IMPROVEMENTS TO FORMVAR TENT FABRICATION USING THE MENISCUS COATER

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The centering of the capsule strongly depends on high quality tents with closely-matched mechanical properties. The relevant properties are tent stiffness and relaxation behavior. Tent stiffness is matched by choosing tents of equal thickness. Here, we describe recent advances in tent fabrication that have increased the quality and production rate of tents. The most significant improvement comes from the use of a meniscus coater to produce Formvar tents of high uniformity and good control of tent thickness with good yield. Other improvements include a switch to silicon wafers as deposition substrate and standardized tent holders. The improvements have resulted in a sixfold increase of the production rate while increasing the yield by a factor of two, despite a tighter quality control.

I. INTRODUCTION

In ignition targets, the capsule is suspended in the center of the hohlraum by two 110 nm thin polymer films called "tents".[1] The positioning of the capsule is critical for a successful experiment on NIF and thus there are tight tolerances (20 µm axial position tolerance, 25 µm radial position tolerance)[2]. The tents play a crucial role in positioning the capsule: the capsule is placed between the two tents, each of which pushes against the capsule. Centering is only achieved if both tents push with equal force. To meet the specification, the force constant of both tents has to be evenly matched, and thus their thickness and internal stress have to be matched.

The specified material for the tents is Formvar, or polyvinyl formal. There are three major challenges in the production of thin films for NIC targets: producing films of sufficient uniformity, removing those films from the substrate and mounting them on holders without compromising their quality, and finally producing films in sufficient numbers with high yield.

Here, we describe improvements to the production of tents that enhance our ability to build targets with two well-matched tents and thus achieve good capsule centering. The major change is the use of a meniscus coater[3] to achieve high thickness uniformity across a large area. The meniscus coater functions similarly to a dip-coater but has several advantages. We further address process changes that have improved film lift-off and overall yield.

In the overall process, we increased our maximum production rate from 12 tents/day to 72 tents/day, and the net yield of tent pairs is typically greater than 60%.

II. EXPERIMENTAL

II.A. The Meniscus Coater

The meniscus coater is a coating device that works under the same operating principles as a dip-coater: the substrate is moved across the meniscus of a polymer solution. A liquid film remains on the substrate after it leaves the meniscus, and the solvent in this film eventually evaporates to yield a thin film of the dissolved polymer. The thickness of the film depends on several parameters, including the solution viscosity, the concentration of the polymer, and the speed at which the substrate is moved.

The meniscus coater setup shown in Fig. 1 consists of a tank for the solution, a chassis that moves the substrate across the solution, and a motor with controls that drives the chassis. Within the tank, there is an air knife-shaped well through which the solution is pumped from below to wet the well uniformly with solution. The unused solution is collected in the tank below the well. The drain in the tank is connected to a pump, which pumps the solution through a 1 µm filter back to the well and completes the cycle. The chassis is currently set up to receive 4" wafer substrates.

The meniscus coater has several advantages compared to a dip-coater that result in more uniform coatings. The substrate motion is horizontal rather than vertical, which removes gravity effects on the film. The meniscus is formed with an air knife-shaped well through which solution is pumped continuously, which prevents skin formation and concentration gradient formation. It also allows continuous filtration of the solution just before it reaches the substrate, enabling a cleaner deposition. Further, because the substrate does not have to be completely immersed in the solution, scaling up the substrate does not require a larger volume of solution and is comparatively simple.

