MICROSCOPIC IMAGE PROCESSING SYSTEM FOR MEASURING NONUNIFORM FILM THICKNESS PROFILES: IMAGE SCANNING ELLIPSOMETRY

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ABSTRACT

The long-term objective of this research program is to determine the stability and heat transfer characteristics of evaporating thin films. The current objective is to develop and use a microscopic image-processing system (IPS) which has two parts: an image analyzing interferometer (IAI) and an image scanning ellipsometer (ISE). The primary purpose of this paper is to present the basic concept of ISE, which is a novel technique to measure the two dimensional thickness profile of a non-uniform, thin film, from several nm up to several μm, in a steady state as well as in a transient state. It is a full-field imaging technique which can study every point on the surface simultaneously with high spatial resolution and thickness sensitivity, i.e., it can measure and map the 2-D film thickness profile. The ISE was tested by measuring the thickness profile and the refractive index of a nonuniform solid film.

I. INTRODUCTION

The effects of interfacial stress (which is a function of **film shape** and interfacial intermolecular forces) and temperature on the stability and dynamics of nonisothermal thin liquid films have been extensively studied because of their generic importance to multiphase flow technology. See, for example, the extensive review of the literature on the effects of interfacial forces on the dynamics and stability of thin evaporating liquid films on heated surfaces by Bankoff [1]. In these small thin film systems, the complex microscopic details of fluid flow and film shape are of paramount importance to the change-of-phase heat transfer process [2]. Needless to say, many experimental studies have been reported in the literature. For example, in a companion paper to the theoretical paper on thermocapillary flow by Tan et al. [3], Burelbach et al. [4] experimentally studied thermocapillary flow in a thin liquid layer. The electrical impedance measurement of the film thickness was accurate to 1 μm. In small nonisothermal systems, the interfacial intermolecular force field controls fluid flow and change of phase heat transfer. This was discussed by Wayner et al. [2,5] who used classical change-of-phase kinetics and interfacial concepts like the Kelvin, Young-Dupré, and augmented Young-Laplace equations to evaluate and compare stress (shape) and thermal effects. These results demonstrate the central importance of the augmented Young-Laplace equation to the understanding of the dynamics of both completely wetting and partially wetting thin liquid films. Experimental confirmation of the central importance of the augmented Young-Laplace to describing the stress field in thin liquid films was obtained using the image analyzing interferometer, IAI [e.g., 5]. However, these and other recent results demonstrate that more powerful and convenient microscopic experimental techniques are needed to decrease the lag in the development of evaluative experimental techniques. Thinner and smaller dynamic systems need to be studied at higher magnification. For this purpose, a new experimental technique, ISE, which uniquely combines ellipsometry, image processing, and a specific algorithm, has been developed and is described herein.

If the physical properties of the evaporating liquid films are known, the film thickness profile, measured experimentally, can be used to calculate the heat transfer characteristics of thin liquid films. Therefore, one of the major experimental objectives of thin film heat transfer research is to determine the film thickness profiles as a function of the experimentally varied evaporation rate. In order to experimentally measure the film thickness profile of an evaporating, non-uniform thin film, the following equipment characteristics are required: (1) The instrument should be able to measure non-uniform films as well as uniform films from several nm up to several µm in thickness; (2) The instrument should be able to measure the thickness profile in the transient state as well as in the steady state; (3) The instrument should be able to measure every point on the liquid surface simultaneously instead of an average value over the measured surface. Many different optical and non-optical techniques have been developed to measure film thickness profiles. However, none of these techniques had all the required characteristics mentioned above. One disadvantage of non-optical methods is that they can disturb the films during measurement. The optical methods based on interferometry, in general, can measure non-uniform films but cannot accurately measure film thicknesses under 1000 Å nor are they appropriate for uniform films. Image Analyzing Interferometry (IAI) [5] is a new technique based on interferometry which was developed to measure non-uniform film thickness profiles. IAI not only has very good thickness sensitivity and lateral resolution but can also be used to study every point on a surface simultaneously by using image processing technology and a low-light-level CCD camera. However, IAI is not well suited for transient state studies because it uses a standard, time consuming, null ellipsometer, to extrapolate the film thickness beyond the first dark fringe. The optical methods based on standard ellipsometry, in general, can measure uniform film thicknesses from 1 Å up to several µm but not spatially varying film thicknesses. They are also not suitable for transient state studies. Dynamic Imaging Microellipsometry (DIM) [6] is the only new technique based on ellipsometry developed to measure a film thickness profile in a transient state. However, with DIM, the optimal settings of the ellipsometric components change with the film thickness and so accurate measurements over wide thickness ranges are difficult. Due to this complication, DIM is basically restricted to the measurement of more uniform films with thickness variations within certain, narrow ranges, but not for thickness profiles ranging from several nm up to several µm.

