OP-R: Optical Thin Film Measurement

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## Interference-based measurement of semi-transparent thin film thickness

(Adapted from product literature Filmetrics F20 Thin Film Measurement System, copyright 1998 Filmetrics, Inc., San Diego, CA. The F20 measurement system in our lab was donated by Filmetrics through the generosity of Dr. Scott Chalmers.)

### Introduction

Very thin layers of material that are deposited on the surface of another material (thin films) are extremely important to many technology-based industries. Thin films are widely used, for example, to provide passivation, insulating layers between conductors, diffusion barriers, and hardness coatings for scratch and wear resistance. The fabrication of integrated circuits consists primarily of the deposition and selective removal of a series of thin films.

Films typically used in thin-film applications range from a few atoms (<10Å or 0.001 µm) to 100 µm thick (the width of a human hair.) They can be formed by many different processes, including spin coating, vacuum evaporation, sputtering, vapor deposition, and dip coating.

To perform the functions for which they were designed, thin films must have the proper thickness, composition, roughness, and other characteristics important to the particular application. These characteristics must often be measured, both during and after thin-film fabrication.

The two main classes of thin-film measurement are optical and stylus based techniques. Stylus measurements measure thickness and roughness by monitoring the deflections of a fine-tipped stylus as it is dragged along the surface of the film. Stylus instruments are limited in speed and accuracy, and they require a “step” in the film to measure thickness. They are often the preferred method when measuring opaque films, such as metals.

Optical techniques determine thin-film characteristics by measuring how the films interact with light. Optical techniques can measure the thickness, roughness, and optical constants of a film. Optical constants describe how light propagates through and reflects from a material. Once known, optical constants may be related to other material parameters, such as composition and band gap.

Optical techniques are usually the preferred method for measuring thin films because they are accurate, nondestructive, and require little or no sample preparation. The two most common optical measurement types are spectral reflectance and ellipsometry Spectral reflectance measures the amount of light reflected from a thin film over a range of wavelengths, with the incident light normal (perpendicular) to the sample surface. Ellipsometry is similar, except that it measures reflectance at non-normal incidence and at two different polarizations. In general, spectral reflectance is much simpler and less expensive than ellipsometry, but it is restricted to measuring less complex structures.

### n and k Definitions

Optical constants ***(n*** and *k)* describe how light propagates through a film. In simple terms, the electromagnetic field that describes light traveling through a material at a fixed time is given by:

$$A \bullet \cos\left(n\frac{2\pi}{\lambda}x\right) \bullet \exp\left(-k\frac{2\pi}{\lambda}x\right),$$

where x is distance, lamda is the wavelength of light, and ***n*** and *k* are the film's refractive index and extinction coefficient, respectively The refractive index is defined as the ratio of the speed of light in a vacuum to the speed of light in the material. The extinction coefficient is a measure of how much light is absorbed in the material.

**Single Interface**

Reflection occurs whenever light crosses the interface between different materials. The fraction of light that is reflected by an interface is determined by the discontinuity in ***n*** and ***k. .*** For light reflected off of a material in air, $R=\frac{(n-1)^2+k^2}{(n+1)^2+k^2}$ .To see how spectral reflectance can be used to measure optical constants, consider the simple case of light reflected by a single nonabsorbing material (k=0). Then:

$$R=\left|\frac{n-1}{n+1}\right|^2$$

Clearly, ***n*** of the material can be determined from a measurement of R. In real materials, ***n*** varies with wavelength (that is to say, real materials exhibit dispersion), but since the reflectance is known at many wavelengths, ***n*** at each of thesewavelengths is also known, as shown here.

**Figure 1: Single interface reflection.**

**Multiple Interfaces**

Consider now a thin film on top of another material. In this case both the top and bottom of the film reflect light. The total amount of reflected light is the sum of these two individual reflections. Because of the wavelike nature of light, the reflections from the two interfaces may add together either constructively (intensities add) or destructively (intensities subtract), depending upon their phase relationship. Their phase relationship is determined by the difference in optical path lengths of the two reflections, which in turn is determined by thickness of the film, its optical constants, and the wavelength of the light. Reflections are in-phase and therefore add constructively when the light path is equal to one integral multiple of the wavelength of light. For light perpendicularly incident on a transparent film, this occurs when 2nd = iλ where ***d*** is the thickness of the film and i is an integer (the factor of two is due to the fact that the light passes through the film twice.) Conversely, reflections are out of phase and add destructively when the light path is one half of a wavelength different from the in-phase condition, or when ***2nd*** = (i+1/2)λ. The qualitative aspects of these reflections may be combined into a single equation:

From this, we can see that

$$R \approx A + B\cos\left(\frac{2\pi}{\lambda} nd\right)$$

the reflectance of a thin film will vary periodically with 1/wavelength, which is illustrated below. Also, thicker films will exhibit a greater number of oscillations over a given wavelength range, while thinner films will exhibit fewer oscillations, and oftentimes only part of an oscillation, over the same range.

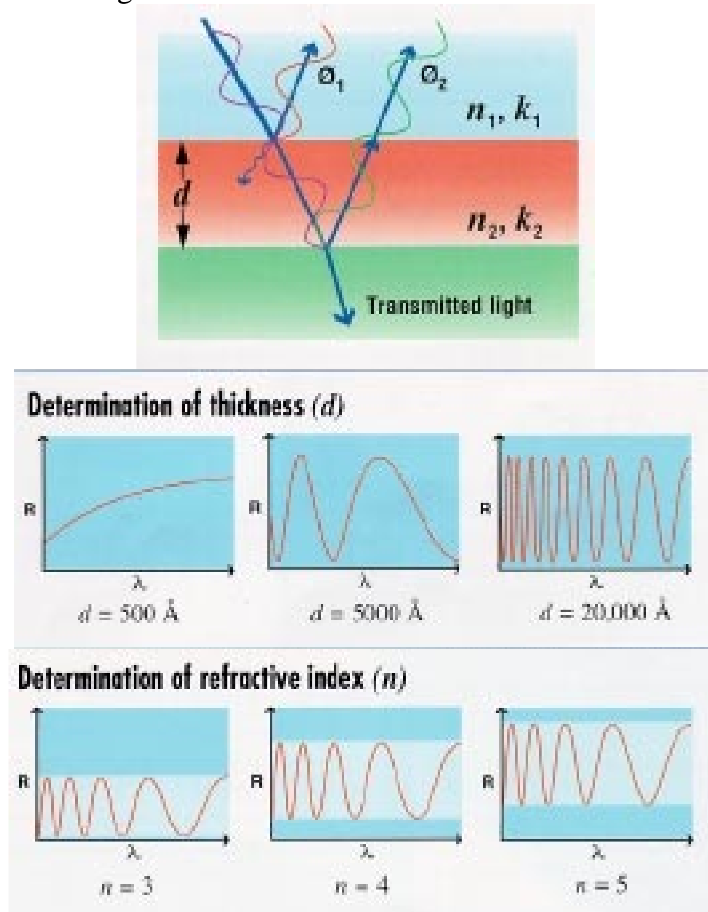


**Figure 2: Multi-interface reflections.**

**Determining Film Properties from Spectral Reflectance**

The amplitude and periodicity of the reflectance of a thin film is determined by the film's thickness optical constants, and other properties such as interface roughness. In cases where there is more than one interface, **it** is not possible to solve for film properties in closed form, nor is **it** possible to solve for ***n*** and ***k*** at each wavelength individually. In practice, mathematical models are used that describe ***n*** and ***k*** over a range of wavelengths using only a few adjustable parameters. A film's properties are determined by calculating reflectance spectra based on trial values of thickness and the ***n*** and ***k*** model parameters, and then adjusting these values until the calculated reflectance matches the measured reflectance.

**Models for *n* and *k***

There are many models for describing ***n*** and ***k*** as a function of wavelength. When choosing a model for a particular film, **it** is important that the model be able to accurately

describe ***n*** and ***k*** over the wavelength range of interest using as few parameters as possible. In general, the optical constants of different classes of materials (e.g., dielectrics, semiconductors, metals, and amorphous materials) vary quite differently with wavelength, and require different models to describe them (see below.) Models for dielectrics (k=0) generally have three parameters, while nondielectrics generally have five or more parameters. Therefore, as an example, to model the two-layer structure shown below, a total of 18 adjustable parameters must be considered in the solution.

