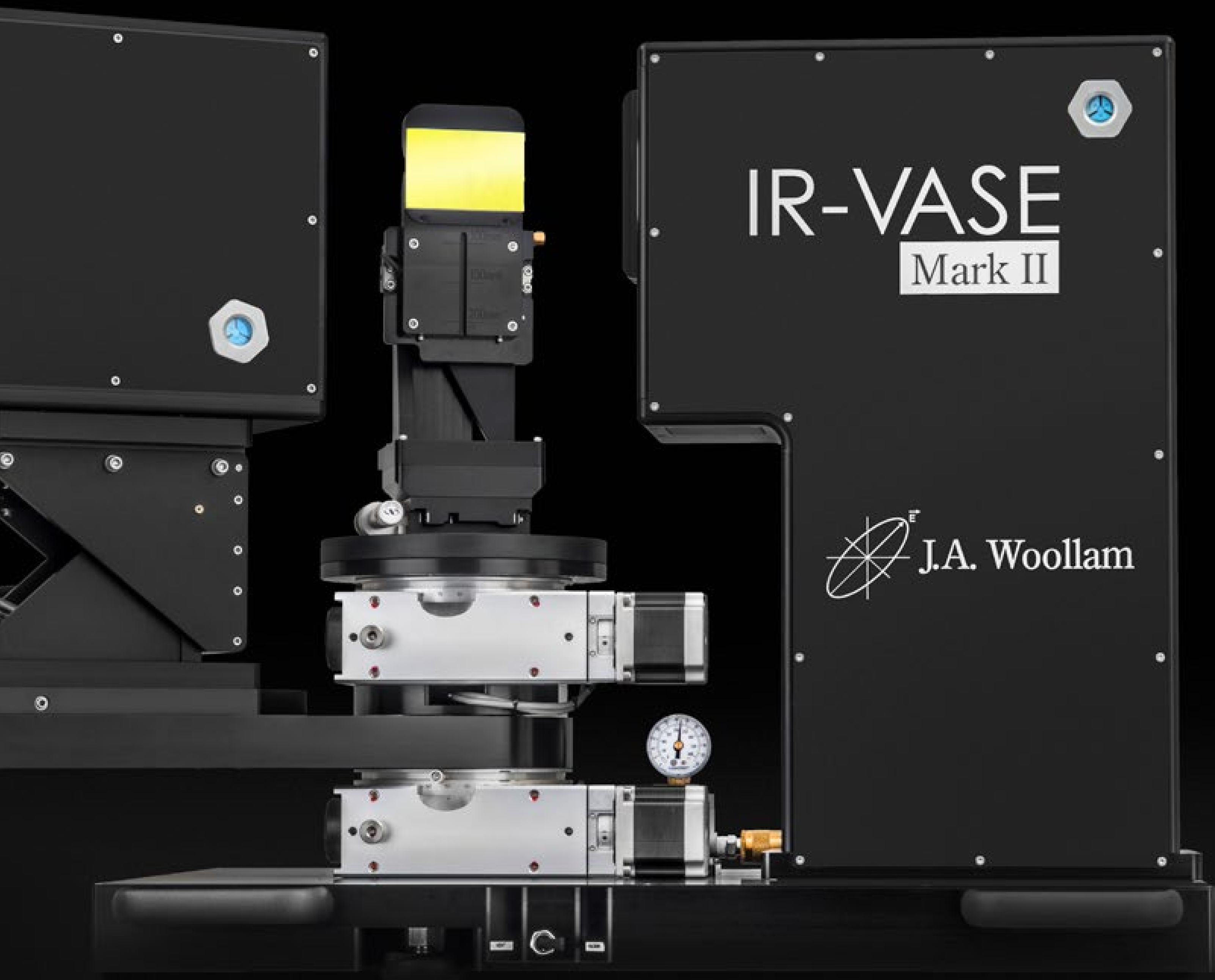
IR-VASE



J.A. Woollam

Ellipsometry Solutions


$\vec{E}$
Accurate

Overview

The J.A. Woollam IR-VASE was first introduced in 1998 and has undergone incremental improvements over the past 25 years. It is recognized worldwide as the premier thin film characterization tool in the infrared spectral region. In 2013, the IR-VASE Mark II was released, which completely updated its functionality and improved its design.

The new Mark II design is half the size of its predecessor, requiring less benchtop space and using less purge gas. The globar light source and scanning laser inside the FTIR are designed for a much longer lifespan, reducing the need for maintenance. The Mark II design is also easier to construct and service.

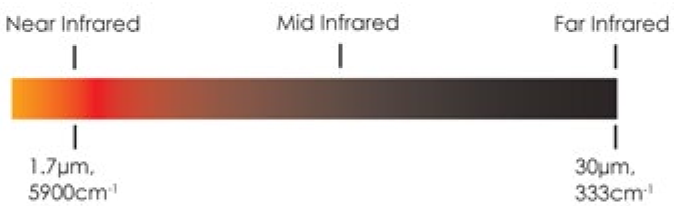
Performance advantages include shorter optical path length and smaller measurement spot size. The Mark II obtains more accurate and repeatable data than ever before.

The IR-VASE Mark II remains capable of measuring both ellipsometry and intensity data in reflection or transmission mode and is compatible with popular IR-VASE accessories such as a cryostat and heat stages.

Why IR-VASE

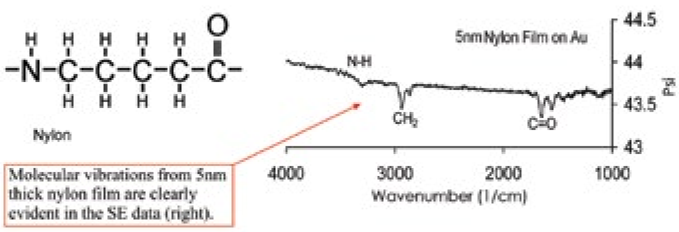
+Wide Spectral Range

Near to far infrared: 1.7 to 30 microns (333 to 5900 cm^{-1})
User-specified resolution from 1 to 64 cm^{-1}



+High Sensitivity to Film Thickness

Spectroscopic ellipsometry data contains both "phase" and "amplitude" information from reflected or transmitted light. IR ellipsometry offers higher sensitivity to film thickness than intensity-based FTIR reflection or absorbance measurements, while retaining sensitivity to chemical composition.



