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# Nondestructive Determination of Thickness and Refractive Index of Transparent Films

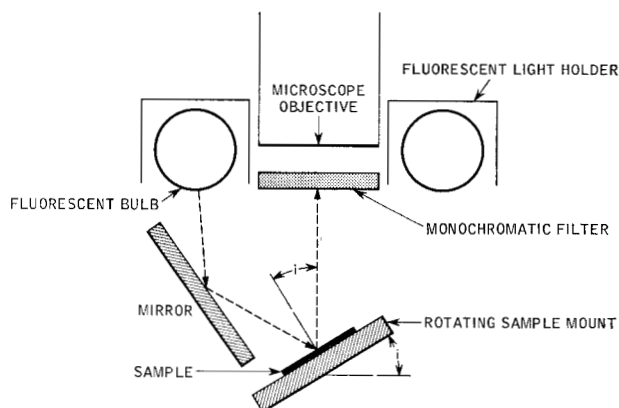
**Abstract:** A simple nondestructive method of measuring the refractive index and thickness of transparent films on reflective substrates has been developed. The technique involves the use of a microscope equipped with a monochromatic filter on the objective and a stage that can be rotated so that the reflected light is observed at various angles. The film thickness,  $d$ , is given by  $d = [\Delta N \lambda] / [2\mu(\cos r_2 - \cos r_1)]$ , where  $\lambda$  is the wavelength of the filtered light,  $\mu$  is the refractive index, and  $\Delta N$  is the number of fringes observed between the angles of refraction  $r_2$  and  $r_1$ .

This technique is especially suited for films thicker than one micron. Techniques are also described for obtaining accurate thicknesses of films less than one micron by the combined use of monochromatic filters and an interference pattern chart. These techniques can be used to determine film thicknesses ranging from several hundred angstroms to several microns with accuracies of 0.2% on films thicker than  $2\mu$ , and accuracies of tens of angstroms on thinner films. Since visual comparisons of color can be used fairly easily for film thickness determinations, the techniques were used to construct a color chart for thermally grown  $\text{SiO}_2$  films up to  $1.5\mu$  thick.

## 1. Introduction

Various optical techniques have been used for thickness and refractive index measurements of thin films.<sup>1</sup> The advantage of the new technique described here is that it is a relatively easy, nondestructive method of measuring transparent film thicknesses and refractive indices without the use of a step. This is accomplished by using a rotatable stage for the film and substrate so that reflected light is observed at various angles.<sup>2</sup> In order to ensure that only reflected monochromatic light is observed, either the microscope objective is covered with a monochromatic filter or the film is illuminated with monochromatic light. The former method has been found to be very convenient. A schematic sketch of the essential portions of the apparatus is shown in Fig. 1. The technique to be described is known as Variable Angle Monochromatic Fringe Observation (VAMFO). 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 rotated by hand and positioned to maintain the proper reflected light on the sample. With a vertically illuminated

*Figure 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  $10\times$ ) can be maintained in the center of the field of view. An American Optical Company microscope, Model 56-M3, was used. The filter is Bausch & Lomb, Series 33-79-XX.



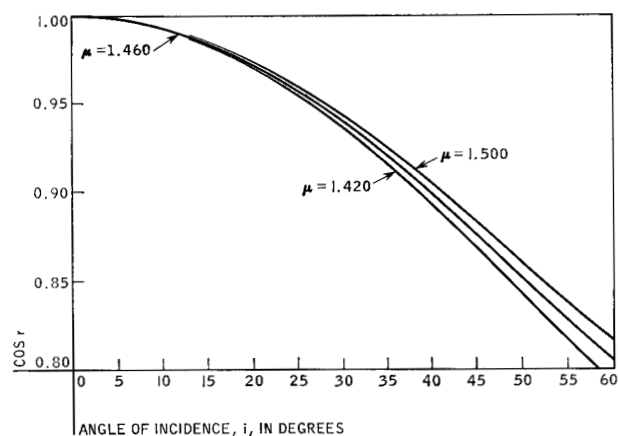


Figure 2 Variation of  $\cos r$  with angle of incidence for refractive indices of 1.420, 1.460, and 1.500.

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. Similarly with the apparatus described herein acceptable results can be attained with a fixed mirror mounting on the stage, if the mirror is mounted close to the point under examination and inclined at about  $100^\circ$ . 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

$\lambda$  is the wavelength of filtered light

$\mu$  is the refractive index of the film

$r_i$  is the angle of refraction at that fringe for which the angle of incidence is  $i_i$  and  $\sin r_i = (\sin i_i)/\mu$

$\Delta N$  = number of fringes observed between  $i_1$  and  $i_2$

$\Delta \cos r = (\cos r_2 - \cos r_1)/\Delta N$  averaged for both maxima and minima.

