Figure 1. Light scattered by a silicon wafer has a characteristic polarization that is a signature of the scattering mechanism, in this case microroughness.
optics has a wide range of applications in industry and science, and the properties of the materials that make up optical instruments are key to their effectiveness. Over the past century, optical instruments have increased in sophistication and performance. The National Institute of Standards and Technology (NIST) plays an essential role in optics development by providing information on the optical properties of various materials. These include glass for telescopes and cameras, semiconductors for electronics, ultraviolet materials for photolithography, and infrared materials for remote-sensing and defense applications.

NIST has developed instruments and measurement methods to obtain data of the highest level of accuracy. It provides industry, government, and academia with calibration services and makes the physical standards available to a wide variety of users. As part of its mission, NIST deals with the determination of optical properties of materials. NIST continues to refine and improve its capabilities, including the development of a theoretical basis for understanding optical properties, to meet the future critical needs of science and industry.

This article will highlight steps NIST has taken to advance the field of optical sciences and technology and give readers a sense of the wide range of activities relevant to the broad category of "optical properties of materials." Specifically, the article will address NIST’s efforts in four areas:

- optical scatter measurements;
- optical constants measurements in the ultraviolet region that are critical for photolithography;
- infrared filter characterization for remote sensing and defense applications;
- improved theoretical methods that can help predict optical constants from first-principles calculations.

These examples comprise a small portion of NIST’s work in the field.

**Optical scatter measurements**

Light scattering has numerous negative effects and positive applications, in that it can degrade the performance of optical systems, enable diagnostic instruments to locate and identify defects, and determine the visual appearance of paints and coatings. The resolving power of high-quality optical systems is often limited by scattering from optical elements as well as from the supporting structure and baffles. Commercial software can help predict optical performance if the scattering function for all the materials in an optical system are known. The semiconductor industry uses light scattering to rapidly scan silicon wafers for defects and particulate contaminants. As device get smaller, detecting defects becomes harder. But finding defects—and detecting the source of the problem—is key to producing products with high yield.

For many years, NIST has conducted research on the measurement and understanding of light scattering. During the 1970s, Nicodemus defined the concept of the bidirectional reflectance distribution function (BRDF), which encapsulates the intensity scattered from a surface, as a function of incident and scattering directions. He further defined various concepts, such as directional-hemispherical reflectance, in terms of the BRDF. Standard materials, which behave like uniform scatterers over various wavelength regions, were developed. While light scattered in the plane of incidence is easy to measure, measurement of light scattered out of the plane of incidence usually requires more complicated instrumentation.

It is not enough to characterize the intensity of scattered light. Scattering also affects polarization, which can yield specific information about the scattering sources. Recently, NIST has led development of theoretical models and experimental techniques showing how different scattering sources—interfacial roughness, subsurface defects, or particulate contaminants—can be differentiated by analyzing polarization. Based on the Rayleigh approximation, such analysis can be viewed in a simple manner. When light is incident obliquely on a surface with its electric field in the plane of incidence (i.e., s-polarized), the electric field above the surface tends to be more normal to the surface, while that below the surface tends to be more parallel to the surface. Therefore, a small particle above the surface tends to radiate like an antenna normal to the surface, while that below the surface radiates like an antenna parallel to the surface. A viewer observing the scatter from a direction out of the plane of incidence need only rotate a polarizer to determine the location of the scattering source.

Scattering from small amounts of roughness can be understood in a similar way; it behaves as if dipoles are created by the field above the surface yet radiate from below the surface. Figure 1 shows the behavior of light scattered by microroughness when the substrate has the optical constants of silicon. Experimental measurements have confirmed that the light scattered by different sources indeed follows the predictions of the respective theories. The technological implications of the NIST findings have resulted in im-
proven detection of defects on silicon wafers. Future work in scatterometry includes the development of methods to characterize coatings and films. Recent results, for instance, have shown that light-scattering ellipsometry can be used to extract the roughness of both interfaces of a dielectric film.3

Optical constants measurements in the ultraviolet region

Since it was founded in 1901, NIST* has served U.S. national needs related to optical materials. Early on, NBS was responsible for much of the domestic optical glass production and for characterizing various applications, including bomb sights.4 NIST’s current mission—to enhance American economic competitiveness and technological development—has placed the agency at the heart of the semiconductor industry, in the area of photolithography.

