

Film Thickness of 'Thick Thin Films' by Spectroscopy

Fred Goldstein, FTG Software Associates, Princeton, NJ

ABSTRACT

Reflection and transmission spectroscopy are popular methods for determining the thickness of 'thick thin films' such as those used for protective coatings, media coatings, base coats, etc. Relatively thinner films are analyzed by counting fringes while thicker films with numerous fringes are better suited to Fourier analysis. These methods can work well when little is known about the optical properties of the substrate.

If the film thickness is independently calibrated, it is not necessary to know the film index accurately when the film is non-dispersive. Problems can occur, however, when film dispersion is neglected. The Fourier transform may be severely distorted if the spectrum is not corrected. These methods have been encapsulated in Windows program *filmthk.exe* which may be downloaded from the Internet. The program includes the two calculation algorithms, calibration, dispersion and smoothing routines for removing noise or spurious high frequency fringes.

INTRODUCTION

Optical spectroscopy is widely utilized for determining the thickness of films, lacquers and other clear coatings. The technique is often used by chemists who have only approximate values for film indices and even less knowledge about the substrate ("It's shiny").

How is it possible to utilize this technique when so little is known about the optical properties? As we could find nothing about the subject in the literature, we decided to investigate for ourselves.

FRINGE COUNTING

Optical thin film engineers frequently use 'fringe counting' for determining thickness and index of films. A rather thick film, say 15 quarter waves (physical thickness 908.6 nm) of TiO₂ at 550 nm, is deposited on a test glass. The position and height of the peaks are used to calculate index and thickness. Adjacent peaks differ by one quarter wave. Ignoring dispersion, $w_1(m) = w_2(m+1)$ where w_1 and w_2 are wavelengths at adjacent peaks. In former times a slide rule was used to search for peaks differing by integral quarter waves.

The technique is illustrated in Figure 1 where peaks are labeled according to quarter wave optical thickness.

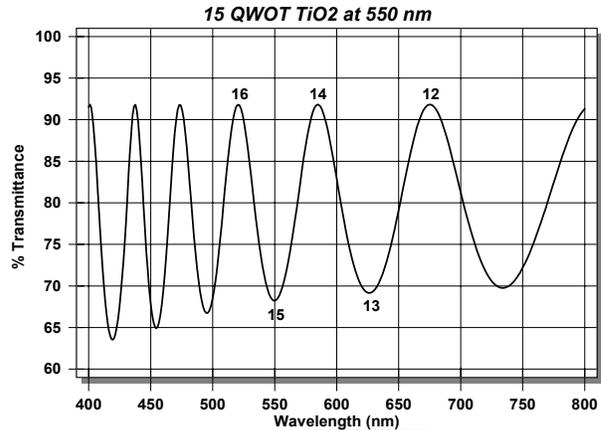


Figure 1. TiO₂ on glass in transmittance

Consider the same film deposited on aluminum and silver.

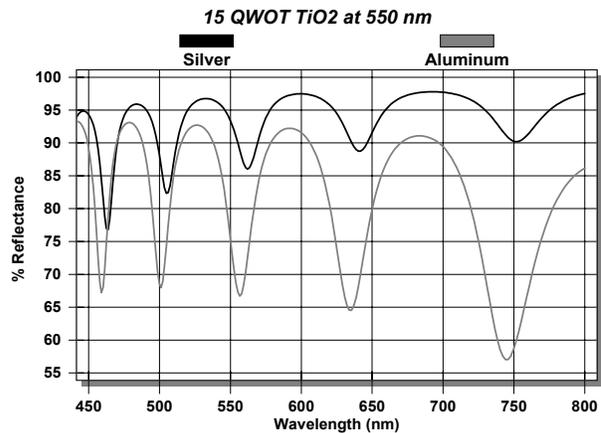


Figure 2. TiO₂ on silver and aluminum

While the spectra are quite different fringe positions appear roughly similar. Can we determine film thickness without knowing the optical properties of the substrate?

To investigate this, consider four calculated (using *Film-Star* design software) spectra for TiO₂ films with varying thickness (0.5 μm , 1.0 μm , 1.5 μm , 2.0 μm) deposited on Al and Ag substrates. This example was chosen because TiO₂ is quite dispersive over the visible region.

'Freeware' program *Film Thickness Module* (FTM) then calculates film thickness without using substrate index. The Calculation Parameters screen illustrated in Figure 3 has no provision for entering substrate index except for checking whether the substrate is metallic or has higher index than the substrate. Film index is specified in index file TIO2. The FTM includes an editor for setting up dispersion files.