**Number of Variables, Limitations of Spectroscopic Reflectance**

Spectral reflectance can measure the thickness, roughness, and optical constants of a broad range of thin films. However, if there is less than one reflectance oscillation (ie. the film is very thin), there is less information available to determine the adjustable model parameters. Therefore, the number of film properties that may be determined decreases for very thin films. If one attempts to solve for too many parameters, a unique solution cannot be found; more than one possible combination of parameter values may result in a calculated reflectance that matches the measured reflectance.

An example of the reflectance from a very thin film, 50Å of $SiO_2$ on silicon is shown below, where it is compared to the reflectance from a bare silicon substrate. In this case, measuring the thickness, roughness, and ***n*** of the $SiO_2$ requires five parameters to be determined. Clearly, the change in the spectra caused by adding 50Å of $SiO_2$ does not require five parameters to describe, and a unique solution cannot be found unless some additional assumptions are made.

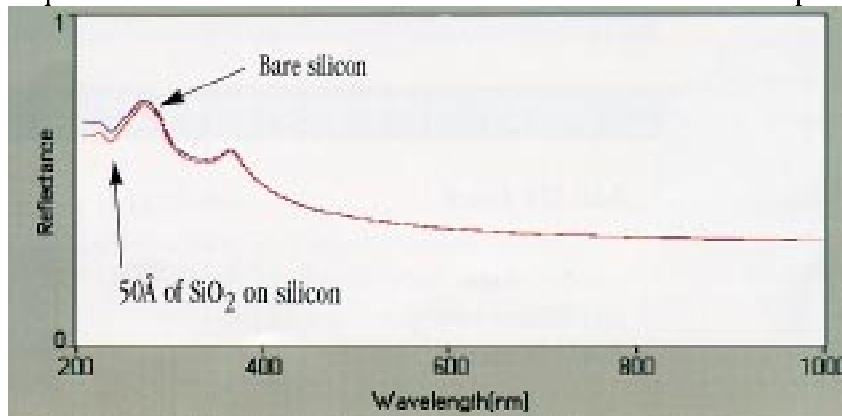


**Figure 3: Sample reflectance trace.**

Depending upon the film and the wavelength range of the measurement, the minimum single-film thickness that can be measured using spectral reflectance is in the 30Å to 300Å range. If one is trying to measure optical constants as well, the minimum thickness increases to between 100Å and 2000Å, unless minimal parameterization models can used. When solving for the optical properties of more than one film, the minimum thicknesses are increased even further.

**Spectroscopic Reflectance versus Ellipsometry**

Given the restrictions listed above, spectral reflectance can be used to measure a large percentage of technologically important films. However, when films are too thin, too numerous, or too complicated to be measured with spectral reflectance, oftentimes they can be measured with the generally more powerful technique of spectroscopic ellipsometry By measuring reflectance at non-normal incidence (typically around 75˚ from normal) ellipsometry is more

sensitive to very thin layers, and the two different polarization measurements provide twice as much information for analysis. To carry the idea even further, variable-angle ellipsometry can be used to take reflectance measurements at many different incidence angles, thereby increasing the amount of information available for analysis.

The following pages of this brochure describe spectral reflectance systems available from Filmetrics. If you are uncertain whether spectral reflectance or ellipsometry is appropriate for your film measurements, please call us to discuss your application. If spectral reflectance cannot satisfy your needs, we will be happy to refer you to a reputable source for ellipsometry