Equation (1) is derived from the following considerations. Assuming that the phase change on reflection at the air-film and film-substrate interfaces are the same,<sup>3</sup> then for maxima<sup>4</sup>

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

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

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

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.

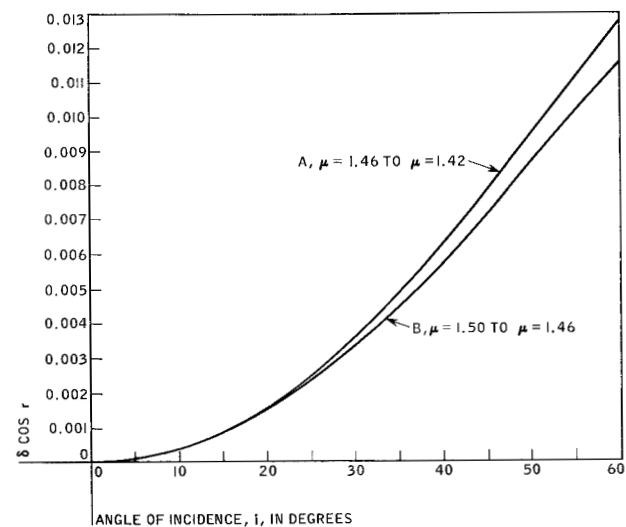
The variation of  $\cos r$  with the angle of incidence,  $i$ , is shown in Fig. 2 for refractive indices of 1.420, 1.460, and 1.500. This refractive index range is useful for silicon dioxide films, both thermally grown and pyrolytic, and for some glass films. For interpolation purposes, two curves relating  $\delta \cos r$  as a function of  $i$  are included in Fig. 3, where  $\delta \cos r = (\cos r)_{\mu=1.46} - (\cos r)_{\mu=1.42}$  and  $\delta \cos r = (\cos r)_{\mu=1.50} - (\cos r)_{\mu=1.46}$ . With such curves it is possible to obtain  $\cos r$  for any refractive index in the range 1.420 to 1.500.

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.

## 2. Experimental results

Various detailed techniques can be used with VAMFO for determining film thicknesses. The best method for a particular film depends on the film thickness and the accuracy desired. A few of these techniques will be described here briefly for films of thick, thin, and intermediate thicknesses, and for multiple films. A more detailed example of a refractive index determination is given in Appendix 1.

Figure 3 Interpolation curves for intermediate refractive indices.  $\delta \cos r = (\cos r)_{\mu_2} - (\cos r)_{\mu_1}$ .



• Thick films

a. Accurate film thickness and refractive index determinations

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. A comparison of some typical results is given in Table 1. The fringes at a step were determined with vertical illumination using a Bausch and Lomb metallurgical microscope, model DMET, equipped with a 5460 Å monochromatic filter. Under such conditions the thickness can be determined to about 0.1 fringe accuracy or about 0.02  $\mu$ .

Note that in the case of pyrolytic oxide, there was a 4% difference between the two determined thicknesses if it is assumed that the refractive index is the same as that for the thermal oxide, but if the refractive index were assumed to be 1.43<sub>7</sub> then the two determinations 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

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

The apparent fringe order  $N'_i$  should be integral for maxima and half integral for minima on the assumption of  $\pi$  phase changes at the interfaces. An approximate  $N_0(i = 0^\circ)$  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.

It is not always necessary to make a step. If the film is not too thick and an approximate refractive index is available from Table 1 for a similar type film or from 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, gives the proper integral and half-integral values of  $N_i$ . An example of the details of this technique are given for a Pyrex film on silicon in Appendix 1.

By this technique the refractive index of Corning 7740 glass at 5450 Å was found to be 1.476. The refractive index of this glass is given by Corning as 1.474 at 5890 Å, and because of dispersion a refractive index of 1.476 at 5450 Å is to be expected.

Table 1 Comparison of film thicknesses determined by VAMFO and by counting fringes at a step.

Film Substance on Silicon	Refractive Index 5460 Å	Method	d (microns)
Pyrex* (Corning 7740)	1.476	VAMFO	3.20
		Step	3.18
Thermal oxide (cycles of steam followed by dry O <sub>2</sub> at 970° C)	1.46 <sub>3</sub>	VAMFO	1.97
		Step	1.96
725°C pyrolytic SiO <sub>2</sub>	1.46 <sub>3</sub>	VAMFO	4.08
		Step	3.92
725°C pyrolytic SiO <sub>2</sub>	1.43 <sub>7</sub>	VAMFO	3.99
		Step	3.99

