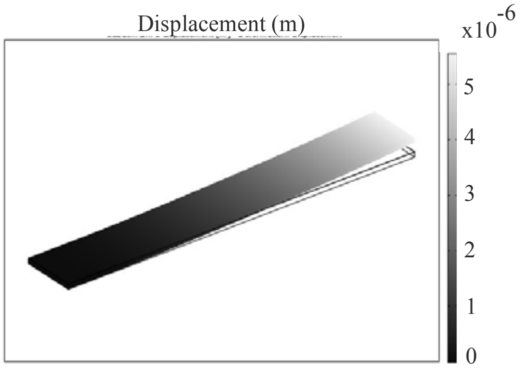

Figure 1 : Cantilever curvature due to the thin film, modelled in COMSOL

II-3 ERROR ANALYSIS

Any error in measurement of the physical parameters or the frequency will affect the final calculated value of the Young's modulus of the thin film. Error analysis using equation (2) shows that the results are sensitive to the geometry of cantilever. The error in the Young's modulus of thin film was calculated using equation (4):

$$\frac{\Delta E_f}{E_f} = \frac{\partial E_f}{\partial x}\left(\frac{x}{E_f}\right)\left(\frac{\Delta x}{x}\right) \qquad (4)$$

where x is any of the concerned parameters used in the right hand-side of equation (2). The same procedure was used to calculate the error in the residual stress by using equation (3).

III- MEASUREMENTS

The cantilevers used in the experiments were made from (100) single crystal silicon wafers and were aligned parallel to the <110> direction. The resonance frequency was measured both before and after deposition by using a MSA-400 Micro System Analyzer scanning laser-Doppler vibrometer under ambient conditions.

Pulsed laser deposition was used to deposit a 100 nm thickness (calibrated by SEM and determined by deposition time) of PZT whereas 30 nm (determined by a quartz crystal thickness monitor) of $Co_{80}Ni_{20}$ was deposited by e-beam evaporation.

The length, width and thickness of the cantilever used for the PZT deposition were 224, 22 and 2.5 µm respectively. For $Co_{80}Ni_{20}$ the corresponding values were 222.5, 28 and 2.5 µm. The difference in the resonance curves measured for the cantilever with and without a PZT thin film is shown in Figure 2. The resonance frequency was obtained by curve fitting with the theoretical expression for a second order mass-spring system with damping. The resonance frequency drops from 70.79 to 68.46 kHz, a drop of 2.33 kHz. For $Co_{80}Ni_{20}$, these values were 72.47 and 71.85, so a drop of 0.62 kHz. The errors in the measured frequencies are of the order of 1 Hz.

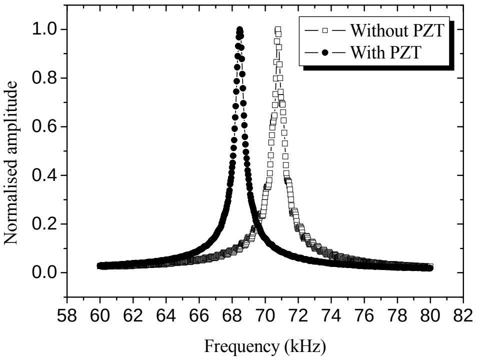

Figure 2 : Measured resonance frequency before and after the deposition of PZT

The MSA-400 micro system analyzer was used for the static topography measurements to obtain the deflection of cantilever. For the PZT deposited cantilever, the upward deflection (ξ) was measured to be 6.2 µm as shown in Figure 3.

IV- RESULTS AND DISCUSSION

With the measured change in resonance frequency, the Young's modulus of thin films was calculated as 83 ± 10 GPa for PZT and 138 ± 11 GPa for $Co_{80}Ni_{20}$ using equation (2). Biaxial Young's modulus of 180.4 GPa was used for single crystal silicon in the analytical model [6,9].

An analysis of the propagation of errors in the value of Young's modulus due to the experimental measurements was performed. There was a cumulative error of 12 % for the realistic values of errors in the various parameters used in equation (2) for PZT and a 8 % error was calculated for $Co_{80}Ni_{20}$. Individual values of errors are listed in Table 3. As can be seen, the error for the film thickness is dominant.

Table-3: Error analysis for Young's modulus

	Error in Parameter	Error in Young's modulus of PZT	Error in Young's modulus of $Co_{80}Ni_{20}$
L	0.5 %	1.2 %	0.64 %
t_s	3 %	0.5 %	0.1 %
t_f	10 %	10.5 %	6.1 %
E_s	2 %	3.2 %	2.6 %
ρ_f	2 %	4.2 %	3.2 %

For the measured bending of the cantilever covered with PZT, a tensile residual stress of 465 ± 57 MPa was calculated.

Topography measurements of the bent cantilever with additional PZT film was compared with the bending of cantilever obtained from FEM simulation as shown in Figure 3. The measured bending was 2 % lower than predicted by COMSOL, most likely caused by the uncertainties about the film thickness.

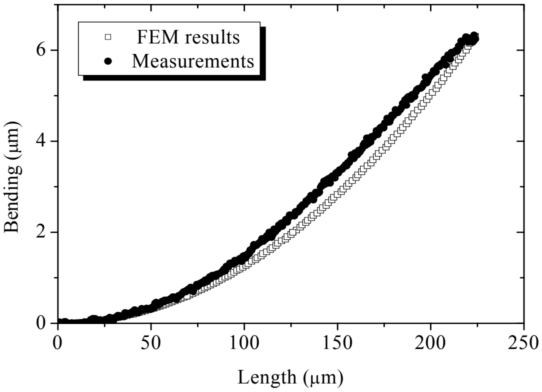

Figure 3 : Curvature of the PZT deposited cantilever

V- CONCLUSIONS

The Young's modulus and residual stress of thin films can be determined by deposition on a cantilever and by then measuring both its change in resonance frequency and beam curvature.

An analytical model based on the Euler-Bernoulli equation was developed and used to relate the change in resonance frequency to the Young's modulus. The validity of the assumptions made in the analytical model was confirmed by 3D finite element simulations, using the anisotropic Young's modulus of a single crystal silicon.

Deposition on soft cantilevers also allows for the simultaneous determination of the residual stress in the film, using the curvature of cantilever bending both before and after deposition of the thin film with the help of Stoney's equation.

Readily fabricated micro-cantilevers were used as test structures. Accurate determination of the Young's modulus depends on the precise values of the physical dimensions of cantilever, thickness of the thin film and experimental measurement of the resonance frequency of cantilever. A thorough error analysis was performed to calculate the propagation of errors in these parameters to the calculated values for the Young's modulus and stress. Uncertainty about the thickness of the deposited thin film is the dominant source of error.

A $Co_{80}Ni_{20}$ alloy, used in MFM probe fabrication, was investigated using this method. The Young's modulus was calculated as 138 ± 11 GPa, which is lower than values previously reported in the literature for bulk cobalt and nickel (210 GPa and 200 GPa respectively [10]).

The Young's modulus of pulsed laser deposited PZT was determined as 83 ± 10 GPa, which lies in the range of values quoted in the literature varying from 25 to 260 GPa [11,12]. The residual stress in the PZT thin film was determined to be 465 ± 57 MPa.

VI- ACKNOWLEDGEMENTS

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VII- REFERENCES

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