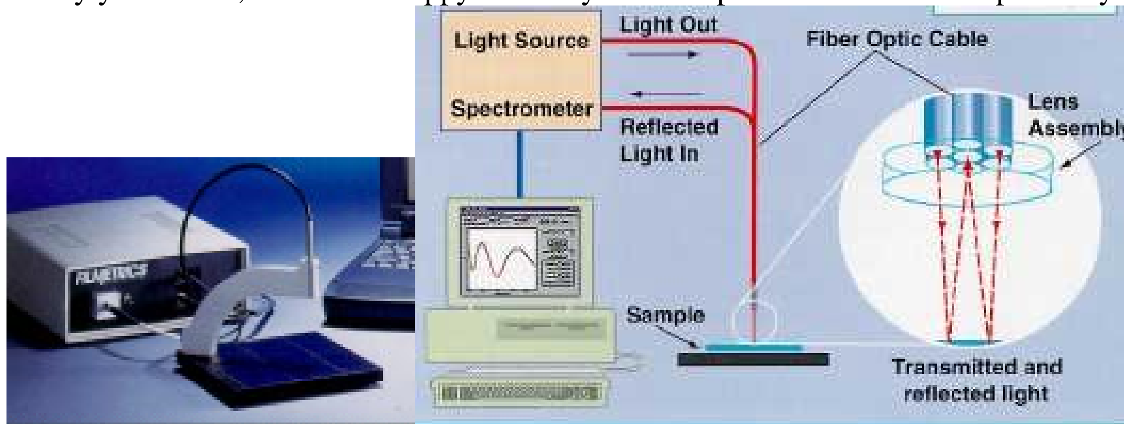


**Figure 4: Filmetrics system.**

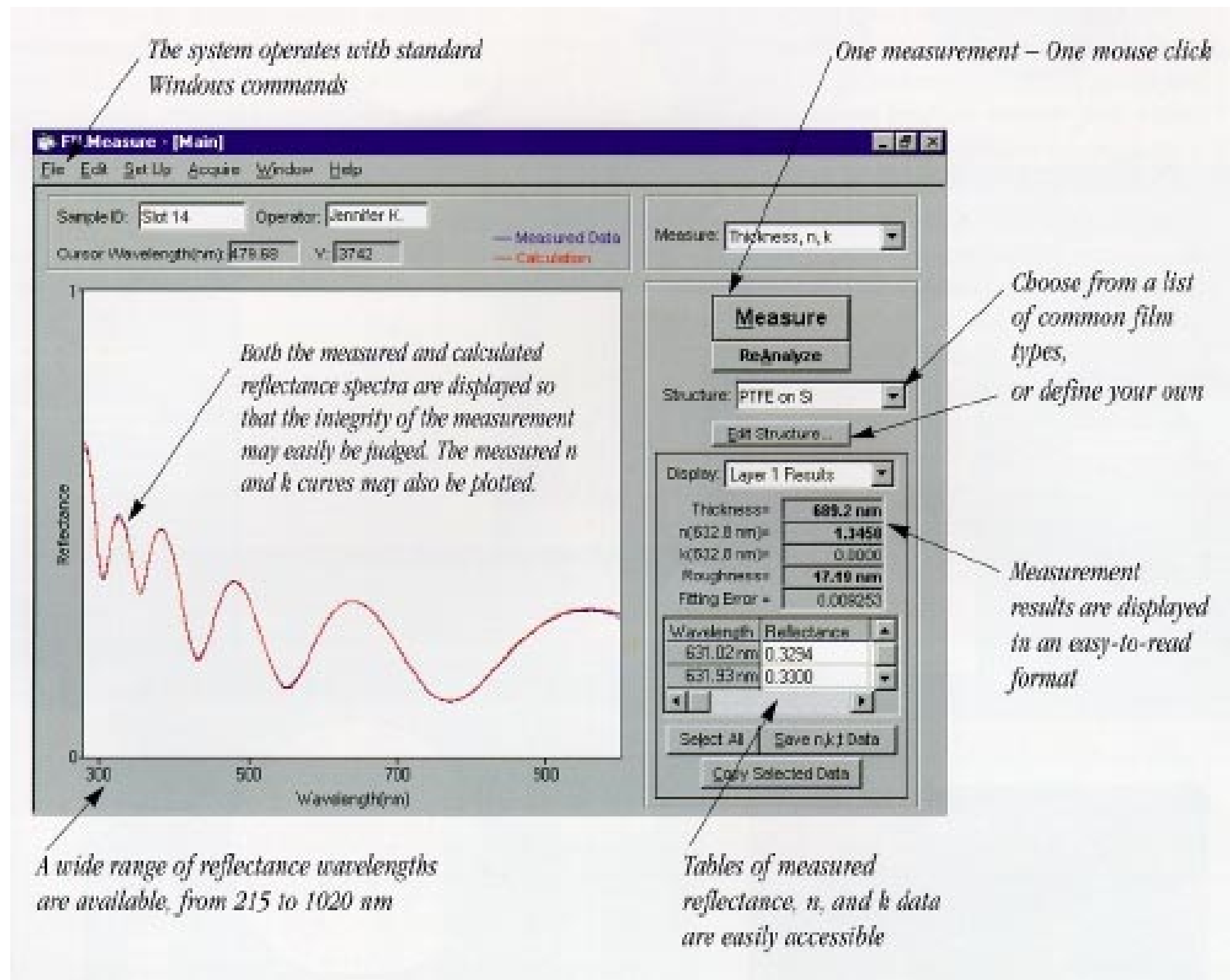


**Figure 5: Main Filmetrics data analysis window.**

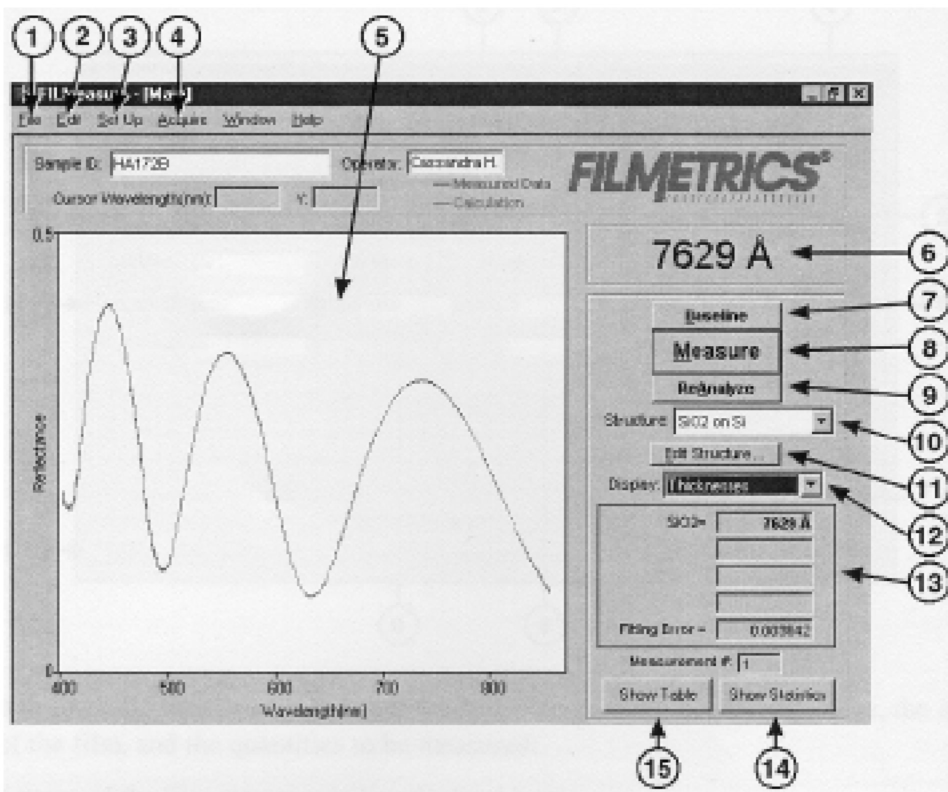


**Figure 6: FILMeasure Main Window:**

1. Standard Windows File menu for saving and retrieving data, printing, etc.
2. The Edit menu is used for copying measured spectra and measurement results, as well as selecting thickness units and editing the material library.
3. Used to setup reflectance acquisition parameters and the graphic display.
4. For starting and stopping continuous reflectance acquisition. Convenient for setting up hardware.
5. Graphical display for measured and calculated reflectance, as well as measured optical constants. Change display limits by double-clicking in the display area.
6. The measured film thickness is displayed here.
7. The baseline measurement sequence, which is required before measurements are made, is initiated by pressing the Baseline button.
8. This button causes a reflectance spectrum to be acquired and then analyzed in one step.
9. Analysis only on the displayed reflectance spectrum. Usually used when trying different analysis settings on a previously acquired spectrum.
10. Used to select the film structure that is to be measured.
11. This is where the selected film structure is described, and the analysis parameters are set.
12. Used to select the information displayed in the Results Box below.
13. The Results Box summarizes the most recent measurement results.
14. Statistical tabulation of all measurement results are accessed by pushing this button.
15. Complete results of the most recent measurement are accessed by pushing this button.