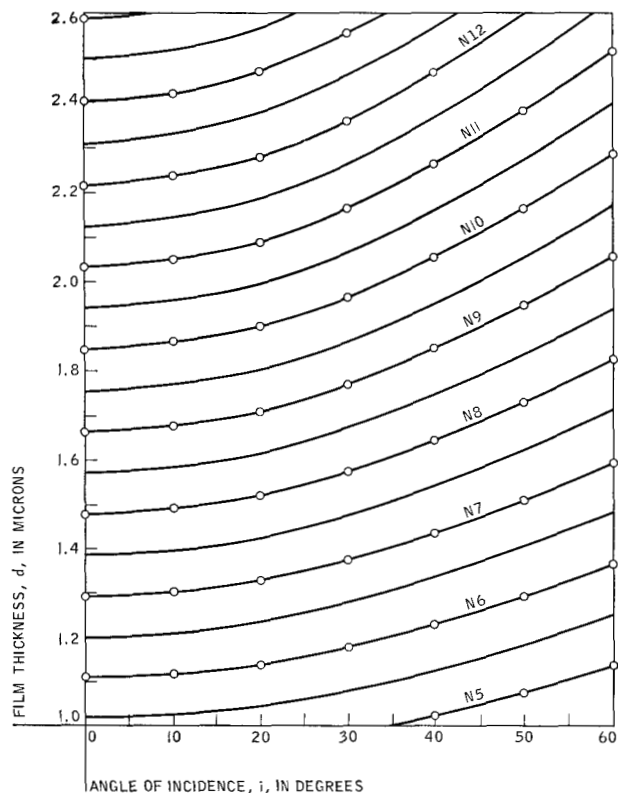
\* The glass films discussed in this report were formed by special sedimentation techniques. (Unpublished results of W. A. Pliskin and E. E. Conrad. See also Ref. 5.)

Another glass whose refractive index is known and which has been examined in detail is Corning 3320 uranium glass. Using 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 5890 Å is 1.481, and thus at 5450 the index should be 1.483, in excellent agreement with the experimentally determined index. The agreement of the refractive indices determined by VAMFO with the known or expected indices provides 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, and used also in the study of various pyrolytic and thermally grown silicon dioxide films.<sup>6</sup>

When the refractive index is known, it is not necessary to make detailed calculations as shown in Appendix 1 to obtain accurate film thicknesses. After determining  $\Delta \cos r$ , it can be substituted into Eq. (1) to obtain the film thickness. In the determination of the maxima and minima, it should be noted that the minima points are generally more accurate than the maxima. (See Appendix 3 for further details). 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, and is 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 becomes washed out at large angles of incidence.

Figure 4 Positions of interference maxima (—○—) and minima (—) for a pyrex film ( $\mu = 1.476$ ) on silicon at 5450 Å.



A chart for determining film thicknesses by VAMFO is shown in Fig. 4. The curves with circles represent the positions of maxima, and the curves without circles represent the minima. On a pyrex film, for example, if minima are observed at  $14.8^\circ$ ,  $38.7^\circ$ , and  $56.1^\circ$ , and maxima are observed at  $28.5^\circ$  and  $47.2^\circ$ , then the film thickness would be determined as  $2.34 \mu$ . The curves in Fig. 4 are based on Eq. (2); each curve represents an integral value of  $N$  for maxima and half-integral values for minima.

#### b. Approximate film thicknesses

Another very useful method for determining film thicknesses with VAMFO is quite accurate for thick films and relatively accurate for intermediate films. Examination of the  $\cos r$  vs  $i$  curves shows they approach linearity in the range  $i = 35^\circ$  to  $60^\circ$ . Thus, approximate film thicknesses can be obtained merely by determining the average angular fringe separation in the  $35^\circ$  to  $60^\circ$  range. Letting  $\Delta i$  be the average fringe separation in the  $35^\circ$  to  $60^\circ$  interval,  $\Delta N = 25^\circ/\Delta i$ , and substituting in Eq. (1), we obtain

$$d = D/\Delta i, \quad (5)$$

where  $D = 12.5\lambda/\mu[(\cos r)_{i=35^\circ} - (\cos r)_{i=60^\circ}]$ . The variation of  $D$  with refractive index for  $\lambda = 0.545 \mu$  is plotted in Fig. 5.

