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UNE TECHNIQUE SIMPLE POUR MESURER L'ÉPAISSEUR ET L'INDICE DE RÉFRACTION DE COUCHES TRANSPARENTES SANS LES ALTÉRER

Par W. A. PLISKIN et E. E. CONRAD,

Résumé. — On expose une méthode non destructive et simple pour mesurer l'indice de réfraction et l'épaisseur de couches transparentes sur des supports réfléchissants. La technique nécessite l'emploi d'un microscope équipé d'un filtre monochromatique et d'un ensemble qui peut tourner de telle sorte que la lumière réfléchie soit observée suivant des angles différents. On peut utiliser cette technique dans la détermination de l'épaisseur de couches de quelques centaines d'angströms à plusieurs microns avec des précisions de 0,2 % par les épaisseurs supérieures à 2 μ et d'une dizaine d'angströms pour les plus faibles.

Abstract. — A simple non-destructive method of measuring the refractive index and thickness of transparent films on reflective substrates has been developed. The technique involves the use of a microscope equipped with a monochromatic filter and a stage that can be rotated so that the reflected light is observed at various angles. The technique can be used for the determination of film thicknesses from several hundred angstroms up to several microns with accuracies of 0.2 % on films thicker than 2 μ , and tens of angstroms on thinner films.

1. Introduction and theory. — A relatively easy, non-destructive method of measuring transparent film thicknesses and refractive indices without the use of a step has been developed. This is accomplished by using a rotatable stage for the film and substrate so that reflected light is observed at various angles. Only reflected monochromatic light is observed by either covering the microscope objective with a monochromatic filter or by illuminating the film with monochromatic light. The former method has been found to be very convenient. A schematic sketch of the essential portions of the apparatus are shown in figure 1; it is referred to as Vamfo (Variable Angle Monochromatic Fringe Observation).

As the stage and sample are rotated, one observes maxima (bright) and minima (dark) fringes on the sample film. During stage rotation the mirror is hand rotated and positioned to maintain the proper reflected light on the sample. With a vertically illuminated microscope, a fixed mirror mounted perpendicular to the rotating stage can be used if it is close to the point on the substrate under examination. The angular positions (angle of incidence or reflection, i) of the sample associated with the observed maxima and minima are read off a calibrated dial attached to the shaft of the rotating stage. The film thickness, d , is given by

$$d = \frac{\Delta N \lambda}{2\mu (\cos r_2 - \cos r_1)} = \frac{\lambda}{2\mu (\Delta \cos r)} \quad (1)$$

where

λ is the wavelength of filtered light,
 μ is the refractive index of the film,
 r_1 is the angle of refraction at that fringe for which the angle of incidence is i_1 and

$$\sin r_1 = (\sin i_1) / \mu,$$

ΔN = number of fringes observed between i_1 and i_2 ,

$\Delta \cos r = \frac{\cos r_2 - \cos r_1}{\Delta N}$ averaged for both maxima and minima.

This formula is derived from the following considerations. Assuming that the phase change on reflection at the air-film and film-substrate interfaces are the same ⁽¹⁾, then for maxima :

$$N_1 \lambda = 2\mu d \cos r_1 \quad (2)$$

$$N_2 \lambda = 2\mu d \cos r_2 \quad (3)$$

$$\Delta N = N_2 - N_1.$$

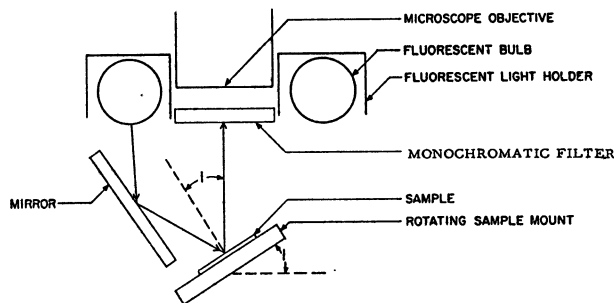


FIG. 1. — Schematic sketch of the essential portions of Vamfo. The rotating stage was attached to an x-y stage so that the reference point under examination (generally at 10X) can be maintained in the center of the field of view. An American Optical microscope model 56-M3 was used. The filter is a Bausch & Lomb series 33-79-XX.

