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MICROCOMPUTER ENHANCED OPTICAL INVESTIGATION OF SPREADING AND EVAPORATIVE PROCESSES IN ULTRA THIN FILMS

Progress Report

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MASTER

NOTICE

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ABSTRACT

The general objective of the research is to determine the heat transfer characteristics of evaporating ultra-thin films. The immediate objective is to develop a microscopic image-processing system (IPS to measure the film thickness profile. The IPS has two parts: an image analyzing interferometer (IAI) and an image analyzing ellipsometer (IAE).

An IAI was designed and used to measure the thickness profiles of an extended meniscus in the contact line region of completely wetting liquid films. Both steady state and oscillating menisci were studied. We find that the convenience and efficiency of microcomputer enhanced video microscopy naturally leads to a better understanding of the transport processes in the contact line region. We also find that the processes of change-of-phase heat transfer and fluid flow in thin films are intrinsically connected because of their common dependence on the intermolecular force field.

The design of an image analyzing ellipsometer (IAE) was completed and the system was successfully tested by measuring the thickness and refractive index profiles of various solid films of known thicknesses. The profiles of draining isothermal films will be measured next.

REVIEW OF PROGRESS

The IPS has two parts: an image analyzing interferometer (IAI) and an image analyzing ellipsometer (IAE). The updated design of the combined IAI-IAE was presented in DOE/ER/14045-2. The design and use of the IAI was discussed in the numerous publications listed below. The use of the IAE to measure the thickness and refractive index profiles of various solids is discussed herein.

The IAE is a rapid full-field imaging technique developed for high spatial resolution that will enable measurement of the changes in the thin liquid film thickness profile associated with fluid flow, evaporation and drying. Conventional ellipsometry is limited to single detector operation which requires slow scanning of the sample to create two-dimensional images of the liquid film. However, greatly increased speed and resolution are required to study changes in the liquid film profile in the transient state.

The IAE described in our initial report (DOE/ER/14045-1), which was designed for proof of principle testing, was configured using an available null ellipsometer modified with laboratory optical equipment. However, the images were small and insufficient for adequate analysis. Therefore, we added an autocollimator to obtain accurate alignment and a long working distance microscope to improve resolution and magnification. Using a computer control system for the analyzer so that precise intensity measurements at three selected analyzer angles can be obtained for analysis, we have obtained a quantitative proof of principle. The new system was described in DOE/ER/14045-2.

To utilize multiple simultaneous detectors (each pixel) a Polarizer, Compensator, Specimen, and Analyzer (PCSA) ellipsometer arrangement is used in an intensity (radiometric) measurement mode rather than in a conventional null measurement mode. Eq.(1) gives the intensity of the light leaving the analyzer:

$$
\begin{aligned}
I(A) = (G^2/2) \{ & [\ 1 + (\cos 2C)(\cos 2(P-C))\ ] \cos^2 A \tan^2\psi \\
& + [\ 1 - (\cos 2C)(\cos 2(P-C)) \sin^2 A] \\
& + [\ \sin 2C\ (\cos 2(P-C)) \cos\Delta - \sin 2(P-C) \sin\Delta] \sin 2A \tan\psi
\end{aligned}
\qquad (1)
$$

When the polarizer angle, P, and the compensator angle, C, are fixed , the ellipsometric parameters Δ and ψ can be evaluated using only intensities recorded at known analyzer angles, A. Since the analyzer settings are independent of the film parameters, measurements may be made using multiple detectors, thereby obtaining full-field imaging. A minimum of three precise intensity measurements at the same film field location are required to define the three unknowns in Eq. (1) at selected analyzer angles: G (the system gain); Δ; and ψ. Therefore, the computer controlled stepping motor is needed. Multiple field locations are obtained using the pixel array in a video camera. The image processing system automates data analysis.

The conventional null ellipsometer can also measure refractive index and film thickness with good accuracy, but the result is an average value over the whole area, i.e. the whole light spot which could be a relatively large area. This is not sufficiently accurate for certain applications such as research in evaporating thin films, monitoring IC fabrications and optical waveguide manufacturing. These applications need good resolution in a micro-region or even in a submicro-region. Furthermore, since it takes several minutes to measure the refractive index or film thickness by null ellipsometry, the instrument cannot be used in real time or in a transient state study. Alternatively, IAE is an instrument that combines the conventional ellipsometer with a high resolution camera, image processing system, and high speed computer. Due to the use of a high resolution camera with pixel number of 512*480, IAE can be used to measure the refractive index and the film thickness within a micro-region. This is because each pixel on the camera functions like a photodetector in the null ellipsometer, so each pixel can acquire the ellipticity of the film which is used to calculate refractive index and film thickness. Due to the real time characteristic of the image processing system, IAE can be used in a transient state study during the formation of thin films, solid or liquid.

Using the design presented in DOE/ER/1405-2, we have successfully measured the refractive index profile of a Si substrate and the film thickness profile of SiO_2 on a Si substrate. Initially, we note that the presence of dust particles, non-uniform optical filters and other non-uniformities caused a variation in the results obtained by each pixel. However, the absolute value of the variation remained constant with a variation in oxide thickness. We feel that these variations can be removed and the system optimized with improved components. Therefore, we believe that the basic procedures have been demonstrated. The IAE results presented in Figures (3-9) can be compared with the average results obtained using a null ellipsometer which are presented in Figures 1 and 2. In Figure 3, the refractive index of the Si substrate obtained using the

Image Analyzing Ellipsometer is presented. The values are as good as those obtained with the null ellipsometer but with higher resolution: 6 micrometers in vertical and 23 micrometers in horizontal directions. In Figure 4 the measured film thickness profile for native SiO_2 on a bare Si substrate is presented. It ranges from 20 to 30 Å. The variation might be due to the porous structure of SiO_2. This needs to be checked with another instrument and improvements in our IAE. In Figure 5 the measured film thickness profile of an 85 Å film of SiO_2 on a Si substrate measured using the Image Analyzing Ellipsometer is presented. In Figure 6 the measured film thickness profile of an 140 Å film of SiO_2 on a Si-substrate is presented. In Figure 7 the measured film thickness profile of an 195 Å film of SiO_2 on a Si substrate is presented. In Figure 8 the measured film thickness profile of a 2250 Å film of SiO_2 on a Si substrate is presented. In Figure 9 the measured thickness profile of a **non-uniform** film of SiO_2 varying from 2340 Å to 2460 Å on a Si substrate is presented whereas the conventional null ellipsometer result of a uniform thickness profile of an average value of 2400 Å is presented in Figure 10. The average value for the non-uniform film is far from accurate.