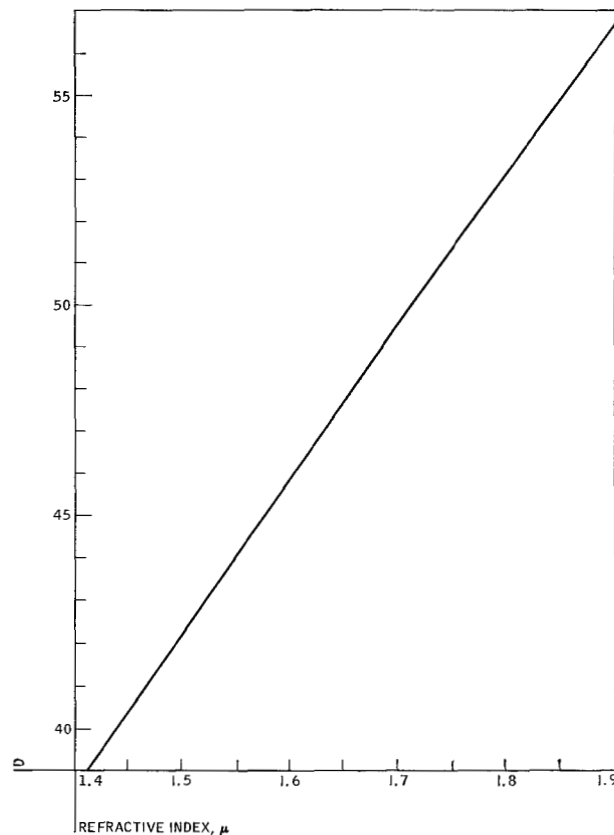
### • Thin films

#### a. Technique of multiple monochromatic filters

The technique using only one monochromatic filter is inaccurate for films less than one micron thick, the reason being 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 Fig. 4 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 Fig. 6 on the page following.

Figure 5 Variation of  $D$  from the formula for approximate film thickness,  $d = D/\Delta i$ , where  $\Delta i$  is the average fringe separation in the  $35^\circ$  to  $60^\circ$  interval ( $\lambda = 0.545 \mu$ ).



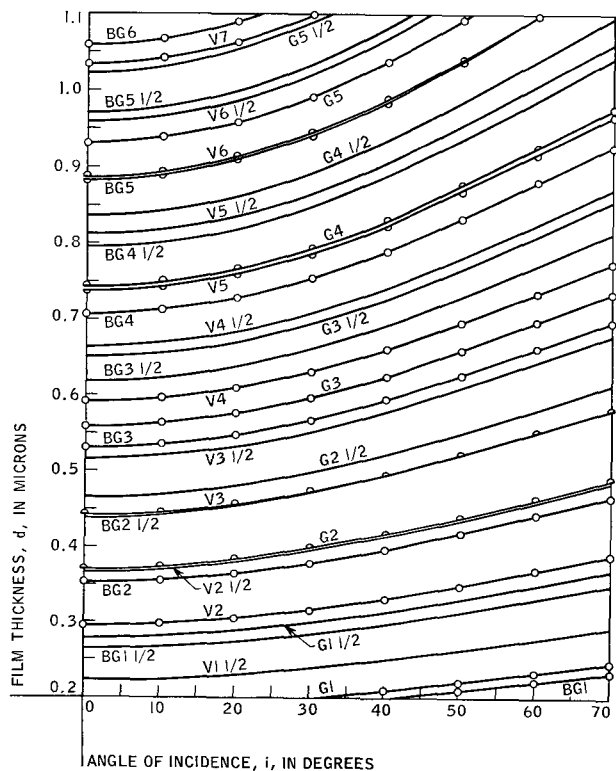


Figure 6 Positions of interference maxima (—○—) and minima (—) for a silicon dioxide film on silicon with filters 4340 Å (V,  $\mu = 1.470$ ), 5190 Å (BG,  $\mu = 1.464$ ) and 5450 Å (G,  $\mu = 1.463$ ).

In Figure 6 the curves for the maxima and minima positions are drawn for filters 4340 Å (V for violet), 5190 Å (BG for blue-green), and 5450 Å (G for green) with refractive indices for  $\text{SiO}_2$  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 2250 Å. The chart we have used with considerable success in actual practice includes the maxima and minima curves of the aforementioned filters together with the minima curves for a blue filter (4850 Å), a yellow filter (5830 Å), and a red filter (6230 Å). This degree of redundancy is not always necessary physically, but is very satisfying psychologically to determine from the minima positions of several filters the same value within 10 Å for a film several thousand angstroms thick.

#### b. Color comparison

With films less than one micron thick the film thicknesses can also be determined by removing the filter and observing the interference colors at various angles. Qualitative measurements of a similar type have been used in the past by many investigators for determining the color and film

thickness by comparison with a GE barium stearate step gauge<sup>7</sup> developed by K. Blodgett but these gauges are made for films only up to 0.4  $\mu$  thick. Step gauges for thicker films can be made by controlled etching of thick, thermally grown silicon dioxide films on silicon. The films were grown in steam followed by oxygen at 970°C.