⁽¹⁾ This is a reasonable assumption of transparent films on high refractive index materials such as silicon. From the optical constants of silicon (DASH (W. C.) and NEWMAN (R.), *Phys. Rev.*, 1955, **99**, 1151) a phase change of 179.6° was calculated for a SiO₂ — silicon interface (non-degenerate silicon).

Solving the above equations for d , we obtain eq. (1).

A similar set of equations, with N_1 and N_2 as half integers, is obtained for the minima.

Values of $\cos r$ as a function of the angle of incidence have been tabulated for refractive indices from 1.05 to 2.05 in increments of 0.05. For interpolation purposes values of $\delta \cos r$, where $\delta \cos r = (\cos r)_\mu - (\cos r)_{\mu-0.05}$, have also been tabulated. Thus $\cos r$ can be determined for any refractive index up to 2.05.

II. Experimental results. — There are various detailed techniques which can be used with Vamfo for determining film thicknesses. The best technique for a particular film depends on the film thickness and the accuracy desired. A few of these techniques will be described briefly.

1. **THICK FILMS.** — Excellent agreement between film thicknesses determined by eq. (1) and by the method of counting fringes at a step of the transparent film has been obtained. Some typical results are given in Table I.

TABLE I

COMPARISON OF FILM THICKNESSES DETERMINED
[BY COUNTING FRINGES AT A STEP ⁽²⁾ AND BY VAMFO

FILM SUBSTANCE ON SILICON	REFRACTIVE INDEX 5 460 Å	METHOD	d (MICRONS)
Pyrex ⁽³⁾ (Corning 7 740)	1.476	Vamfo	3.20
		Step	3.18
Thermal oxide (stem fol- lowed by dry O ₂ grown at 975 °C)	1.46 ₃	Vamfo	1.97
		Step	1.96
725 °C pyrolytic SiO ₂	1.46 ₃	Vamfo	4.08
		Step	3.92
	1.43 ₇	Vamfo	3.99
		Step	3.99

Note that with the pyrolytic oxide, there was a 4 % difference between the two determined thicknesses on the assumption that the refractive index

⁽²⁾ The fringes at a step were determined with vertical illumination using a Bausch and Lomb metallurgical microscope model DMET equipped with a 5 460 Å monochromatic filter. Under such conditions the thickness can be determined to about 0.1 fringe accuracy or about 0.02 μ .

⁽³⁾ The glass films discussed in this report were formed by special sedimentation techniques. (Unpublished results of W. A. Pliskin and E. E. Conrad. See also J. A. Perri, H. S. Lehman, W. A. Pliskin and J. Riseman, "Surface Protection of Silicon Devices with Glass Films" presented to the Electrochemical Society Semiconductor Symposium, Detroit Michigan, October 2, 1961 and J. L. Langdon, W. E. Mutter, R. P. Pecoraro, K. K. Schuegraf, "Hermetically Sealed Silicon Chip Diodes and Transistors" presented to the 1961 Electron Devices Meeting, Washington, D. C., October 27, 1961.)

is the same as that for the thermal oxide, but if the refractive index were assumed to be 1.43₇, then the two determination agree. Thus, with the combination of the two methods, it is possible to determine an approximate refractive index of the film.

The thickness measurement determined by counting fringes at a step is less accurate than that determined by Vamfo and a more accurate refractive index measurement can be made by further refinement of the technique. Substituting eq. (1) into eq. (2) or (3) or into the similar set for minima, one obtains

$$N' = \cos r_i / \Delta \cos r. \quad (4)$$

The apparent fringe order N_i should be integral for maxima and half integral for minima on the assumption of π phase changes at the interfaces. An approximate N_0 ($i = 0$) can be determined by counting fringes at a step. The refractive index is then determined by the value which would give agreement between the N_i set and N_0 . This technique is capable of determining refractive indices within 0.2 % accuracy, depending on the film thickness and sharpness of the fringe pattern.

Making a step is not always necessary. If the film is not too thick and an approximate index is available from Table I for a similar type film or by some prior knowledge of the composition of the film, then the proper N_i set is given by the closest integral and half-integral values to those calculated from eq. (4). The refractive index is then determined by that value, which, when used in the calculations, given the proper integral and half-integral values of N_i .

