THE INDENTATION FRACTURE TECHNIQUE FOR MEASUREMENTS OF FILM STRESSES

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ABSTRACT

The present work summarizes the indentation fracture technique for the assessment of residual stresses in thin films deposited on brittle substrates by using a Vickers indenter tip. Based on a simplified analysis and experimental observations, we proposed a semi-empirical formula, i.e., $\frac{p}{c^{3/2}} = \frac{K_{IC}}{\chi} + \frac{\gamma - \delta\sigma_r t}{c^{1/2}}$, to characterize film residual stresses. In the semi-empirical formula, c and p denote the crack length and the indentation load, respectively, t and σ_r are the film thickness and film stress, respectively, χ, γ, and δ are three constants, and K_{IC} is the fracture toughness of the system and approximately equals the fracture toughness of the substrate when the crack length is much larger than the film thickness. Using the stress data from the curvature measurements, we have calibrated the dimensionless parameters introduced. δ has a value of about 59. The dimensional parameter χ changes from 0.11 to 0.145 with a mean of 0.124. The semi-empirical formula introduces a physical parameter, γ, in units of *N/m*, to represent the nature of indentation fracture on the bare silicon wafers. The experimental results have shown that γ ranges from 1.87 to 2.52 x10^4 *N/m* with a mean 2.26 (±0.28) x10^4 *N/m*. Experimental results on various thin films deposited on Si wafers verify the semi-empirical formula.

1 INTRODUCTION

Thin films on semiconductor substrates are of special interest to the microelectronic industries. Characterizing mechanical properties of thin films has become a very active area of research. The US Materials Research Society has organised ten symposia on "Thin Films: Stresses and Mechanical Properties" since 1988 [1]. Thin films have, in general, properties that are different from their bulk counterparts. An important issue in thin film research is residual stress that is usually induced during fabrication. Residual stress may damage a microelectronic device during its fabrication and/or reduce its service life. The reliability of device performance depends, to a large extent, on residual stresses in the thin films. Therefore, it is imperative to be able characterize residual stresses in thin films. A range of methods is currently available for the estimation of residual stresses [2-7], including X-ray and neutron diffraction, strain/curvature measurements, hole drilling, layer removal, Raman spectroscopy, optical fluorescence, indentation and indentation fracture, etc. Recently, we have developed novel methods to characterize residual stresses in thin films. These methods include the indentation fracture technique [8], the micro-rotating-structure indicator [9], the microbridge test [10, 11, 12], and the micro Raman Spectroscopy method [13]. This paper briefly summarizes the indentation fracture technique [8].

2 ANALYSIS

When the film thickness is much smaller than the substrate thickness, bi-axial residual stress exists in the film and no residual stresses are induced in the substrate. Furthermore, if the film thickness is much smaller than the indentation crack length, it will be reasonable to assume that the fracture toughness of the film/substrate system is approximately equal to the fracture toughness of the substrate. Following Lawn and Fuller's approach [7], we assume that the median crack, formed in the indentation fracture test, has a half-penny shape and the residual stresses in the film do not change the crack shape. We introduce a dimensionless term, β, to incorporate the necessary correction of the free surface and crack interaction. The fundamental and unique argument in our method is that the mean value of the stress intensity factors averaged over the entire crack front of the half-penny crack should correspond to the equilibrium length of the median crack. Then, the analysis of fracture mechanics of such an indentation crack yields the formula of

$$\chi \frac{p}{c^{3/2}} + \frac{\beta \sigma_r t}{c^{1/2}} = K_{IC} , \quad (1)$$

where $\chi = \lambda(E/H)^{1/2}$, H and E are, respectively, the hardness and Young's modulus, λ is a geometric factor [14], p is the applied indentation load, c is the median crack length, t is the thickness of the thin film, σ_r is the residual stress of the film, and K_{IC} is the fracture toughness of the substrate. Equation (1) shows that a linear relationship between $p/c^{3/2}$ and $1/c^{1/2}$ and the slope of $p/c^{3/2}$ versus $1/c^{1/2}$ should be zero if no film is deposited on the substrate. However, experimental tests on the bare silicon wafer show that the slope of $p/c^{3/2}$ versus $1/c^{1/2}$ has a nonzero value of $2.00\text{x}10^4$ *N/m*, which may be induced by the surface stress and/or the native oxide layer of the silicon wafer and/or other reasons. According to these experimental results, we modify Eq. (1) and propose a semi-empirical formula for characterization of residual stresses in thin films by the indentation fracture technique. The semi-empirical formula takes the form:

$$\frac{p}{c^{3/2}} = \frac{K_{IC}}{\chi} + \frac{\eta}{c^{1/2}} , \quad (2a)$$

$$\eta = \gamma - \delta \sigma_r t , \quad (2b)$$

where $\delta = \beta/\chi$ and η and γ are the slopes of $p/c^{3/2}$ versus $1/c^{1/2}$ for the film/substrate and pure substrate samples, respectively.

