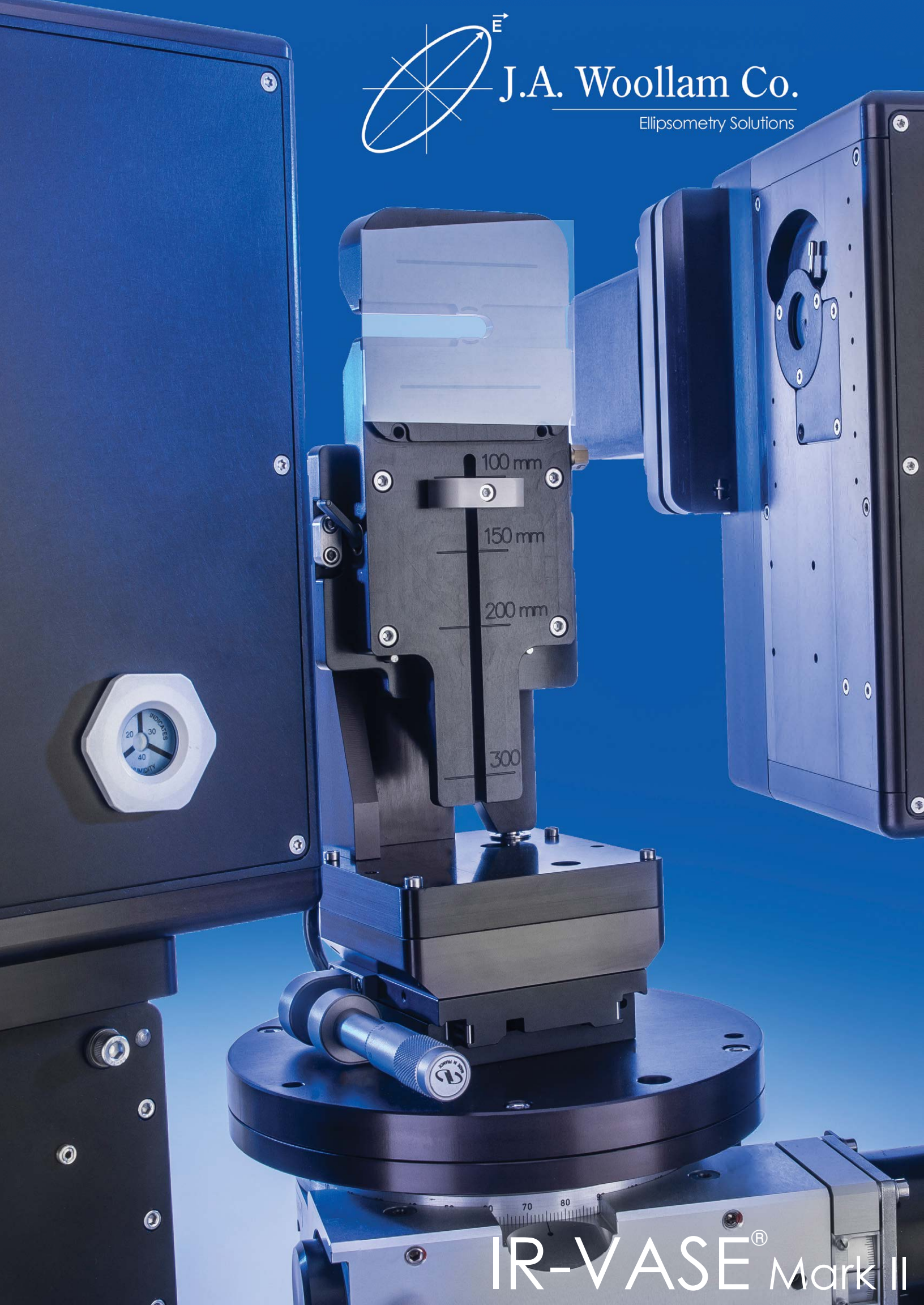




J.A. Woollam Co.

Ellipsometry Solutions



IR-VASE[®] Mark II

Mark II Features

The J.A. Woollam IR-VASE was first introduced in 1998, with incremental changes through the years. It is recognized world-wide as the premiere thin film characterization tool in the infrared. After more than 15 years, the IR-VASE has been given a complete update.

The new Mark II design is half the size of its predecessor, requiring less benchtop space and using less purge gas. The global 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 cryostat and heat stages.