II. THEORY AND DESIGN OF ISE

ISE is a new optical technique based on null ellipsometry for studying non-uniform film thickness profiles in the transient state as well as in the steady state. It is a full-field imaging technique which can simultaneously study every point on the surface with high spatial resolution and great thickness sensitivity. Moreover, ISE uniquely results in a 2-D mapping of the film thickness profile. The theoretical development of this approach is based on the null ellipsometer. The measurements of null ellipsometry are quite routine, involving the rotation of a polarizer and an analyzer to cause extinction of a beam of polarized light reflected from the surface of the sample. The extinction point is called the null point. At the null point the values of the measured azimuths of the polarizer (P), the analyzer (A), and the compensator (C), measured from the plane of incidence, are recorded. All these angles are measured positive counter-clockwise from the plane of incidence when looking into the light beam.

The relative phase, Δ, and relative amplitude, Ψ, of the two orthogonal electric field components of the polarized light can be determined from the measured angles of P, A, and C using the standard equation for ellipsometry:

$$\rho = \mathrm{Tan}(\Psi) \times \mathrm{Exp}(i\,\Delta) \qquad (1)$$

where i is $(-1)^{0.5}$, ρ is the ellipticity, the ratio of reflection coefficients of parallel components over perpendicular components, Tan (Ψ) is the ratio of the amplitude of reflection coefficients of parallel components over perpendicular components, and is the phase shift between the parallel and perpendicular components. The values of Ψ and Δ can then be used to calculate the refractive index of the substrate or the film thickness and film refractive index. Due to multiple reflections of light across the film-vapor and the substrate-film interfaces, the measured values of Ψ and Δ exhibit an interference effect. In other words, Ψ and Δ are cyclic functions of the film thickness. The cyclic behavior is governed by

$$\delta = \lambda/2\ [n_F^2 - n_m^2\ \mathrm{Sin}^2(\Phi_1)]^{0.5} \qquad (2)$$

where λ is the wavelength of the light source, n_F is the refractive index of the film, where n_m is the refractive index of medium and Φ_1 is the angle of incidence. When the measured thin film is non-uniform, rotating the polarizer and analyzer can only partly extinguish the beam of polarized light reflected from the surface. The image appears as a set of destructive fringes representing various film thicknesses. In Fig. (1), the locations of the destructive fringes are presented as a function of film thickness and polarizer setting for A=0.00 and C=45.00. Combining this null ellipsometer with an image processing system, a long-working-distance microscope, and a low-light-level CCD camera with manual gain control, forms a system capable of easily measuring the thickness profile of a non-uniform film. By fixing the analyzer and compensator settings and rotating the polarizer, the ellipsometric null points, which have a minimum reflectivity, can be observed to sweep across the whole measuring area. Based on a knowledge of the pixel intensity and magnification of the system, the exact position of the null points can be determined. Using a series of ellipsometric images the film thickness profile from several nm up to several µm can be calculated. This thickness profile includes the thin film region, under 1000 Å, that the IAI cannot measure. The measurement process varies the polarizer setting, recording an ellipsometric image for each point. The more settings we use on the