By this technique the refractive index of Corning 7 740 glass at 5 450 Å was found to be 1.476. The refractive index of this glass is given as 1.474 at 5 890 Å by Corning and due to dispersion a refractive index of 1.476 at 5 450 Å is to be expected.

Another glass whose refractive index is known and which has been examined in detail is Corning 3 320 Uranium glass. By the above techniques the refractive index of a film on silicon was found to be 1.481. The given refractive index for this glass at 5 890 Å is 1.481 and thus at 5 450 the index should be 1.483 in excellent agreement with the experimentally determined index. The excellent agreement of the refractive indices determined by Vamfo with the known or expected indices is verification of the accuracy obtainable by this technique.

The technique has been used for determining film thicknesses and refractive indices of glasses with refractive indices as high as 1.91. It has also been used in the study of various pyrolytic and thermally grown silicon dioxide films ⁽⁴⁾.

In the determination of the maxima and minima, it should be noted that the minima points are

⁽⁴⁾ Unpublished work W. A. Pliskin and H. S. Lehman.

generally more accurate than the maxima. Greater accuracy, especially at angles that exceed Brewster's angle for the film, can be obtained by using a polarizing filter in conjunction with the monochromatic filter such that only the senkrecht component (the vibration perpendicular to the plane of incidence) is observed. For all practical purposes, the phase difference between the senkrecht components reflected from the air-film surface and the film-silicon surface is zero independent of the angle of incidence. However, for the parallel component, the phase difference is zero at angles of incidence less than Brewster's angle and is 180° at angles of incidence greater than Brewster's angle; thus without the polarizer, the fringe system gets washed out at large angles of incidence.

2. THIN FILM THICKNESS DETERMINATION. —

a. *Technique of multiple monochromatic filters.* — The technique using only one monochromatic filter is inaccurate for films less than one micron thick. The reason is that the maxima and minima are rather broad and $\Delta N < 1$. In such cases, greater accuracy can be obtained by finding the maxima and minima associated with more than one wavelength. For example, curves for maxima and minima such as shown in figure 2 can be calculated for three or more significantly different wavelengths and for thicknesses in the neighborhood of one micron or less. The traces may all be plotted on one chart using specific colors for differentiation. Then by proper identification of maxima or minima for each wavelength, the thickness can be determined.

An example of a chart useful for silicon dioxide is shown in figure 2; the curves for the maxima and minima positions are drawn for filters 4 340 Å (V for violet), 5 190 Å (BG for blue-green), and 5 450 Å (G for green) with refractive indices for SiO_2 of 1.470, 1.464, and 1.463, respectively. Since in actual practice it is found that the minima positions can be determined more accurately than the maxima, it is advisable to include a filter in the yellow region so at least one minima will be observed for any thickness greater than 2 250 Å. The chart which we have used with considerable success in actual practice includes the maxima and minima curves for the aforementioned filters together with the minima curves for a blue filter (4 850 Å), a yellow filter (5 830 Å), and a red filter (6 230 Å). This degree of redundancy is not always necessary physically, but is useful as a check on the accuracy of the film thickness determinations.

The positions of maxima and minima at a specific point on a film can be determined very accurately with films of non-uniform thickness. On non-uniform films a fringe pattern can be seen and