+Non-destructive Characterization

The IR-VASE offers non-contact, non-destructive measurements of many different material properties. Measurements do not require vacuum and can be used to study liquid/solid interfaces common in biology and chemistry applications.

+No Baseline Sample Required

Ellipsometry is a self-referencing technique that does not require reference samples to maintain accuracy. Samples smaller than the beam diameter can be measured because the entire beam does not need to be collected.

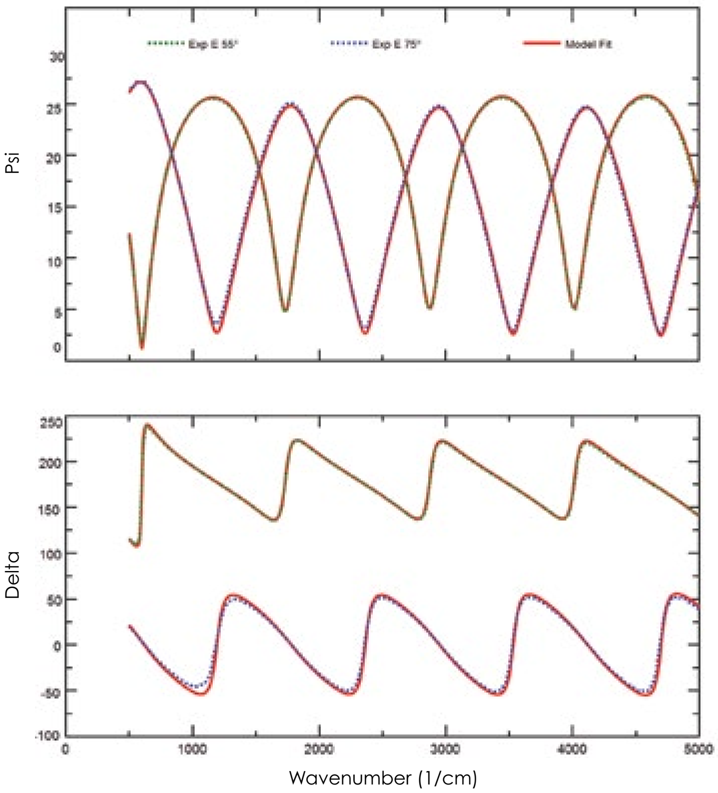
+Highly Accurate Measurement

Patented calibration and data acquisition procedures provide accurate measurements of Ψ and Δ over the full range of the instrument. The IR-VASE can determine both n and k for materials over the entire spectral range from 1.7 to 30 microns without extrapolating data outside the measured range, as with a Kramers-Kronig analysis. Perfect for thin films or bulk materials including dielectrics, semiconductors, polymers, and metals.

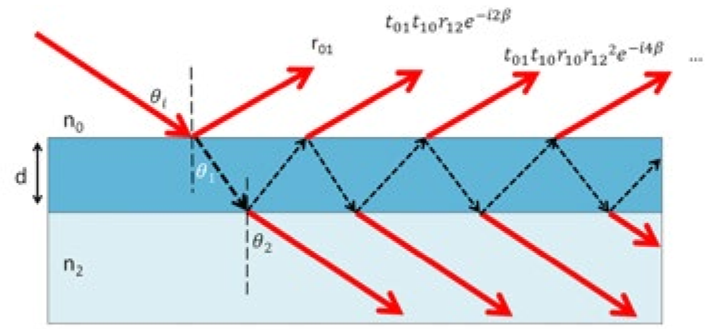
Basic IR Ellipsometry: Thickness + Index

Determining the thickness and index of a transparent film is one of the easiest measurements to make with an IR-VASE. The thickness and refractive index of the film can be determined by simply matching the amplitudes and periods of the oscillations seen in the Ψ and Δ vs. wavenumber spectra, assuming that the index of the substrate is already known.

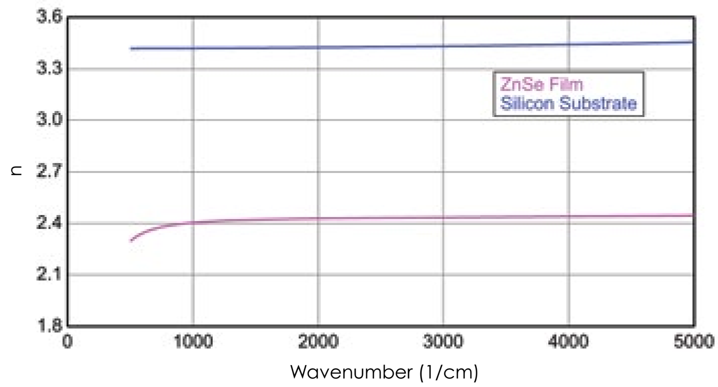
During measurement, the incident light separates into multiple rays that reflect from the top and bottom interfaces of the film. Each of the rays travels with a different path length, causing each to have a different phase. The phase differences produce constructive or destructive interference depending upon the wavelength, film thickness, index, and angle of incidence. The amount of light reflected from each interface depends upon the refractive index difference (or "index contrast") of the materials on either side of the interface. Larger index contrast results in higher reflection at that interface. The phase of the reflected electric fields depends upon the ratio of two indices. The strength and phase of interface reflection also depends upon the angle of incidence and polarization.



Measured ellipsometric data and corresponding model data showing interference oscillations caused by constructive and destructive interference in thin films (both graphs shown above).



Schematic ray tracing of the electromagnetic wave through a sample.



Refractive index of the ZnSe film, obtained by analysis of the raw Psi and Delta data shown on the left. For comparison, the index of the silicon substrate is also shown.