IR-VASE Mark II Specifications

Spectral Range

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

Angle of Incidence

32° to 90° Standard

System Overview

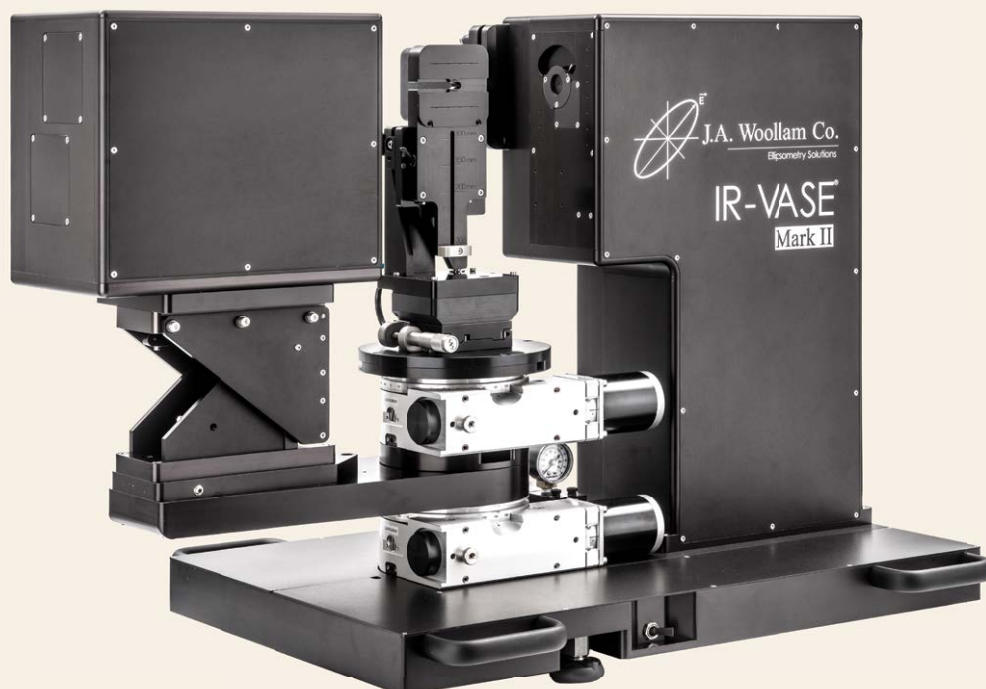
Patented rotating compensator ellipsometry, simultaneous data collection with Fourier Transform Method (FTIR). Vertical sample mounted with vacuum-assist for flexible measurement configurations.

Data Acquisition Rate

1 to 30 minutes, typical

(1 angle of incidence at 16 cm^{-1} resolution)

**Finer resolution will require longer time.*



Capabilities

The IR-VASE is the first spectroscopic ellipsometer to combine the chemical sensitivity of FTIR spectroscopy with 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 rapidly growing technology is finding 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 multilayers)
- Material composition (alloy fraction)
- Chemical bonding - molecular vibrations
- Phonon absorption - crystalline materials
- Surface and interfacial layers
- Doping concentration (resistivity)
- Free carrier absorption
- Anisotropy - uniaxial and biaxial

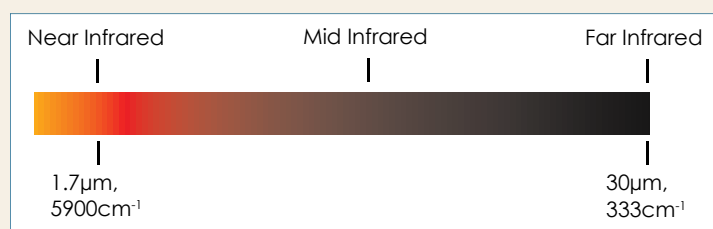
Why an IR-VASE?

Wide Spectral Range

Near to far infrared:

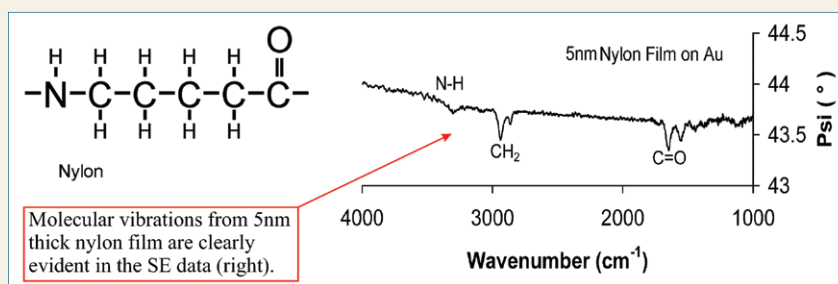
1.7 to 30 microns (333 to 5900 cm^{-1})

User-specified resolution from 1 to 64 cm^{-1}



High Sensitivity to Film Thickness, Even Ultra-thin Films

Spectroscopic ellipsometry data contain both “phase” and “amplitude” information from reflected or transmitted light. IR ellipsometry offers higher sensitivity to film thickness even for ultra-thin films a few nanometers thick than FTIR reflection/absorbance, 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 or Reference 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.