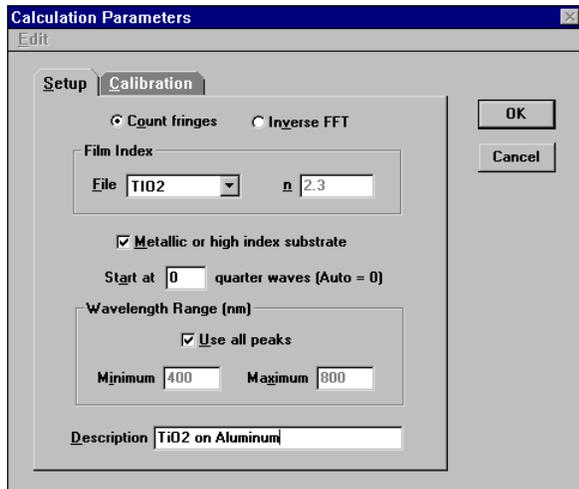


Figure 3. Calculation Parameters

When imported into the FTM, the spectrum is displayed as shown in Figure 4. The program automatically displays peak and valley positions. This helps users determine that the program is operating correctly.

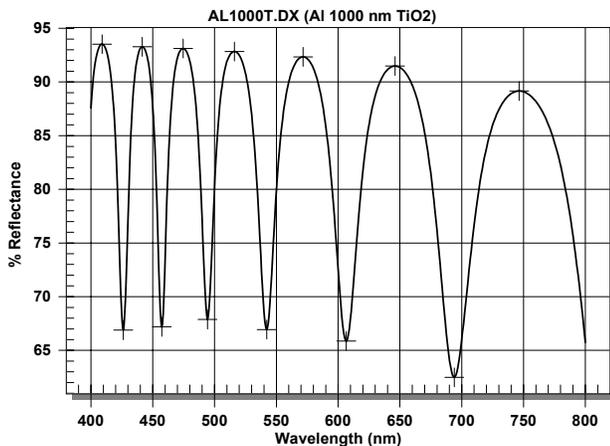


Figure 4. Spectrum for fringe counting

Calculated results are shown in Table 1 below. All fringes in the range 400 – 800 nm were utilized.

Actual	Al	Ag
0.5 μm	0.5113	0.5251
1.0 μm	1.0125	1.0243
1.5 μm	1.5131	1.5243
2.0 μm	2.0134	2.0245

Table 1. Results for fringe counting

The next step is to deduce calibration functions for each material. This is accomplished by least-squares fitting. If there is only one calibration point, the function is a constant; for two points the program provides linear calibration and for three points or more points a quadratic function.

In these cases the following functions are obtained where t_m is measured thickness in microns:

$$\text{Al: } t = -.0097 + .9964*t_m + .0009*t_m^2$$

$$\text{Ag: } t = -.026 + 1.0029*t_m - .001*t_m^2$$

What if the film indices are not known accurately? This appears to be unimportant under the following conditions:

1. The film is not dispersive in the measured wavelength range,
 2. At least two calibration points are available.
- As an example we calculated spectra with $n = 2.3$ and then assumed its index was $n = 2.0$ in the Film Thickness Module. This leads to correction function such as:

$$\text{Ag: } t = -.0241 + .8698*t_m$$

FFT (FAST FOURIER TRANSFORM)

As films become thicker there are too many fringes for reliably locating peaks and valleys. Instead we use the Fast Fourier Transform (FFT). This mathematically converts spectral response to film thickness. An ideal example is 10 μm of material $n = 2.3$ deposited on glass:

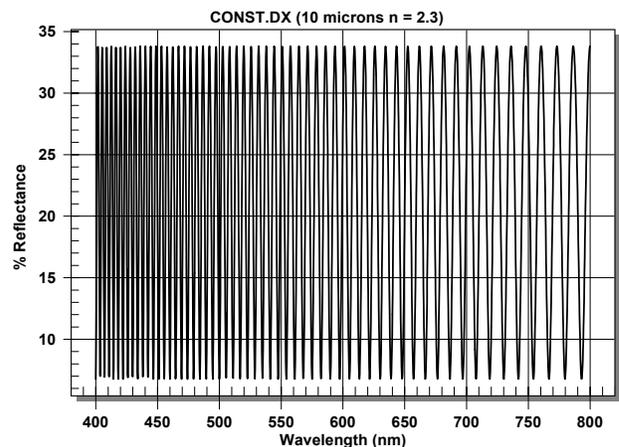


Figure 5. 10 μm $n = 2.3$ on glass

The Fourier transform provides the following curve:

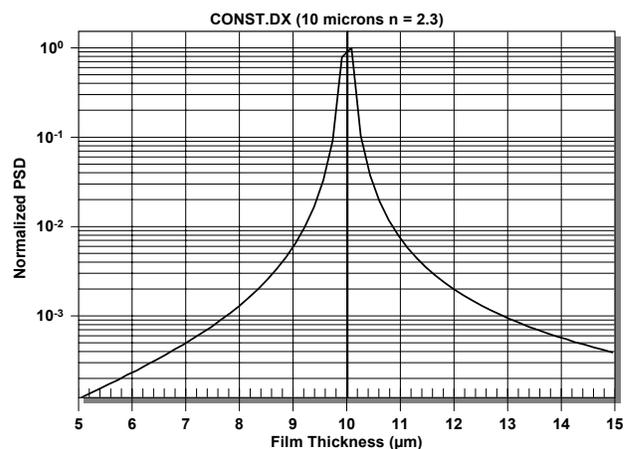


Figure 6. Fourier transform of Figure 5 spectrum

Here PSD (power spectral density) is plotted vs. film thickness. Ideally the Fourier transform is a perfect spike centered at the film thickness. Since this requires an infinitely wide spectrum, it is fortunate that measurable spectral ranges are sufficient to provide unambiguous results.

Repeating the above for 10 μm of TiO₂ on Silver we obtain a more complex Fourier spectrum.

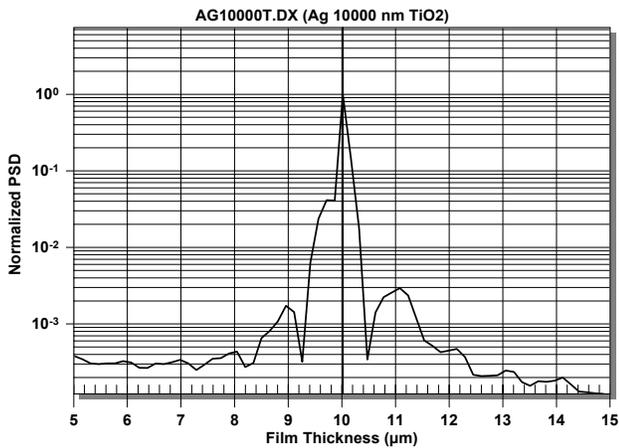


Figure 7. Fourier transform 10 μm TiO₂ on silver

Although distorted by dispersion the PDS peak unambiguously provides the correct answer. Calculation Parameters are illustrated below.

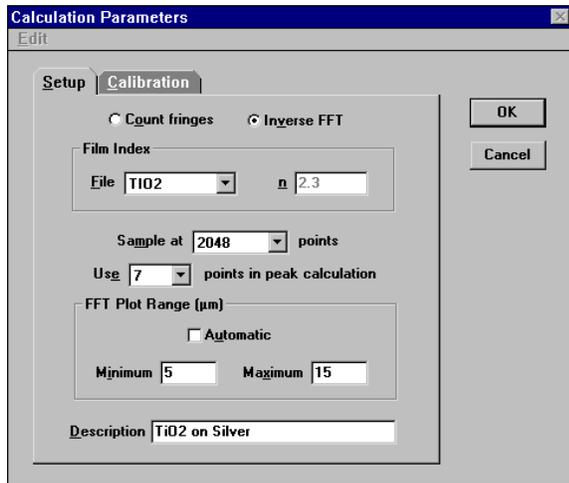


Figure 8. Calculation Parameters for FFT

What happens when we don't know the substrate index? As an example, we replace TiO₂ by $n = 2.3$. As illustrated in the above screen shot, this is readily accomplished by setting the Film Index File name to blank and entering 2.3 as constant index.

This leads to serious trouble as shown in Figure 9. The program provides an inaccurate solution. In this case spectra need to be acquired at longer wavelengths where film dispersion is negligible.

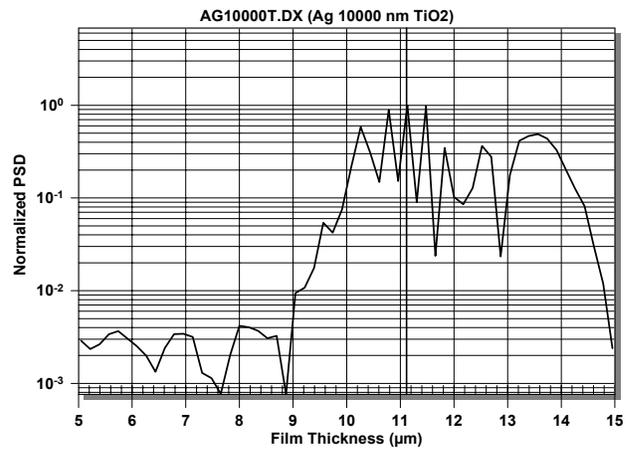


Figure 9. Problem when film dispersion neglected

SPURIOUS FRINGES

Spurious fringes may occur in transmission when films are deposited on a thin substrate. This is simulated in Figure 10 for 500 nm film of TiO₂ deposited on 0.2 mm glass. The calculation is performed by assuming the glass layer is a thin film and then spectral averaging with slightly different glass thickness. Actual fringe heights depend on the uniformity of the glass as well as monochromator bandwidth.

