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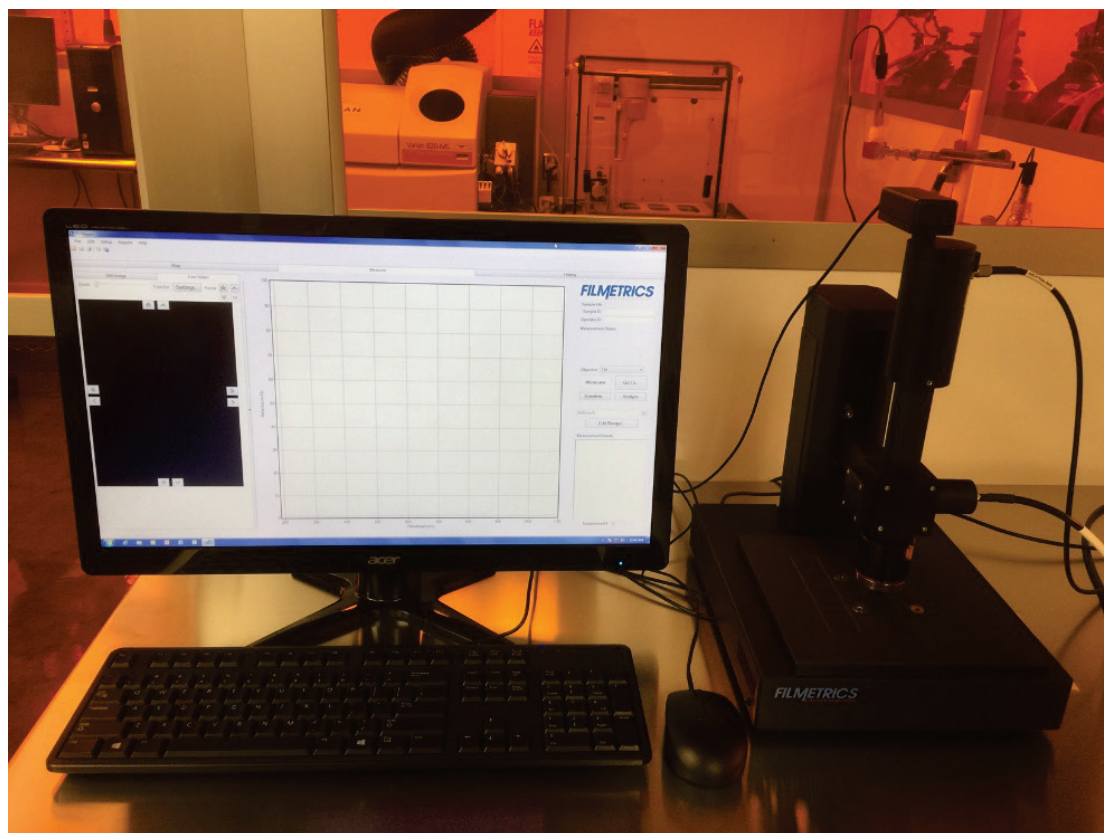
Environmental Consequences of Nanotechnologies

Determination of Nanomaterials' Film Thickness Using Filmetrics F40-UV Thin-Film Analyzer

Standard Operating Procedure Series: Characterization (C)

Qihua Wu, Kathryn Kremer, and Stephen Gibbons

July 2019



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Final report

Approved for public release; distribution is unlimited.

Prepared for U.S. Army Corps of Engineers
Washington, DC 20314-1000

Under Contract number W912HZ-15-2-0032

Monitored by Environmental Laboratory
U.S. Army Engineer Research and Development Center
3909 Halls Ferry Road, Vicksburg, MS 39180-6199

Abstract

Optical measurement techniques have been widely used for the determination of thin film thicknesses and optical constants. This standard operating procedure (SOP) presents a specific protocol for the determination of nanomaterial thin-film thicknesses and their optical constants — including the refractive index (RI) and the extinction coefficient — using a Filmetrics F40-UV Thin Film Analyzer. Procedures and recommendations of instrument preparation and parameters, sample measurement, and results analysis are included. The procedure applies to a wide range of nanomaterial thin films in the thickness range from approximately 4 nm to 25 μm .