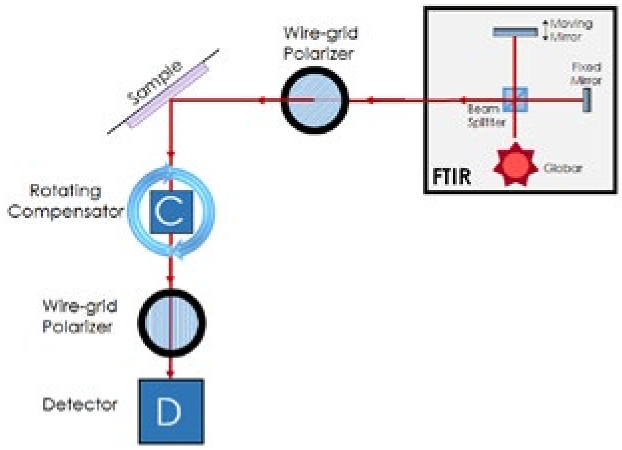
Capabilities + Technology

The IR-VASE spectroscopic ellipsometer combines the chemical sensitivity of FTIR spectroscopy with the thin film sensitivity of spectroscopic ellipsometry. Covering a wide spectral range from 1.7 to 30 microns (333 to 5900 wavenumbers), it is used to characterize both thin films and bulk materials in research and industry. This analytical technique is finding a growing number of applications in the optical coatings, semiconductor, biological, and chemical industries, as well as material research labs. The IR-VASE is often used to characterize:

- Bulk materials
- Optical constants (n and k , ε_1 and ε_2)
- Film thickness (single and multiple layers)
- Material composition (alloy fraction)
- Chemical bonding and molecular vibrations
- Phonon absorption in crystalline materials
- Surface and interfacial layers
- Doping concentration (resistivity)
- Free carrier absorption
- Anisotropy (uniaxial and biaxial)

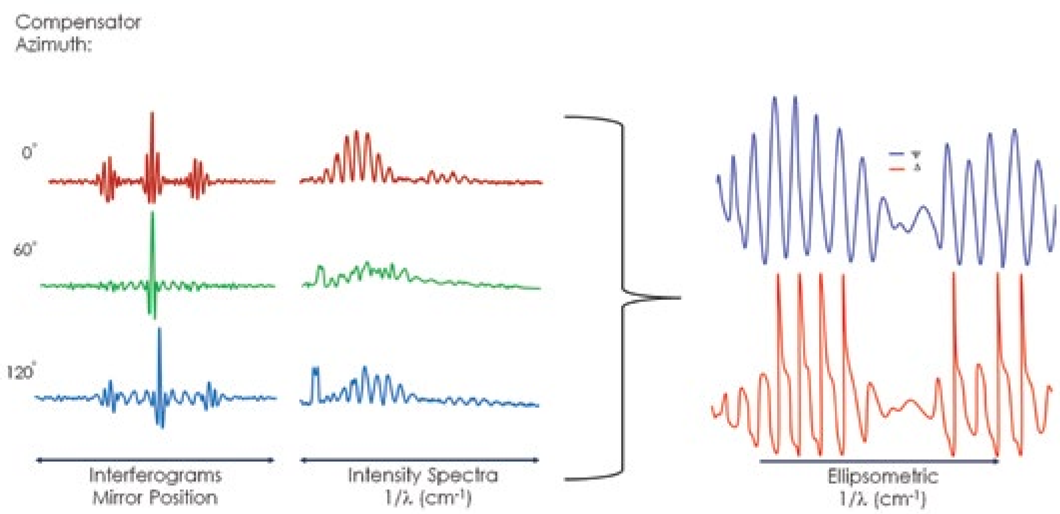
Optics

The IR-VASE Mark II integrates a Fourier Transform Infrared (FTIR) interferometer source with a rotating compensator ellipsometer to provide accurate ellipsometric measurements. Wideband polarizers and patented compensator design are combined with an optimized beam splitter, collimators, and a DTGS detector to provide the widest available spectral range in a commercial infrared ellipsometer.



Measurement

During measurement, the compensator is rotated 360° in a series of steps, and an intensity spectrum is recorded at each step as shown below. The intensity spectrum is the result of the optical properties of the sample in combination with the compensator and polarizers. The Psi and Delta spectra (or other ellipsometric quantities) for the sample can then be calculated from a combination of intensity spectra from each position of the compensator.



Advanced

This advanced technology also offers unambiguous relative phase (Delta) measurements from 0° to 360°, automated angle of incidence from 32° to 90° for optimum data on any material, and advanced measurement capabilities including anisotropy, Mueller-matrix, and depolarization data. A patented calibration routine produces the highest data quality available. These advances allow the IR-VASE to excel under nonideal measurement conditions including sample nonuniformity, patterned regions, incomplete film coverage, and double-side-polished substrates.

Information in the Infrared

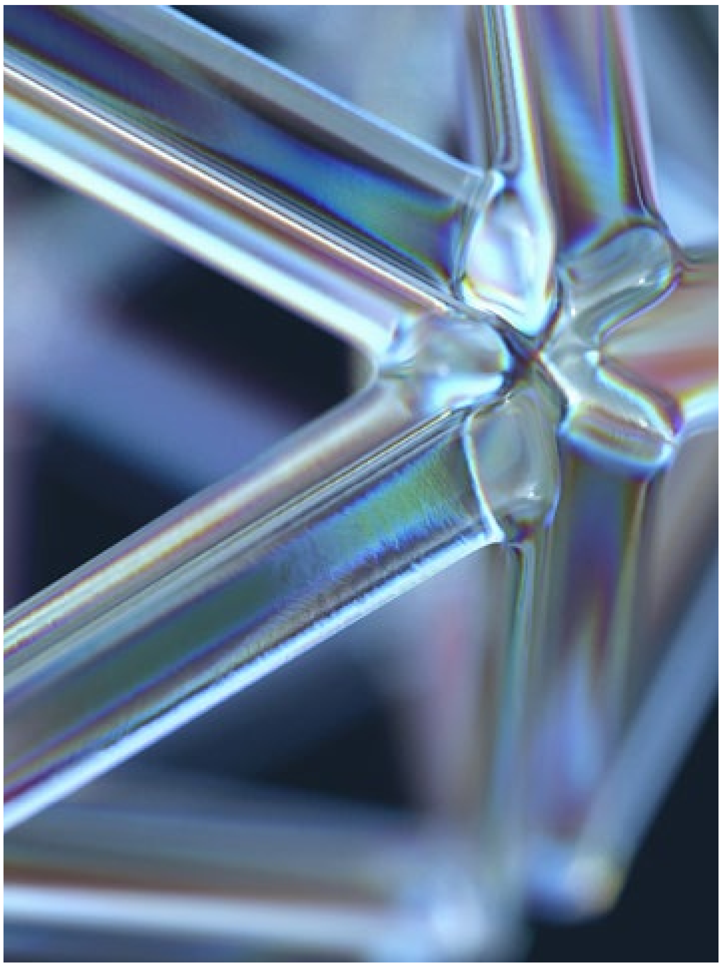
Many of the features observed by infrared spectroscopic ellipsometry are related to the following absorption mechanisms:

1. Vibrational Absorption

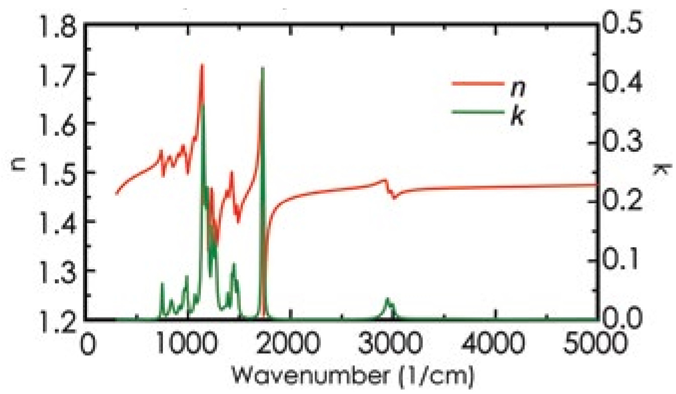
Most materials have regions of transparency that are separated by one or more absorbing regions, which are the result of vibrational resonances (see figure to the right for the Acrylic film).

Vibrational absorption occurs when molecules and lattices resonate at infrared frequencies. These absorptions can act as fingerprints of the materials, as the frequencies of resonance depend on the weight of resonating atoms or molecules as well as the types of bonds between them. Organic materials exhibit well-defined molecular vibrations. Vibrational absorptions also occur in many dielectrics and semiconductors that have polar bonds.

For amorphous and other glassy materials, the atomic bond lengths and angles are distributed randomly, so these absorptions tend to have a Gaussian character (see lower right figure for silica). In highly-ordered materials such as crystalline dielectrics and semiconductors, the vibrational resonances are no longer localized to individual molecules and thus become extended lattice vibrations. These tend to have strong Lorentzian line shapes with very narrow broadening (see figure on the right for crystalline SiC).

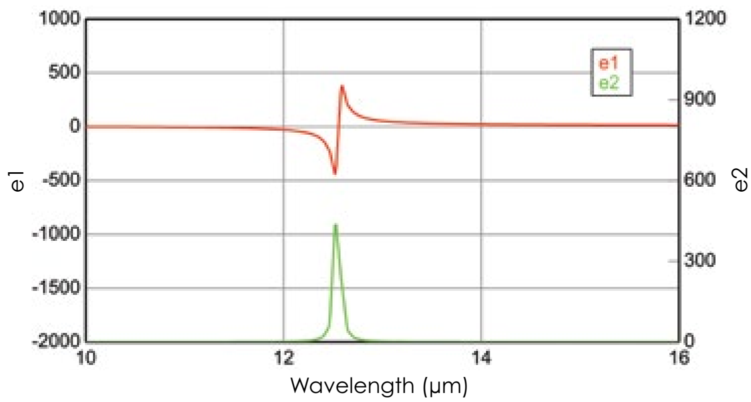


ACRYLIC OPTICAL CONSTANTS



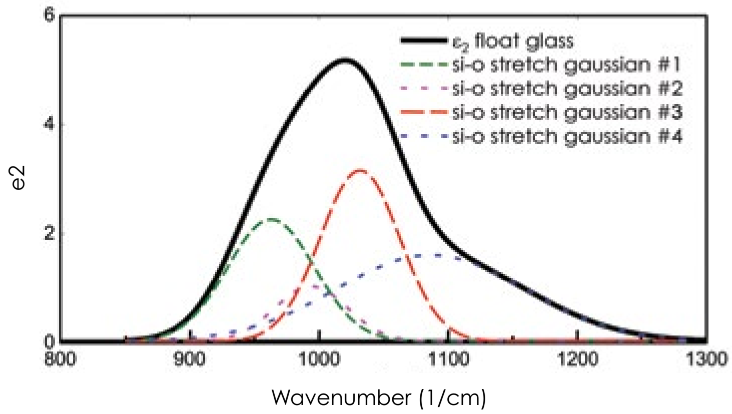
This organic material exhibits a large number of molecular vibrations related to different bonds within the material.

SILICON CARBIDE OPTICAL CONSTANTS



Strong lattice vibration absorption of crystalline silicon carbide.

