

# Thermal expansion coefficient of polycrystalline silicon and silicon dioxide thin films at high temperatures

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The rapid growth of microelectromechanical systems (MEMS) industry has introduced a need for the characterization of thin film properties at all temperatures encountered during fabrication and application of the devices. A technique was developed to use MEMS test structures for the determination of the difference in thermal expansion coefficients ( $\alpha$ ) between poly-Si and SiO<sub>2</sub> thin films at high temperatures. The test structure consists of multilayered cantilever beams, fabricated using standard photolithography techniques. An apparatus was developed to measure the thermally induced curvature of beams at high temperatures using imaging techniques. The curvatures measured were compared to the numerical model for multilayered beam curvature. The model accounts for the variation in thermomechanical properties with temperature. The beams were designed so that the values of Young's moduli had negligible effect on beam curvature; therefore, values from literature were used for  $E_{\text{Si}}$  and  $E_{\text{SiO}_2}$  without introducing significant error in curvature analysis. Applying this approximation, the difference in thermal expansion coefficients between  $\alpha_{\text{Si}}$  and  $\alpha_{\text{SiO}_2}$  was found to increase from  $2.9 \times 10^{-6}$  to  $5.8 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$  between room temperature and 900 °C. These results suggest that the  $\alpha$  for poly-Si thin films may be significantly higher than values for bulk, crystalline Si. © 2000 American Institute of Physics. [S0021-8979(00)04109-8]

## INTRODUCTION

The microelectromechanical systems (MEMS) industry is rapidly growing in quantity and diversity. As the applications of MEMS devices become more demanding, there is a need for accurate characterization of thermal and mechanical properties of thin film materials that are used in the devices. Silicon based thin films are the material of choice for many of these applications due to the excellent mechanical properties of silicon, as well as the readily available fabrication technologies developed in the microelectronics industry.<sup>1</sup> Applications of MEMS range from chemical and electrical sensors, inertial sensors, to micromotors and actuators. When the films are used as mechanical components in MEMS, mechanical properties (such as Young's modulus, fracture strength, Poisson's ratio) are needed for accurate modeling and prediction of the structure behavior. Furthermore, modeling of MEMS for high temperature applications requires that these properties be known as functions of temperature. An additional concern at high temperatures is the thermal expansion coefficient  $\alpha$  of the materials, which becomes critical in accurate prediction of device behavior. Thermal expansion coefficient also plays a role in predicting the thermal stress generation within the structure, both during fabrication and in application. Neglecting the effects of thermal

stress may result in deformation of device due to thermal strain, delamination, or fracture in extreme cases.

Due to the well established micromachining technology for silicon, studies of thin film properties thus far have concentrated on the mechanical properties (Young's modulus, fracture strength, and Poisson's ratio) of crystalline or polycrystalline silicon films at room temperature. Techniques vary widely, and many are still under development. In fact, the lack of a "standard" and consistent technique for thin film material property measurement has become a growing concern in the MEMS industry.<sup>2,3</sup>

For example, much research has been conducted to determine the Young's modulus of thin films, and techniques used include mechanical deflection of cantilevers,<sup>4,5</sup> resonance method for cantilevers or comb structures,<sup>6-9</sup> bulge-testing of membranes,<sup>10,11</sup> and tensile testing of micromachined specimens.<sup>12-14</sup> The large scatter in the results is often attributed to differences in sample fabrication technique; however, films made under the same condition show similar inconsistencies in results when tested using different techniques.<sup>3,15</sup> The scatter in data suggests the need for innovative techniques for determining the properties of thin films. Furthermore, most of this work was done at room temperature, and application of the technique at high temperature is difficult due to the delicate instruments required for testing. A technique for finding the mechanical properties of thin films at high temperatures needs to be developed.