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Preface

This SOP was developed under Task 1: “Materials evaluation and characterization” of project “Advancing Carbon Nanomaterials-Based Device Manufacturing through Life Cycle Analysis, Risk Assessment and Mitigation.” This program was funded by the Engineer Research and Development Center (ERDC). The program manager was Mr. Alan J. Kennedy and the contract number was W912HZ-15-2-0032. This work was directed by Rishi J. Patel, Senior Research Scientist at Missouri State University’s Jordan Valley Innovation Center. And this task is under the direct supervision of Dr. Wu-Sheng Shih, of Brewer Science, Inc. The technical monitor was Mr. Jerry Miller (CEERD-EL-EMJ).

The work was performed by the Environmental Risk Branch (EPR) of the Environmental Processes Division (EP), Environmental Laboratory (EL). At the time of publication, Dr. William Nelson was Branch Chief, CEERD-EPR; Mr. Warren P. Lorentz was Division Chief, CEERD-EP; and Dr. Elizabeth A. Ferguson, CEERD-EMJ was the Technical Director for Environmental Quality and Installations. The Deputy Director of ERDC-EL was Dr. Jack E. Davis and the Director was Dr. Ilker R. Adiguzel.

The Commander of ERDC was COL Ivan P. Beckman and the Director was Dr. David W. Pittman.

Acronyms

CCD: charge-coupled device

GOF: goodness of fit

µm: micrometer

min: minute

nm: nanometer

QC: quality control

QA: quality assurance

RI: refractive index

SOP: standard operating procedure

Unit Conversion Factors

Multiply	By	To Obtain
angstroms	0.1	nanometers
Microns (μm)	1.0 E-06	meters
Nanometers (nm)	1.0 E-09	meters

1 Introduction

Background

Very thin layers of materials (thin films) that are applied to a variety of surfaces are extremely important for many technology-based industries. Thin films are widely used for a range of purposes, such as for insulating layers between conductors, creating diffusion barriers, and providing protective coatings for scratch-and-wear resistance. Thin-film thickness is typically in the range from sub-nanometer (<1 nm, or $<0.001\mu\text{m}$) to hundreds of microns thick ($>100\mu\text{m}$) (Adams 1990). To ensure the film functions properly, thin films must have the proper characteristics, such as thickness, composition, and roughness. Measurements of those characteristics are essential for materials development and process evaluation in thin film-related applications.

Objective

This standard operating procedure (SOP) describes how to determine nanomaterial film thickness using a Filmetrics F40-UV Thin Film Analyzer. This SOP was developed under Task 1: “Materials evaluation and characterization” of the project “Advancing Carbon Nanomaterials-Based Device Manufacturing through Life Cycle Analysis, Risk Assessment, and Mitigation.”

Approach

There are several techniques for measuring thin film thickness, including optical techniques (Adams 1990). Generally, optical techniques determine the thin film thickness based on how light propagates through, reflects, or is absorbed by the thin film (Poelman and Smet 2003). Optical techniques are usually the preferred method for measuring thin films because they are accurate, nondestructive, and require little to 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 perpendicular to the sample surface (Poelman and Smet 2003). Ellipsometry measures the change of polarization upon reflection or transmission and compares it to

a model (Tompkins and Irene 2005). In general, spectral reflectance and ellipsometry are similar techniques. Spectral reflectance is much simpler and less expensive than ellipsometry, but it is restricted to measuring less complex structures only.

In a spectral reflectance technique, the amplitude and numbers of the reflectance of a thin film are determined by the film's thickness, optical constants, and roughness. In cases where there is more than one interface, it is not possible to determine 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, as well as the n and k model parameters, and then adjusting these values until the calculated reflectance matches the measured reflectance.

This SOP has been developed based on the manufacturer's operating manual and it applies to thin films of nanomaterials with thicknesses in the sub-micron or nanometer (nm) range. The nanomaterial thin film must be transparent or semi-transparent in order to have enough reflectance to measure.

2 Terminology

2.1 Related Documents

- Filmetrics F40 Thin-Film Analyzer Operation Manual

2.2 Definitions

- Refractive index (n): RI is defined as the ratio of the speed of light in a vacuum to the speed of light in the material.
- Extinction coefficient (k): This is a measure of how much light is absorbed in the material.

3 Materials and Apparatus

3.1 Materials

- Nanomaterial sample thin films
- NIST-traceable thickness standard (available from various vendors)
- Filmetrics-angled block (Filmetrics part number 230-0247)
- Wafer-handling tweezers

3.2 Apparatus

- Filmetrics F40-UV Thin-Film Analyzer equipped with LS-DT2 light source (deuterium and tungsten-halogen light), Filmetrics F40 spectrometer, optical light microscope with adjustable stage, and a CCD camera

4 Procedure

4.1 Experiment Preparation

4.1.1 Sample preparation

Typically, no sample preparation is needed. The nanomaterial sample film should be on a flat, clean substrate. Most samples can be directly tested as-is. If samples need to be pretreated prior to the test, record the treatment in the proper notebook.

