REAL-TIME MONITORING OF STRIATION DEVELOPMENT DURING SPIN-ON-GLASS DEPOSITION

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ABSTRACT

A commercial spin-on-glass (SOG) solution was monitored during its deposition across silicon wafers in order to determine the moment at which coating defects, specifically striations, develop within the spinning solution. The radially oriented thick film ridges of striations act as a diffraction grating for an incident laser beam. Video monitoring of this diffraction pattern throughout the spin coating process allows us to determine the exact moment at which the striation defects begin to develop within the spinning solution. Simultaneous interferometric analysis has been performed to determine the fluid thickness at this instant in time. Performing this simultaneous video monitoring and interferometry during the spin coating of several Si wafers at various rotation rates we were able to confirm that striation growth is an evaporation driven phenomenon, taking place near the end of the "spin off" stage of spin coating.

INTRODUCTION

Spin coating is a common method for depositing thin, uniform films across planar substrates. Rapid wafer rotation causes the precursor fluid to spread radially and (theoretically) evenly across the surface. The coating fluid consists of solutes in solution with more volatile solvents that serve to lower the fluid viscosity and that steadily evaporate throughout the spin coating process leaving behind a thin, solute-rich film.

Spin coating is used in the microelectronics industry primarily for the deposition of photoresists in lithographic circuitry definition, although it has become increasingly important for the deposition of spin-on-glass (SOG) solutions. These SOGs are used primarily to provide planarization of substrates. The SOG solution can coat and fill in topographically complex surfaces that result after metallization of a particular level. The SOG also serves as a passivation and insulating dielectric layer. In addition, spin-on-glass solutions may be used to replace thermally grown gate and field effect oxide passivation layers in the manufacturing of various metal-oxide semiconductor (MOS) devices [1].

The spin coating process is traditionally broken into a number of stages [2,3] describing the physical mechanisms that dominate the thinning behavior over a period of

time. Essentially these can be summarized as the "spin-off," or "flow dominated," and "evaporation dominated" stages. "Spin-off" refers to the bulk removal due to centrifugal acceleration of fluid at the beginning. This stabilizes into a regime where the viscous effects control the thinning. This is followed immediately by the "evaporation" stage in which any further thinning occurs mainly due to the evaporation of solvents.

Although it is generally quite an efficient process, a number of coating defects can occur as a result of the spin coating process. One common type of surface defect is called "striations". These are thick ridges that develop during spinning and that emanate spoke-like from the center of the substrate. As feature sizes and layering depths decrease in IC and VLSI devices, the necessity for any SOG layers to be uniform and strictly planar increases. For these reasons it is imperative that spin on glass layers be as uniform and planar as possible. This requires intimate knowledge of the parameters governing striation growth during the spin coating process.

In the present work we are concerned with determining the precise moment at which striations begin to develop during the spin coating of a particular striation-prone SOG solution as well as the thickness of the film at this instant. Our overall objective is to attain a complete understanding of the factors governing spin-on processing of films.

EXPERIMENTAL PROCEDURE

In recent work [4] we have presented a simple diffraction experiment with which the characteristic spacing of the striations in a thin coating can be determined. In performing the present tests we have utilized the diffraction effect, but have applied it to wafers that are spinning rather than stationary; the motion of the wafer does not interfere with the striation diffraction effect. Thus, we have observed the light pattern from a laser reflecting from the surface of the wafer (with SOG solution flowing over it) throughout the spin coating process. This has allowed us to monitor when the striations develop. The independence of the diffraction effect between stationary and rotating wafers was confirmed by the fact that the diffraction pattern was not changed when the wafer stopped at the end of spinning. In earlier work we had shown that the average striation wavelength was dependent on the spinning rate [5], but it is also true that simultaneously the coating will be reaching a different final thickness. Independent of what the final coating thickness will be, the interesting thing to note is that for spin coating using a striation-producing solution, the monitoring of the reflected laser light reveals a distinct period during which the diffraction pattern develops – and by inference it indicates the time when the striation defects are formed and printed into the coating thickness.