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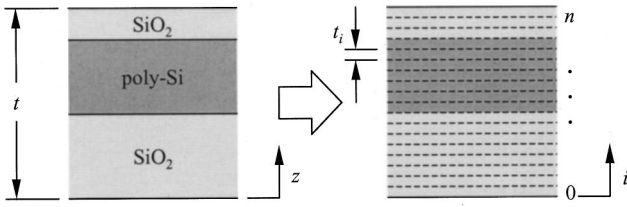


FIG. 1. Discretization of multilayered beam.

Despite their importance in analyzing MEMS response at high temperatures, thermal expansion coefficients of thin films have been less widely studied. The lack of data in literature regarding this property is due to the difficulty in experimentally determining the property for thin films. The main issue in measuring  $\alpha$  of thin films is the small dimension involved; due to the small size of thin film samples, any deflection caused by thermal expansion is often too small to detect directly. Further challenge is added by the nature of the property; thermal expansion coefficients must be studied at very high temperatures to induce any measurable change in test structure. Experimental design and implementation for such systems have proven to be very difficult.

An innovative technique for determining  $E(T)$  and  $\alpha(T)$  of thin films using multilayered cantilever beams has been developed.<sup>16</sup> The technique is based on the thermally induced curvature of the multilayer that results from the difference in thermal expansion coefficients of the layers. The curvature is measured at temperatures of up to 850 °C using an optical curvature measurement system.<sup>17</sup> Determination of property results from comparing the beam response to a numerical model for curvature of multilayers.<sup>18</sup> The technique has been demonstrated on multilayers consisting of polycrystalline Si and silicon dioxide for the determination of  $(\alpha_{\text{Si}} - \alpha_{\text{SiO}_2})$  as a function of temperature.

## NUMERICAL MODEL

The test structure consists of multilayered cantilever beams having layers of poly-Si and SiO<sub>2</sub>. Multilayered cantilevers consisting of materials having different thermal expansion coefficients deflect as they undergo temperature change. A numerical technique for determining the thermally induced curvature of multilayers has been developed by Townsend,<sup>18</sup> and is used in analysis of the structures.

The numerical formulation begins with dividing the layers into  $n$  sublayers, which must have a thickness that is much smaller than the actual layer thicknesses (Fig. 1). The curvature is found by first applying appropriate forces to the layers such that two requirements are met: (1) the dimensions of layers must be the same at the interface, and (2) the sum of forces must equal zero. Next, a moment is applied to the structure in order to balance the internal moment generated by the forces applied in the previous step. The resulting expression for curvature is

$$K = \frac{\sum_{i=1}^n E_i \gamma_i \frac{t_i}{2} \left( -\varepsilon_{0i} + \frac{\sum_{j=1}^n E_j t_j \varepsilon_{0j}}{\sum_{j=1}^n E_j t_j} \right)}{\sum_{i=1}^n E_i t_i \left[ \left( \frac{\pi t}{2} - \frac{t^2}{3} \right) + (t - \pi) \frac{\gamma_i}{2} - \frac{1}{12} (3 \gamma_i^2 + t_i^2 - t^2) \right]}, \quad (1)$$

where  $t_i$  is the thickness of  $i$ th sublayer,  $t$  is the total thickness of all layers,  $E_i$  is the Young's modulus of each layer, and  $\pi$  is the location of neutral axis measured from the bottom of the structure as seen in Fig. 1. The neutral axis is the location through the thickness of the layers about which the total moment is zero, and is defined as

$$\pi = \frac{t}{2} - \frac{\sum_{i=1}^n \frac{E_i t_i \gamma_i}{2}}{\sum_{i=1}^n E_i t_i}. \quad (2)$$

