Measurement of Polymer Mechanical Properties Using Microfabricated Resonant Structures

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Introduction

Polyimides have been widely used in microelectronics applications. For example, polyimides have found application as interlayer dielectrics, chip encapsulants, and packaging materials for integrated circuits [1]. More recently, polyimides have been used as mechanical elements in microsensors and microactuators, on-chip elements which possess the ability of mechanical motion [2]. In either application, accurate knowledge of the mechanical properties of the polyimide is of great importance in the success or failure of the microfabricated device. Mechanical properties of interest include residual film stress, Young's modulus, Poisson's ratio, and adhesion, both of the polyimide to a substrate and of thin films to the polyimide itself.

In selecting a mechanical property measurement test, it is important to remember that the mechanical properties of interest must be measured in-situ; that is, on as-deposited films. A variety of tests are available for this in-situ measurement such as wafer curvature [3], membrane load-deflection [4], and bending-beam techniques [3]. Resonant methods have also been used to determine the Young's modulus of microfabricated silicon cantilever beams [5]. In this work, we apply resonant techniques to determination of residual stress in polyimide films by measuring the mechanical resonant frequency of thin polyimide 'strings'.

Theoretical

The mechanical resonant frequency of a structure can be related to the structure mechanical properties by using the Rayleigh method [6]. In this method, the maximum kinetic energy of a vibrating structure is equated to the maximum potential energy stored in the deformation of the structure. For a string under residual tensile stress σ, it can be shown that the resonant frequency of the string can be related to the residual stress by the equation:

$$f = \frac{1}{2L}\left(\frac{\sigma}{\rho}\right)^{0.5} \qquad (1)$$

where σ is the tensile stress in the string, L is the string length, ρ is the material density, and f is the string resonant frequency in Hz. Thus, if the string length and density are known, measurement of the string resonant frequency will directly yield the residual tensile stress in the string. For typical stresses in polyimide films of 30 MPa, application of equation (1) suggests that mechanical resonances in the audio regime can be obtained with string lengths on the order of 1 cm, easily attainable on a silicon wafer.

Fabrication

The resonant string structures were fabricated using standard micromachining techniques as shown in Figure 1. The starting material was a single-side polished <100> n-type silicon wafer two inches in diameter, heavily boron-doped on the front side [4]. A 3500 Å oxide film was grown on the wafer. This oxide was patterned using photolithography to open a window in the backside and the wafer was anisotropically etched in 20 wt% potassium hydroxide solution at 56 °C for 15 hours. This etch stopped on the boron-doped layer, forming a square silicon diaphragm 8 mm on a side and 5 μm thick. The masking oxide was then removed in hydrofluoric acid and the polyimides of interest (DuPont PI-2555 and PI-2611) were spun on the front side of the wafer in multiple coats at a speed of 2500 rpm with a prebake of 120 °C for 10 minutes in air between each coat. The final cure was carried out at 300 °C for one hour in air. A total of two coats was used, resulting in an after-cure thickness of approximately 5 μm. An aluminum film 1000 Å thick was then evaporated onto the front of the wafer and patterned into strings. This film was used as a hard mask during an O_2/CF_4 plasma etch of the underlying polyimide. After etching, the aluminum was removed in a wet etch and the thin silicon diaphragm was removed in a CF_4/O_2 plasma etch from the back, releasing the strings. Figure 2 shows a photograph of the fabricated structure.