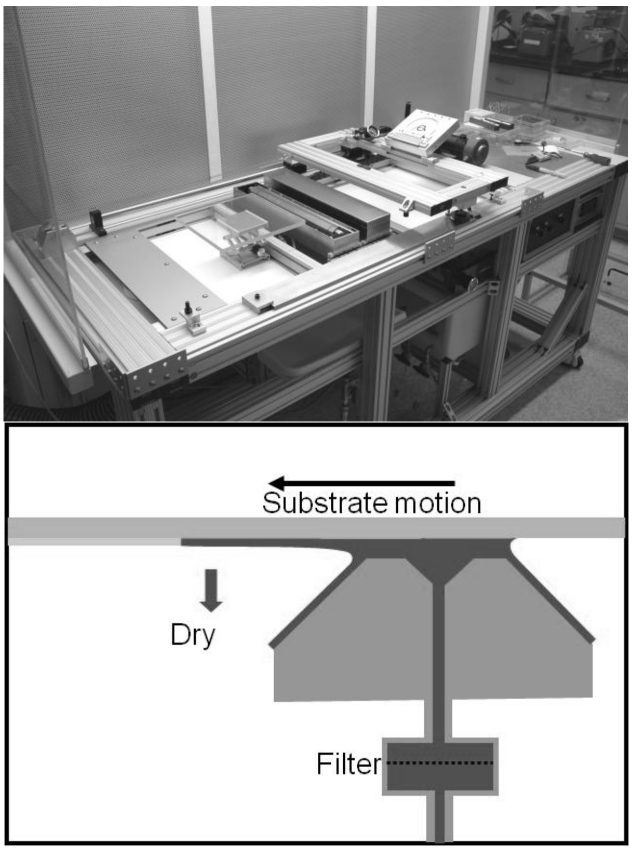

Figure 1. top: the meniscus coater setup. bottom: schematic of the meniscus coater deposition.

II.B. Film Production

A 4” silicon <100> wafer is placed into a petri dish,covered with piranha solution (3:1 H_2SO_4 (95%) : H_2O_2 (30%)) and then heated to 100°C on a hotplate. After 30 mins, the wafer is removed and rinsed thoroughly with distilled water. When the wafer has dried, it is sprayed with Windex®. The Windex foam is kept on the wafer for 60 s before it is rinsed off with distilled water. The wafer is then dried completely before the film is deposited.

The wafer is placed in the meniscus coater and moved over the solvent well at a constant velocity. The solution used for coating is a 1% by weight solution of Formvar in ethyl lactate. After coating, the wafer dries in air for 15 mins, then is baked at 50°C for 1 min.

The film is cut into squares by scribing it on the substrate with a razor blade. Then, the wafer is slowly lowered into water. The film lifts off from the wafer as the wafer is immersed and floats on the surface. When the film has been removed from the wafer, the film squares are fished out of the water with custom-made holder and then dried for 2 hrs.

The film thickness is characterized with a Filmetrics F20 reflectance spectrometer. Some tents are destructively tested with a ball indenter to verify that they will hold up to the assembly process.

III. RESULTS AND DISCUSSION

III.A. Meniscus coater characterization

Thin films fabricated by meniscus coating required two process development steps. In the first step, the optimal solution concentration and substrate velocity range was determined. We may note here that is easier to perform this step on a dip coater than on the meniscus coater, because switching solutions between depositions is simpler in the dip coater, and a smaller volume of solution is required

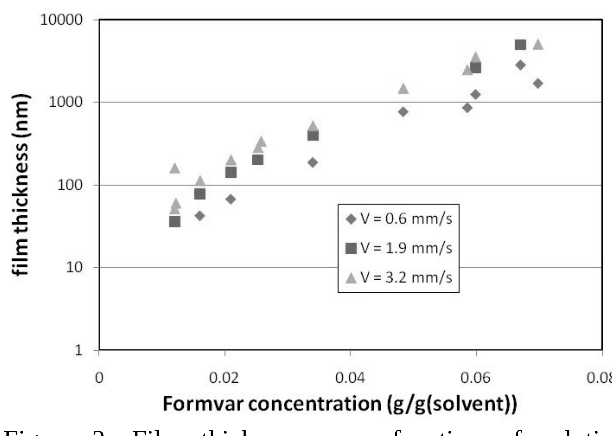

Figure 2: Film thickness as a function of solution concentration. Depositions were performed at three different substrate velocities.

Figure 2 shows the result of the first test. Ten different concentrations of Formvar in ethyl lactate were deposited at three different speeds to find an optimal deposition concentration. The thickness t relates to the viscosity η of the solution through[4]

$$t \sim \eta^{2/3} \tag{1}$$

The viscosity, in turn, depends on temperature and solution concentration, as given by the Huggins equation:

$$\frac{\eta_{sp}}{c} = |\eta| + k'|\eta|^2 c \tag{2}$$

$$\eta_{sp} = \frac{\eta - \eta_0}{\eta_0} \tag{3}$$

, where c is the solution concentration, η_{sp} is the specific viscosity, η0 is the viscosity of the pure solvent, η is the viscosity of the solution, $|\eta|$ is the intrinsic viscosity of the solvent/polymer pair and k' is a constant that is dependent on the solvent/polymer pair as well as the temperature. While we have not measured the viscosities of the individual solutions or maintained constant temperature during the calibration measurements, we find that the dependence of the film thickness on the concentration generally follows the theoretically expected trend.