4.1.2 Instrument Start-up

Ensure the instrument has been correctly set up, and that all optical cables are not bent. First, turn on the cooling fan on the backside of the LS-DT2 light source unit. Turn on the tungsten-halogen lamp by pressing the red button on the front panel. The button will latch in place (toggle button) and the status indicator will illuminate continuously. Next, turn on the deuterium (D2) lamp by pressing the blue button on the front panel. The D2 lamp will enter an automated start-up sequence where the indicator will flash slowly at the beginning. When the lamp ignites, the indicator will illuminate continuously. Switch on the computer with F40 software installed. The F40 spectrometer and microscope always stay on.

The F40-UV Thin-Film Analyzer has measurement specifications as follows:

- Wavelength range: approximately 200 to 1100 nm
- General Precision: 0.1 nm
- Stability: 0.07 nm
- Thickness range: approximately 4 nm to 2 μm
- Spot size: 33 μm (with 500- μm Aperture)

4.2 Sample Measurement

4.2.1 Select Film Recipe

A film recipe is the defined model that will be used to process the reflection data of the captured image and thus to calculate the target film thickness.

Select the film structure to be measured from the recipe list on the main screen. For unknown materials, n and k files need to be created for the recipe. Refer to Filmetrics F-40 operation manual for detailed procedure of obtaining n and k values for unknown materials.

4.2.2 Edit the Film Recipe

To edit the film stack, open the “**Measure**” Tab and click on “**Edit Recipe**.” The “**Edit Recipe**” dialog box will be opened as shown in Figure 1. This dialog box shows the stored specifications of the structure from the given recipe. Check to see that the film stack matches that of the current sample. Different films can be selected using either the drop-down menu next to the material or through the “**Search**” function.

A new layer of material can be added by clicking the “+” button. An existing layer can be deleted by clicking the “x” button. Note that the “substrate” and “medium” always need to be defined. Once the changes have been made, the recipe can be saved by clicking on the save icon.

If n and k values also need to be measured, enable the optical-constants-solving function under the “**Composition**” options.

Figure 1. Material library page.

The screenshot shows the 'Edit Recipe' dialog box with the 'Film Stack' tab selected. The recipe name is 'SiO2 on Si'. The film stack is defined by three layers: 'Medium' (Air), '1' (SiO2), and 'Substrate' (Si). The SiO2 layer has a nominal thickness of 10000 Angstroms with a range of +/- 100. The 'Analysis Options' tab is selected, and the 'Composition' section is visible. The 'Units' are set to 'Angstroms (Å)'. The 'Thickness' section shows 'Nominal (Å)' as 10000, 'Range (%)' as +/- 100, and 'Meas.' as checked. The 'Refine via' dropdown is set to 'Grid'. The 'OK', 'Cancel', and 'Apply' buttons are at the bottom.

Layer	Composition	Nominal (Å)	Range (%)	Meas.	Refine via
Medium	Air				
1	SiO2	10000	+/- 100	☒	Grid
Substrate	Si				

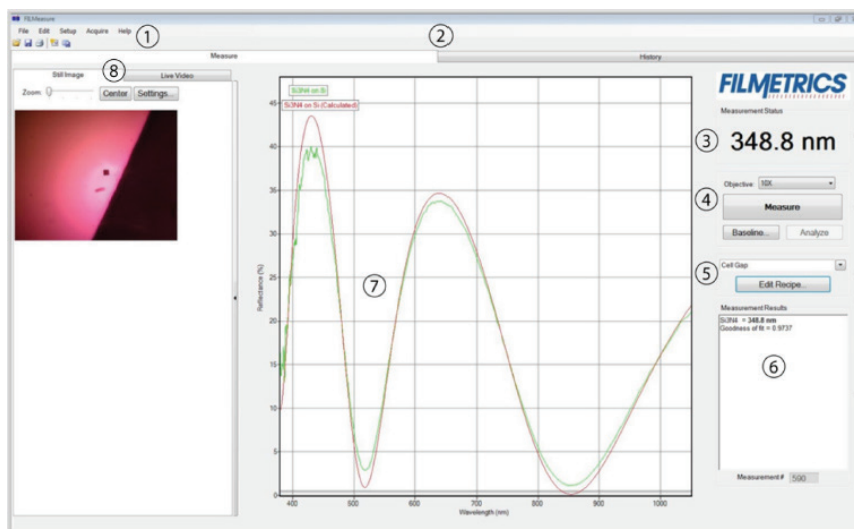
4.2.3 Baseline Measurement

A baseline measurement should be performed before measuring any sample. Obtain a baseline measurement by clicking on the “**Baseline**” button on the main screen. A dialog box will appear. Follow the steps on the screen to perform a baseline measurement. Be sure to use the Angled BK7 reflectance standard when the dialog asks to do so.

