Mechanics analysis of femtosecond laser induced blisters produced in thermally-grown oxide on Si(100)

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Abstract: Blister features produced by laser-induced delamination of silicon dioxide from silicon substrates are analyzed with thin-film buckling mechanics. These analyses reveal the role of the interaction between the material and the femtosecond (fs) pulsed laser on blister formation. In particular, it was deduced that the magnitude of the compressive residual film stress within the irradiated region appears to exceed the intrinsic residual stress obtained from wafer curvature techniques. This apparent increase in the compressive stress after fs pulsed laser irradiation may be caused by a modification of the oxide which results in a local rarefaction of the film. The results demonstrate important features of the interaction between materials and fs pulsed laser, including the presence of subtle modification thresholds and the limited role of thermal effects.

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1. Introduction

Femtosecond (fs) pulsed lasers have emerged as a powerful tool for high-precision machining a wide range of materials, including dielectrics [1-4], semiconductors [5-16], metals [17-24], and thin films [25-30]. Essential to this precision is the ultrashort nature of the energy impulse, which is shorter than most physical phenomena associated with material ablation [31]. Focused ultrashort energy impulses lead to high intensities, which allow access to non-linear effects of energy absorption [32], providing for micromachining of dielectrics and semiconductors where the bandgap energy exceeds the energy of a single photon. Furthermore, sub-surface micromachining and modification of glasses have been demonstrated for the purposes of producing fluidic networks [3, 33, 34] and waveguides [35-39]. In the present work, the interaction between ultrashort light pulses and materials is further investigated through a mechanical analysis of thin-film blister features produced by fs-pulsed laser irradiation of silicon dioxide on the surface of silicon [14, 40, 41].

The surface morphology of ablation features produced by fs laser pulses onto Si(100) substrates with thermally-grown and plasma-enhanced chemical-vapor-deposited (PECVD) oxide films have been described earlier [14, 40, 41]. Two primary ablation morphologies were observed; one in which the oxide film was delaminated from the underlying substrate but remained intact, and the other in which an area of the oxide film was completely removed. Here we focus on the case in which the oxide film is delaminated from the underlying substrate producing a thin-film buckling event. Previous work attributed laser-induced thin-film buckling to the phenomenon of wrinkling on a compliant substrate, with a thin film of silicon dioxide buckling while remaining in contact with a silicon surface that had been melted by the laser[42]. This is in contrast to the present work, where delamination of the film from the underlying substrate is clearly observed in the irradiated area. Laser-induced modification of the thin oxide film is revealed from the mechanics analysis, demonstrating that mechanisms typically associated with waveguide writing in oxide materials [35-39] are likely contributing to the observed buckling phenomenon.

Buckling-driven delamination has been extensively used to study the properties of thin films and interfaces [43-45]. In this work, these theoretical methods are applied to circular blisters induced by femtosecond pulsed laser irradiation of Si(100) with thin oxide films. As detailed above, the particular goal of this work was to assess the role of the laser-material interaction on the buckling event. To do this, a buckling mechanics analysis was used to infer the compressive stress within the oxide from measured geometrical quantities, and this stress was compared with the intrinsic values for the film obtained from wafer-curvature techniques.

2. Experimental Details

Oxide (SiO_2) films on Si(100) substrates were grown by thermal oxidation at 1000 °C. Circular blisters, in which the oxide film was delaminated from the underlying Si(100) substrate, were produced by single laser pulses at normal incidence. The laser was focused onto the sample surface with a 20 cm focal length, plano-convex lens yielding a focused beam diameter ($1/e^2$) of 18.3 ± 0.5 μm. Variations in the blister dimensions (diameter and amplitude) were achieved by using a range of incident laser fluences: 0.36 - 0.59 J/cm^2 for blisters in the 300 nm thick oxide and 0.48 - 0.81 J/cm^2 for blisters in the 1200 nm oxide. Once generated, the blister dimensions were measured using an atomic force microscope (AFM), with the diameter of circular blisters being defined as the distance over which the vertical deflection of the film exceeded 10 nm.