3 SiO_2 FILMS AND Cr FILMS ON SILICON WAFERS

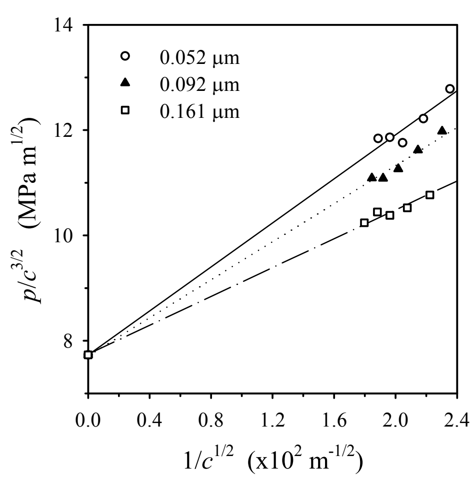

Fig. 1 Plot of $p/c^{3/2}$ vs $1/c^{1/2}$ for the Cr/Si set.

In our tests, chromium and wet-thermal silicon dioxide films were grown on 4 inch (100)-oriented n-type silicon wafers with a thickness of 525 μm. Two approaches were employed to change the SiO_2 film thickness and each approach was applied to one wafer. One approach was named consecutive film growth and testing, where one run contained film growth and testing. After each run, an additional thickness of the oxide film was further grown and five film thicknesses, 0.11, 0.20, 0.32, 0.40 and 0.49 μm, were adopted. The other approach was called consecutive film etching and testing, wherein the film was first grown to a certain thickness and tested. After the test, a thin layer of the film was removed by etching and the second test was conducted, and so on. Four film thicknesses, 0.19, 0.32, 0.40 and 0.50 μm, were employed for the consecutive film etching and testing. Three Cr films of thicknesses, 0.052, 0.092 and 0.161μm, were deposited by ion sputtering on three Si wafers, respectively. The residual stress in each film was also measured by the curvature technique, which resulted in the mean and standard deviation of the residual tresses of -359 (±22), -357 (±14) and 1095 (±24) *MPa* for the consecutive growth and testing SiO_2, the consecutive etching and testing SiO_2 and the C_r films, respectively.

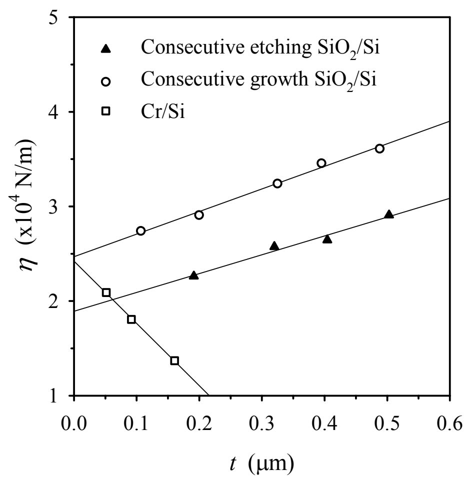

Fig. 2 The parameter, η, versus the film thickness, *t*.

