On the Determination of Thermal Expansion Coefficient of Thermal Oxide

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ABSTRACT

The accurate value of thermal expansion coefficient (α) of thin film is an important thermal property in the design of microelectronic devices and microsystem. This research presents a microbridge buckling deformation caused by the residual stresses to determine the α of thermal oxide (SiO2) film. The extraction of α is supported through experimental means and finite element analysis (FEM) of the buckling profiles of microbridge. Moreover, in order to obtain the α value of thermal SiO2 film in accuracy, the nanoindentation system and the optical microscope with high resolution gauge were used to determine the elastic modulus of thermal SiO2 film and the α of silicon substrate, respectively. The combination of MEMS technologies and FEM with thermo-mechanical analysis approaches, the α value of thermal SiO2 film was calculated. Measurement value presents for α of thermal SiO2 film was 0.24×10-6/degree C with a standard deviation of 0.02×10-6/degree C.

Keywords: thermal expansion coefficient, thermal oxide film, microbridge, buckling deformation, finite element analysis

1. INTRODUCTION

Mechanical thermal properties of thin film are important parameters in the design of both microelectronic devices and microsystem, especially, in the areas of IC package and thermally driven microactuators or thermal type microsensors. Physically, the functional performance of those microdevices will be affected directly by the thermal expansion coefficient (α) of thin film [1]. However, thermal expansion coefficient of thin film material on the order of 1 μm thick or less can be significantly influenced by the fabrication processes [2]. In order to properly design microelectronic devices as well as micromachined components, it is necessary to characterize the thermal expansion coefficient of thin film materials.

Several measurement techniques including X-ray diffraction method [3], optically levered laser beam [4], and deformation of suspension micromeachined structure [2,5] have been applied to measure thermal expansion coefficient of thin films. As described in Ref. [3,4], those methods are exploited to determine the film stress by measuring the changes of the wafer curvature induced by the deposited films, and hence, thermal expansion coefficient can be extracted. However, X-ray diffraction method is only suitable for measuring crystalline structure and the technique of optically levered laser beam requires the understanding of knowledge of the thermal expansion coefficient of substrate and the elastic modulus of thin film. In the case of using deformation behavior of micromeachined structure to determine the thermal expansion coefficient of thin films, such as microcantilever bending and microring bulkling mentioned in Ref. [2] and [5], they are only suitable for characterizing CMOS intermetal dielectric and metal film to be utilized for microelectromechanical systems because the fabrication processes and micromachined structure used in those studies are simplified. But the problem of micromeachined structure test is that the changes of measured magnitude of out-of-plane deformation using optical interferometric techniques on these heat-deformed microstructures is affected by the thermal creep behavior of thin film.

In this paper, microbridge that was fabricated using a standard bulk micromachining process has been presented to determine the thermal expansion coefficient of thin film. According to the Ref. [6], the residual stresses of a thin film can be regarded as

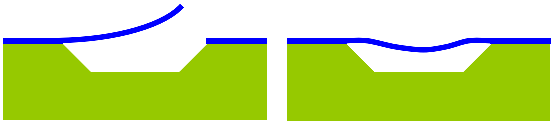
$$\sigma_{total} \approx \sigma_0 + \sigma_1 (\frac{2y}{h}) \tag{1}$$

where h is the thickness of thin film and y ∈ (-h / 2, h / 2) is the across the thickness with its origin at the mid-plane of the film. From formula (1), the residual stresses were comprised of a mean component σ0 and a gradient componentσ1. In which, the bending of the microcantilever is caused by the gradient residual stress as show in Fig. 1a. On the other hand, the buckling of the microbridge as show in Fig. 1b is induced by the mean compressive residual stress. Therefore, through the experimental process, the out-of-plane deformation of those microstructures can be measured using optical interferometric. After that, by contrasting the results of experimental measurement and that of finite element analysis (FEM) simulation, film residual stresses can be determined.