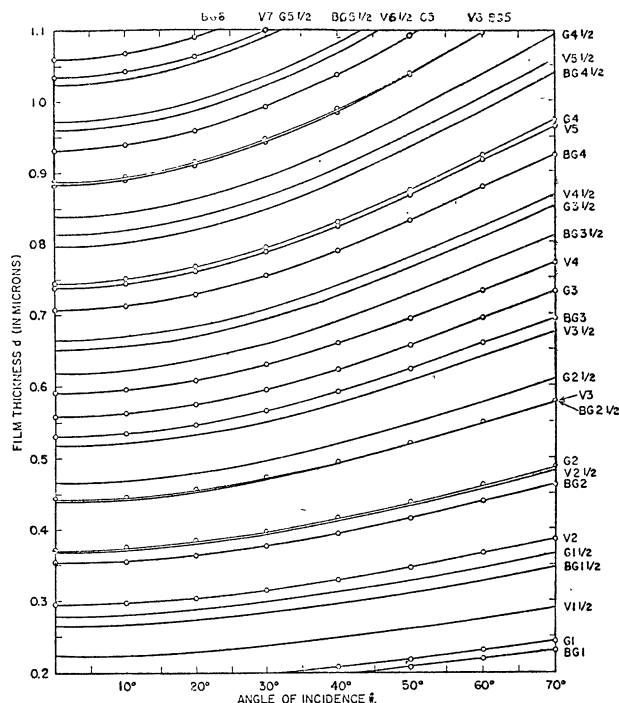


FIG. 2. — Positions of interference maxima (—○—) and minima (—□—) for a silicon dioxide film on silicon with filters 4 340 Å (V, $\mu = 1.470$), 5 190 Å (BG, $\mu = 1.464$) and 5 450 Å (G, $\mu = 1.463$).

the maxima and minima positions at the point in question are easily determined by comparison with the remainder of the film. On perfectly uniform films the problem of increased reflectivity with increased angle of incidence is a complicating factor especially with thinner films.

For film thickness accuracies within 20 Å for uniform films less than 5 000 Å thick, it is often advisable (when good minima can not be observed) to purposely remove by etching a known small amount (~ 100 Å) of the film by use of a controlled dilute etch on a small section of the film. The thickness difference of 100 Å is easily observable in Vamfo except at the maxima and minima positions corresponding to the average thickness between the etched and unetched portions of the film. By adding 50 Å to the determined average thickness an accurate film thickness is obtained for the unetched film.

A comparison of the thicknesses determined in this manner with those determined by Tolansky interferometric techniques and by Talysurf measurements is shown in Table II.

In conclusion the data show that the Vamfo technique can be used to obtain accurate thickness measurements of transparent films on reflective substrates. It can also be used for determining the refractive index of such films providing they have sufficient thickness.

TABLE II
COMPARISON OF THICKNESS MEASUREMENTS OF SiO_2
FILMS ON SILICON

SAMPLE	VAMFO	INTERFEROMETRIC (TOLANSKY)	TALYSURF
A	0.387 μ	0.389 μ	0.382 μ
B	0.573 μ	—	0.564 μ
C	1.932 μ	1.936 μ	1.948 μ

Discussion

M. HEAVENS. — How is the method affected by variation of refractive index of the film in a direction normal to the film? One would not generally expect films produced by the steam/oxygen treatment to be homogeneous.

Réponse: In effect this technique will give a weighted average of the refractive index of the film. A comparison of etch rates and the good agreement in thickness we obtain with those determined by Tolansky interferometer techniques indicates that there is no significant variation in the film properties for films thicker than 3 800 Å. Below about 1 500 Å we have observed slight differences in the infrared spectra and the apparent etch rate of the films. These effects can be attributed to a slight change in the structure of the film.

M. MAYER. — How did you prepare your films?

Réponse: The thick thermally grown SiO_2 films were formed by subjecting a high resistivity silicon wafer to eight cycles of two hours steam followed

by one hour of dry oxygen at atmospheric pressure and 970 °C. The glass films were prepared by special sedimentation techniques, the details of which will be presented by Pliskin and Conrad at the 1963 meeting of the Electrochemical Society in New York.

M. VASICEK. — According to your opinion is your method more convenient than the ellipsometric method or not?

Réponse: The Vamfo technique is more convenient than the ellipsometric technique for the measurement of film thicknesses greater than $\lambda/4$. It is also more convenient for the measurement of refractive index for films greater than 1.5 micron thick. The technique is simpler and cheaper to set up than the ellipsometer method and is more practical for wide use in the laboratory and in productive facilities.

M. HEINBERGER. — The minimum optical thickness which may be determined by this method is $\lambda/4$. In his reply the speaker noted that for accuracy in determining the angle at minimum reflectivity the optical thickness must be somewhat greater than $\lambda/4$.

Réponse: The minimum thickness that can be measured by this technique is slightly greater than $\lambda/4\mu$. At $i = 0^\circ$ a minimum fringe should occur at $\lambda/4\mu$ but in order to be certain that one passes through a minimum, it is necessary to have $i > 0^\circ$ (actually greater than α where α is half the angle subtended by the stereo microscope used) and therefore since $d = \lambda/4\mu \cos r$ for $N = 1/2$, $d > \lambda/4\mu$.