The experimental data, $p/c^{3/2}$ versus $1/c^{1/2}$, for each film thickness were fitted with a straight line of nonzero slope. As an example, Fig. 1 shows the plots of $p/c^{3/2}$ versus $1/c^{1/2}$ for the C_r film/Si wafer set. The results are in good agreement with the theoretical analysis. The slopes are 2.07, 1.84 and 1.38 x10^4 *N/m*, respectively, for film thicknesses, 0.052, 0.092 and 0.161 μm with the common intercept of K_{IC}/χ= 7.7 $MPam^{1/2}$. For the consecutive growth and testing SiO_2/Si set, the slopes from the linear regression are 2.80, 2.90, 3.28, 3.40, and 3.60 x10^4 *N/m*, respectively, for film thicknesses of 0.11, 0.20, 0.32, 0.40 and 0.49 μm and the intercept is K_{IC}/χ=6.2 $MPam^{1/2}$. The experimental results on the consecutive etching and testing SiO_2/Si set lead to the slopes of 2.25, 2.58, 2.65 and 2.92 x10^4 *N/m*, respectively, for film thicknesses of 0.19, 0.32, 0.40 and 0.50 μm and the intercept of K_{IC}/χ= 8.2 $MPam^{1/2}$. The fracture toughness of the silicon substrate is used to calibrate the dimensionless parameter, χ. The mean fracture toughness of silicon is K_{IC} =0.9 $MPam^{1/2}$ [15], and, consequently, the dimensionless parameter χ is 0.145 for the consecutive growth and testing SiO_2/Si set, 0.110 for the consecutive etching and testing SiO_2/Si set, and 0.117 for the Cr/Si set. The average value of the dimensionless parameter, χ, for the three sets of the film/substrate samples is 0.124. In turn, when this average value of χ is used to recalculate the fracture toughness of silicon from the intercepts, K_{IC} ranges from 0.77 to 1.02 $MPam^{1/2}$. These values are also acceptable for silicon [15] because the fracture toughness for a brittle material scatters considerably.

Equation (2) shows a linear relationship between the slope, η, and the film thickness, t. Using the values of residual stresses measured by the curvature technique, we calibrate the dimensionless parameter, δ, by plotting η versus t. Figure 2 shows that η is directly proportional to t, indicating that the product of $-\delta\sigma_r$ is a constant for different film thicknesses in each set. The slopes of η versus t are 2.20, 2.08 and -6.37 x10^4 *MPa*, respectively, for the consecutive growth and testing SiO_2/Si, the consecutive etching and testing SiO_2/Si, and the Cr/Si sets. Consequently, the dimensionless parameter, δ, is calibrated to be 61, 58 and 58, having a mean of 59±1.4. These results show that the dimensionless parameter δ may be invariant for the three sets. The mean values of δ and χ yield a mean value of β=7.316. The intercepts of the curves, η versus t, shown in Fig. 2, are 2.52, 1.87 and 2.40 x10^4 *N/m*, respectively, for the SiO_2/Si sets undergoing the consecutive growth or etching and the Cr/Si set. The average of the three values is 2.26±0.28 x10^4 *N/m*. The slope of $p/c^{3/2}$ versus $1/c^{1/2}$ for the bare silicon wafer, 2.00 x10^4 *N/m*, is very close to the average 2.26±0.28 x10^4 *N/m*, indicating that these nonzero intercepts stem from the silicon substrates.

4 ZnO FILMS DEPOSITED ON Si WAFERS

The developed indentation fracture technique was applied to measure residual stress in ZnO films [16]. The ZnO films with thicknesses ranging from 0.202 to 0.554 μm were deposited by using the magnetron sputtering technique on 525 μm-thick (100) Si substrates. Then, Vickers indentation tests were carried out on the ZnO/Si systems at room temperature, in which the applied load varied from 10 mN to 2.0 N. The ZnO/Si systems with the 0.202, 0.283, 0.443 and 0.554 μm-thick films exhibited only radial cracking under the indentation tests. No delamination occurred in these ZnO/Si systems. Figure 3 shows the images of the 0.554 μm-thick-ZnO/Si system after the indentation tests under different loads ranging from 0.05 N to 2.0 N. The 0.05 N and 0.2 N loads caused only impressions at the film surface. Radial cracks were observed as the load increased to and beyond 0.5 N, indicating that a threshold indentation load for radial cracking must exist between 0.2 N and 0.5 N for the 0.554 μm-thick ZnO/Si system. The radial cracks emanate from the corners of the impression and extend parallel to the <110> directions of the silicon wafer. The radial crack length increases with the applied load for a given film thickness. Take the 0.554 μm-thick-ZnO/Si system as an example. When the load increased from 0.5 N to 2.0 N, the corresponding radial crack length also increased from 9 μm to 27 μm. The other three ZnO/Si systems with different film thicknesses also exhibited the similar indentation behavior. However, for a given load, the radial crack length decreased with the film thickness. For instance, the radial crack length decreased from 30 μm to 27 μm as the film thickness increased from 0.202 μm to 0.554 μm for the load of 2.0 N.