4.2.4 Sample Measurement

Open both the field diaphragm and the aperture diaphragm located between the light source and the microscope column. In addition, turn on the shutter by depressing the “**Shutter**” button on the light source. Focus the image by adjusting the distance from the sample to the objective. To obtain a sample measurement, place the sample on the stage and click on the “**Measure**” button on the main page. The software will then acquire the reflectance spectrum and calculate the corresponding thickness. If the measurement was successful, the calculated reflectance will coincide with the measured reflectance.

Figure 2. Main page: (1) standard menus, (2) measurement and history tab, (3) calculated thickness result, (4) Measure button, (5) Edit Recipe button, (6) detailed information, and (7) reflectance spectra.



4.2.5 Shut-Down

When the measurement is completed, save or record all results and close the software. Turn off the D2 lamp and halogen lamp, and leave the cooling fan on for an additional 15 min.

5 Reporting

5.1 Analysis of Results

After testing the sample, the measured and calculated reflectance spectra will be displayed on the graph, and the measured film thickness will be listed in the results box in bold numbers. Report the calculated thickness and the reflectance spectra.

5.2 QA/QC Considerations

The quality and accuracy of the result is determined by how well the measured reflectance spectrum matches with the calculated spectrum. This can be judged by the goodness-of-fit (GOF) value, which is a number between 0 and 1. A GOF close to 1 indicates a good match between measured and theoretical spectra.

References

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- Poelman, D., and P. F. Smet. 2003. Methods for the determination of the optical constants of thin films from single transmission measurements: a critical review. *Journal of Physics D: Applied Physics* 36(15): 1850.
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REPORT DOCUMENTATION PAGE					Form Approved OMB No. 0704-0188	
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1. REPORT DATE July 2019		2. REPORT TYPE Final			3. DATES COVERED (From - To)	
4. TITLE AND SUBTITLE Determination of Nanomaterials Film Thickness Using Filmetrics F40-UV Thin Film Analyzer: Standard Operating Procedure Series: Characterization (C)				5a. CONTRACT NUMBER W912HZ-15-2-0032		
				5b. GRANT NUMBER		
				5c. PROGRAM ELEMENT NUMBER		
6. AUTHOR(S) Qihua Wu, Kathryn Kremer, and Stephen Gibbons				5d. PROJECT NUMBER		
				5e. TASK NUMBER 4		
				5f. WORK UNIT NUMBER		
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Brewer Science 2401 Brewer Drive, Rolla, MO 65401				8. PERFORMING ORGANIZATION REPORT NUMBER ERDC/EL CR-19-1		
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES) U.S. Army Corps of Engineers Washington, DC 20314-1000				10. SPONSOR/MONITOR'S ACRONYM(S) USACE		
				11. SPONSOR/MONITOR'S REPORT NUMBER(S)		
12. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution is unlimited.						
13. SUPPLEMENTARY NOTES						
14. ABSTRACT Optical measurement techniques have been widely used for the determination of thin film thicknesses and optical constants. This standard operating procedure (SOP) presents a specific protocol for the determination of nanomaterial thin-film thicknesses and their optical constants — including the refractive index (RI) and the extinction coefficient — using a Filmetrics F40-UV Thin Film Analyzer. Procedures and recommendations of instrument preparation and parameters, sample measurement, and results analysis are included. The procedure applies to a wide range of nanomaterial thin films in the thickness range from approximately 4 nm to 25 μm.						
15. SUBJECT TERMS extinction coefficient, nanomaterial thin-film, optical constant, optical measurement techniques, refractive index						
16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT SAR	18. NUMBER OF PAGES 19	19a. NAME OF RESPONSIBLE PERSON Alan Kennedy	
a. REPORT Unlimited	b. ABSTRACT Unlimited	c. THIS PAGE Unlimited			19b. TELEPHONE NUMBER (Include area code)	