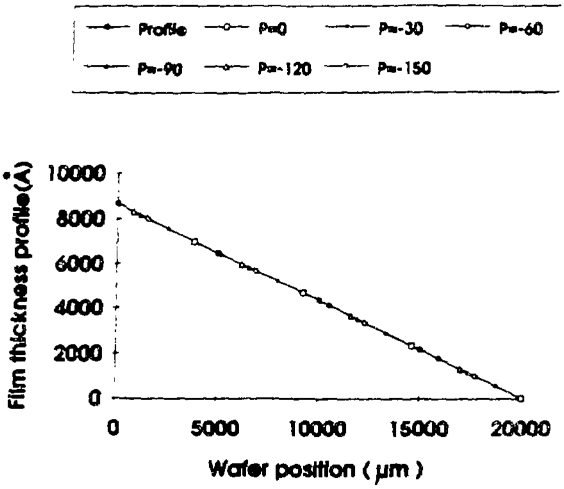

Figure 1. Calculated film thickness at the null point versus position for various polarizer settings. System was a particular ThF_4 solid wedge deposited on a Si wafer.

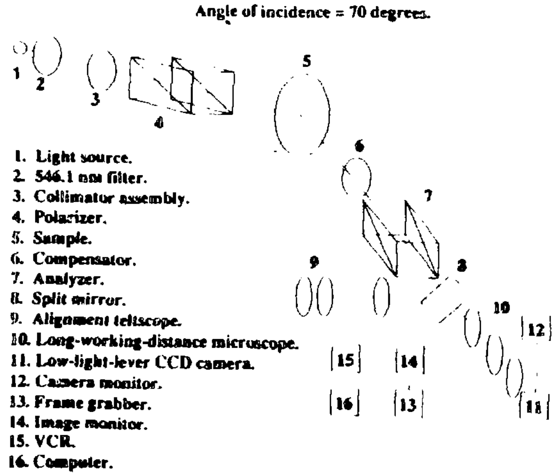

Figure 2. A schematic sketch of the Image Scanning Ellipsometer.

polarizer, the more points we have in the film thickness profile, i.e., the more detailed our measurements will be. The image processing system enables us to study the transient state phenomena and to find all the null points in the measured area. We can then construct 2-D contour maps to describe the non-uniform film thickness profiles.

The ISE measures the profile only using the null points instead of the intensity itself. By considering only reflectivity minima, there will be little or no interference from imperfections in the optical components, instability of the light source, sensitivity variations of the camera, or the presence of dust particles, finger prints, and other contaminants in the optical path. The accuracy and spatial resolution of ISE depends on the resolution of the imaging microscope and the slope of the thin film profile.

III. EXPERIMENTAL DESIGN

ISE includes the following major components which are organized schematically in Fig. (2): a null ellipsometer, step motor driver and step motor, long-working-distance (LWD) microscope, low-light-level(LLL) CCD camera with manual gain control, PC compatible computer, Frame grabber with 4 MB onboard memory, and Frame coprocessor for accelerating image processing.

Our ISE was constructed using a standard, manually-operated, null ellipsometer, with vertical sample holder, manufactured by Rudolph and Sons, Inc. in 1970's. In order to monitor transient liquid films in situ from the TV screen, magnification of the light spot after the analyzer is needed. The minimum distance between the analyzer and surfaces of the samples is 22" in this system and so an ordinary microscope could not be used. A K2-single port LWD microscope from Infinity Photo-Optical Company was used because the working distance of this instrument is 12" to 96". The actual magnification also depends on the distance between the eyepiece and the camera. Ideally the longer this distance, the higher the magnification. Unfortunately, the resolution decreases dramatically as the distance increases due to an overall decrease in the intensity of the images projected onto the camera. The best spatial resolution in our system was found to be 2 μm (vertical) x 7.5 μm (horizontal). The actual film thickness resolution depends on the nature of the film measured.