3. Results

The series of optical microscope images in Figure 1 shows how the ablation morphology for a 300 nm thermal oxide varies with the fluence of the incident laser pulse. The accompanying schematics similarly illustrate how the resultant cross sections of film vary with the laser fluence. For low laser fluences (0.21 J/cm^2), contrast is visible in the optical microscope image but no change in surface morphology is observed by AFM. It is likely that this change in contrast is associated with laser-induced amorphization of the underlying silicon, which leads to an increase in reflectivity of the interface [46]. For laser fluences between 0.36 and 0.59 J/cm^2, blister features were produced in which the oxide film was delaminated and forced upward from the underlying substrate [40]. The modification to the film/substrate interface, as observed by optical microscopy was always about 2 to 5 μm beyond the circumference of the blister as defined by the AFM measurements. Finally, for laser fluences exceeding 0.7 J/cm^2, spallation of the oxide film is apparent, leaving a crater with a depth approximately equal to the thickness of the film. The spalled section is within the region in which the film/substrate interface appeared to have been modified. Spalling occurs when the tip of a crack kinks into the film and extends up to the surface. Therefore, the fact that spalling occurred inside the region where interface modification was observed, rather than at the perimeter of this region, lends strong support to the notion that the modification to the interface extended beyond the region of delamination.

There are three mechanisms by which a delaminated film may form a blister: (i) the debonded region may buckle under the influence of a residual compressive stress once a critical size of debond has been formed [44, 47-50], or (ii) an internal gas pressure within the debond region may cause an upwards displacement of the film [51, 52], or (iii) both effects may act simultaneously [53]. In the present case, it is believed that the laser causes delamination, and that the blisters form only by buckling above this damage, with no effect of an internal gas pressure. The reason for this statement is illustrated in Fig. 2. A linear blister was formed by overlapping a series of circular blisters, as discussed in reference [41]. After the linear blister was formed, a crack developed through the film part way along one edge. As a result of this fracture, the compressive stress in the oxide was relaxed in one portion of the blister and it was released from the constraint of the substrate. AFM images of the film surface showed that the released/semi-fractured portion of the film was flat and retained no curvature representative of the film in the nearby blistered state. The fact that the fractured film was flat demonstrated that very little or no plasticity and/or creep was associated with the laser ablation process. Furthermore, the buckling amplitudes of linear blister with open ends (i.e. written off the edge of the substrate) were identical to un-opened or isolated blister channels of the same width. The fact that the open blisters did not collapse indicates that the blistering was not associated with the development of a gas pressure during laser ablation as this gas would have been released through the open end of the blister. Finally, it should be noted that the AFM measurements suggested that blisters did not form below a critical size of delamination; this is consistent with a buckling model. These three pieces of evidence support the use of a linear-elastic buckling analysis for the phenomenon investigated in this paper.

3.1 Buckling Mechanics Analysis

The radius, R, and amplitude, δ, of the circular blisters formed by single laser shots in the 300 and 1200 nm thermal oxides are shown in Figure 3 as a function of laser fluence. Each datum point represents the average for the dimensions of three blisters created at a given fluence, and the error bars represent the maximum and minimum values. The two sets of data from the different thicknesses collapse into a single master curve (Fig. 4) when plotted in a normalized fashion as δ/h against R/h, where h is the film thickness. Therefore, the data from the two experiments with different thicknesses of film were regarded as providing a single set of dimensionless data, and the subsequent calculations were performed under this assumption [54, 55].

Modeling the blister as a clamped circular plate of radius a on a rigid substrate, the critical stress for buckling is given by [47]:

$$\sigma_C = 1.2235 \frac{E}{1-\nu^2} \left(\frac{h}{R} \right)^2 \quad (1)$$

where E (assumed to be 70 GPa) is the Young's modulus of the oxide film, and ν (assumed to be 0.3) is the Poisson ratio of the oxide film. If the residual stress in the film is less than the critical value given by Eqn. (1), no blister will form by buckling. Therefore, the intrinsic compressive stress in a film can be deduced from this equation by determining the minimum radius, R_o, at which buckling occurs. Extrapolation of the data in Fig. 4, indicates that the amplitude of the blister would be zero at a normalized blister radius of approximately $R_o/h = 8$. Substituting this value of radius back into Eqn. (1), produces a value for the residual stress in the film of about 1.5 GPa. As will be addressed later, this value is an upper bound on the residual stress. It will be shown that 900 MPa is a lower bound on the stress.

This value of 1.5 GPa is significantly higher than expected, as other work has indicated that the intrinsic compressive stress in thermally grown SiO_2 films on crystalline silicon substrates is about 0.3 GPa when the thickness exceeds 150 nm [56]. Given the discrepancy between inferred and expected stress, additional samples with the thermally-grown oxide were characterized using wafer curvature techniques. These experiments indicated stress levels consistent with Refs. [56-

58]: a compressive stress of 380 MPa. It is clear that the residual stress inferred from the minimum radius for buckling is a factor of about four greater than might have been expected.