Table 2 is a color chart for  $\text{SiO}_2$  films up to 1.5 $\mu$  thick. It was determined by the methods discussed in this paper. This chart gives the color of thermally grown (steam followed by oxygen at 970°C)  $\text{SiO}_2$  films ( $\mu_0 = 1.46_3$  at 5460 Å) when observed perpendicularly under daylight fluorescent lighting. The thickness of a film under angular observation is given by

$$d_F = d_0(\mu_0/\mu_F)/\cos r, \quad (6)$$

where  $d_0$  is the film thickness on the color chart corresponding to the observed color and  $\mu_F$  = refractive index of the film. By varying the angle of observation several determinations of  $d_F$  are made and, if the correct fringe order was selected, consistent film thickness values will be obtained. The fringe order or an approximate thickness can also be determined by comparison with either a commercial or a "homemade" step gauge for films thicker than 0.4 micron or by counting fringes at a step, if etching a part of the film is feasible. An accurate film thickness can then be determined from Eq. (6) and the color chart of Table 2, or by use of a monochromatic filter and Eq. (2) (and its counterpart for minima), or by use of Fig. 6.

Although color charts have been used by various investigators, this color chart is included to show and emphasize the color differences between various orders. For example, near Order IV we have blue-green instead of green and past Order IV at 8000 Å the film color is a broad orange which is much more pronounced than for the thinner films. The most interesting observation is that the color sequence from Order VI to VII is the opposite of that usually observed. Other differences can be seen by close examination of Table 2. The importance of these differences is that they can be utilized in establishing the fringe order or approximate film thickness and then a more exact thickness can be determined by Eq. (2) or Fig. 6.

#### • Films of intermediate thickness

If the refractive index is known and if the film thickness is not large enough to obtain an accurate value of  $\Delta \cos r$ , but is large enough to determine an approximate value, then Eq. (2) can be used to obtain an accurate film thickness. Using the approximate value of  $\Delta \cos r$  in Eq. (4), relevant  $N_i'$  values are obtained. Exact values for  $N_i'$  are then assumed by taking the closest integral and half-integral values to the calculated  $N_i'$  and are substituted into Eq. (2) to obtain accurate values of the film thickness  $d$ .

Table 2 Color chart for thermally grown SiO<sub>2</sub> films observed perpendicularly under daylight fluorescent lighting.

Film Thickness (microns)	Order (5450 Å)	Color and Comments	Film Thickness (microns)	Order (5450 Å)	Color and Comments
0.05 <sub>0</sub>		Tan	0.63 <sub>9</sub>		Violet-red
0.07 <sub>5</sub>		Brown	0.68		"Bluish." (Not blue but border- line between violet and blue green. It appears more like a mix- ture between violet-red and blue- green and over-all looks greyish).
0.10 <sub>0</sub>		Dark violet to red-violet			Blue-green to green (quite broad)
0.12 <sub>5</sub>		Royal blue			"Yellowish"
0.15 <sub>0</sub>		Light blue to metallic blue	0.72	IV	Orange (rather broad for orange)
0.17 <sub>5</sub>	I	Metallic to very light yellow- green	0.77		Salmon
0.20 <sub>0</sub>		Light gold or yellow—slightly metallic	0.80		Dull, light red-violet
0.22 <sub>5</sub>		Gold with slight yellow-orange	0.82		Violet
0.25 <sub>0</sub>		Orange to melon	0.85		Blue-violet
0.27 <sub>5</sub>		Red-violet	0.86		Blue
0.30 <sub>0</sub>		Blue to violet-blue	0.87		Blue-green
0.31 <sub>0</sub>		Blue	0.89		Dull yellow-green
0.32 <sub>5</sub>		Blue to blue-green	0.92	V	Yellow to "yellowish"
0.34 <sub>5</sub>		Light green	0.95		Orange
0.35 <sub>0</sub>		Green to yellow-green	0.97		Carnation pink
0.36 <sub>5</sub>	II	Yellow-green	0.99		Violet-red
0.37 <sub>5</sub>		Green-yellow	1.00		Violet
0.39		Yellow	1.02		Blue-violet
0.41 <sub>2</sub>		Light orange	1.05		Green
0.42 <sub>5</sub>		Carnation pink	1.06		Yellow-green
0.44 <sub>3</sub>		Violet-red	1.07		Green
0.46 <sub>5</sub>		Red-violet	1.10		Violet
0.47 <sub>5</sub>		Violet	1.11		Red-violet
0.48 <sub>0</sub>		Blue-violet	1.12	VI	Violet-red
0.49 <sub>3</sub>		Blue	1.18		Carnation pink to salmon
0.50 <sub>2</sub>		Blue-green	1.19		Orange
0.52 <sub>0</sub>		Green (broad)	1.21		"Yellowish"
0.54 <sub>0</sub>		Yellow-green	1.24	VII	Sky blue to green-blue
0.56	III	Green-yellow	1.25		Orange
0.57 <sub>4</sub>		Yellow to "yellowish." (Not yel- low is to be expected. At times it appears to be light creamy grey or metallic.)	1.28		Violet
			1.32		Blue-violet
			1.40		Blue
			1.45		Dull yellow-green
			1.46		
0.58 <sub>5</sub>		Light-orange or yellow to pink borderline	1.50	VIII	
0.60 <sub>0</sub>		Carnation pink	1.54		