Even though we can currently measure the refractive index and film thickness profiles, many improvements are still needed. First we should be able to obtain resolution in a sub-micro region with a new low light level CCD camera and a better light source of higher intensity. We also note from the figures that there is a decrease in the variation in the measured profile with an increase in the film thickness. This is because the adsorbed dust particles from the air have a larger relative effect on thinner films. Therefore, the second improvement we plan to focus on is to reduce the effects of dust particles during the measurement. Imperfect prisms will also have a large impact on the accuracy of the measurements because the IAE is based on intensity changes. Therefore, the quality of the prisms will be increased. After these improvements we will begin the transient state study of the liquid film profile in the real time scale. This will be a much more difficult study because liquid is more apt to be contaminated and more sensitive to dust particles on the surface of the substrate. However we are strongly convinced that we will be able to accomplish the proposed transient state study of thin liquid films.

Until we find other references to the contrary, we believe that our Image Analyzing Ellipsometer is the first setup in the world to offer the possibility of measuring the complete refractive index and film thickness profiles within the resolution of a micro-region. Therefore, there are many potential applications for this Image Analyzing Ellipsometer. In summary: although much additional work needs to be done, we conclude that we have an operating IAE.

LIST OF PUBLICATIONS/PRESENTATIONS/DOE REPORTS

PUBLICATIONS

P1) "Microcomputer Enhanced Optical Investigation of Transport Processes in Thin Liquid Films", M. Sujanani and P.C. Wayner, Jr., pp. 79-86, DOE CONF-9005183, Proceedings of Eighth Symposium on Energy Engineering Sciences, Argonne National Laboratory, Argonne, IL, May 9-11, 1990.

P2) "Interfacial Phenomena in an Evaporating Thin Film", M. Sujanani, *Ph.D. Thesis*, Rensselaer Polytechnic Institute, Troy, NY, 1990. Abstract in Appendix A of DOE/ER/14045-2.

P3) "Microcomputer Enhanced Optical Investigation of Transport Processes with Phase Change in Near-Equilibrium Thin Liquid Films", M. Sujanani and P.C. Wayner,Jr., *J. Colloid and Interface Science*,**143**, 472-487,1991. (DOE/ER/1045-3)

P4a) "Transport Processes and Interfacial Phenomena in an Evaporating Meniscus", M. Sujanani and P.C.Wayner,Jr., paper preprint AIAA 91-4002 presented at 1991 *ASME/AIChE/AIAA National Heat Transfer Conference*, Minneapolis, MN. (sent to DOE as manuscript)

b) "Transport Processes and Interfacial Phenomena in an Evaporating Meniscus" , M Sujanani and P. C. Wayner,Jr. rewritten version of P4a, accepted for publication by *Chemical Engineering Communications*.

P5a) "Evaporation and Stress in a Small Constrained System, P.C. Wayner, Jr., Extended Abstract, pp. 269-272, in Paper Summaries, *IS&T's 44th Annual Conference*, May 12-17, 1991, St. Paul, MN, DOE/ER/14045-4.

b) P5a will be published as part of a book of selected articles from the *IS&T's 44th Annual Conference*.

P6) "Spreading of a Liquid Film on a Substrate by the Evaporation-Adsorption Process" Submitted for publication to *J. Colloid and Interface Science*. The initial reviews were positive and the suggested modifications are being made. (sent to DOE as a manuscript)

PRESENTATIONS

PR1) "Fluid Flow in Ultra Thin Films" M. Sujanani and P.C.Wayner,Jr., Presentation 167C-b, 1989 Annual Meeting of A.I.Ch.E., Nov. 5-10, San Francisco, CA.

PR2) P1 above

PR3) P4a above

PR4) P5a above

DOE REPORTS

DOE/ER/14045-1) "Microcomputer Enhanced Optical Investigation of Spreading and Evaporative Processes in Ultra Thin Films", P.C. Wayner, Jr., M. Sujanani and A.H. Liu, DOE Progress Report DOE/ER/14045-1, January 1990.

DOE/ER/14045-2) "Microcomputer Enhanced Optical Investigation of Spreading and Evaporative Processes in Ultra Thin Films", P.C. Wayner, Jr., M. Sujanani and A.H. Liu, DOE Progress Report DOE/ER/14045-2, December 1990.

DOE/ER/14045-3) P3 above

DOE/ER/14045-4) P5a & P5b above

DOE/ER/14045-5) "Microcomputer Enhanced Optical Investigation of Spreading and Evaporative Processes in Ultra Thin Films", P.C. Wayner, Jr. and A.H. Liu, DOE Progress Report DOE/ER/14045-5, December 1991.

BUDGET INFORMATION

We do not anticipate an unexpended balance at the end of the current funding period. The principal investigator charged the following time to the contract (this is less than the actual time spent): Summer 1989 (1 month); 1989-90 academic year (9%); summer 1990 (1 month); 1990-91 academic year(10%); summer 1991 (100 hrs.). Although the PI has been working on the contract during the initial part of the 1991-92 academic year, he has not charged release time to the contract because of insufficient funds. He has used these funds for graduate student support.