Linear-elasticity indicates that the magnitude of the residual compressive stress in the film, $\sigma_{o,}$ can also be deduced from the amplitude of a buckled blister and its radius. The relationship between the three parameters is given by the following approximate analytical expression [44]:

$$\sigma_0 = \sigma_C \left[c_1 \left(\frac{\delta}{h} \right)^2 + 1 \right] = 1.2235 \frac{E}{1-\nu^2} \left[c_1 \left(\frac{\delta}{R} \right)^2 + \left(\frac{h}{R} \right)^2 \right] \quad , \tag{2}$$

where c_1 is a function of the Poisson ratio ν:

$$c_1 = 0.2473(1+\nu) + 0.2231(1-\nu^2) \tag{3}$$

The residual stress, calculated from this expression, is plotted as a function of normalized blister radius for the combined data sets (both 300 nm and 1200 nm thermal oxide) in Figure 5. This figure shows that the stress drops in a monotonic fashion from the 1.5 GPa calculated in the previous paragraph as the normalized blister radius increases.

3.3 Energy-release rate

It is possible that the delamination observed in response to the laser is caused either by the direct interaction of the laser with the material at the interface or by buckling-driven delamination. An indication of which of these two mechanisms is responsible for the delamination can be obtained by investigating the energy-release rate at the perimeter of the blisters. If a fracture-mechanics type of buckling-driven delamination [44, 47, 50, 59] is responsible, then calculations of the energy-release rate at the edge of the blisters should give reasonably consistent values comparable to the appropriate interface toughness[44, 59] if the delamination is caused by direct action of the laser, perhaps by the propagation of a stress wave exceeding the cohesive strength of the interface [60] or by local melting of the interface, so that buckling occurs *after* delamination, rather than being associated with it, then the calculations of the energy-release rates at the perimeter of the blisters will merely give a lower bound for the interfacial toughness.

An approximate, asymptotic expression for the energy-release rate associated with buckle-driven delamination of a circular blister is given by [44, 47]:

$$\frac{\mathcal{G}}{\mathcal{G}_o} = c_2\left[1-\left(\frac{\sigma_c}{\sigma_o}\right)^2\right] \quad , \tag{5}$$

where,

$$\mathcal{G}_o = (1-\nu)h_1\sigma_o^2/E\,, \tag{6}$$

and

$$c_2 = \left[1+0.9021(1-\nu)\right]^{-1}. \tag{7}$$

The two stresses, σ_c and σ_o, are obtained from the geometries of the blisters using Eqns. 1 and 2, respectively. As shown in Fig. 6, the results indicate little consistency between the different values of the energy-release rate, indicating only that the toughness of the interface is greater than about 2 J/m^2, and that the delamination is probably not driven by the buckling process. The results indicate that delamination is induced by the direct interaction of the laser with the interface.

4. Discussion

There are two unexpected conclusions from the results presented in the previous section that need further discussion. First, the magnitude of the compressive stress within the fs laser-induced blisters can be much higher than would be expected for a thermally-gown SiO_2 film on silicon. Second, the magnitude of this compressive stress is not constant, but appears to decrease as the normalized blister dimensions increase. These results suggest that the laser is interacting with the oxide film and changing its internal structure to induce these stresses [37, 61]. While we are describing the changes in terms of an induced stress, it should be noted that the observed phenomena could also be described in terms of a reduced modulus. However, it would appear to be much easier to induce stresses of an appropriate magnitude to explain the results than to induce a large enough change in modulus.

Other possible explanations for the results have been considered, and have been eliminated. First, root-rotation effects [62-64] (the notion that the assumption of a clamped boundary at the edge of the delamination is invalid) were considered, and are expected to be negligible for this system, given the relative stiffnesses of the film and substrate. However, the possible existence and potential role of a plastic hinge at the root of the blister [65] will be investigated in future works.[1] Second, the experiment involving fracture of a linear blister demonstrated that the apparently excessive deformation of the film was not caused by an internal gas pressure. Third, the experiment also demonstrated that the excessive deformation was not induced by flow processes at high temperatures, since the blister relaxed elastically when the constraint was removed. Fourth, it was recognized that the region of delamination was defined as that over which the deflection of the blister exceeded 10 nm. Clearly, the delamination extends beyond this region. The extent to which the radius of delamination is greater than that deduced by the AFM measurements can be estimated by assuming the profile of a clamped buckled plate, and