• Multiple films

The thicknesses determined from fringes observed for films of glass on a silicon dioxide covered wafer are those of the glass plus the oxide. In this case, it can be shown that the glass film thickness is given by:

$$d_g = \frac{\Delta N \lambda}{2\mu_o (\cos r_{2o} - \cos r_{1o})} - \frac{\mu_o d_o (\cos r_{2o} - \cos r_{1o})}{\mu_o (\cos r_{2o} - \cos r_{1o})} \quad (7a)$$

$$= \frac{\lambda}{2\mu_o \Delta \cos r_o} - \frac{\mu_o d_o \Delta \cos r_o}{\mu_o \Delta \cos r_o} \quad (7b)$$

where the subscript *g* refers to glass and the subscript *o* to oxide. Needless to say, this formula is applicable to other transparent film systems such as one type of glass on a second type of glass film.

An approximation similar to that used in Eq. (5) may be used under proper conditions in place of Eq. (7a):

$$d_g = (D_o/\Delta i) - (D_o/D_o) d_o \quad (8)$$

A relation slightly more complicated than Eq. (4) can be used for determining the refractive index of the glass film above the oxide film:

$$N'_i = \frac{\cos r_{ig}}{\Delta \cos r_o} - \left[ \frac{2\mu_o d_o}{\lambda} \left( \frac{\cos r_{ig}}{\Delta \cos r_o} \Delta \cos r_o - \cos r_{io} \right) \right] \quad (9)$$

Acknowledgments

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## Appendix 1. Pyrex film example

If an approximate refractive index is known and if the film is not too thick, then it is not always necessary to count the fringes at a step to determine the proper  $N'_i$  set. For example, consider a Pyrex film formed on a polished silicon wafer. From Table 1, it is known<sup>8,9</sup> that the refractive index should be close to 1.47<sub>6</sub> at 5460 Å. For a first approximation and for illustrative purposes, assume  $\mu = 1.460$  and utilize Fig. 2 to obtain the  $\cos r$  as shown in Column 3 of Table 3. From Column 3,  $\Delta \cos r$  is found to be 0.0808<sub>3</sub>. Dividing this value into the  $\cos r$ , the apparent fringe orders  $N'$  listed in Column 4 are obtained. Since the  $N'$  are not half-integral and integral numbers for minima and maxima, it is evident that the refractive index is not 1.460. The closest half integer to 12.18 is 12.5. This would occur for a refractive index greater than 1.46 as can be seen by an examination of Figs. 2 or 3. For a given increase in refractive index, the increase in  $\cos r$  is considerably less for small values of  $i$  than for large values of  $i$ . Therefore,  $\Delta \cos r$  will decrease with an increase in refractive index and, consequently,  $N'$  will increase. Similarly for small values of  $i$ , if the correct  $N$  is divided into the first approximation of  $\cos r$ , then, because  $i$  is small, the quotient is a reasonably accurate value of the  $\Delta \cos r$  obtained on the second (and generally last) approximation. The difference between the first  $\Delta \cos r$  and this preliminary calculated  $\Delta \cos r$  can be used in conjunction with the interpolation curves to determine the refractive index to be used in the second approximation. The technique can be understood more clearly by use of a specific example. Since it is known from Table 1 that the refractive index of the Pyrex films is somewhat greater than 1.46, it appears reasonable to assume  $N$  for  $i = 14.8^\circ$  is 12.5. Dividing 12.5 into the corresponding  $\cos r$  (0.9846) one obtains 0.0788, which is 0.0020 less than the  $\Delta \cos r$  obtained in Column 3. From

the interpolation curves given in Fig. 3, for a change in refractive index from 1.460 to 1.500,  $\delta \cos r = 0.0008$  for  $i = 14.8^\circ$  and  $\delta \cos r = 0.0103$  for  $i = 56.1^\circ$ . Since there are two complete fringes between the minima at  $14.8^\circ$  and  $56.1^\circ$ , a refractive index increase of 0.040 will result in a decrease in  $\Delta \cos r$  of  $(0.0103 - 0.0008)/2 = 0.00475$ . Since the calculated decrease in  $\Delta \cos r$  is 0.0020,  $\Delta \mu = (0.0020/0.00475)(0.040) = 0.017$  and the second choice for the refractive index should be  $\mu = 1.460 + 0.017 = 1.477$ . The values for  $\cos r$  for  $\mu = 1.477$  as given in Column 5 were determined by adding the proper proportion,  $(0.017/0.040)$ , of the  $\delta \cos r$  given in Fig. 3 to the  $\cos r$  given in Column 3 for each value of  $i$ .