Figure 1. Average refractive index profile of bare Si substrate measued by NULL Ellipsometer (Area= 90 mm**2)

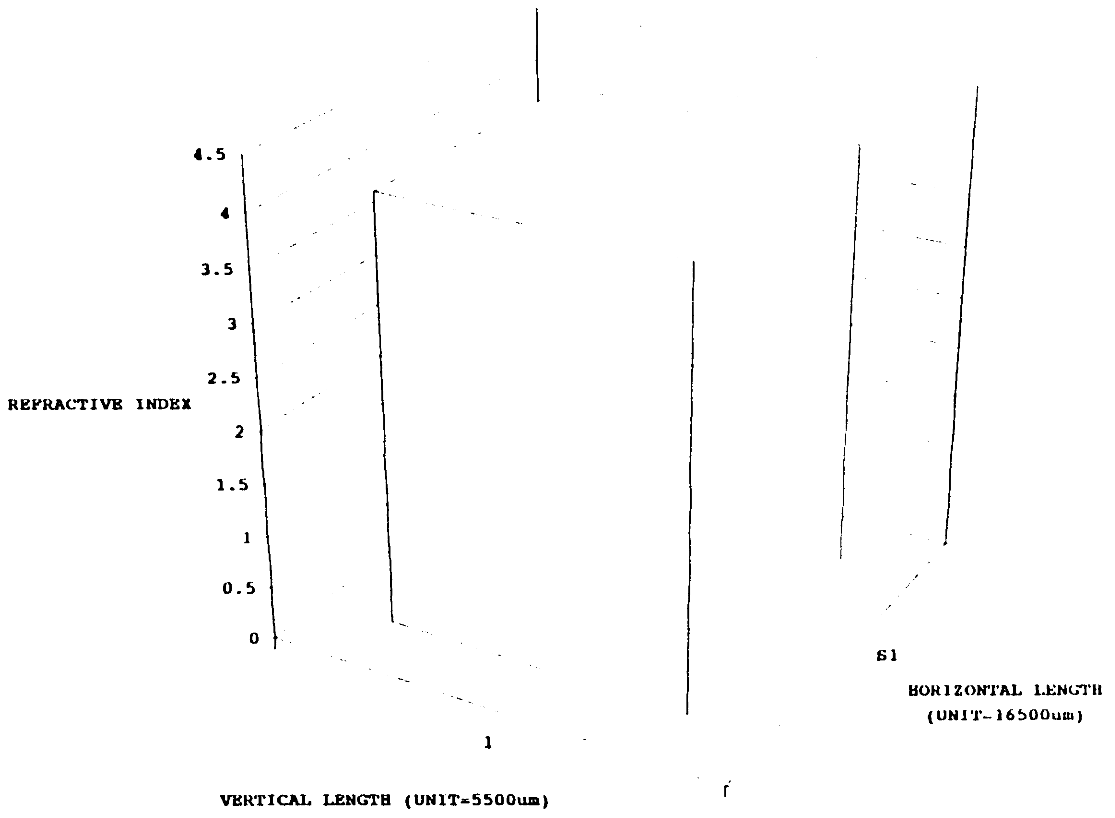


Figure 2. Average film thickness profile of bare Si substrate measured by NULL Ellipsometer (Area= 90 mm**2)

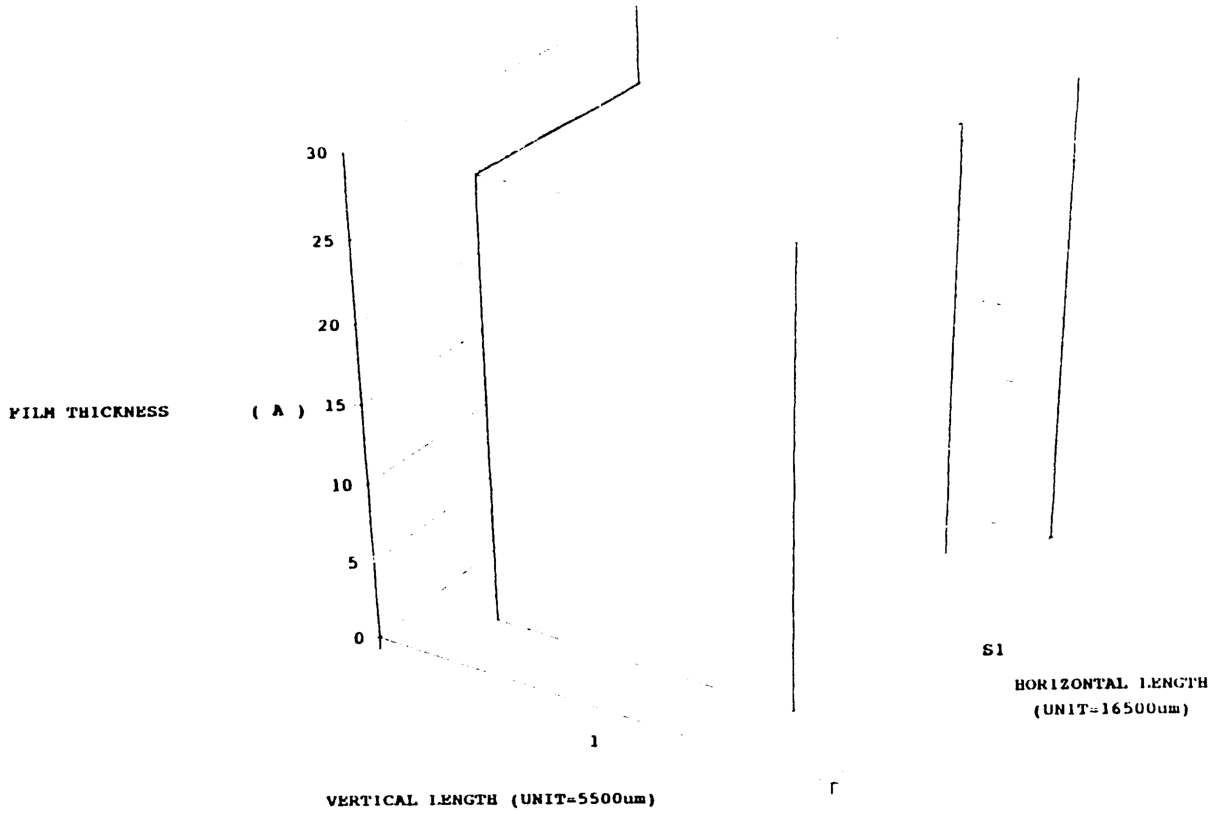


Figure 3. Refractive index profile of bare Si substrate measured by Image Analyzing Ellipsometer (Pixel dimensions = 6um*23um)