The solution concentration increases with time as some solvent invariably evaporates. As[4]

$$t \sim V^{2/3} \tag{4}$$

, where V is the substrate velocity, moving the substrate at higher speeds should provide a finer control of the thickness. We chose a solution with a concentration of 1% by weight to obtain a solution that could be used over four months before needing to be exchanged This also allowed us to coat at substrate speeds between 6-7 mm/s. a thickness vs concentration calibration for the 1% solution is seen in Figure 3.

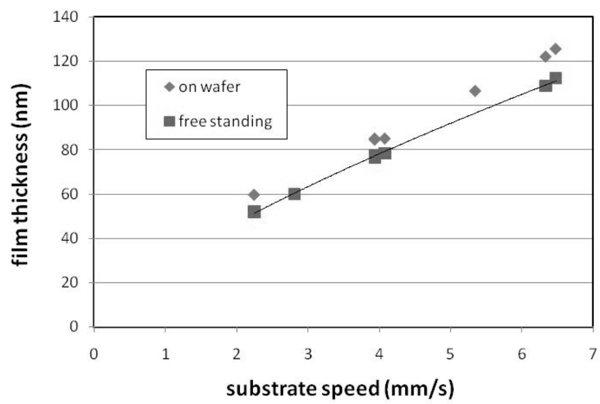

Figure 3: Calibration curves of film thickness vs. substrate speed for a 1% solution of Formvar in ethyl lactate. A power law fit with an exponent of 0.73 was added to the freestanding film data.

The thickness of the film on the wafer is about 10% larger than that of the free standing film. The data was fitted to a power law curve with an exponent of 0.73, close to the expected 0.66. To obtain finer control over the thickness, even higher deposition speeds and lower concentrations would be necessary, but we have found that the thickness control provided by the 1% solution is sufficient to make films with the required tolerance (±5 nm).

Because the solution concentration increases with time, and the thickness of the deposited film also depends quite sensitively on temperature (changing temperature by 1˚C changes the film thickness by 2-3 nm at 110 nm deposition thickness), a calibration run is performed at the beginning of every production day to determine the deposition speed for that day.

III.B. Substrate Preparation improvements

A substrate with well-controlled surface properties is essential for obtaining repeatable coating and lift-off. The surface roughness and functionality play a major role in lift-off, and a high thermal conductance prevents hot or cold spots on the film, which can lead to drying abnormalities, such as the one shown in Fig. 4.

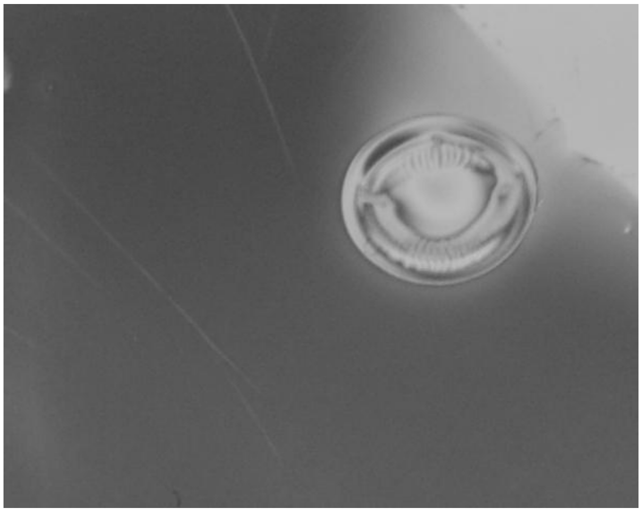

Figure 4: Formvar film with drying spot. The film was transferred to a silicon substrate to increase contrast. The film thickness in the drying spot can be more than twice as high as in the rest of the film.