The intensity and contrast of the ellipsometric image decreases sharply as the film thickness increases. This problem is especially acute for film thicknesses beyond 1 μm. To achieve the highest spatial and film thickness resolution, a high resolution, low-light-level, CCD camera was used in our system. This camera had wide dynamic range, 52 dB, and manual gain control. Adjustable gain is important to control image intensity and contrast as the liquid films drain. The dark current of the camera must also be adjustable to insure accurate detection of the null point. If the dark current is set too low, the intensity minima may be buried below the resolution of the frame grabber/digitizer and accurate null point locations cannot be found.

A computer controlled step motor was used to rotate the polarizer. The rotation was synchronized with the frame grabber so that images were captured at known polarizer settings. Using only the frame grabber's storage capability, a complete film thickness profile, composed of 15 separate images could be obtained in 3 sec. Using a VCR to store the images decreased our capture time to 1.5 sec and increased the number of images to 45. The current speed of our system is limited by the frame processing speed of the frame grabber and camera system and the sheer mass of the ellipsometer components we are using. Significant improvements are possible in those two areas.

To test the ISE, a special custom wedge, ThF_4 on Si wafer substrate, was obtained. The film thickness changed linearly from 0 nm up to 870 nm within a distance of 20 mm. The thickness profile of this wafer was measured by fixing the compensator at 45.00, the analyzer at 0.00, and then varying the polarizer angles. The destructive fringes (minimum pixel value) were thereby moved across the surface. The images of the reflection patterns and therefore the location of the null points were captured and digitized into 480 (vertical) x 512 (horizontal) space pixels and assigned to 256 possible gray values representing an intensity from 0 (black) to 255 (white). In order to reduce the data processing time and the memory needed, non-uniform interval settings on the polarizer were used, 360, 350, 340, 330, 320, 310, 285, 260, 235, 210, and 185. These choices were selected because the relationship between polarizer angle and film thickness is non-linear (See Fig. 3). The choice of the best angles depends on the optical properties of substrate, the film, and the characteristics of the thin film profile. Although the choice of the polarizer angle does not make too much difference for a profile with a constant or nearly constant gradient such as a wedge profile, the choice of the polarizer angles will affect the sensitivity and accuracy of the measurements for a large, variable, gradient such as a draining liquid film. The rule of thumb is to use as many null points as possible and make the null points as equally spaced as possible within the first thickness cycle. This means that polarizer angles should be concentrated in regions I and III of Fig. 3. Beyond the first cycle, the null points are spaced closer to one another because of the increase of the film thickness gradient.

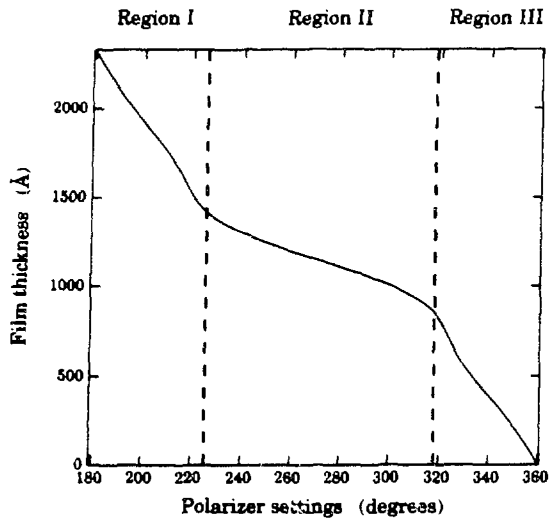

Figure 3. Calculated film thickness at the null point versus polarizer settings.