The experimental setup is as follows: a 633 nm solid-state laser is aimed at a position roughly 1.5 in. from the center of the four-inch diameter Si wafers upon which the SOG is to be deposited. The laser is incident upon the wafer at a glancing angle of about 79^{o} from normal. The reflected light rays then pass through a lens and the resulting diffraction pattern is imaged on a screen located at the focal length of the lens. A commercial Sony® digital video camera, model number DCR-TR7000, was then used to record the evolution of the diffracted light pattern throughout the spin coating process. The standard video frame rate of 30 fps then defined the time frequency at which this diffraction data was recorded. The moment at which the diffraction from striations was first noticeable was then defined as t_s and is tabulated below for several spin speeds.

At the same time, optical interferometry was performed upon the spinning solution. This optical interferometry follows along the precise scheme described elsewhere [6] for the measurement of evaporation rate and kinematic viscosity of spin-on solutions. A second laser is directed at near-normal incidence at a position near the center of the Si wafer. This light is passed through a beam splitter so that half is directed into a ("baseline") linear photo-detector and the other half travels straight down to reflect from the wafer/film surface. This reflected beam upon returning to the beam splitter is then directed into a second ("signal") linear photo-detector. The signal is then normalized using the baseline and the resulting intensity vs. time plot is analyzed. The reflected signal consists of a superposition of the light that is reflected from the fluid surface and that which is reflected from the surface of the Si wafer. These reflected signals are then translated into varying intensity vs. time plots, the peaks of which correspond to fluid thicknesses being an integral number of ¼ wavelengths (within the solution) thick. Interested readers are directed to [6,7] for a more thorough explanation as well as examples of these intensity vs. time plots, or "optospinograms [7]." With knowledge of the final coating thickness, we can use these "optospinograms" to generate a consistent plot of the fluid thickness H throughout the spin coating process. Using this H vs. t plot we can accurately determine the thickness of the fluid at the moment when striation growth gets started, H_s.

Using the processes described above, we monitored the spin coating of a commercially available SOG solution spun at different rotation rates varying from 500rpm to 4000rpm. Each trial began with the deposition of an approximately 5ml puddle of solution across the center of four-inch diameter Si wafers. This volume of fluid corresponds to roughly one millimeter of initial fluid thickness. The wafer was then accelerated at the highest ramping rate available, which brings the substrate from rest to the desired rotation rate within a half a second or less, as determined from independent stroboscopic video analyses performed upon the spin coater itself.

In order to determine the final coating thickness we etched away a small region of the SOG until all that remained was bare silicon and then measured the SOG thickness using a Tencor brand Alpha Step 200 surface profiling device.

RESULTS AND DISCUSSION

Table I and Fig.1 show the thickness H_s of the thinning film at the point when striations begin to develop and its dependence on the rotation rate ω. The exact relationship between H_s and ω is not straightforward. Indeed Fig.1 suggests that H_s takes on two distinct forms depending upon the range of rotation rates being considered. Fig. 1 also demonstrates a similar discontinuity in the amount of time t_s that passes before the thinning fluid attains this H_s depth at any particular spin speed.

The diffraction pattern imaged at a given spin speed reaches its full spread within a few seconds of the onset of striations, again depending upon the rotation rate. That is, once a diffraction pattern starts to become evident at t_s, it takes approximately 1 second for the responsible striation pattern to fully develop when spinning at 4000 rpm and 8 to 10 seconds at 500 rpm. As is depicted in Fig. 2 the fluid thickness H_s at the onset of striations is considerably less than the initial fluid depth. It is interesting to note that the solution depths are much smaller than the corresponding striation wavelengths (see last

Table I: Parameters characteristic of striation growth at several spin speeds, ω. H_s represent the fluid depth at the onset of striation growth; t_s is the time at which this growth begins, as referenced to the start of spin; Δt is the time period through which the striation pattern grows into its final form; H_f is the final thickness of the deposited film and λ_f is the final spacing between striations.