Initially, glass microscope slides were used as substrates. However, lift-off from these slides was not reproducible and varied from lot to lot. XAFS was performed on the slides, and it was found that the chemical composition of the glass and the surface varied substantially between lots. The surface roughness of glass slides was measured with white light interferometry (Wyko) and likewise found to vary strongly between lots (15-30nm RMS roughness). Finally, glass has a low thermal conduction coefficient, and depositions on glass slides where prone to drying spots and thickness variations of several nanometers over a 1 inch square deposition area.

Silicon <100> wafers are commercially available with very low wafer-to-wafer variations for the microelectronics industry. The silicon wafer makes an ideal substrate for the deposition. The surface composition and roughness variations between wafer batches are minimal. The thermal conductance of silicon is higher, reducing the impact of thermal gradients on the back of the substrate. Many well-documented and tested procedures exist for the cleaning and functionalization of these substrates. Therefore, we replaced our glass microscope slide substrates with 4” silicon <100> wafers.

To further enhance lift-off, the wafer is made hydrophilic by immersing it into a piranha solution (3:1 H_2SO_4 (95%) : H_2O_2 (30%)) for 30 mins at 100˚C and then applying a layer of surfactant. Most surfactants improve lift-off, but some surfactants provide smoother lift-off than others. The best surfactant among the tested ones was Windex®. The Windex solution was sprayed onto the wafer, allowed to stand for 60 s, and then rinsed off with distilled water.. When the wafer is immersed in the water, we believe the water permeates between the two hydrophilic surfaces and separates them.

Also, it appears that lift-off works best if the Formvar solution contains minute amounts of water, which it takes up naturally over several days. A fresh, water-free solution leads to a film that is much more difficult to lift off.

III.C. Lift-off improvements

Lift-off is ideally performed as soon as the film has dried. It was observed that completely dry films (dried over many hours) did not lift off. The films can typically handled for 20-30 mins after coating and still be lifted off successfully if the wafer was treated as described above.

During a daily calibration run, the film thickness is measured while the film is still on the wafer. The film uniformity across the entire wafer was found to be usually around 0.5 nm, excluding the edges. The thickness of the film on the wafer is always higher than that of the free-standing film, likely due to residual solvent in the film. If the film is not lifted off of the wafer, but left to dry there instead, the film thickness decreases over the next day before stabilizing.

Figure 5: Film liftoff. The holder is clamped into a rack-and-pinion at an angle of 35° to the water surface. The film is pinned to the holder, and then the holder is withdrawn from the water with a steady motion.

One improvement for lift-off consists of the use of rack-and-pinions with fixed angles both for immersing the water into the wafer and for removing the films from the water surface. The rack-and-pinion provides a steady, well controlled motion. A jerky motion while manually immersing the wafer possibly leads to the film folding back onto itself or becoming partially immersed in the water instead of floating on top, both of which make the film unusable. The film removal from the water surface likewise benefits from a smooth motion and a fixed angle. When the film is removed from the water, interplay between surface tension and the parts of the tent that are fixed on the holder may cause the tent to wrinkle or crease. For our setup, an angle of about 35˚ minimizes this creasing (Fig. 5). The film is pinned onto the holder in the initial phase of the removal by attaching it to the holder and the draining the water between the holder and the tent. After the initial pinning, the tent is withdrawn at a steady pace until the lowest part of the tent holder leaves the water.

Wrinkles and creases, such as the one shown in Figure 6, are undesirable because they indicate or cause asymmetric stress in the tent. This can result in the tent responding differently to loading compared to a unwrinkled film, leading to capsule offset.