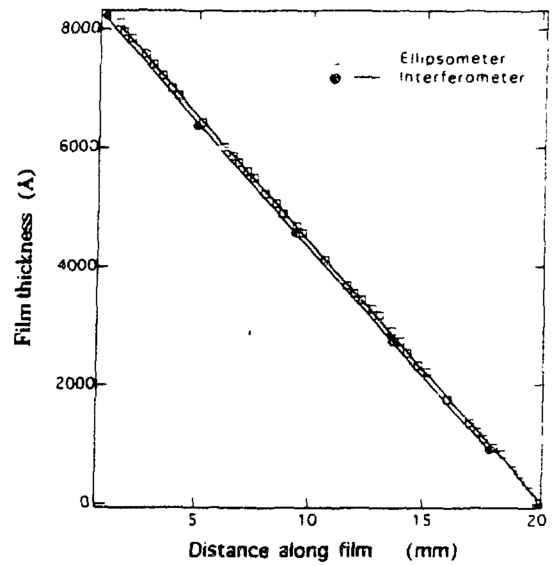

Figure 4. Experimentally measured film thickness profiles.

IV. RESULTS

To demonstrate the capability and accuracy of the ISE, the 2-D thickness profile of a non-uniform solid film of ThF_4 on a Si substrate was measured and compared with the profiles obtained by us using interferometry and measured by the manufacturer of the films (Reynard Company). The measured wedge profiles obtained from each method discussed above were analyzed using a regression program and the results are listed below:

From the Reynard company:

Standard wafer #1: $TH (Å) = -0.432 (Å/\mu m) * X(\mu m) + 8661.1(Å)$

From the interferometer:

Standard wafer #1: $TH (Å) = -0.426(Å/\mu m) * X(\mu m) + 8532.0(Å)$

The measured results obtained using our interferometer are almost the same as those reported by the manufacturer. Therefore, it is certain that the measured film thickness profiles on the standard wafers are correct. By the layout of the colored bands on the standard wafers the film thickness profiles can also be estimated by eye and used to verify our other measurements. A correction of the raw ISE data is required in the horizontal direction, because the imaging system is not oriented perpendicular to the substrate. This results in a non-uniform magnification in the horizontal direction. The vertical direction requires no corrections and the magnification there is uniform across the substrate. The 1-D film thickness profile measured by ISE and our interferometer is

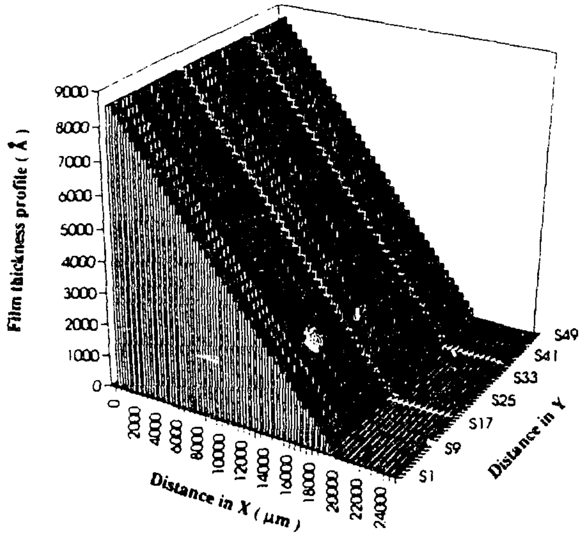

Figure 5. The 2-D film thickness profile of the standard wedge wafer # 1 measured by Image Scanning Ellipsometer.

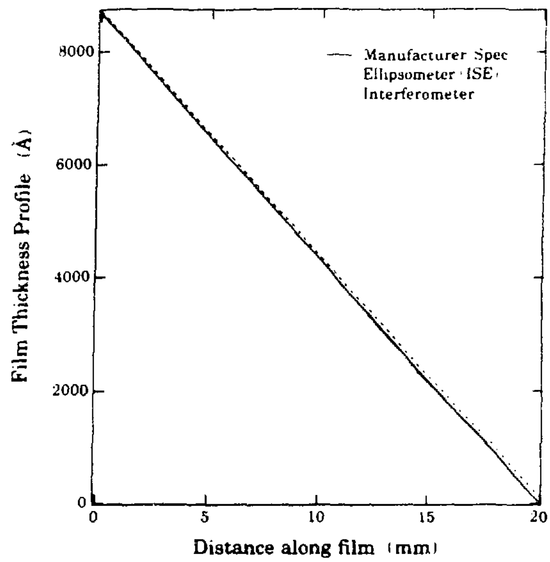

Figure 6. The comparison among different methods for standard wafer # 1, where the measured results from Reynard and the interferometer are overlaid.