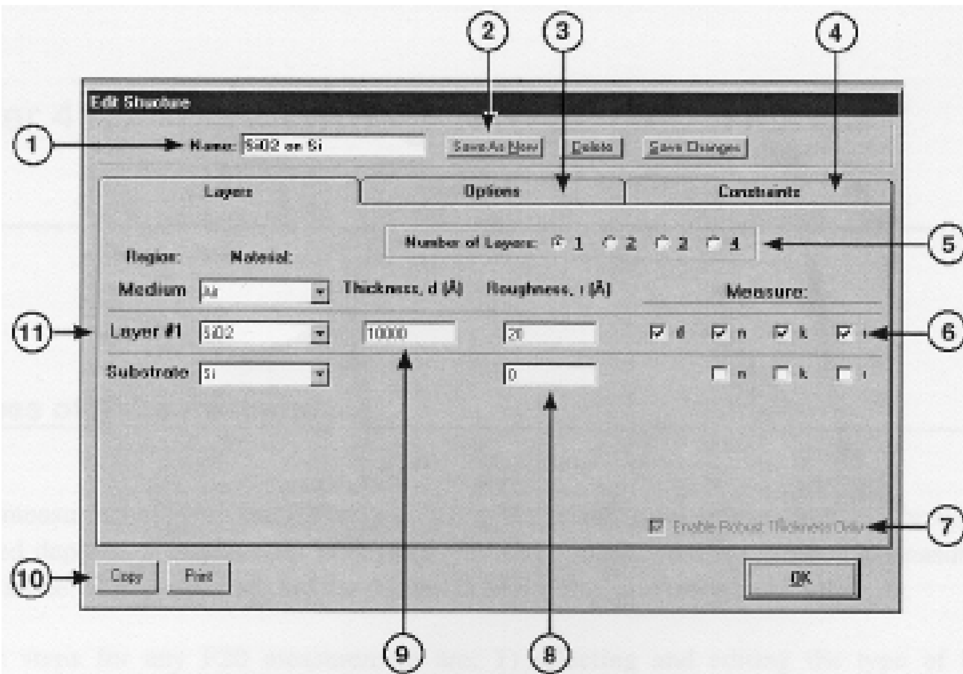


**Figure 7: Edit Structure Window**

A structure defines the film to be measured, the substrate and any underlying films, the approximate thickness of the film, and the quantities to be measured:

1. The name of the film structure is listed/edited here.

2. The structure can be saved as a new structure, deleted, or changes can be saved.

3. Advanced measurement parameters can be accessed by clicking on the Options tab.

4. Constraints on the possible measured values are selected here.
5. The number of film layers is chosen here.
6. The values to be measured are selected by checking the appropriate boxes.
7. A very robust thickness measurement routine is enabled by selecting this box.
8. Known film roughness for films not being measured, and the initial guess for the film(s) being measured are entered in this column.
9. Known film thicknesses for films not being measured, and the initial guess for the film(s) being measured are entered here.
10. All of the Edit Structure parameters can be copied or printed. This is convenient for remembering temporary setups, setting up multiple F20s, or sending measurement parameters to Filmetrics for troubleshooting.
11. The film layers are listed here. Common films can be selected from the pull-down menus.

## Example: Thickness and Optical Constants of Films on Opaque Substrates: $Si0_2$ on Silicon

For this example we will demonstrate only the measurement of $SiO_2$ on silicon, but this type of measurement has an extremely broad range of applications, including the measurement of nitrides, polysilicon, and optical coatings.

**Hardware:** Any standard or rotating sample stage should work fine. The sample frontside must be flat. If the sample backside is not flat and parallel with the frontside, then the contact stage must be used.

### Step 1: Select the film structure

Select the film structure to be measured, in this case "Si02 on Si", from the "Structure:" selection box on the main screen. If the structure to be measured does not exist, a new structure must be defined.

### Step 2: Edit the film structure

**Figure 8: Example Edit Structure:Layers window for measuring the thickness, *n,* and *k* of films on an opaque substrate.**

For this example the film structure will probably not require editing, unless a different film is being measured. To edit the structure, click on "Edit Structure" to open the dialog box. Check to see that the film sequence matches that of the actual sample. If not, different films can be selected. Also enter your best guess for the thickness of the film to be measured. Also check that thickness, n, and k of the $SiO_2$, layer are selected to be measured.