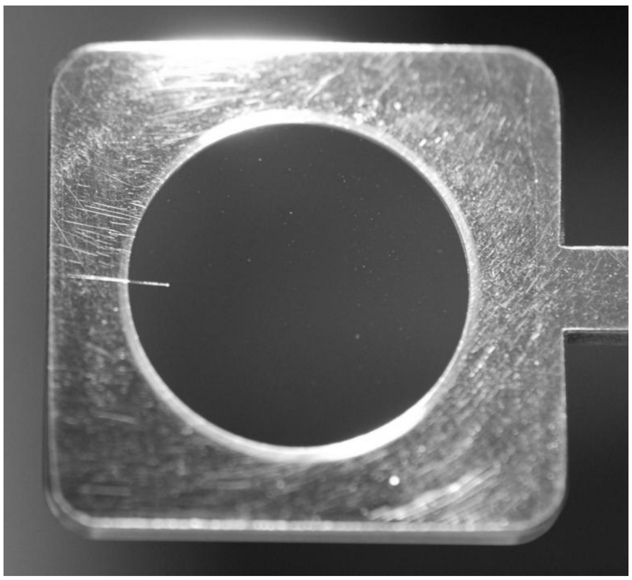

Figure 6: Tent on tent holder. The tent holder is a 19 mm square of stainless steel with a hole in the middle. This tent has a crease on the left side. The white dots inside the circle are dust particles on the tent. The tent edge on the holder can be seen on the right side where the tone of the steel changes slightly.

III.D. Film Characterization and Pairing Criteria

After drying for two hours, the film thickness is measured with a Filmetric F20 reflectance spectrometer. The Filmetrics is a precise instrument, but its accuracy strongly depends on the angle of the measured film. The repeatability between measurements is typically below 0.5 nm. However, the substrate has to be perfectly perpendicular to the incident light to produce and accurate measurement. The fidelity of fit of the data gives an indication of the perpendicularity of the substrate. The holders were designed to provide a level surface perpendicular to the incident light to make as accurate measurements as possible. The tents tend to warp slightly around the edge of the holder, however, which distorts the measurement and makes the tents appear less uniform in thickness than they actually could be. Regardless of whether distortions or film non-uniformities are the cause, films that vary in thickness by more than 2 nm are not used in target assembly.

After characterization, the tents are packaged in sets of two or three. Each set comes from the same batch, and the thickness of the tents in the set is matched to within 1 nm. The films are stored in sealed wafer trays overnight to allow stresses introduced during lift-off to relax before the tent is processed further.

Some tents from each production run are set aside for destructive testing. The testing is performed with a ball indenter[5]: the tent is mounted on a holder with the same dimensions as a hohlraum, and then a steel ball with the dimensions of the capsule is lowered step-wise into the film while the film stress is measured with a balance. The indentation is performed in the same way as the tenting process during target assembly: the ball is moved 100 µm into the tent in 1 min, followed by a pause of 3 mins before the next repeat step. In assembly, the tents are indented about 1 mm deep (radius of capsule). For our test, we require that the tent not fail to at least 2 mm, giving us a safety margin of 100%. The stress response of two tents from the same batch usually deviates by less than 2%. Typical data from such a test is shown in Fig. 7.

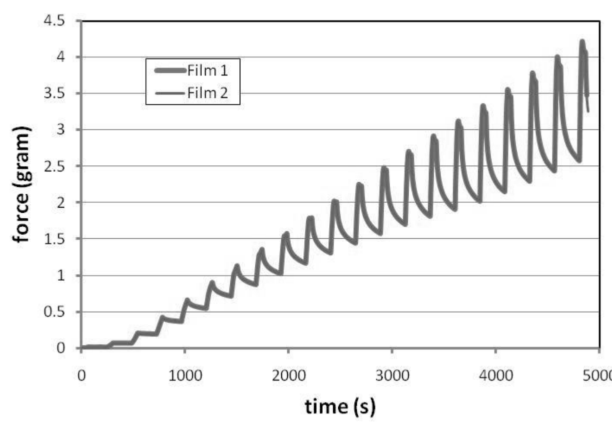

Figure 7: Indentation test of two films from the same batch. The tents are indented 100 µm every 4 mins at a rate of 100 µm/min. The two curves are almost exactly identical.

The tents have a shelf life of three weeks, and are usually processed as soon as possible. A more detailed description of aging effects and mechanisms exceeds the scope of this publication and will be treated elsewhere.[6]

IV. SUMMARY

We have developed a robust fabrication method for Formvar tents for NIF targets that produces well-matching tents with good yield at a high rate. The major improvements come from replacing the dip-coating process with a meniscus-coating process, and switching from glass to silicon substrates. The resulting tents show consistent performance that meets the NIF target requirements.

ACKNOWLEDGMENT

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