presented in Fig. 4. The 2-D film thickness profile measured by ISE after correction is shown in Fig. 5. The regression results from ISE after correction are listed below:

From ISE :

Standard wafer #1: TH (Å) = -0.429(Å/µm) * X(µm) + 8708.7(Å)

Comparison among the three methods, for the standard wafer, is presented in Figs. 6. The percentage differences, assuming that the measurement from the manufacturer is the accurate base, are also listed below:

Standard wafer #1:

Interferometer: Differences in slope = 0.72 %
Differences in intercept = 3.09 %
Accumulated differences in film thickness within 20,000 µm distance = 82 Å

ISE: Differences in slope = 0.69 %
Differences in intercept = 0.55 %
Accumulated differences in film thickness within 20,000 µm distance = 47 Å

Based on the above differences calculated from the regression program, the measured results from the ISE after correction (calibration) due to non-uniform magnification are well within the accuracy of any other techniques used. Two identical standard wafers were purchased and analyzed. The results from the ISE for wafer #2 were nearly identical to the results presented above for wafer #1.

V. CONCLUSIONS

A new experimental technique for measuring film thickness profiles, Image Scanning Ellipsometry, was developed and tested. The technique is based on conventional null Ellipsometry. The experimental results, presented in this paper, established and confirmed the accuracy of full-field imaging for measuring the film thickness profile of a non-uniform film by sweeping the null points across the sample surface. The ISE system has been shown to require an additional correction (calibration) in the measured horizontal distance, which might be due to four factors: (1) the imaging system is not oriented perpendicular to the substrate and so the horizontal magnification varies in this direction, (2) the allocation of the exact position of the dark fringes is limited by the ability to accurately digitize and analyze the images, (3) the sensitivity of the polarizer settings with respect to the film thickness limits the number of null points possible, and (4) the limited magnification of the ISE hinders the exact location of a null point. Factors (2) and (4) limit the location of the null points to $\pm$ 1 pixel width. In this study that is $\pm$ 7.5 μ

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V. REFERENCES

1. Bankoff, S.G., "Dynamics and Stability of Thin Heated Liquid Films," *ASME J. of Heat Transfer*, **112**, 538-546 (1990).
2. Wayner, Jr., P.C., "The Effect of Interfacial Mass Transport on Flow in Thin Liquid Films", *Colloids and Surfaces*, **52**, 71-84 (1991).
3. Tan, M.J., Bankoff, S.G., and Davis, S.H., "Steady Thermocapillary Flows of Thin Liquid Layers, I" *Physics of Fluids A, Fluid Dynamics*, **2**, 313-321, (1990).
4. Burelbach,J.P., Bankoff,S.G., and Davis,S.H., "Steady Thermocapillary Flows of Thin Liquid Layers, II" *Physics of Fluids A, Fluid Dynamics*, **2**, 322-333, (1990).
5. S. DasGupta, J. Schonberg, and P. C. Wayner, Jr., "Investigation of an Evaporating Extended Meniscus Based on the Augmented Young-Laplace Equation", J. of Heat Transfer, Vol. 115, 201-208(1993).
6. R. F. Cohn, J. W. Wagner, and J. Kruger, "Dynamic Imaging Microellipsometry: Theory, System, Design, and Feasibility Demonstration", Applied Optics, Vol. 27, **22**, 4644-4671 (15 November, 1988).