From these values of  $\cos r$ ,  $\Delta \cos r$  corresponding to  $\mu = 1.477$  was found to be 0.07880. Dividing the  $\cos r$  by  $\Delta \cos r$  one obtains the fringe orders  $N$  given in Column 6. These are half-integral and integral values for minima and maxima. This chosen  $N$  set is the only set which would result in a refractive index in the vicinity of that to be expected for Pyrex ( $\mu \sim 1.47 - 1.48$ ), and therefore  $\mu = 1.477$  is a reasonably accurate value for the refractive index of the Pyrex film.<sup>10</sup> The  $\cos r$  values given in Column 5 are approximate since they were obtained by adding to the  $\cos r$  from Fig. 2 ( $\mu = 1.460$ ) values determined from the interpolation curves in Fig. 3. More accurate values of  $\cos r$  were obtained directly from a  $\cos r$  vs  $i$  curve for  $\mu = 1.476$  and are listed in Column 7. The calculated  $N$  in Column 8 shows proper half-integral and integral values. Therefore, it is concluded that  $\mu = 1.476$ . The close agreement with the index (1.477) obtained by interpolation shows that the interpolation curves can be used with reasonable accuracy for intermediate refractive indices. Similarly the film thicknesses calculated from Eq. (1) and given at the bottom of Table 3 are in agreement. The refractive index of Corning 7740 at 5890 Å is 1.474 and, because of dispersion, the refractive index at 5450 Å should be 1.476, as observed.

Table 3 Example of determination of the refractive index of a Pyrex film (Corning 7740 Glass) on silicon ( $\lambda = 5450$  Å).

Fringe	$i$	$\mu = 1.460$		$\mu = 1.477$ (int)		$\mu = 1.476$	
		$\cos r$	$N'$	$\cos r$	$N$	$\cos r$	$N$
min.	14.8°	0.9846	12.18	0.9850	12.50	0.9849	12.49
max.	28.5°	0.9451	11.69	0.9463	12.01	0.9463	12.00
min.	38.7°	0.9037	11.18	0.9059	11.50	0.9059	11.49
max.	47.2°	0.8645	10.70	0.8678	11.01	0.8677	11.00
min.	56.1°	0.8227	10.18	0.8271	10.50	0.8269	10.49
		$\Delta \cos r = 0.0808_3$		$= 0.0788_0$		$= 0.0788_7$	
		$d = 2.31 \mu$		$= 2.34_1 \mu$		$= 2.34_1 \mu$	

## Appendix 2. Angular correction for stereomicroscopes

If a stereomicroscope is used, a small angular correction should be applied to  $i_0$ , the observed angle of incidence. This correction is not large and in many cases can be neglected. If  $2\alpha$  is the angle subtended by the microscope, then the correct angle of incidence  $i$  is given by

$$\cos i = \cos \alpha \cos i_0. \quad (\text{A-1})$$

Thus for  $2\alpha = 10^\circ$ , the correction is  $+5^\circ$  at  $i_0 = 0^\circ$  but is only  $+0.6^\circ$  at  $i_0 = 20^\circ$  and  $+0.1^\circ$  at  $i_0 = 60^\circ$ .

## Appendix 3. Accuracy of fringe positions

The positions of maxima and minima at a specific point on a film can be determined very accurately with films of nonuniform thickness. On nonuniform films a fringe pattern can be seen and 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, and is especially so for thinner films.

There are two surfaces from which reflectivity must be considered: the air-oxide (or glass) film interface and the oxide-silicon (or substrate) interface. The observed light intensity from the film and substrate can be represented by

$$I = f_1(x) + f_2(x) \cos(2\pi x/P), \quad (\text{A-2})$$

where  $x = \cos r$  and  $P = \Delta \cos r$ . For silicon dioxide on silicon  $f_1$  represents the reflectivity from the silicon dioxide-silicon interface and  $f_2$  the reflectivity from the oxide-air interface. The proper maxima are given by integral values of  $x/P$  and the proper minima by half-integral values of  $x/P$ . The observed maxima and minima are given by:  $dI/dx = 0 = f_1' + f_2' \cos(2\pi x/P) - (2\pi/P)f_2 \sin(2\pi x/P)$ .