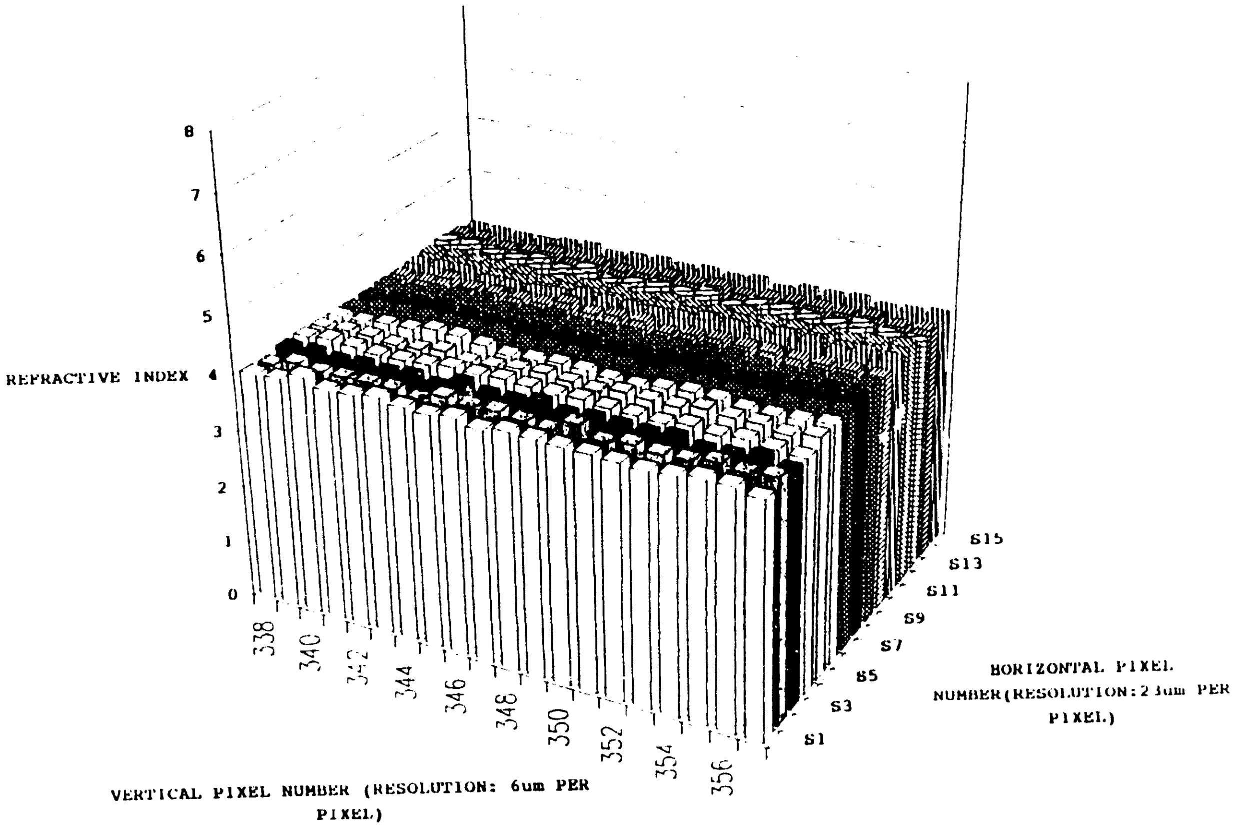


Figure 4. Film thickness profile of SiO2 on bare Si substrata measured by Image Analyzing Ellipsometer (Pixel dimensions=6um*23um)