SILICA GLASS OPTICAL CONSTANTS



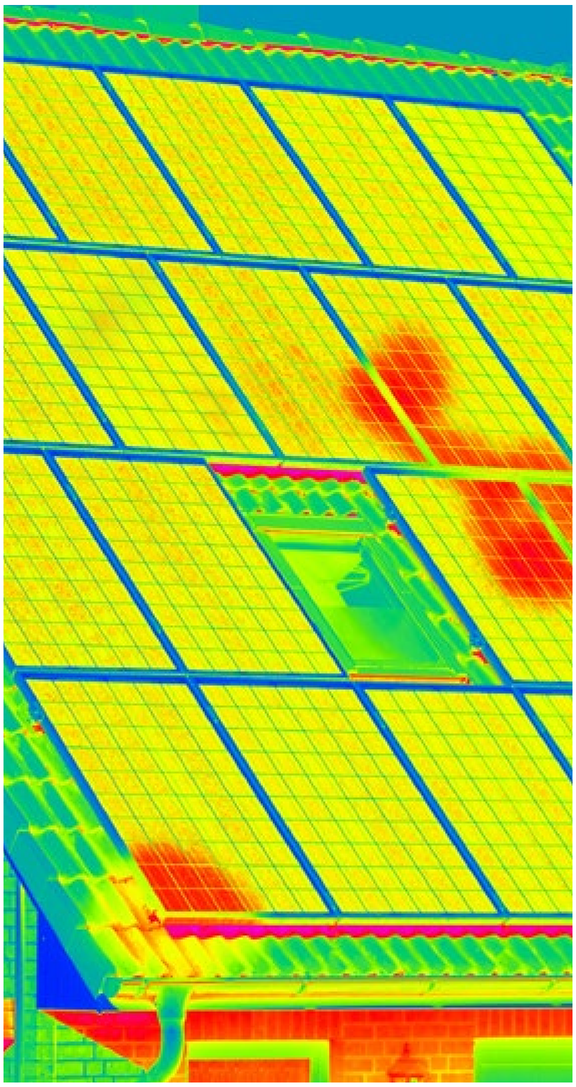
Absorption shape in silica glass can be modeled as a combination of various Si-O Gaussian stretch vibrations.

2. Free Carriers

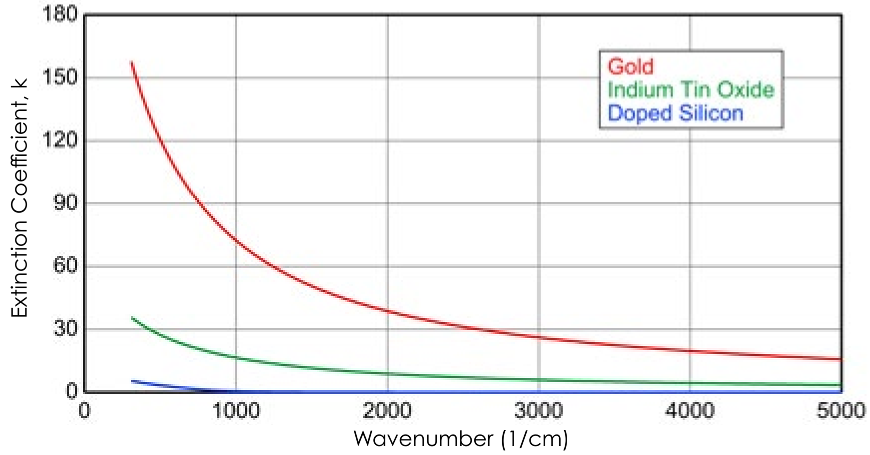
Absorption occurs when free charge carriers (electrons and "holes") are accelerated by the electric fields of a light beam. At infrared wavelengths, free-carrier absorption can be detected in metals, heavily doped semiconductors, and conductive oxides such as Indium Tin Oxide (ITO). This absorption is readily modeled with a Drude oscillator function, which provides information about the material conductivity.

3. Electronic Transitions

There are also a few semiconductors (such as HgCdTe) with bandgaps in the infrared. The figures below show a Ψ spectrum and a fit to HgCdTe on a CdZnTe substrate, as well as the resulting dielectric function (results were reported in Daraselia, et al, J. Elect. Mats. 34 (2005) 762). Metals can also have interband transitions in the infrared.



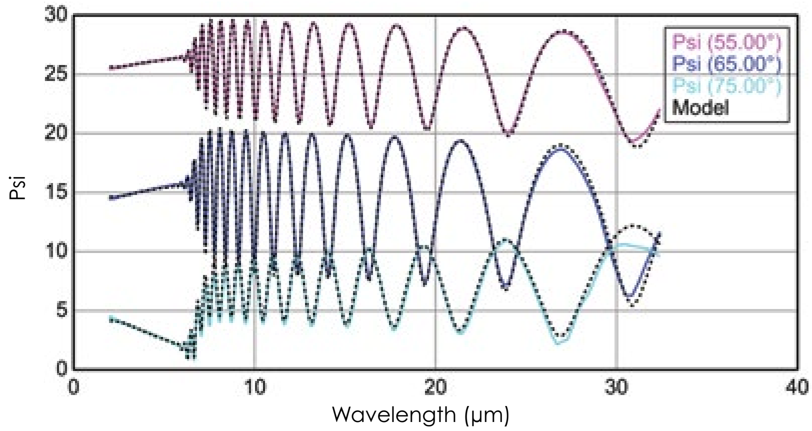
FREE CARRIER ABSORPTIONS



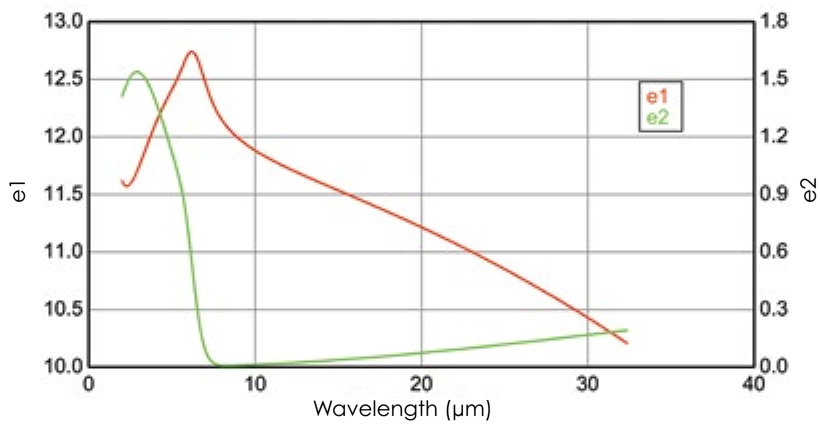
Comparison of the free-carrier absorption for different conductive materials.