ω (rpm)	H_s (μm)	t_s (s)	Δt (s)	H_f (μm)	λ_f (μm)
500	8.0	4.1	9.6	1.5	143
1000	4.0	2.9	3.0	1.0	107
1500	2.6	2.4	1.9	0.80	89
2000	2.5	2.1	1.8	0.62	56
2500	4.2	1.1	1.8	0.56	44
3000	5.3	0.8	1.3	0.52	28
3500	5.7	0.7	1.3	0.46	29
4000	6.7	0.6	1.1	0.45	29

column in Table 1). In fact, the coating depths measured at the point when striations just get started is very small suggesting that our coating solutions have already reached a point where viscous outflow has effectively ceased and evaporation of the solvents controls the thinning of the solution.

We have demonstrated elsewhere [5] that the spacing between the ridges on a given striated wafer is constant across the entire substrate surface. Thus unlike the simple spoke analogy, the distance between these radially oriented thick film ridges remains essentially unchanged at any position on the film surface. That is, looking out along a given radius we see that striations appear to bifurcate locally once sufficient

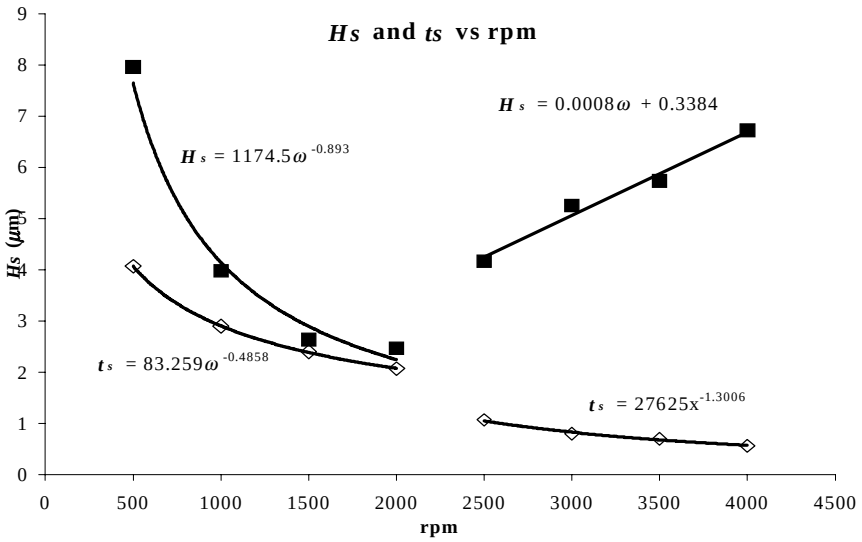

Fig.1: The variance in fluid thickness at the onset of striation growth H_s with spin speed ω as well as the variance in the amount of time which passes from the start of spin coating process to the onset of striation growth, t_s with spin speed ω.