During thermal growth of amorphous dielectric film, the mean residual stress σ is generated by the mismatch Δα between the film and substrate. In the case of uniaxial sample, the thermal stress is given by

$$\sigma = E \Delta\alpha \Delta T \tag{2}$$

where E is the film's elastic modulus. Since the temperature change ΔT in microfabrication process can be substantial, and the elastic modulus of thin film was known, the averaging Δα over the process range can be calculated using the measured value of residual stresses. Thus the



(a) (b)

Figure 1. Out-of-plane deformation of: (a) microcantilever bending; and (b) microbridge buckling

thermal expansion coefficient of thin film can be extracted using the known thermal expansion coefficient of substrate. For the microbridge with compressive residual stress to have enough buckling, thermal SiO2 film was used as thin film test structure in this study. Besides, in order to eliminate the error margin of correlation material parameters while implementing thermal stress analysis, this paper also uses nanoindenation system and optical microscope to determine the elastic modulus of thermal SiO2 film and the thermal expansion coefficient of silicon substrate, respectively.

2. EXPERITENT

To accurately measure the thermal expansion coefficients of thermal SiO2 film, the experiments of this research mainly include three aspects:(1) Determine elastic modulus of thermal SiO2 film using nanoindenation technique, and then this measured value is used as a data point in calculating gradient residual stress; (2) Fabricate the micromachined test structure using bulk micromachining process and then measure the out-of-plane deformation magnitude using the interferometric profilometry; (3) Use the optical microscope to observe the heated silicon substrate and to obtain the thermal expansion coefficient of the silicon. In the following, the detailed content of each experiment is described.

2.1 Elastic modulus of SiO_2

Experiments for the elastic modulus measurement of thermal SiO2 film with thickness 1 μm grown on (100) silicon wafer were performed using a commercial nanoindention system with 0.0002 nm displacement resolution and 1 nN force resolution. The Berkovich indenter with a triangular pyramid tip was used in this experiment. During the thin film indentation test, the commercial indentation system was used to continuously record the load and displacement of the indenter head. Hence, the elastic modulus of the thin film materials for various different thicknesses was measured, as shown in Fig. 2. A safekeeping rule of thumb is that the depth of the contact should be less than 10% of the thin film thickness. Thus, the elastic modulus of SiO2 under consideration is in the range at 5% and 10%, which is 72.2 GPa.

2.2 Beam deformation

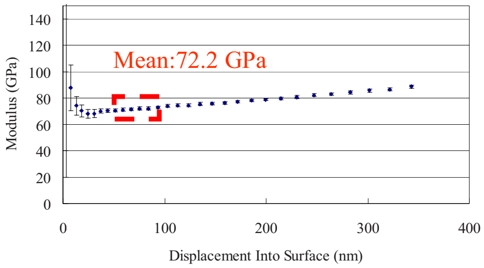


Figure 2. The elastic modulus of the thermal SiO_2 film under different indentation depths.

The effect of residual stresses on the micromachined test structure was investigated in this experiment. The SiO_2 microcantilever and microbridge with 1 μm thickness, 10 μm in width, and 20-140 μm in length were fabricated by a standard bulk micromachining process. First, the (100) single crystal Si substrate was placed in the furnace at 1050 degree C for 150 minutes to grow a 1 μm thick thermal oxide layer. After the oxide layer was patterned, the substrate was etched anisotropically by KOH. The test beams were all released from the substrate after their being etched for 30 minutes. However, due to the influence of the etching selectivity of KOH, the actual thickness of the suspending microcantilever and microbridge being etched is 0.98 μm. The microbridge is buckled downward as caused by compressive residual stress. To determine the effect of residual stresses on micromachined test structure, the out-of-plane deformation of different beams was measured by means of a non-contact interferometric profilometry. The out-of-plane deflection profile of microbridge shown in Fig. 3b was measured along the line AA′ in Fig. 3a. Hence, the maximum deflection amplitude of the beams at the point c was also determined. The measured deformation amplitude of microbridge with different length *L* is shown in Fig. 4. The data points in Fig. 4 denote the average value of the measured upward buckling deformation for ten different arrays. In addition, according to the above-mentioned measurement method, the deflection profile for micro- cantilevers was also measured, as shown in Fig. 5. The deflection profile of the microcantilever gradually increases with the increase of