[1] In this context, it is noted that the top of the broken film of Fig. 2c is at a height that is the equivalent of exactly one film thickness above what would have been expected. This was attributed to the possible existence of debris, but it is recognized that it could be a symptom of a local plastic hinge induced under conditions of high heat flux during the blistering process. This is an issue that will be addressed in future work.

extrapolating the deflection to zero. This adds up to 1.5 μm to the radius in some cases, but does not affect the conclusions significantly.

The effect of this additional uncertainty is illustrated in Fig. 7. This figure shows the amplitude of the blisters as a function of normalized radius; the uncertainty on the radius now includes the effect of using an AFM to define the blister dimensions. Superimposed on this figure are predictions of how the deflection would vary if the residual stress in the film was constant. These plots have been determined by rearranging Eqn. (2) into the following form:

$$\frac{\delta}{h} = \sqrt{\frac{1}{c_1}\left[\sigma_0\left(\frac{1-\nu^2}{1.2235E}\right)\left(\frac{R}{h}\right)^2 - 1\right]} \qquad (4)$$

It is clear from this figure, that while the peak stress may, perhaps, not be as high as 1.5 GPa, the compressive stress in the blister must be at least 900 MPa, which is considerably in excess of the value obtained by wafer curvature. Furthermore, this figure also indicates that the compressive stress drops as the normalized blister size increases. A possible mechanics explanation for this is given in the next section.

4.1 Calculation of compressive stress induced by the laser pulse

The lack of any other explanation leads to the conclusion that the laser is interacting with the film, and directly affecting its structure as to induce an extra compressive stress within the delaminated region. As is explained in this section this modification must be seen in terms of a rarefaction of the film. A calculation of the additional compressive stress that would be induced by a laser interacting with the oxide film can be done by assuming that the blistered region consists of a circular disk of radius *R*, clamped around its edges. The film is assumed to have isotropic linear-elastic properties with Young's modulus E, and Poisson's ratio ν. The laser pulse induces a volumetric strain (expansion) of Δ in a central cylindrical region of radius a.

The calculation proceeds by imagining that the delaminated region is separated from the rest of the film, so that it is relaxed from the constraint of the substrate. It can be shown that a volumetric strain of $(\delta V/V)$ within the central region would cause the outer radius of the disc to expand by

$$u_R = \frac{(\delta V/V)a^2}{3R} \tag{8}$$

In order for this expanded cylinder to fit back into the film from where it had been removed, a biaxial compressive stress would need to be exerted on the cylinder to shrink it back to a radius of R. Hooke's law can be used to show that the magnitude of this additional compressive stress is given by

$$\Delta\sigma_o = \frac{(\delta V/V)a^2 E}{3R^2(1-\nu)} \tag{9}$$

This results in an effective residual stress within the blister of

$$\sigma_o = \sigma_i + \frac{\Delta a^2 E}{3R^2(1-\nu)} \tag{10}$$

where, σ_i is the intrinsic stress within the thermal oxide before any interaction with the laser (as measured by wafer curvature measurements, for example, to be 380 MPa). It is this effective residual stress that has been determined by the experiments to be in the range of 1.5 GPa to 500 MPa. In Eqn. (10), the effective compressive stress would drop with delamination radius if both the size of the central region undergoing a volume change and the volume change are relatively fixed.

These calculations demonstrate the important mechanics principle that *if the magnitude of the observed compressive stress is greater than the magnitude of the intrinsic compressive stress, then the structural change that must have occurred within the oxide film is a rarefaction.* Conversely, if the material modification results in densification, then the compressive stress would be lowered. In the present case, where there is apparently a rarefaction of the material, the compressive stress indicates that the atoms of the oxide film are being compressed to be closer than their equilibrium position until buckling occurs, when they can expand back towards their equilibrium separations. However, this new equilibrium separation is greater than it would have been before the interaction with a laser. It is further noted that in the present experiments there is an indication that the extent of structural modification increases with film thickness: for a given radius of delamination, the induced compressive stress is larger for the thicker film (as indicated by the stress increasing with R/h (see Fig. 5). This suggests an increased interaction between the film and laser as the thickness of oxide increases.