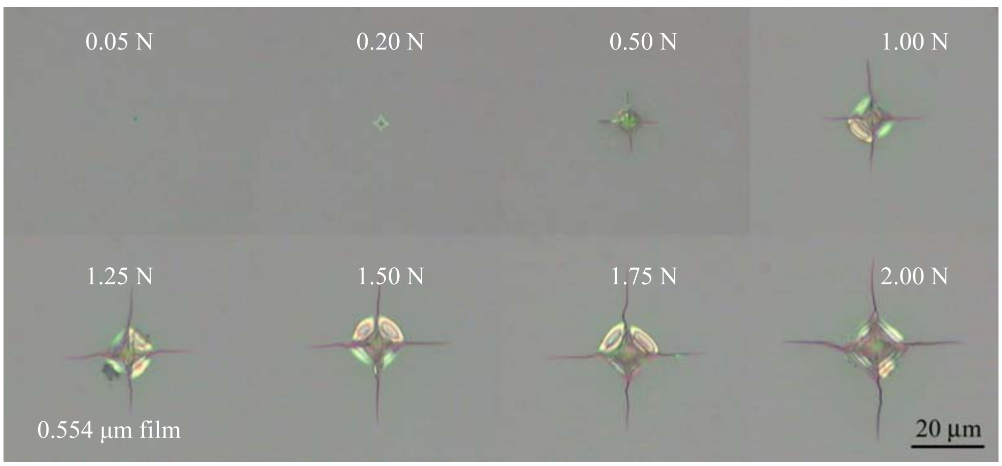

Fig. 3 The optical microscopic images of the 0.554 μm-thick-ZnO/Si system after the indentation tests under different loads ranging from 0.05 N to 2.0 N.

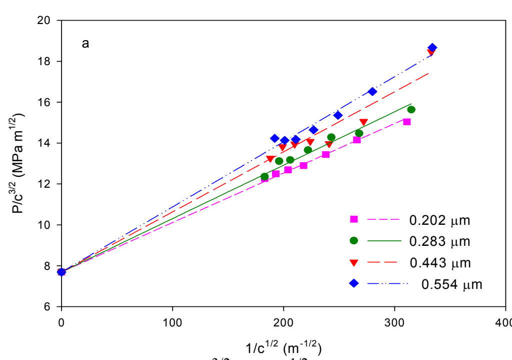

Fig. 4 Plot of $p/c^{3/2}$ vs $1/c^{1/2}$ for the ZnO/Si set.

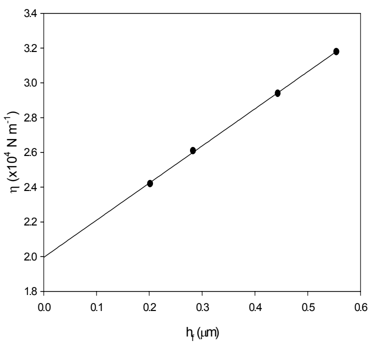

Fig. 5 The parameter, η, versus the film thickness, h_f, for the ZnO/Si systems.

Using Eq. (2a), we plot $p/c^{3/2}$ versus $1/c^{1/2}$ in Fig. 4 for the ZnO/Si systems with the 0.202, 0.283, 0.443 and 0.554 μm-thick films. From the common intercept of the four fitting lines and using K_{IC} =0.9 $MPam^{1/2}$ [15], we calculate the parameter χ to be 0.117, which is within the range of (0.110, 0.145) reported previously [8]. The slope, η, is plotted in Fig. 5 versus the film thickness, which shows that the data are located on a straight line, indicating the residual stress independent of film thickness. Thus, Eq. (2b) is applicable to the ZnO/Si systems. The intercept of the line in Fig. 5 is 1.99×10^4 N m^{-1}, which is almost the same as 2.00×10^4 N m^{-1} for the bare silicon wafer [8]. Furthermore, from the slope of the regression line in Fig. 5, we estimate the residual stress, σ_r, of the ZnO films to be 387 MPa in compression.

CONCLUDING REMARKS

The present work summarizes the indentation fracture technique to measure residual stresses in thin films deposited on brittle substrates. The proposed semi-empirical formula is simple and easy to use in the indentation fracture tests with a Vickers indenter. The formula indicates that the ratio of the indentation load to the cubic of square root of the crack length is linearly proportional to the reciprocal of the square root of the crack length, the magnitude of the residual stress, and the film thickness. Wafer curvature measurements have been conducted to calibrate the dimensionless parameters in the proposed formula. Experimentally, micro-indentation tests were conducted on the silicon dioxide films, Cr films and ZnO films deposited on silicon wafers. The experimental results verify the semi-empirical formula.

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