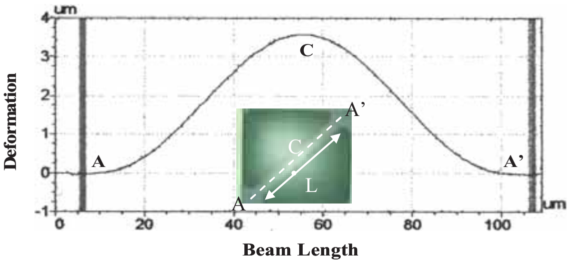


Figure 3. The optical microscope photograph of the SiO_2 microbridge to be measured and (a) along the path A-C-A'(b) the measured deflection profile of the beam show in.

beam length since the beam was bent by the gradient residual stress. Therefore, with the change of the out-of-plane deformation along the beam length, the radius of curvature of the microcantilever was obtained automatically from measurement software. The measured average value for the several microcantilevers with a beam length of 100 μm and width of 10 μm was 3.26 mm.

2.3 Thermal expansion coefficient of Si

To eliminate the parameters from the reference material in producing an error margin on analyzed process, this study also carries out the measurement on the thermal expansion coefficient of silicon substrate. This experiment was designed to directly cut wafer into a rectangle sample of size of 2 cm × 0.8 cm and then use the diamond pen to engrave two parallel caves on the rectangle sample. The distance between these two caves can be treated as an equivalent length to be used as a reference value later on when measuring the thermal expansion coefficient. Then, the sample was heated on the heating stage of which it had a controller to maintain deviations in a temperature range within 0.1 degree C, and an optical microscope with 0.1 μm high position resolution was used to observe the equivalent length change of sample. In dilatormetry, the linear thermal expansion coefficient α is given by a well know formula

$$\alpha(T) = (\frac{\Delta L}{L})(\frac{1}{\Delta T}) \tag{3}$$

where L is the sample's original length (at room temperature), T is the sample's temperature and ΔL is the change in sample's length due to the change of ΔT in its temperature. Consequently, according to above formula and the results measured, the thermal expansion coefficient of silicon substrate was determined, and the value is 2.67×10^{-6} /degree C within a temperature range of 25~125 degree C.

3 ANALYSIS AND DISCUSSION

This research is based on the out-of-plane buckling behavior of microbridge to calculate the thin film mean compressive residual stress, further, the thermal expansion coefficient of thin film was determined using thermal stress analysis. In which, the creation of the gradient residual stress can be regarded as caused by the localized effects including interstitial or substitutional defects and atomic peening and mean residual stress can be regarded as caused by a mismatch of thermal expansion coefficient between film and substrate. In this section, the out-of-plane deformation caused by the mean stress and gradient stress on the microbridge is studied. In addition, the deformation amplitudes of the microbridge caused by various mean compressive stress is predicted using a finite element model.

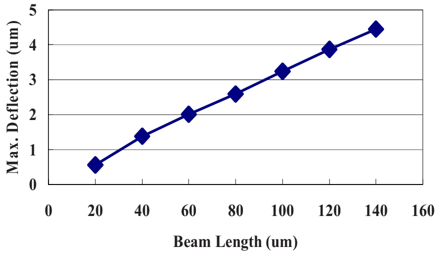


Figure 4. The measured deflection amplitudes of the SiO_2 microbridge against beam length.

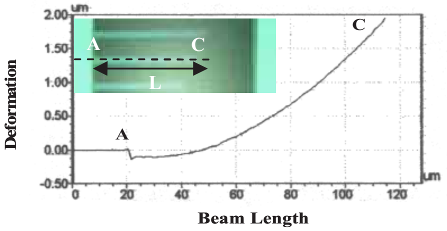


Figure 5. The optical microscope photograph of the SiO_2 microcantilever to be measured and the measured deflection profile of the beam along the path A-C.