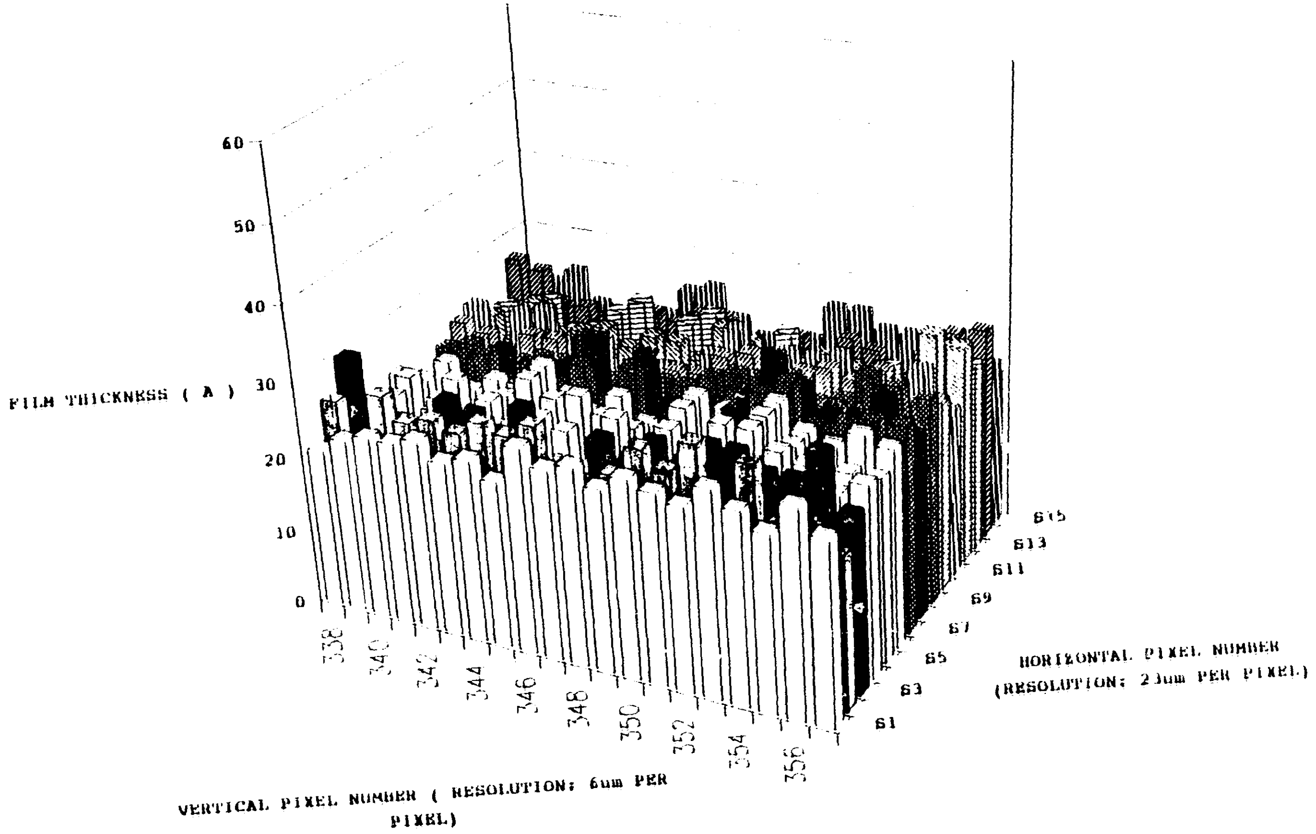


Figure 5. Film thickness profile of 85A SiO2 on Si substrate measured by Image Analyzing Ellipsometer (Pixel dimensions=6um*23um)

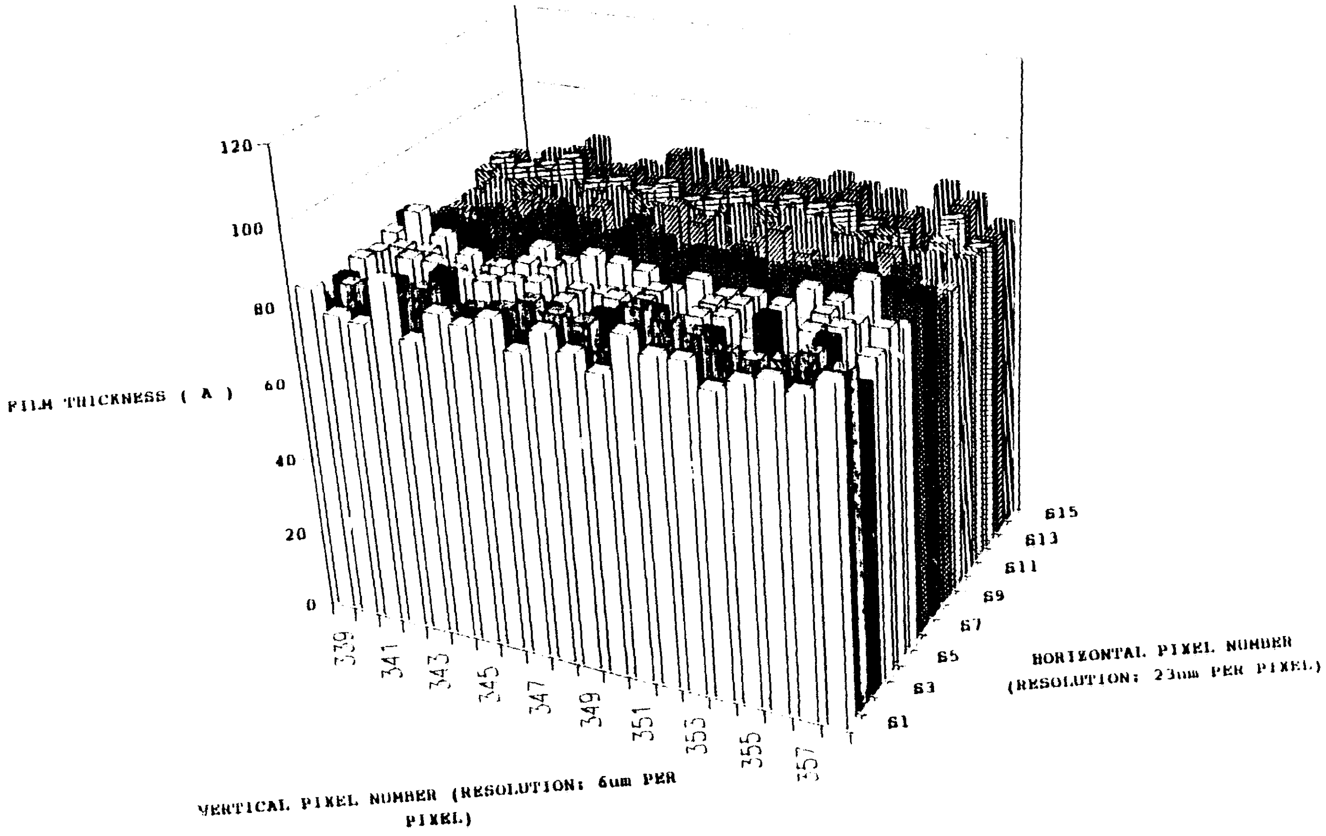


Figure 6. Film thickness profile of 140A SiO2 on Si substrate measured by Image Analyzing Ellipsometer (Pixel dimensions=6um*23um)