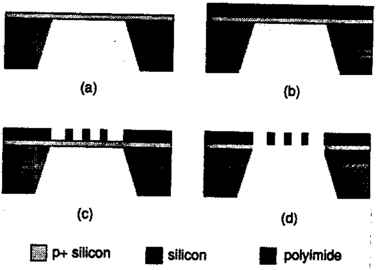

Figure 1. Fabrication sequence for resonant strings (side view). (a) after anisotropic etch; (b) after polyimide deposition and cure; (c) after polyimide patterning; (d) after removal of silicon membrane

Testing and Results

The wafer with strings was mounted in a test jig as shown schematically in Figure 3. As the expected mechanical resonances were in the audio regime, a piezoelectric audio speaker driven by a variable-frequency (500 Hz - 20 kHz) function generator was used to generate the acoustic excitation. The sound waves thus generated were passed through the strings where they were sensed by a high-sensitivity ceramic microphone. The output of the microphone was fed to an oscilloscope

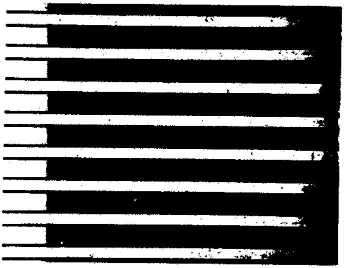

Figure 2. Microphotograph of fabricated resonant strings. The strings shown above are 50 microns in width and 8 millimeters in length. The strings are shown supported on the substrate at left, and suspended over the hole in the substrate at right.

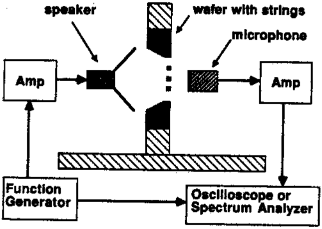

Figure 3. Test apparatus used for resonant frequency measurement (schematic side view)

which recorded the amplitude of the transmitted wave. When the frequency of the excitation is exactly matched to the mechanical resonant frequency of the strings, an energy transfer occurs which causes the strings to vibrate. Some of this vibration energy will be lost to the substrate. The transmitted energy will therefore be reduced, resulting in a decreased signal at the sensing microphone at the resonance frequency. By sweeping the incident frequency from low to high and locating the point where the transmitted energy (microphone output) is a minimum, the mechanical resonant frequency of the strings can be determined.

In order to reduce errors associated with extraneous resonances, two approaches were taken. First, the speaker, strings, and microphone were shielded with acoustic absorption foam to minimize outside interference as well as to reduce echoes and chamber resonances not associated with the mechanical resonances of the strings. Second, a baseline measurement of transmission as a function of frequency was taken on a wafer which had no strings, only the 8x8 mm hole. This spectrum was used to correct and normalize the spectrum taken on the wafer with the strings suspended over the hole.

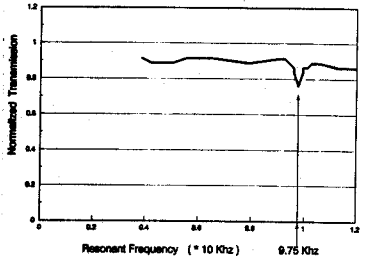

Figure 4. Typical normalized transmission data for the strings shown in Figure 2. The observed resonance frequency is 9.75 kHz.

Figure 4 shows a typical spectrum (acoustic transmission as a function of frequency) normalized as described above, obtained for DuPont PI-2555. The observed resonant frequency for this material was 9.75 kHz. Use of equation (1), along with an estimated value for the polyimide density of 1.4 g/cm^3 [1], yielded a value for residual stress of 34 MPa. This value is in good agreement with literature values [4] published for this material.

Conclusions

A microfabricated test structure for the in-situ measurement of thin polymer film residual stress has been described. Preliminary results indicate that residual stresses for polyimide films measured by this method are in good agreement with other in-situ methods. Current work involves extending this method to quantitative measurement of Young's modulus and Poisson's ratio, qualitative assessment of adhesion and fatigue, and measurement of the properties of various other thin film polymer materials.

Acknowledgement

This work was supported in part by Digital Equipment Corporation, IBM, and Motorola through the Georgia Tech Manufacturing Research Center. The donation of the polyimides studied in this work by DuPont is also gratefully acknowledged. Microfabrication was carried out in the Georgia Tech Microelectronics Research Center.

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