3.1 Gradient residual stress

In the film fabrication process, the gradient residual stress will cause the microcantilever deformed. However, the peak value of the gradient residual stress can be found by measuring the curvature of microcantilever and is given by [7]

$$\sigma_1 = \frac{Eh}{2R} \tag{4}$$

where R is the curvature radius of microcantilever. Correspondingly, substituting the actual thickness h (0.98 μm) of the microcantilever and its elastic modulus E (72.2 GPa) measured by indentation test as well as the curvature radius R (3.26 mm) into the formula (4), the calculated peak value of the gradient residual stress of thermal SiO_2 film is determined approximately to be 10.85 MPa. The value of this gradient stress σ_1 will be used as an initial loading condition for the FEM model followed by a thermo-mechanical analysis.

3.2 Mean residual stress

The nonlinear analysis of FEM was used to simulate the buckling behavior of the microbridge caused by the mean compressive stress, in that the applied residual stress can cause a temperature effect on the film. Consequently, the out-of-plane deformation configuration of the microbridge resulting from the residual stresses can be predicted and characterized. From experiment, the constant value of stress gradient can be extracted using the curvature radius of the microcantilever. Therefore, in the following buckling

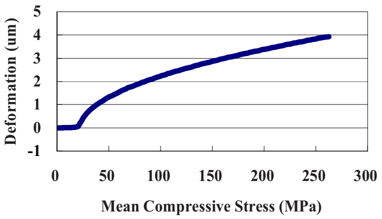


Figure 6. the variation of the maximum out-of-plane deflection with the mean compressive stress.

simulation process, the applied residual stresses are superposing the effects by using various mean residual stress and the measured constant stress gradient.

Fig. 6 shows the variation of the maximum out-of-plane deflection of the microbridge with the applied mean compressive stress of which having contributed by a loading condition of constant stress gradient with a peak value of 10.85 MPa. This figure acts as a calibration curve for calculated values of the thermal expansion coefficient of SiO_2. Thus, according to the simulation result shown in Fig. 6 in comparison with the experiment result shown in Fig. 4 under the same deformation of 3.24 μm, the extracted mean residual stresses is 180 MPa. With all measuredexperimental results being substituted into the formula (1) of thermal stress analysis, a thermal expansion coefficient value of thermal SiO_2 equal to 0.24×10^{-6}/degree C was obtained.

According to above measurement mechanism, the microbridge with the length L = 60 μm, 80 μm, 100 μm, 120 μm, and 140 μm respectively was also analyzed in this study. Fig. 7 shows the variation of the microbridge length with the calculated $\Delta\alpha$ from formula (1). It is obviously that the variation of α mismatch with the beam length L exhibits a state value at the longer beam. However, the shorter beam can be affected more significantly by the beam's imperfections and it is not suitable for beam theory analysis [7]. On the other hand, a reliable and accurately value of α mismatch in this experiment should be considered from the longer beam corresponded the approach constant value. Therefore, considering the microbridge with a length L = 100 μm, 120 μm, and 140 μm respectively, the calculated thermal expansion coefficient of thermal SiO_2 film is 0.24×10^{-6}/degree C with a standard deviation of 0.02×10^{-6}/degree C.

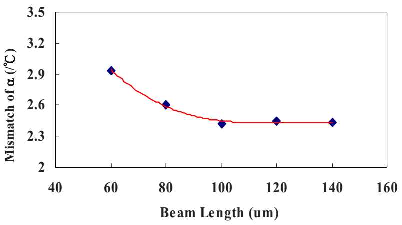


Figure 7. The variation of the mismatch of α with the microbridge length L

4 CONCLUSION

In this work, the usage of microbridge buckling deformation to extract thermal expansion coefficient of thin film is demonstrated. Both experimental and FEM approaches were used to determine the thermal expansion coefficient of thermal SiO_2 film. In addition, the elastic modulus of thermal SiO_2 film and the thermal expansion coefficient of silicon were also measured in this experiment to eliminate the error margin of the related material parameter in the thermal mechanical analysis. The extraction of thermal expansion coefficient of thermal oxide was determined to be 0.24×10^{-6}/degree C with a standard deviation of 0.02×10^{-6}/degree C. More importantly, the measurement mechanism presented here can be also applied to the measurement of other thin film materials or/and used as a test key for characterizing CMOS intermetal dielectric film, but, only on the condition that it must be limited with a compressive prestres

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