In Eqs. (1) and (2),  $\gamma_i$  is given by

$$\gamma_i = \sum_{j=1}^n \beta_{ij} t_j, \quad (3)$$

where  $\beta_{ij}$  is  $-1$  for  $i > j$ ,  $0$  for  $i = j$ , and  $1$  for  $i < j$ .<sup>18</sup> The term  $\varepsilon_{0i}$  is the initial strain in each term from some reference length; for convenience the reference can be chosen to be the length of one of the layers. In the case of thermally induced curvature, this term is the thermal strain ( $\varepsilon^{\text{thermal}}$ ), measured with respect to the expanded length of one of the layers. For a temperature-dependent thermal expansion coefficient, the thermal strain due to a temperature rise from  $T_1$  to  $T_2$  is given by

$$\varepsilon^{\text{thermal}} = \int_{T_1}^{T_2} \alpha(T) dt. \quad (4)$$

To find the thermal strain in the poly-Si layer using the SiO<sub>2</sub> layer as reference,  $\alpha(T)$  is replaced by  $\Delta\alpha(T)$ , where  $\Delta\alpha$  is defined as  $(\alpha_{\text{Si}} - \alpha_{\text{SiO}_2})$ . Width variation in the layers is treated by replacing the Young's modulus of a layer by an effective modulus  $E^*$

$$E_i^* = \frac{w_i}{w_0} E_i, \quad (5)$$

where  $w_i$  and  $w_0$  are the widths of  $i$ th layer and the bottom layer, respectively.

The thermally induced curvature of multilayers, given by Eq. (1), depends on the ratio between Young's moduli of materials, as well as the difference between their thermal expansion coefficients. For a beam consisting of poly-Si and SiO<sub>2</sub>, the curvature is a function of  $E_{\text{Si}}/E_{\text{SiO}_2}$  and  $\Delta\alpha$ . By using published data for  $E_{\text{Si}}$  and  $E_{\text{SiO}_2}$ , the difference in thermal expansion coefficients between poly-Si and SiO<sub>2</sub> thin films can be determined.

## MEMS TEST STRUCTURE

The test structure used for the experiment is a three-layered cantilever beam, consisting of (from top) 0.19  $\mu\text{m}$  SiO<sub>2</sub>, 0.54  $\mu\text{m}$  poly-Si, and 1.03  $\mu\text{m}$  SiO<sub>2</sub>, suspended over

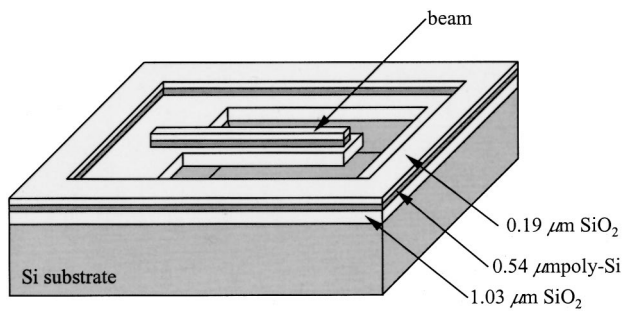


FIG. 2. Schematic of the MEMS test structure.

Si substrate. The beam, shown in Fig. 2, is 100  $\mu\text{m}$  long, and has a top-to-bottom layer width ratio of 0.54. The beams are fabricated on an *n*-type, 3 in. Si wafer. The bottom  $\text{SiO}_2$  layer is thermally grown on the wafer at 1100  $^\circ\text{C}$ . The poly-Si layer is deposited by low pressure chemical vapor deposition (LPCVD) at 610  $^\circ\text{C}$ . The top  $\text{SiO}_2$  layer is deposited by LPCVD at 420  $^\circ\text{C}$ . The two top layers are patterned photolithographically to form the top beam layer. A thin layer of thermal  $\text{SiO}_2$  is grown on the layers to protect the poly-Si layer during the final etching process. The layer is approximated to be 0.2  $\mu\text{m}$  thick on the sides of the poly-Si layer; however, the growth on the  $\text{SiO}_2$  layers are believed to be negligible. The bottom  $\text{SiO}_2$  layer is patterned photolithographically to form the bottom layer, and the beams are released from the substrate by etching the Si substrate. The final etch process creates a 23  $\mu\text{m}$  well under the beam. The completed beams have an upward curvature at room temperature due to residual stress, as shown in the SEM micrograph in Fig. 3. The beam tip deflection due to residual stress typically ranges between 10 and 7  $\mu\text{m}$  for a 100  $\mu\text{m}$  beam.