A key problem in fabricating high-precision optics that transmit in the ultraviolet (UV) is characterizing the optical properties of transmissive materials such as the index of refraction, its dependence on wavelength and temperature, and transmittance. The need for accurate data continues to increase, especially as optical systems are designed to achieve higher resolution and to operate at shorter wavelengths (e.g., 157 nm). Accurate data have become indispensable, especially to today’s microelectronics industry, which is rapidly extending photolithography to the deep ultraviolet. Projection optics for lithography must be able to achieve diffraction-limited performance with high numerical apertures and extremely wide fields of view. Under these design rules, optical constants of materials must be known to a few parts per million.

The most accurate measurements of the index of refraction of solid materials in the UV can be performed using the minimum deviation method. This method works by fabricating a prism with an accurately known apex angle, and measuring the minimum deviation angle of a monochromatic beam of light that passes through the prism. An NIST team, for instance, used this method to obtain high-accuracy values of the index and its dependence on wavelength and temperature. The accuracy of the measurements made around the wavelength of 193 nm was sufficient to distinguish between grades of fused silica that were produced by different manufacturers, pointing the industry to the need for better material specifications5 (Fig. 2). The technique has been extended to shorter wavelengths to make critically needed measurements of the index of refraction for calcium fluoride and other materials around the wavelength of 157 nm.

As the wavelength of interest for photolithography becomes shorter, all the design parameters become more stringent—requiring increasingly accurate characterization of the optical properties of transmissive materials. The NIST team, using an interferometric technique based on a UV Fourier transform spectrometer and the broadband radiation from an in-house synchrotron source, is at the forefront of index of refraction measurements. An accurate count of the fringes produced in a flat, plane-parallel piece of optical material has the potential of yielding a value of the index of refraction with an accuracy of a few parts per million.

Infrared filter characterization

Fourier Transform (FT) methods for optical properties characterization in the infrared offer a number of well-known advantages including high spectral resolution and signal-to-noise ratio. However, FT methods are also prone to numerous sources of measurement error6 that generally require correction prior to FT processing. Hence, FT spectrophotometry’s application to accurate characterization of optical materials and components, and generation of reference data and standard artifacts, requires a thorough evaluation of all the important sources of error as well as the subsequent development and implementation of methods for reduction of those errors.

Standard artifacts for infrared transmittance (neutral-density filters) and wavelength/wavenumber calibration have been developed at NIST’s FTIR (Fourier transform infrared) Spectrophotometry Laboratory.7 In the process, primary sources of error, including detector system non-linearity and sample-interferometer and sample-detector inter-reflections, have been evaluated and minimized.8

Several systems employing different methods for the determination of the complex index of refraction measurements have been developed and established. These include absolute spectral transmittance and reflectance, ellipsometry, and spectral fringe analysis techniques (also used in the ultraviolet, as described

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* Originally known as the National Bureau of Standards (NBS).

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Figure 3. Transmittance of a 4 µm narrow-band filter at 25 K. Measurements were required from 1.6 µm to 80 µm for use with a cryogenic cavity radiometer. This plot is the combined result of six separate measurements covering different sections of the spectrum, as determined by the spectral ranges of the detectors, beam splitters, sources, blocking filters, and regions of high transmission of the sample filter. Regions where the noise level determines the base level of the measurement appear as structure that extends to the bottom of the plot (i.e., to 0).
The laboratory uses multiple methods of measurement for much of its work because this allows for method comparisons, validation of uncertainties, and method refinement—all of which yield higher-accuracy results.

