

## *Stress Gradients in Annealed Tetrahedral Amorphous-Carbon Films*

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**Motivation**—We have fabricated simple tetrahedral amorphous-carbon (ta-C) Micro-ElectroMechanical Systems (MEMS) devices where the components were made wholly from ta-C – not just as a coating. One critical issue in materials for MEMS technologies is out-of-plane bending caused by stress gradients through the thickness of the device. Indeed, we have found that released ta-C devices can show out-of-plane bending for films that have zero average biaxial stress. This indicates that non-zero stress gradients are embedded in ta-C films. Yet, little is known about the origin of these stress gradients and how to control them. These gradients cannot be measured during growth because the final stress state is determined by a post-growth anneal. Furthermore, the through-thickness stress profile may be nonlinear due to nonuniform processing conditions and/or intrinsic interfacial stresses.

**Accomplishment**—We have integrated a laser based wafer curvature technique (Fig. 1) into a commercial etch tool to record continuous measurements of curvature versus etch time. Stress gradients were measured during etch-back of as-grown as well as annealed (stress relieved) ta-C thin films deposited by pulsed-laser deposition. Figure 2a shows a representative data set for an annealed sample. The curvature data were converted into average and instantaneous stress curves versus thickness (Fig. 2b), assuming that the etch rate was linear with time.

The as-grown compressively stressed films display a uniform, constant (~6 GPa) stress

profile (not shown) through the bulk of the film. At the silicon interface, the stress increases quickly from zero to the bulk value, but at the film surface there is a thin layer (~20 Å) with a large tensile (5-10 GPa) stress.

The data for the annealed film (Fig. 2a) show a complicated stress profile, even though the nominal in-plane average stress is near zero (no net change in curvature from start to finish in Fig. 2a). The bulk film stress is actually 78 MPa tensile, the silicon/ta-C interface is in compression (1 – 5 GPa), and the ta-C growth surface remains in tension (5-10 GPa). Clearly, the stress relaxation dynamics (that relieves the bulk film stress) are not the same at the silicon interface and the top surface of the film. Annealing relaxes the stress near the Si interface only partially and has almost no effect on the surface tensile stress.

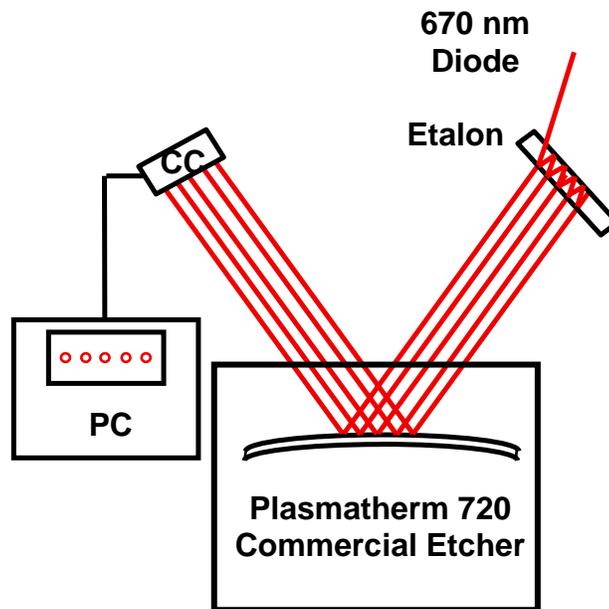
**Significance**—The information we have obtained on stress gradients in ta-C thin films now allows us to control out-of-plane bending in released devices, mainly by depositing compensating stressed layers on devices. This technique is generally useful for other materials systems where knowledge of stress gradients is important.

For ta-C thin films, it has been generally known that the surface layer is not the same as the bulk due to the growth energetics. Molecular dynamics simulations of film growth have predicted a tensile surface layer. We have now provided the first experimental confirmation of that result.

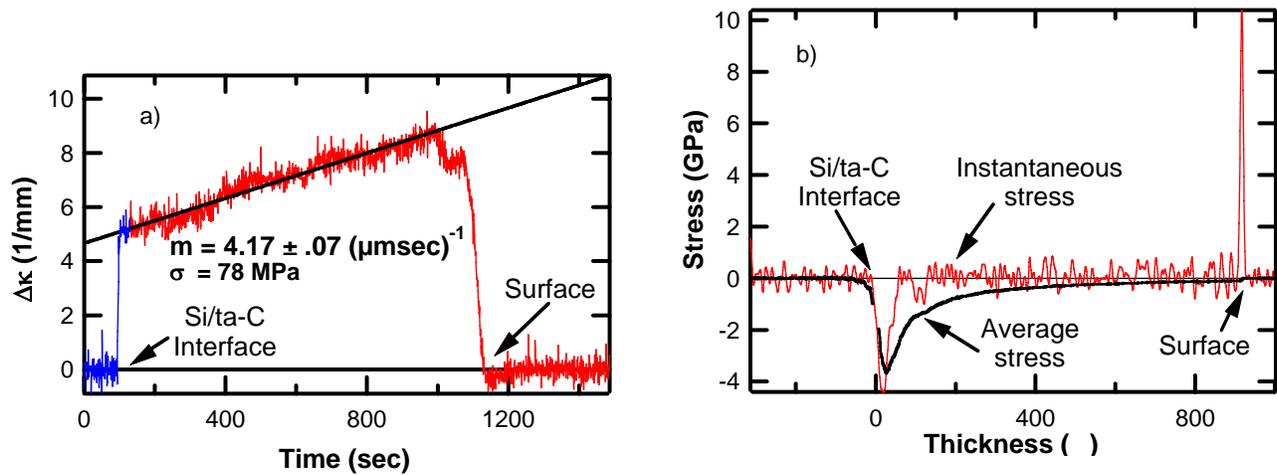
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**Figure 1.** Schematic of the apparatus used to measure radius of curvature *in situ* during etch-back. The system is capable of measuring a change in radius of curvature of 5 km, corresponding to a stress change of  $\pm 5$  MPa for the 250  $\mu\text{m}$  thick silicon substrates.



**Figures 2a & 2b.** Figure 2a shows change in curvature versus etch time for a sample with nominal zero-average stress as determined by conventional wafer curvature measurements. Note the large excursions both at the sample surface and ta-C/silicon boundary associated with interfacial stresses. The slope of the curvature change versus time corresponds to a bulk stress of 78 MPa tensile. In Fig. 2b the average and instantaneous stress derived from the curvature data in Fig. 2a are plotted versus sample thickness. Note the high surface stress ( $\sim 10$  GPa tensile) and the high interface stress ( $-4$  GPa compressive).