The effect of  $E$  on the thermally induced curvature of beams varies dramatically with the type of structure used. A parametric study using the numerical model indicates that the curvature of a beam with its neutral axis passing through or near a layer interface experiences negligible effect from variations in  $E$ .<sup>16</sup> This characteristic allows the use of approximate values for  $E$  without introducing significant error in the curvature analysis. Such customized beam design is ideal for determining  $\Delta\alpha$ , since error due to uncertainties in

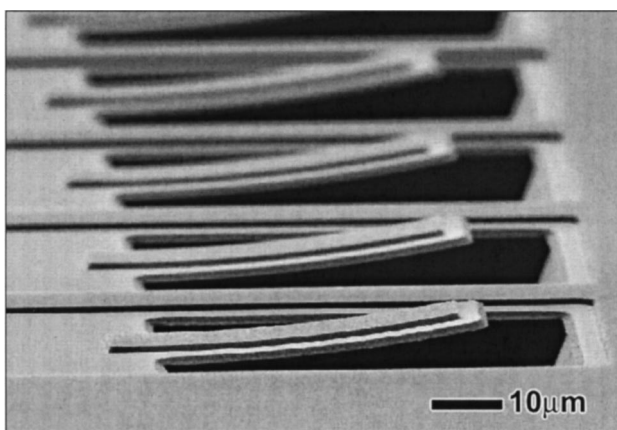


FIG. 3. SEM micrograph of test structure.

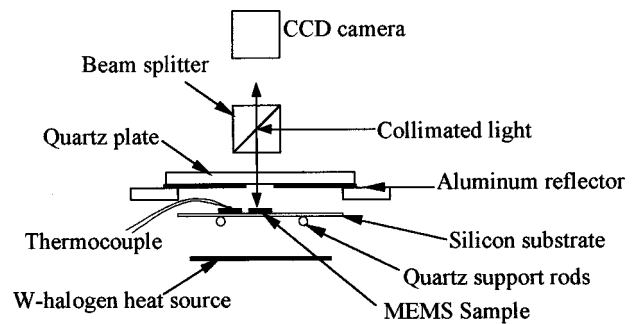


FIG. 4. Schematic of the experimental setup.

$E$  values are negligible. The MEMS test structure used in this study was designed to have its neutral axis pass near the interface between the bottom  $\text{SiO}_2$  and poly-Si layer; the layer thicknesses are such that  $\pi$  lies in the bottom  $\text{SiO}_2$  layer at approximately 0.8  $\mu\text{m}$  from the bottom of the beam. This design effectively reduces the number of unknown properties used in the curvature analysis by allowing the use of literature values for  $E$  for the analysis.

## EXPERIMENTAL SETUP

A novel imaging technique for the measurement of thermally induced curvature of microcantilevers was developed.<sup>17</sup> The schematic of the setup is shown in Fig. 4. The apparatus consists of a tungsten-halogen lamp for heating, collimated light source for illumination, and charge-coupled device (CCD) camera for the visualization of the beams. The curvature is monitored optically by the CCD camera, which is equipped with a telescopic lens with a field-of-view of approximately 400  $\mu\text{m}$ . Collimated light reflecting off the sample is scattered by the curved beams. The numerical aperture of the lens is limited; therefore, only the nearly flat portion of the beam is seen by the camera, resulting in an "apparent length" of the beam ( $l_{\text{beam}}$ ). The curvature is found by relating  $l_{\text{beam}}$  to the beam geometry. The system allows the measurement of both positive and negative curvatures, and reaches a temperature of up to 850  $^\circ\text{C}$ . The temperature limit is defined by the heater; the system is capable of reaching higher temperatures if a more powerful heat source was used. The temperature during the experiment is monitored by a  $\sim 5\text{ mm} \times 5\text{ mm}$  Si wafer with embedded thermocouple (Sensarray), which is placed next to the MEMS sample. Aluminum reflectors and a quartz window are placed above the sample to enhance sample heating by reflecting emitted radiation back onto the sample. The system is characterized by an uncertainty in  $K$  that is a function of the magnitude of curvature, resulting from  $\pm 2$  pixel uncertainty in  $l_{\text{beam}}$ . The uncertainty in  $K$  is greater for large magnitudes of curvature.