Roughness = 0.0070 µm (fit)
+ Layer # 1 = HgCdTe HgCdTe Thickness = 15.0000 µm (fit)
Substrate = CdZnTe Substrate

ELLIPSOMETRIC DATA FROM MERCURY CADMIUM TELLURIDE



OPTICAL CONSTANTS OF MERCURY CADMIUM TELLURIDE

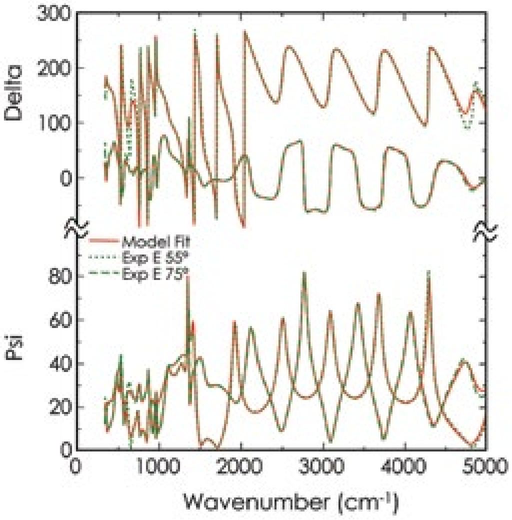


Top: Schematic of the optical model for Mercury Cadmium Telluride (MCT) sample. Middle: measured and model-generated ellipsometric Psi spectra for three incident angles for the MCT sample. Bottom: Real and Imaginary parts of the dielectric function for the MCT layer.

Applications

Optical Coatings

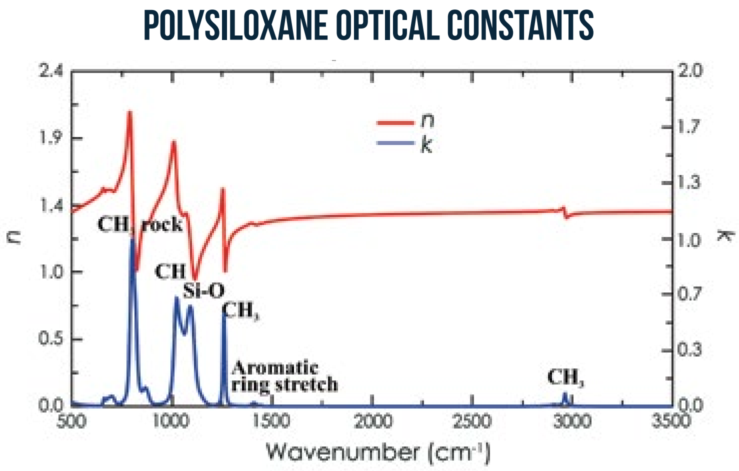
Ellipsometry plays an effective role in both the design and manufacturing stage of optical coatings. Anti-reflection coatings, notch filters, and highly reflective film stacks are all examples of material systems composed of high/low-index films. The optical properties and thickness of each layer in a coating stack play a pivotal role in determining the performance of a coating stack within a given wavelength range.



Multi-layer Characterization

SiO_2	4.975µm
MgF_2-Al_2O_3	1.488µm
ZnS	0.72µm
Al_2O_3	0.014µm
Silicon	

Multi-layer films can be studied in detail using the wide spectral range and variable angle capability of the IR-VASE. Measurements at multiple angles provide additional information by varying the path length through each layer. The ellipsometric spectra on the left are sensitive to all 4 layers shown in the model. The difference in index and absorption from layer to layer allows precise measurement of each thickness.

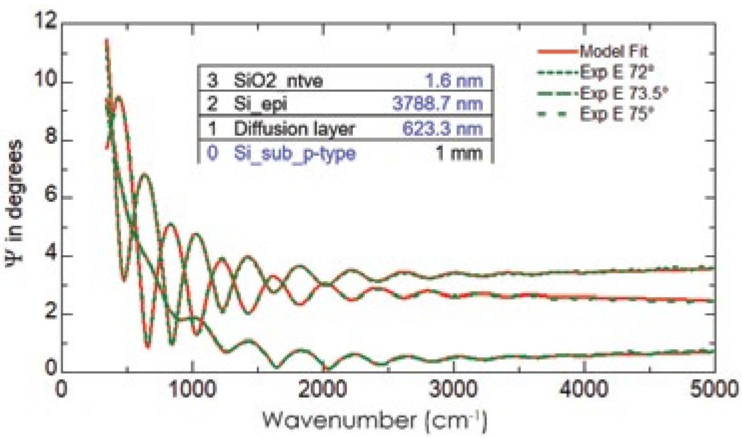


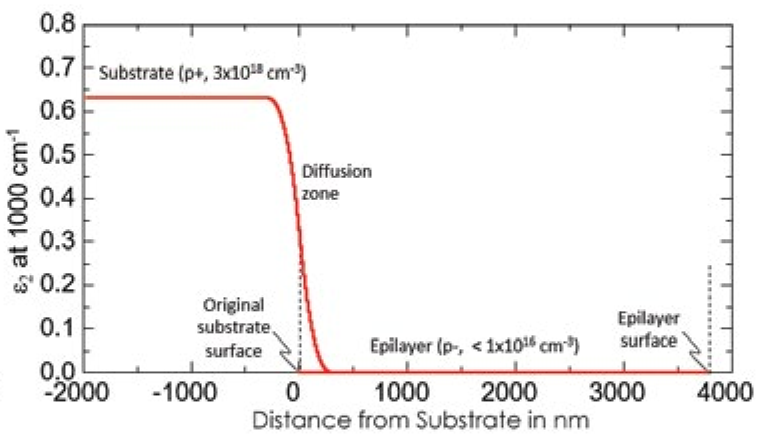
Chemical Composition via Molecular Bond Vibrational Absorptions

Ellipsometry is similar to standard FTIR spectroscopy, in that it contains information about molecular bonds via vibrational absorptions. These absorptions can be studied in bulk and thin film materials. For thin films, IR ellipsometry offers increased sensitivity over FTIR spectroscopy and yields quantitative values for both n and k rather than just absorbance. The figure on the left shows measured optical constants of a polysiloxane thin film with the vibrational absorptions labeled.

Epitaxial Layers, Doping Concentration, and Doping Profiles

At infrared wavelengths, differences in free-carrier density produce optical contrast between epitaxial or implanted layers and the underlying substrate. This results in measurement sensitivity to epitaxial layer thickness and substrate doping concentration, which cannot be detected at shorter wavelengths. The IR-VASE is also sensitive to carrier gradients at interfaces. Carrier profiles vs. depth that are generated from non-destructive IR-VASE analysis compare well with those generated by SIMS and SRP measurements, which are both destructive techniques.