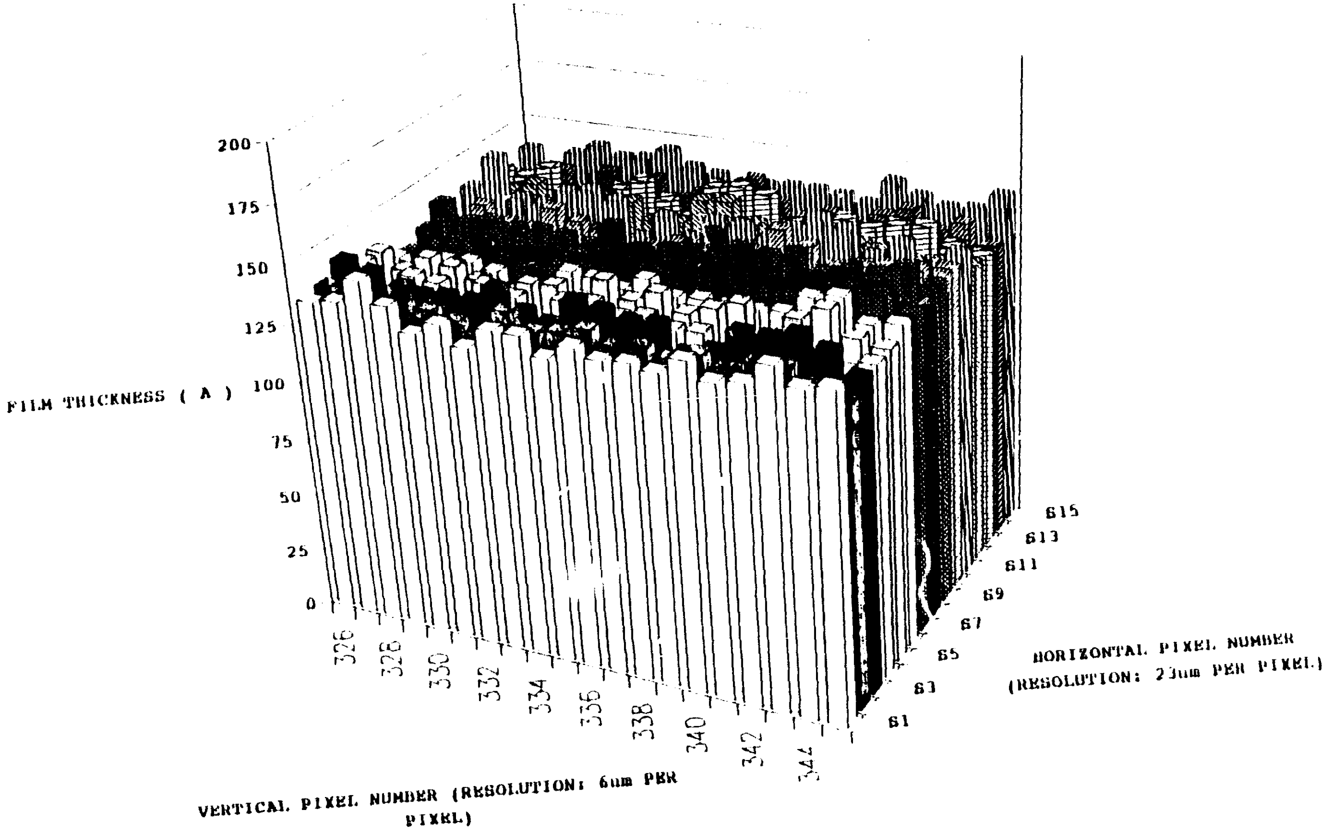


Figure 7. Film thickness profile of 195A SiO2 on Si substrate measured by Image Analyzing Ellipsometer (Pixel dimensions=6um*23um)