## RESULTS AND DISCUSSION

As discussed earlier, the three-layered MEMS structures were designed so that  $E(T)$  of the three films had a negligible effect on the curvature of the beams. Therefore, previously published data are used as input parameters for these properties. High-temperature  $E_{\text{Si}}(T)$  was studied by Kahn

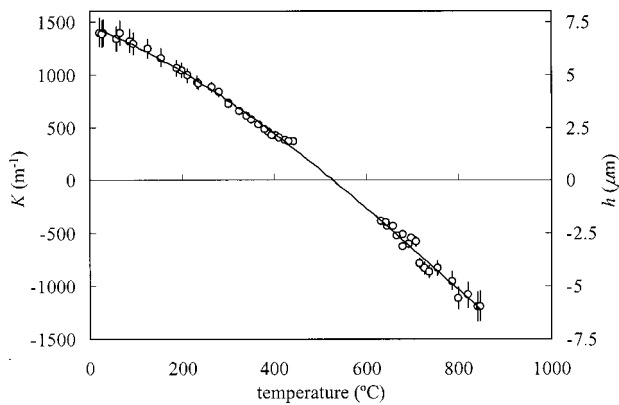


FIG. 5. Curvature and deflection of multilayered beam at high temperatures. (○) experimental, (—) third-order curve fit. The vertical bars represent the uncertainty.

*et al.*<sup>9</sup> for temperatures up to  $\sim 450^\circ\text{C}$ . Values reported are used to generate a second order polynomial fit:

$$E_{\text{Si}}(T) = 1.6806 \times 10^{11} - 8.2225 \times 10^6 T - 5.9816 \times 10^3 T^2 \text{ (Pa)}. \quad (6)$$

This curve is extrapolated for all temperature ranges included in this study (up to  $850^\circ\text{C}$ ).  $E_{\text{SiO}_2}$  of 64 GPa is used as reported by Weihs *et al.*,<sup>19</sup> based on the study of the load-deflection curve of micromachined  $\text{SiO}_2$  samples, mechanically deflected by a nanoindenter. This property is approximated to be a constant through all temperature ranges.

Thermally induced curvature measured for the three-layered cantilever beam is shown in Fig. 5. The secondary  $y$  axis of Fig. 5 indicates a beam tip deflection of  $100 \mu\text{m}$  beam corresponding to the curvature shown on the primary  $y$  axis. The results shown are for one heating-cooling cycle. The absence of data near zero curvature indicates the region where the beam curvature was below the minimum curvature limit of the experimental system. The uncertainty of the curvature, resulting from  $\pm 2$  pixel image uncertainty, is represented by the vertical bars; the uncertainty is higher for large magnitudes of curvature. A third-order curve fit, shown by the solid line, was generated through the curvature data for further analysis.

Using the third-order polynomial fit for  $K(T)$ ,  $\Delta\alpha$  was found between room temperature and  $900^\circ\text{C}$  in a piecewise manner, as an average over some temperature range. The temperature range used was typically  $100^\circ\text{C}$ , with the exception of the first range which was from  $20$  to  $100^\circ\text{C}$ . The resulting  $\Delta\alpha$  are shown in Fig. 6, plotted at the midpoints of the temperature range used. The temperature ranges are indicated by the horizontal bars, while the vertical bars show the uncertainty at each point. The uncertainty is larger at regions where the magnitudes of curvatures are high. The results show that  $\Delta\alpha$  increases between  $2.9 \times 10^{-6}$  and  $5.8 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$  from room temperature to  $900^\circ\text{C}$ .