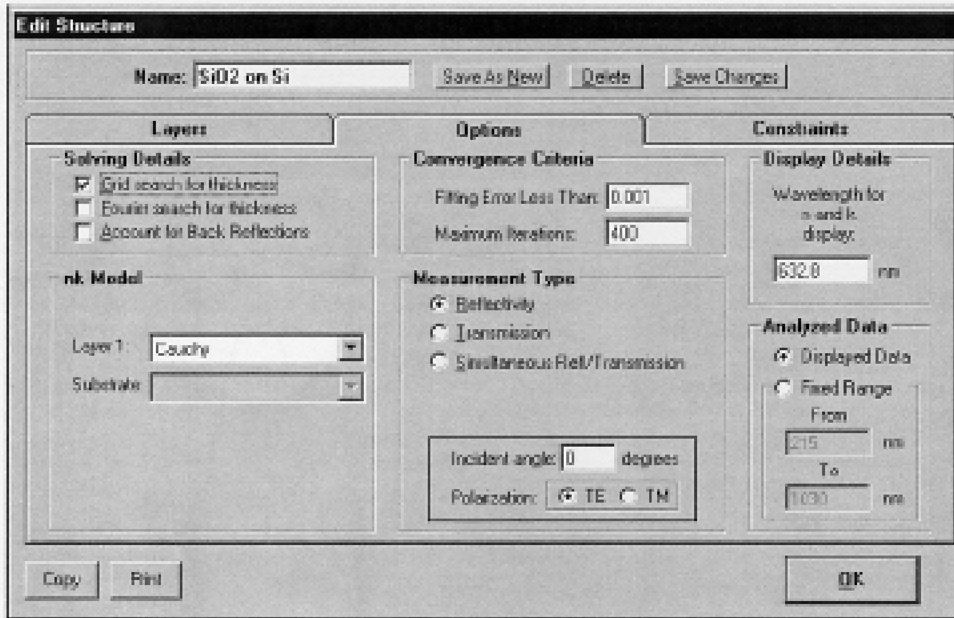


**Figure 9: Example Edit Structure:Options window for measuring the thickness, *n,* and k of films on an opaque substrate.**

**Step 3: Take a Baseline Measurement**

Take a Baseline measurement by first clicking on the Baseline button on the main screen. On the Take Baseline dialog box, make certain the "Autoscale Integration Time" option is selected, and choose the Baseline Sample that will be used (Si in this case). Then put the Silicon Baseline sample on the measurement stage and click on "Take Baseline". Then remove the Baseline sample from the stage and click "Take Zero". (In the case of a contact probe, place the probe on the Baseline sample and click on "Take Baseline", and then remove the probe from the sample and click "Take Zero".)

**Step 4: Make the Measurement**

Make the measurement by placing your sample on the stage (or the contact probe on your sample) and click on the "Measure" button. FILMeasure will then acquire the reflectance spectrum and calculate the corresponding thickness. If the measurement was successful, the calculated reflectance (the red line on the graph) will coincide with the measured reflectance (the blue line on the graph.)

If the measured and calculated spectra do not fall on top of each other, the resulting thickness, n, and k values are incorrect. If the mismatch between measured data and calculation is only slight, the results reported will only be off by a small amount. If the measured and calculated spectra match, but the results are implausible, there may be a problem with the sample positioning and light collection. Causes and corrective actions to improve the measurements are listed in cases #5

and #6 in the troubleshooting section.