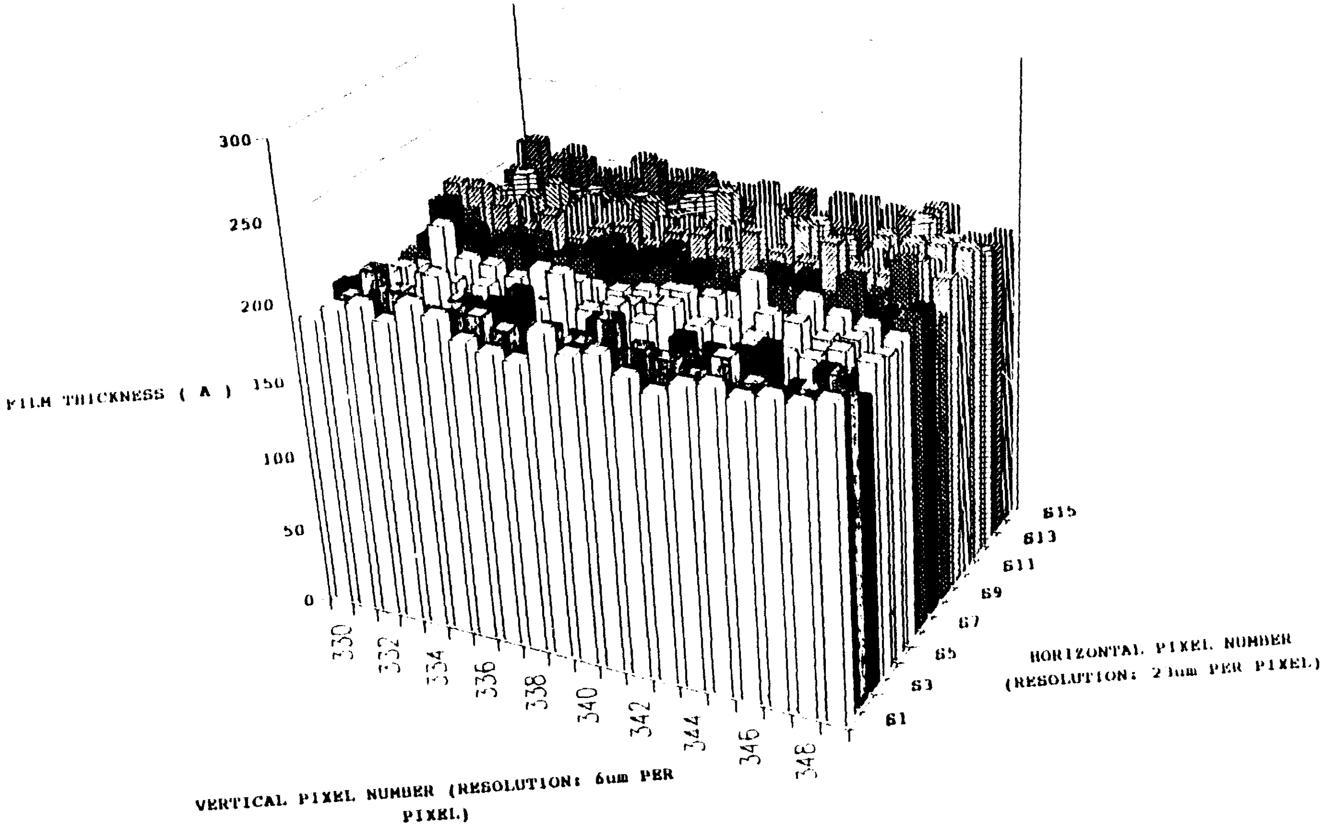


Figure 8. Film thickness profile of 2250A SiO2 on Si substrate measured by Image Analyzing Ellipsometer (Pixel dimensions=6um*23um)

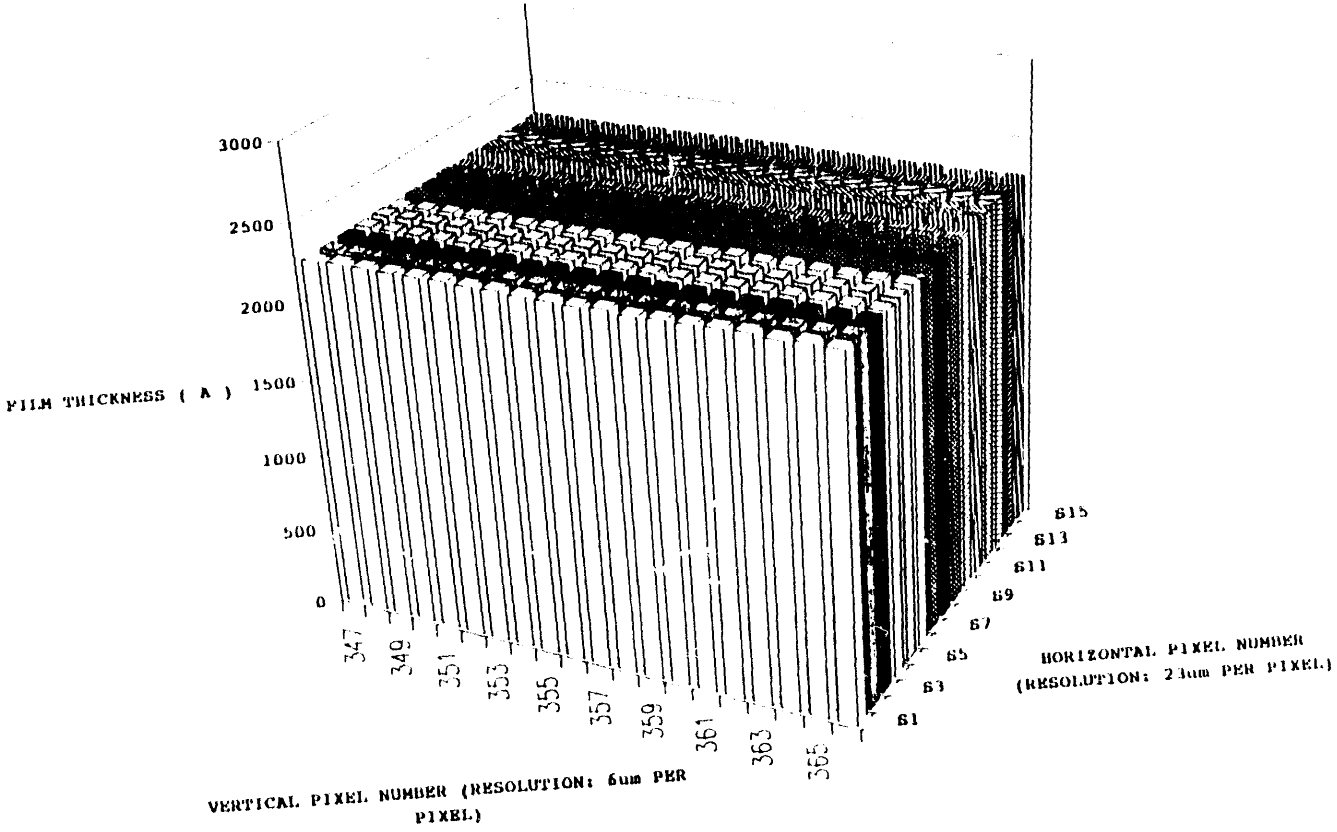


Figure 9. NON-UNIFORM film thickness profile of SiO2 on Si substrate measured by Image Analyzing Ellipsometer (Pixel dimensions=6um*23um)