Accepted values of  $\alpha_{\text{Si}}$  for bulk, crystalline silicon increase from  $2.6 \times 10^{-6}$  to  $4.3 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$  between room temperature and  $900^\circ\text{C}$ .<sup>20</sup> If the  $\alpha$  of poly-Si thin films were comparable to that of bulk Si crystal, the resulting  $\alpha_{\text{SiO}_2}$  must decrease from approximately  $0.3 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$  at room tem-

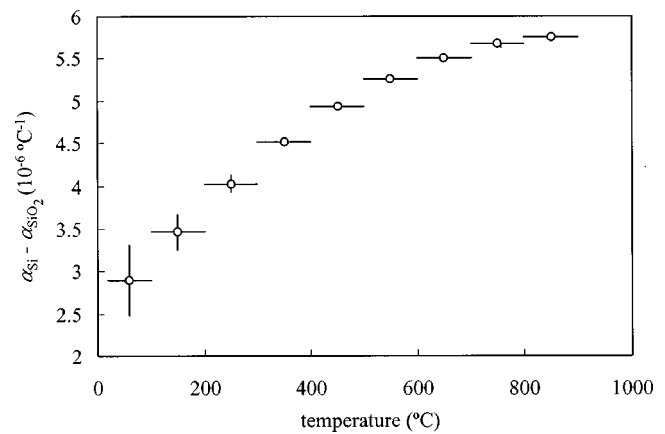


FIG. 6. The average  $(\alpha_{\text{Si}} - \alpha_{\text{SiO}_2})$  over  $100^\circ\text{C}$  temperature intervals, from room temperature to  $900^\circ\text{C}$ . The horizontal bars represent the temperature ranges used; vertical bars correspond to the uncertainty.

perature to a negative value at higher temperatures. Reported values for  $\alpha_{\text{SiO}_2}$  typically range between  $0.5 \times 10^{-6}$  and  $4.1 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$  at room temperature, and are expected to increase following the characteristic of  $\alpha$  for bulk  $\text{SiO}_2$  or other silica glass materials.<sup>21,22</sup> This suggests that  $\alpha$  of poly-Si films may be considerably higher than that for bulk Si crystals. Such values are consistent with other studies, which report values for  $\alpha$  for poly-Si as high as  $4.4 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$  for temperatures under  $300^\circ\text{C}$ .<sup>23</sup> It should also be noted, however, that negative values of  $\alpha_{\text{SiO}_2}$  have been reported in some cases for  $\text{SiO}_2$  thin films.<sup>23</sup>

## CONCLUSIONS

Multilayered cantilever beams were used to determine the thermal expansion coefficient of thin films at high temperatures. Films of polycrystalline silicon and silicon dioxide were used in this study. Beam layers were designed so that the thermally induced curvature of the multilayer was insensitive to changes in  $E$ ; therefore, the error introduced in the analysis from approximating values for  $E$  was negligible. The curvature of the cantilever beams for temperatures up to  $850^\circ\text{C}$  was measured using an innovative optical technique. The resulting curvature was compared to a numerical model to determine the difference in thermal expansion coefficient of the two films. The resulting values for  $(\alpha_{\text{Si}} - \alpha_{\text{SiO}_2})$  varied between  $2.9 \times 10^{-6}$  and  $5.8 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$  from room temperature to  $850^\circ\text{C}$ , respectively. These values suggest that the  $\alpha$  of poly-Si thin films may be significantly higher than that of bulk Si crystals. However, no definite conclusions can be drawn due to lack of consistency in reported values for  $\alpha_{\text{SiO}_2}$ , which range from a typically used value of  $0.5 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$  to values as high as  $4.1 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$ . In some cases, negative values of  $\alpha_{\text{SiO}_2}$  have also been found, which will result in values of  $\alpha_{\text{Si}}$  that are comparable to bulk crystalline  $\alpha$  values.

## ACKNOWLEDGMENT

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