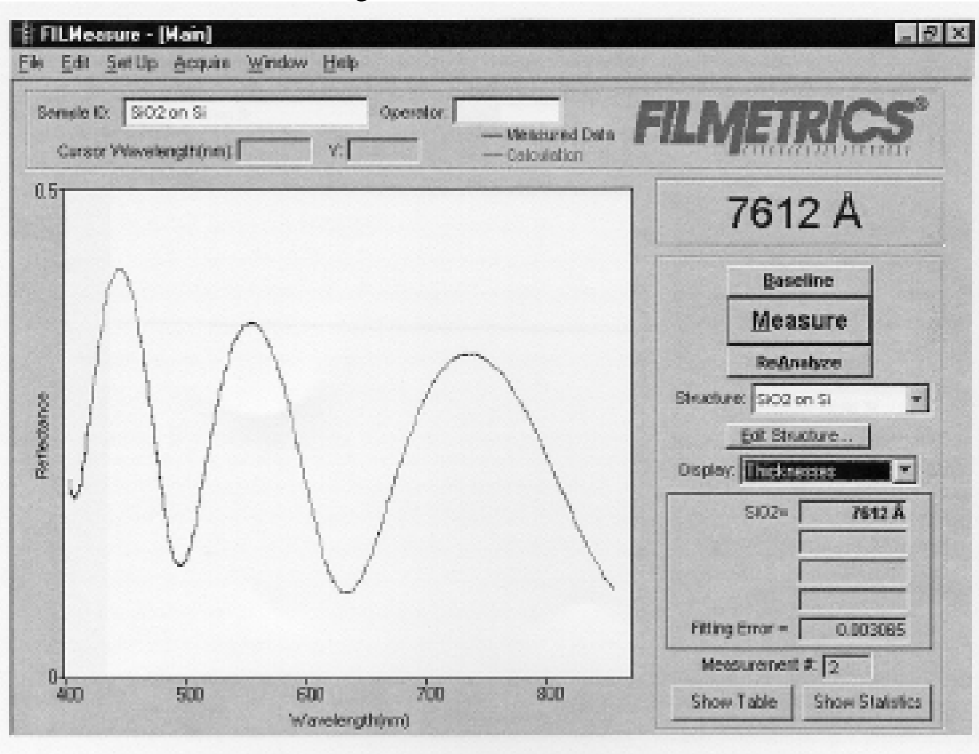


**Figure 10: Measured and calculated reflectance spectra when measuring the thickness, n, k, and roughness of $SiO_2$ on silicon.**

If the calculated (red) and measured (blue) minima and maxima do not coincide, then the measurement was not successful. There are several possible causes of an unsuccessful measurement. The most common for this type of measurement are described in cases #1, #2, #3, and #4 in the troubleshooting section of the Filmetrics manual.

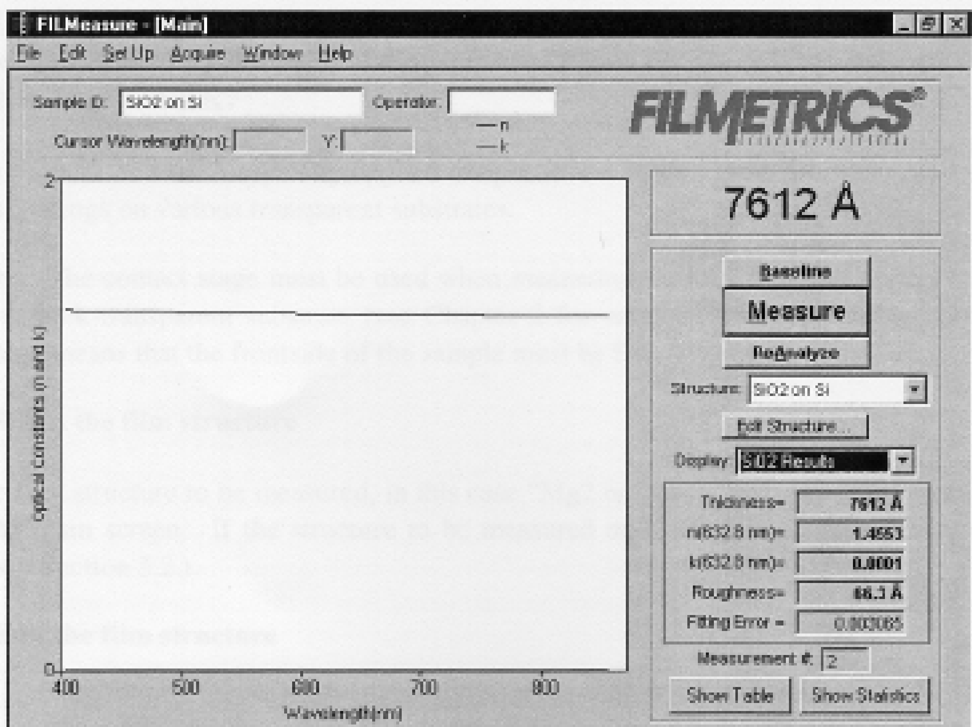


**Figure 11: Calculated n and k spectra when measuring the thickness, n, k, and roughness of $Si0_2$ on silicon.**