A critical component of many sensor and radiometer systems is the filters used to define the spectral regions of interest. In the infrared, such systems are used in numerous defense and remote-sensing systems on the ground, in the atmosphere, or in space. Often, the detectors are sensitive over a much broader spectral range than the filter passband. Even at low levels, for instance, the out-of-band transmittance of the filters can trigger incorrect and misinterpreted results when integrated over the complete detector range. In the worst-case scenario, such transmittance can render the measurement useless. Knowledge of the in-band spectral transmittance is equally important for sensors looking at infrared flux that varies rapidly within the filter passband—in the short-wavelength part of a Planckian blackbody distribution, or adjacent to atmospheric absorption lines. Furthermore, most infrared bandpass filters have both an angular dependence and a temperature dependence.

NIST has developed a methodology for characterizing infrared narrow-band filters at temperatures from 10 K to 300 K for frequently used incident-beam geometries, and employed it to support a number of Department of Defense, NASA, and National Oceanic and Atmospheric Administration programs. An example of combined in- and out-of-band filter measurements in semi-log format is shown in Fig. 3. Through the use of transfer standard neutral density filters and passband blocking filters, the out-of-band transmittance can be measured down to levels of $10^{-5}$ to $10^{-7}$, depending on the wavelength. Use of the supplementary filters allows sample and reference measurements to be made at the same time that operation of the cryogenic detectors in linear regimes is being maintained. The in-band transmittance level is measured to high accuracy at ambient conditions using custom absolute transmittance–reflectance instrumentation. It is important to take the time to select the right measured interferogram type and to pay attention to the details of the phase correction and apodization functions used in the FT processing that produces the final spectra. The ambient results are used to calibrate measurements obtained with optical cryostats at the required temperature and angle. Often, additional measurements are performed to characterize the temperature and incident-angle dependencies.

NIST filter-characterization results are used to help DoD and NASA contractors assess their performance. The results also help filter manufacturers design and fabricate filters critical to several satellite sensor systems. Finally, accurate filter transmittance data are used to improve the quality of the measurements made by the sensor systems themselves.

Improving theoretical methods for optical constants predictions

It is often helpful to compare measurement results with the results of theoretical and numerical modeling. For instance, theoretical analysis can help determine whether features of a given spectrum are intrinsic to a material or are extrinsic artifacts resulting from impurities, defects or the measurement apparatus itself. NIST is working to improve researchers’ ability to predict optical properties of materials (e.g., optical constants) from first principles—an effort that complements laboratory measurements and their refinements.

In contrast, the use of empirical models with adjustable parameters that can be fit to measured optical properties allows for sensible extrapolation of measurement results to infer properties that were not actually measured, e.g., the value of the refractive index at a given wavelength. However, a first-principles approach can enable researchers to anticipate the optical properties of materials before they are measured.

Although the overall systematic trends are impressive, it is doubtful that modeling will ever be able to achieve the same level of accuracy now realized in measurements of optical constants. Consider, for instance, the dielectric function for periclase (magnesium oxide, or MgO) in Fig. 4, where measured and theoretical values are shown. It is important to note the qualitative and quantitative correspondence of spectral features. Even a qualitative accounting for spectral features requires detailed numerical analysis of the optical excitation process that was realized only recently at NIST and independently by others.

Much of the work on first-principles modeling of optical constants has emphasized the visible and ultraviolet regions of the spectrum and the role of electron inter-band transitions. However, the index dispersion throughout the infrared region is equally relevant, especially in view of the rapid pace of developments in infrared technologies. One of NIST’s goals is to develop a predictive model for optical constants in the far-infrared region, where optical absorption is governed by single- and multi-phonon processes.

To achieve this goal, two criteria must be met. First, we must understand the complex, anharmonic inter-atomic forces that govern lattice dynamics. Second, we must be able to solve the equations of motion for the dynamical system of atoms vibrating in a lattice, which is governed by such forces, to determine the response to light in detail. Such a solution presents potentially unsolvable problems that must nonetheless be addressed within wisely chosen approximations. One way to address this could be to treat multi-phonon excitations statistically and to limit one’s evaluation to various moments of the ab-
sorption spectrum instead of attempting to describe the total dynamical system.

The work highlighted in this article illustrates but a few of the challenges facing NIST scientists in the field of optical properties of materials. It represents only a snapshot of NIST’s efforts to maintain a key role in optics in general and to develop the skills, knowledge, and experience necessary to accomplish the task today and in the future.

References