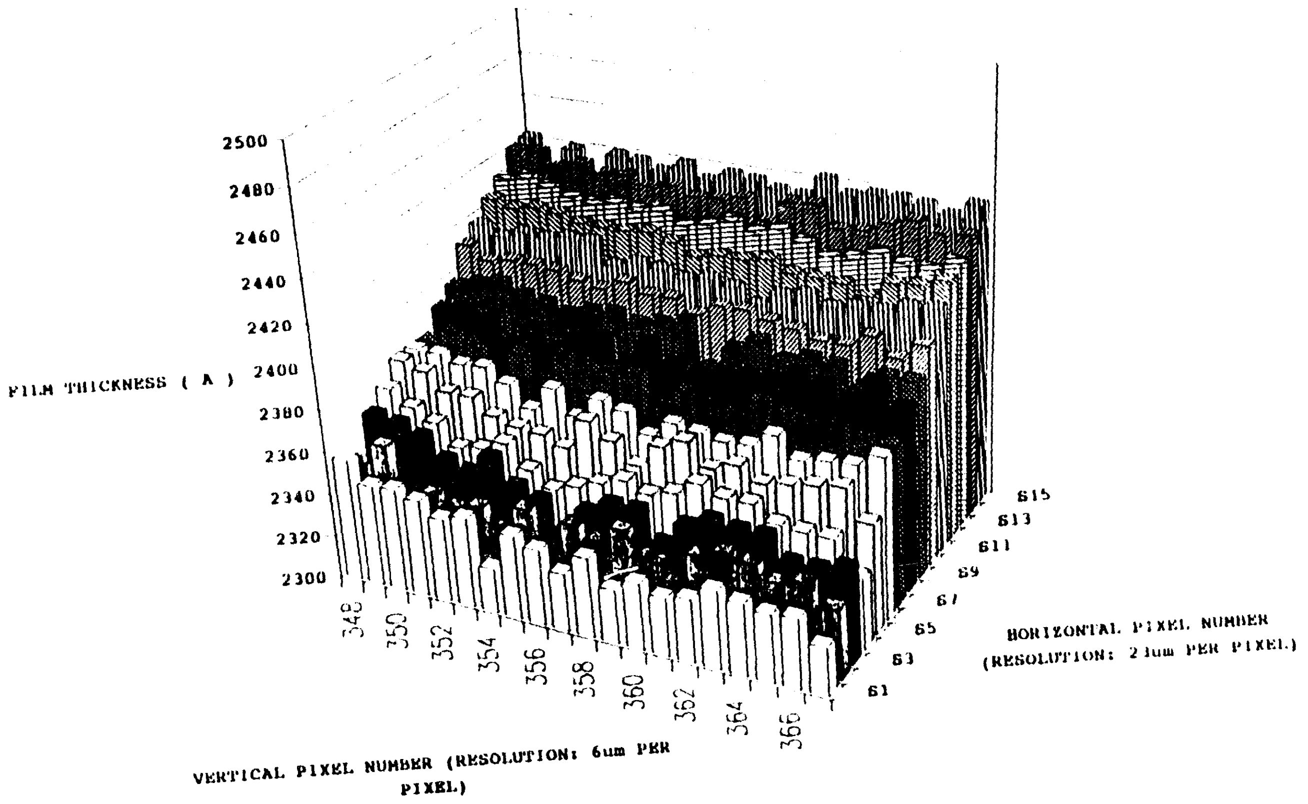
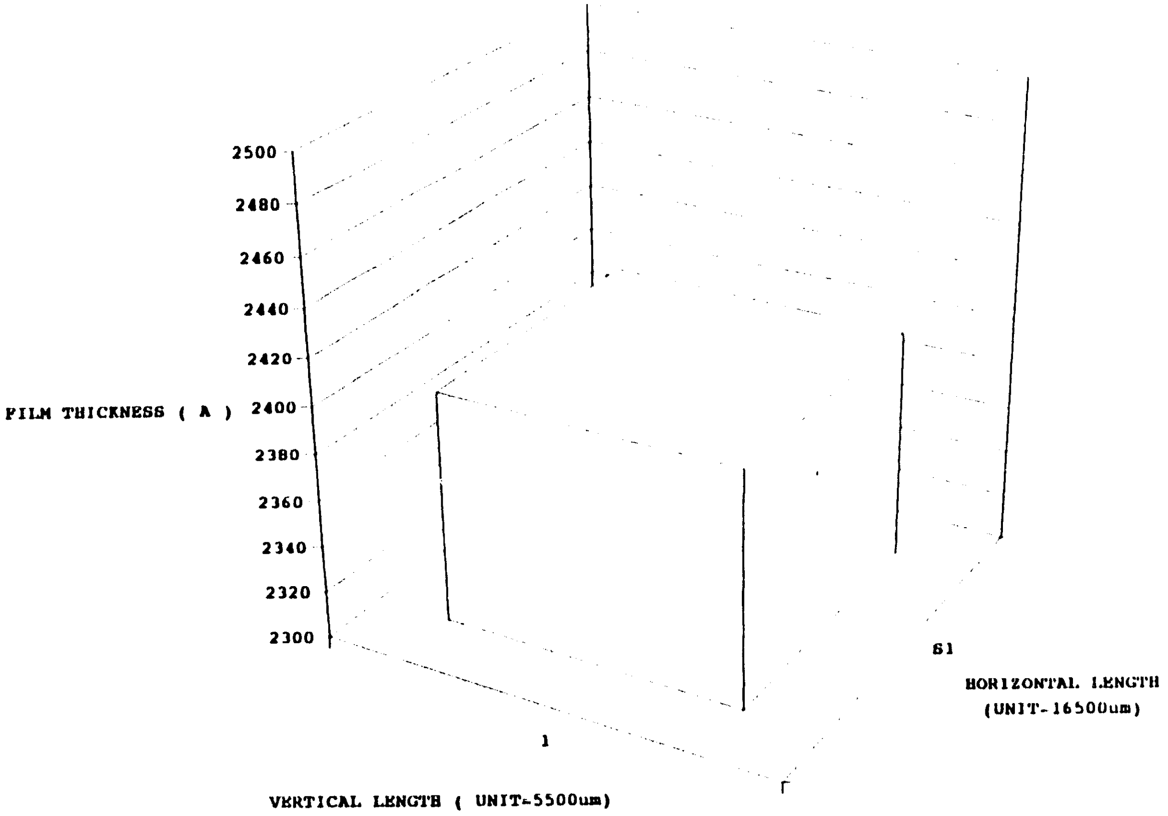


Figure 10. NON-UNIFORM film thickness profile of SiO2 on Si substrate measured by NULL Ellipsometer (Area= 90 mm**2)



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