As detailed in the introduction, fs laser-induced modification of glass has been used to produce waveguides in bulk glasses (see [37] and references therein). Techniques to characterize the stresses induced by this modification in previous works include birefringence measurements [61], measurements of the topography of the waveguide cross sections [66], and Raman spectroscopy [67]. Depending on the details of the system, these stresses are attributed to either a densification of the structure [36, 38] or to a rarefaction [35]. For example, rapid quenching of a bulk silica has been associated with a densification, but other phenomena have also been associated with rarefaction [37]. While we have not identified the mechanism for the structural change, it is apparent that in this system it is associated with a rarefaction, rather than a densification of the oxide layer.

5. Summary/Conclusions

In summary, the dimensions of fs laser-induced circular blisters in 300 nm and 1200 nm thermally-grown oxide films, have been analyzed according to the mechanics of thin-film buckling. Two important conclusions came out of these analyses. First, the delamination at the interface could *not* be described by fracture mechanics. Delamination had to be associated with modification of the interface by the laser or with decohesion from a shock wave. Second, the compressive stress in the film calculated from the dimensions of the blister, clearly indicated that the material modification associated with the interaction of the laser with the oxide film had to be contributing additional residual stresses to the film. The modification is expected to be similar to that exploited for writing of waveguides in bulk glasses. This analysis provides a unique opportunity to quantify and characterize sub-ablation threshold modifications to solids associated with fs pulsed laser-material interactions. Future work will examine the nature of the material modification through micro-Raman analysis of the oxide film in blistered regions and cross sectional imaging of blister features with high resolution electron microscopy.

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Figure Captions:

Figure 1 Optical microscope images of a 300 nm thermally-grown oxide on Si(100) after exposure to single pulses of a fs laser of different fluences. For a laser fluence of 0.21 J/cm^2, any apparent effects of irradiation are confined to the interface between the substrate and film. For laser fluences between 0.36 and 0.59 J/cm^2, blisters are produced in which the oxide film is delaminated from the substrate but remains intact. The blistering is indicated by the concentric rainbow interference pattern within the features (within the dotted line in the 0.44 J/cm^2 image). Finally, for a laser fluence of 1.2 J/cm^2, the oxide film spalls, exposing the substrate. Some debris from the oxide film is present in the center of the crater.

Figure 2 A fractured linear blister produced in a 1200 nm thermally-grown oxide on Si(100). This particular channel was created using two passes at a laser fluence of 0.40 J/cm^2 with a lateral overlap of 10 μm, and a translation velocity of 6 mm/s [41]. **(a)** Optical-microscopy image of a linear blister indicating the location where the oxide fractured. **(b)** Atomic-force microscopy (AFM) topographic image of the partially fractured linear blister, with a schematic of the cross section. **(c)** AFM cross section of the partially fractured portion of the linear blister. **(d)** AFM cross section of the fully intact portion of the linear blister.

Figure 3. Dimensions of the circular blisters used to study the mechanics of the blister formation. a) Blister diameter plotted as a function of the laser fluence. b) Blister amplitude plotted as a function of laser fluence. Error bars represent the

maximum and minimum values for a particular laser fluence from at least 5 distinct blister features.

Figure 4 The amplitude (normalized by oxide thickness) plotted against radius (normalized by oxide thickness) for the circular blisters created by single laser shots. The data sets for both the 1200 nm and 300 nm thermal oxides on Si(100) have been combined for this plot. A power-law fit to the data indicates that the minimum normalized radius for buckling is approximately $R_0/h = 8$.

Figure 5 The apparent residual compressive stress, as calculated from the dimensions of the blisters, decreases as a function of normalized blister radius (R/*h*).

Figure 6 Plot of the energy-release rate for buckling-driven delamination as a function of blister radius for circular blisters with an oxide thickness of 300 nm and 1200 nm. The observation that the energy-release rate is not reasonably constant for different values of *R/h* suggests that the delamination is not driven by the buckling process.

Figure 7 The relationships between the amplitudes and radii of the blisters. The experimental data points have had an additional uncertainty added to them, to reflect the extent of delamination may be under-estimated by defining it as the region over which the deflection exceeds 10 nm. Superimposed on the this plot are calculations of how the deflection would depend on blister size for different values of compressive stress. These results indicate that the effective compressive stress within the film varies with *R/h* and lies between about 1.5 GPa and 500 MPa for the blisters in this study. This compares with an intrinsic compressive stress of about 380 MPa.

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