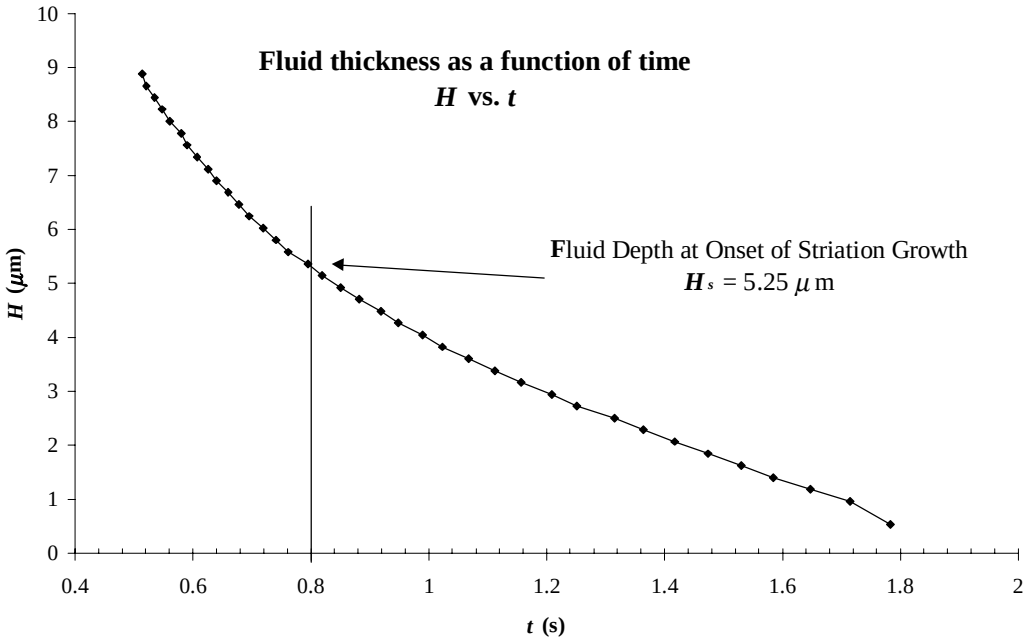

Fig 2: Typical thickness vs. time profile generated from interferometric monitoring of the thinning fluid behavior. This particular curve shows the thinning of an SOG spin coated at 3000 rpm. The vertical line represents the time at which striations began to develop as determined from frame by frame analysis of the video monitoring of the ensuing diffraction pattern which develops as the striations grow. The position at which this line crosses the H vs.t curve represents H_s, the fluid depth at the onset of striation growth.

spreading occurs such that the average spacing between striations remains constant across the entire substrate.

The development of striations as a direct result of solvent evaporation has been demonstrated experimentally [8,9]. The generation of ridges throughout the striation development time Δt which results in a constant striation spacing at all positions across the substrate can be explained in terms of localized feedback processes in solvent vapor evaporation from the SOG solution to the surrounding ambient. As the thinning solution nears the H_s fluid depth evaporation effects become increasingly significant. The evaporation rate becomes large enough that the solution's composition starts to be affected. A condition develops in which solvent diffuses faster out of particular regions near the fluid surface resulting in localized depletion of solvents and thereby localized surface tension variations (i.e. causing the "Marangoni Effect" [10]).

The airflow over these developing striations which is a result of the rotating substrate carries the diffused solvents tangentially "down stream" a brief distance prior to their ultimate bridging of the diffusion layer between the surrounding ambient and the fluid surface. Thus the air directly above the region behind a growing striation ridge is slightly more solvent enriched. This serves to decrease the diffusion of solvents from the solution in this region thereby enhancing the slight surface gradient in solvent concentration that fueled the striation growth in the first place. Furthermore the striation

ridges themselves, being relatively depleted of solvents, possess a slightly higher surface tension which instigates a capillary drawing of more solvents away from these nearby regions which possess slightly greater solvent concentrations due to the air-flow retardation of local solvent diffusion, creating an unstable effect similar to that found for the Rayleigh instability [11].

By monitoring the diffraction pattern from a particular position on the wafer throughout Δt we can observe this process in action. Once the fluid thins to the characteristic H_s depth we see a slight diffraction pattern begin to emerge. The spin on solution passes from the "flow dominated" to the "evaporation dominated" stage and thus solvent evaporation becomes increasingly significant to the thinning process and the striation "seeds" begin to develop as described above.

CONCLUSIONS

Judging from the relative thickness of the SOG solution at the onset of a diffraction pattern it is clear that striations do not develop until the latest stages of spinning, after the so-called "flow dominated" stage is very nearly completed. Further, the full or final striation pattern sets in across the film surface at essentially the same moment the entire fluid layer stops thinning, as indicated by the optospinograms.

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