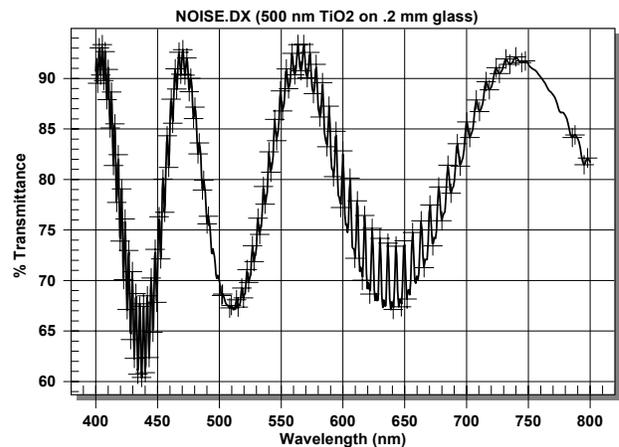


Figure 10. Spurious high frequency fringes

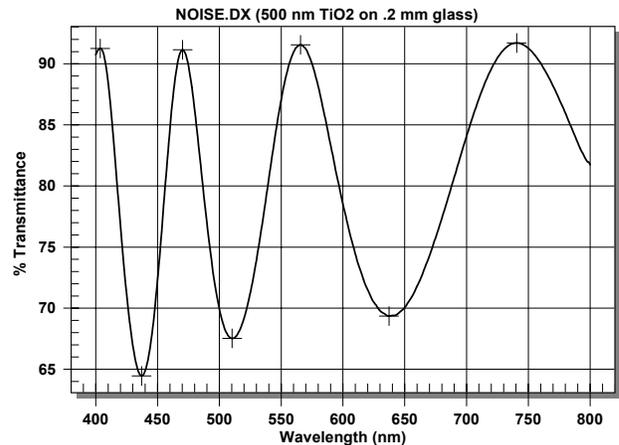


Figure 11. Data after Savitzky-Golay smoothing

Spurious fringes can be eliminated by moving-average or the more mathematically rigorous Savitzky-Golay¹ algorithm illustrated above in Figure 11. Such methods have little effect on peak position. The user repeatedly smoothes the curve until the peaks are unambiguously resolved.

DISCUSSION

Both fringe counting and Fourier methods can determine the physical thickness of 'thick' thin films when the optical properties of the substrate are unknown. If the optical properties of the film are only approximately known, it is crucial that there be no dispersion in the measured wavelength range and that there exists an independent method for determining film thickness.

Dispersion is easy to check for. If there is dispersion, fringe peak and valley wavelength locations deviate from the ratio relationship $w_1(m) = w_2(m+1)$. Perhaps the slide rule is still the best tool for this job!

In working on this paper we have tested this approach for aluminum, silver and gold substrates. FTM users need to ensure validity with substrate materials exhibiting absorption peaks and similar sharp structures in the wavelength range being utilized.

The FFT method requires that the number of data points be a power of two. Using interpolation, FTM converts supplied spectra to the required spacing. Those who wish to understand more about Fourier transform methods will find treatments in any number of text books or even by searching the Internet for 'Fourier transform'.

DOWNLOAD

The Film Thickness Module includes both fringe counting and the FFT technique. It may be downloaded freely and without restrictions from web site

<http://www.ftgsoftware.com>

Measured spectra can be pasted from the Windows clipboard (Excel format) or loaded in two file formats: comma-separated .csv and JCAMP .dx (a widely utilized format for exchanging spectroscopic data). Spectra plots are easily exported in .wmf format.

Moving-average and Savitzky-Golay smoothing are included. The program can also be operated via DDE (dynamic data exchange) as part of automatic test procedures.

Users who find an implementation of JCAMP which cannot be opened should send the file via e-mail to the author at

support@ftgsoftware.com

¹ Savitzky, A. and Golay, M.J.E., *Analytical Chemistry*, Vol. 36, pp. 1627-1639 (1964)