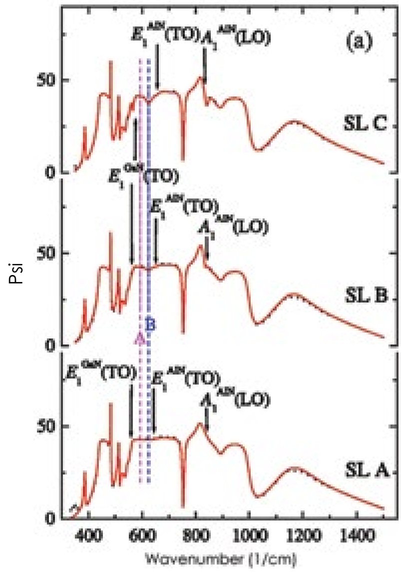


Advanced Applications

Phonon Structure (Compound Semiconductors)

The wide spectral range of the IR-VASE allows for phonon absorption studies of compound semiconductors and other crystalline solids. The figure on the right shows phonon modes of three 10-period AlN/GaN superlattice (SL) structures, each having a different AlN/GaN thickness ratio [1]. Phonon modes originating from the SL constituents are also visible, as well as two delocalized modes that are not predicted by the theory (dashed lines A and B).

[1] V. Darakchieva et al, Phys. Rev. B 71, 115329 (2005)



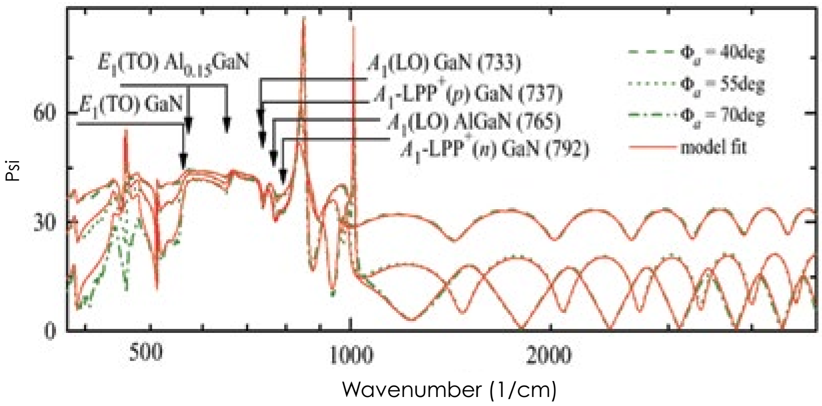
Above: Phonon modes of three 10-period AlN/GaN superlattice structures.

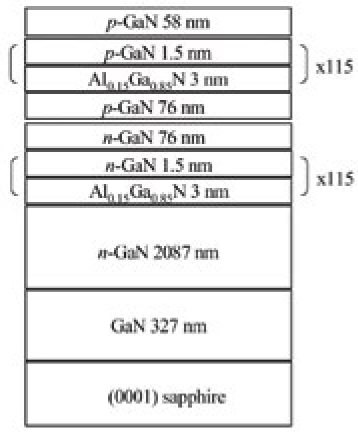
Anisotropy

For more complex sample structures (those that have multiple layers, optical absorption, anisotropy, etc.), the comparatively simple interference oscillations of a single transparent film described on page 4 are replaced by intricate, information-rich spectra. An example is the Ψ spectra for the GaN/AlGaN laser structure [2]. Because the IR-VASE spectra are highly accurate and precise, careful line-shape analysis of multi-angle data can yield quantitative information about many aspects of a complex structure. For instance, from the GaN/AlGaN laser data shown below, the researchers were able to determine the phonon modes, alloy ratios, doping concentrations, film thicknesses, and film quality.

[2] M. Schubert et al., SPIE Vol. 4449-8 (2001).

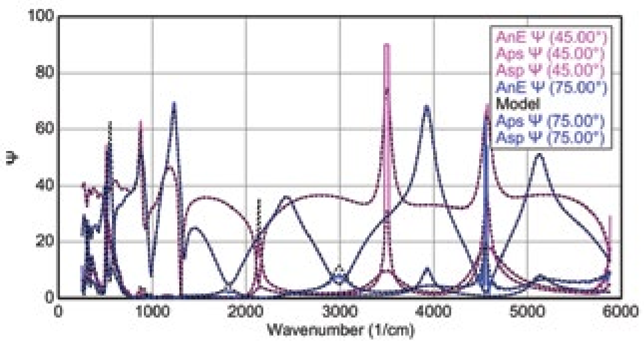
Ellipsometric data from GaN/AlGaN laser structure showing phonon modes and alloy concentrations at multiple orientation angles.

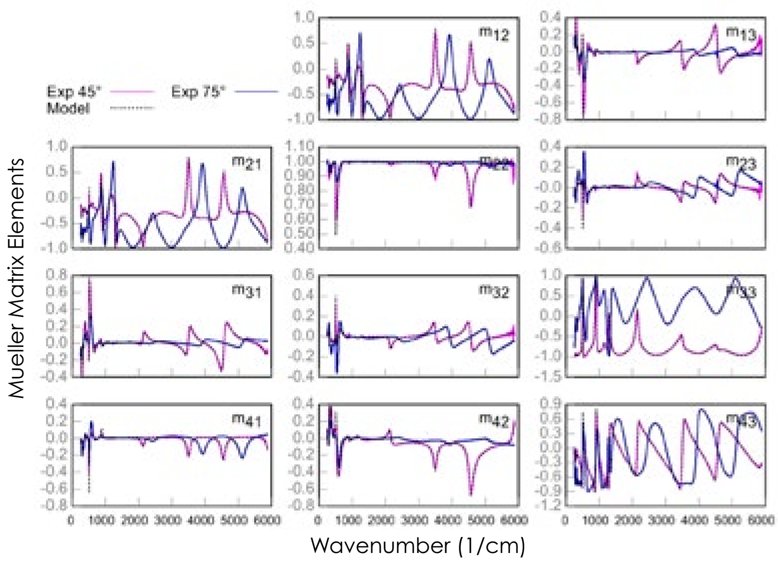




Generalized & Mueller Matrix Ellipsometry

Many anisotropic samples exhibit mode conversion, meaning that some of the p-polarized light is converted to s-polarized and vice versa. The IR-VASE can fully quantify mode conversion by measuring the three generalized-ellipsometry Psi and Delta spectra – AnE, Aps, and Asp – instead of a single Psi-Delta pair. The IR-VASE can also measure 12 of the 16 Mueller Matrix elements, allowing full characterization of anisotropic samples that exhibit both mode-conversion and depolarization. The figures below show generalized ellipsometric Ψ data (below left), Mueller Matrix ellipsometric data (below right), and fit results for an x-cut Lithium Niobate substrate.