Letting  $\delta x$  be a small difference between the position of the observed maximum or minimum and the proper maximum or minimum, we have:

$$\delta x = \frac{P^2}{4\pi^2} \left( \frac{f_2' \pm f_1'}{f_2} \right), \quad + \text{maxima}, \quad - \text{minima}.$$

Since  $P = \lambda/2\mu d$ ,

$$\delta x = \frac{\lambda^2}{16\pi^2 \mu^2 d^2} \left( \frac{f_2' \pm f_1'}{f_2} \right), \quad (\text{A-3})$$

where  $\mu$  is the refractive index of the film and  $d$  its thickness. Calculations, based on reflectivity formulae for the perpendicular component in the silicon-silicon dioxide system,<sup>11</sup> show that  $f_1'$  and  $f_2'$  are both negative (for the parallel component  $f_1'$  and  $f_2'$  are positive) and therefore  $\delta x$  is smaller for minima than for maxima, and the thicknesses determined from minima are more accurate. Fur-

thermore, Eq. (A-3) shows that the corrections are smaller with thicker films. At small  $i$ ,  $f_1' < f_2'$ ; at  $26^\circ$ ,  $f_1' = f_2'$ ; and at  $i > 26^\circ$ ,  $f_1' > f_2'$ . Thus with  $i$  near  $26^\circ$  the observed minima are close to the proper minima and accurate thickness values are obtained.

The greater accuracy obtained with minima can best be shown by a specific example. For a 4120 Å, silicon dioxide film using a 5450 Å filter the perpendicular component maximum was at  $46.7^\circ$ , corresponding to a thickness of 4290 Å, and the parallel component maximum was at  $30.1^\circ$ , corresponding to a thickness of 3960 Å. Using a 4340 Å filter, the minima for the perpendicular and parallel components gave thicknesses of 4140 and 4080 Å, both of which are much closer to the actual thickness.

To obtain film thickness accuracies within 20 Å for uniform films less than 5000 Å thick, it is often advisable (when good minima can not be observed) to purposely remove a known small amount ( $\sim 100$  Å) of the film by using a controlled, dilute etch on a small section. The thickness difference of 100 Å is easily observable with 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.

## References and footnotes

1. O. S. Heavens, *Optical Properties of Thin Solid Films*, Butterworth Scientific Publications, London, 1955.
2. See Appendix 2 regarding small angular corrections for stereomicroscopes.
3. This is a reasonable assumption for transparent films on high-refractive-index materials such as silicon. From the optical constants of silicon [W. C. Dash and R. Newman, *Phys. Rev.* 99, 1151 (1955)] a phase change of  $179.6^\circ$  was calculated for a  $\text{SiO}_2$ -silicon interface (nondegenerate silicon).
4. F. A. Jenkins and H. E. White, *Fundamentals of Physical Optics*, McGraw-Hill Book Company, Inc., New York, 1937.
5. 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, October 2, 1961 and J. L. Langdon, W. E. Mutter, R. P. Pecorara and K. K. Schuegraf, "Hermetically Sealed Silicon Chip Diodes and Transistors," presented to the 1961 Electron Devices Meeting, Washington, D. C., October 27, 1961.
6. Unpublished work, W. A. Pliskin and H. S. Lehman.
7. Available from Sunshine Scientific Instrument, Philadelphia, Pa.
8. A 5450 Å filter was used in VAMFO.
9. Approximate refractive indices can also be determined by obtaining Brewster's angle in a simplified set-up by inserting a polarizer in addition to the monochromatic filter on the microscope objective in VAMFO. Brewster's angle can then be determined quite easily for films which are non-uniform by the angle at which the interference fringes (due



to nonuniformity of the film) disappear. It is difficult to determine Brewster's angle for uniform films on highly reflective substrates, but with some of our glass films on silicon the thickness variation over a wafer is about one or two fringes and thus an approximate refractive index can be determined by  $\mu = \tan \phi$ , where  $\phi$  is Brewster's angle.

10. If the  $N$  set were taken such that  $N = 11.5$  for  $i = 14.8^\circ$ , then  $\mu$  would be 1.424, and if the  $N$  were 13.5 then  $\mu$  would be 1.528. From Table 1 and from the refractive index expected for Pyrex, it is obvious that these choices are incorrect and that  $N$  should be 12.5. A check by etching a

step revealed that for perpendicular illumination with 5450 Å light, the thickness corresponded to about 12.7 fringes and therefore at  $i = 14.8^\circ$ ,  $N = 12.5$  was the proper choice.

11. See J. A. Stratton, *Electromagnetic Theory*, Chapter IX, McGraw-Hill Book Co., Inc., New York, 1941; or F. A. Jenkins and H. E. White, *Fundamentals of Physical Optics*, Chapter 18, McGraw-Hill Book Co., Inc., New York, 1937.

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