Generalized ellipsometry data (above) and Mueller Matrix data of x-cut Lithium Niobate (right).

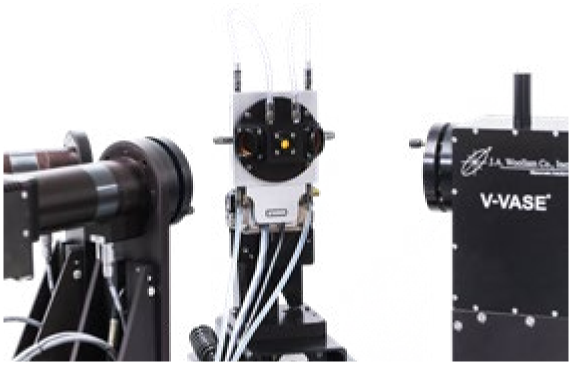
Accessories

Temperature Control

By adding a cryostat or heat stage to the IR-VASE for variable temperature studies, measurements can be conducted at temperatures as low as 4.2 K and as high as 1000° C. These stages have Zinc Selenide or Potassium Bromide optical ports.

Linkam Standard Heat Stage

The Linkam Standard Heat Stage enables temperature-controlled ellipsometry measurements from -70° to 600° C with stability of 0.1° C. The heating chuck is 22 mm in diameter and can accommodate similarly sized samples. The heat stage features a custom lid with optical ports configured for ellipsometry measurements at 70° (AOI) to promote a controlled environment and enable purging when necessary.



Linkam High-Temperature Heat Stage

The Linkam High-Temperature Heat Stage is intended for applications that require temperature above 600° C. This heat stage enables measurements from room temperature to 1000° C. The crucible design is compatible with samples up to 17 mm in diameter and is sold with a custom lid that enables ellipsometry measurements at 50° (AOI). Customers with primary interest in temperature measurements below 600° C should consider the standard heat stage.

Lake Shore Cryostat

The Lake Shore Cryostat provides an ultra-high-vacuum, continuous-flow cryostat for variable temperature studies from 4.2 to 800 K. The cryostat can accommodate samples up to 25 x 25 mm and has optical viewports for ellipsometry measurements at 70° (AOI). The cryostat includes a temperature controller, turbo vacuum pump, custom table, and software for coordinating temperature control with ellipsometric measurements.

Sample Mounting Options

We offer a variety of sample stage mounts to accommodate your measurement needs. Our standard and small sample stages are provided with every ellipsometer, while the others are available as upgrades.

Standard Stage

Rotation Stage

Manual XY Stage

Specifications

Spectral Range

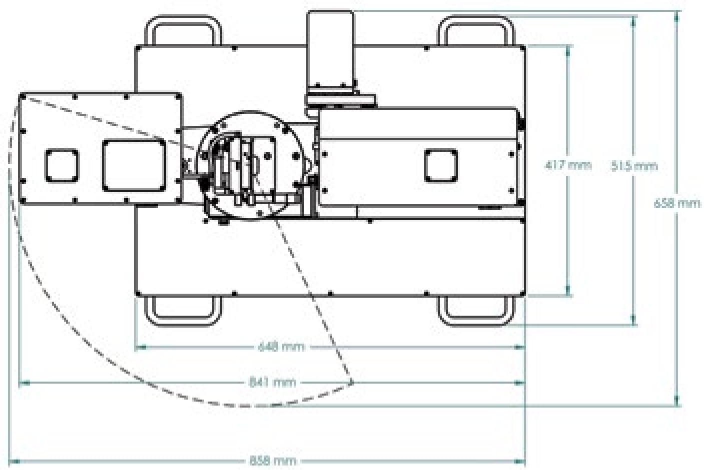
1.7 µm to 30 µm (333 cm^{-1} to 5900 cm^{-1})

Angle of Incidence

32° to 90°

Light Source

Silicon Carbide Globar with
FTIR Spectrometer (Michelson style)
Resolution: variable (1, 2, 4, 8, 16, 32, 64 cm^{-1})



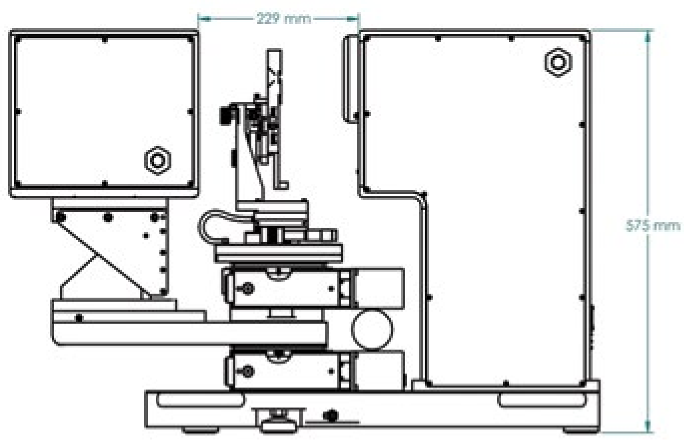
Top View

Software

WVASE for data acquisition, data analysis, and optical simulations CompleteEASE for additional data analysis

Power

100-120/220-240 VAC, 47-63 Hz, 9/4.5 Amps



Front View

Facility Requirements

To protect the instrument's optics, the system must be purged at all times with dry air or nitrogen.

For more information:

J.A. Woollam

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