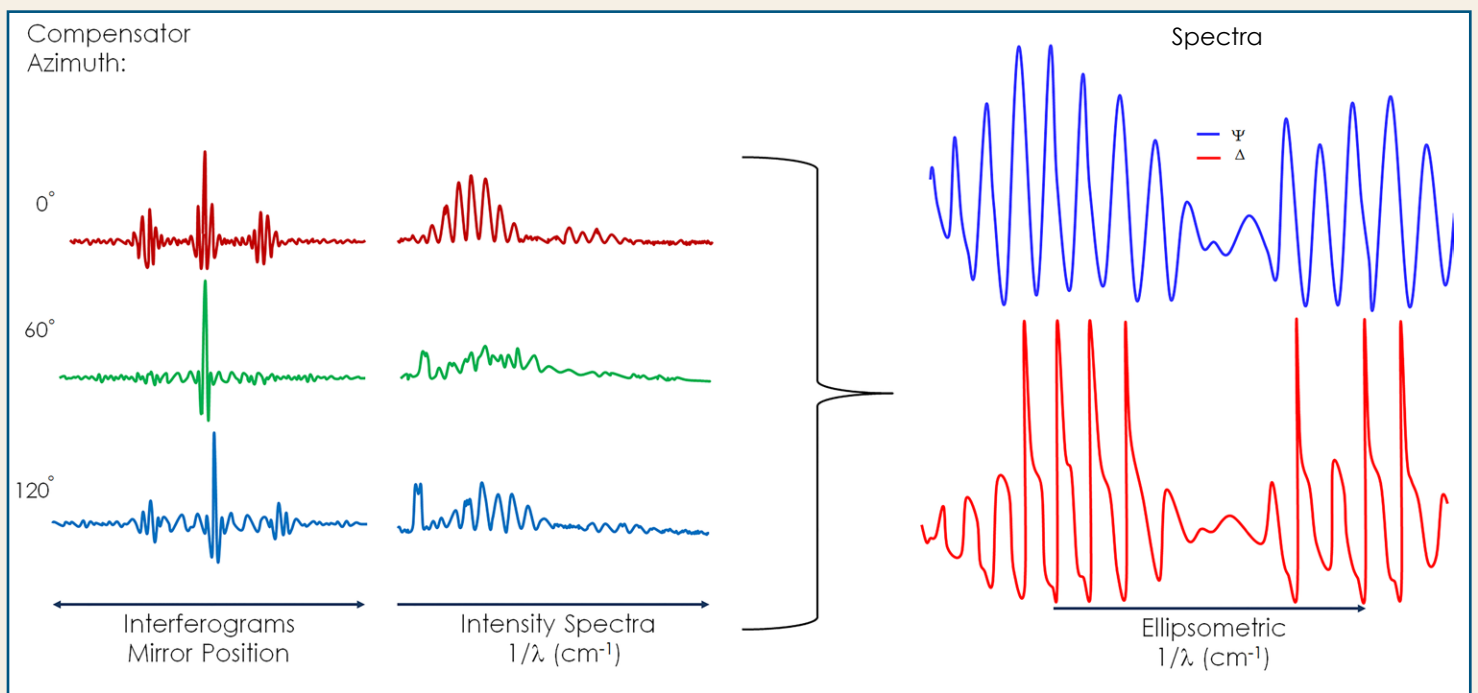
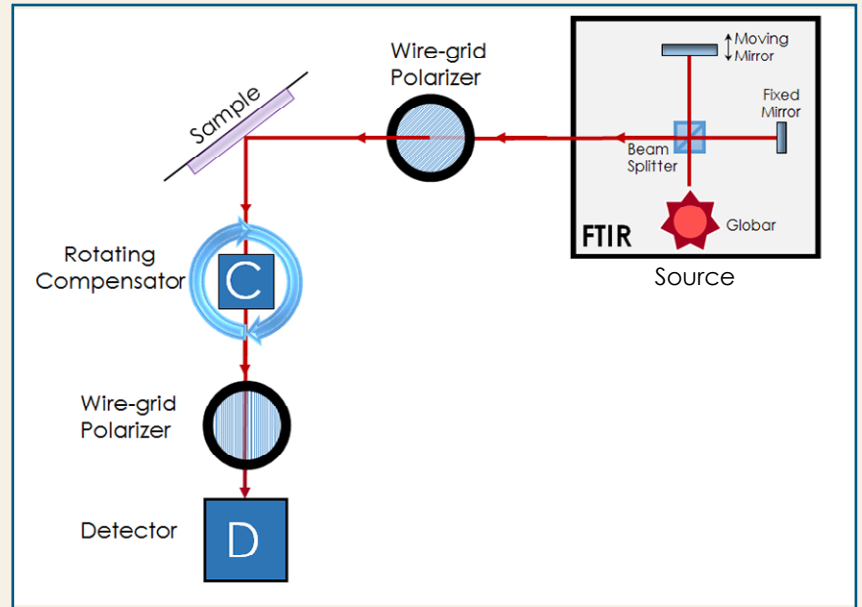
Technology: How It Works

Optics

The IR-VASE Mark II integrates a Fourier Transform Infrared (FTIR) interferometer source with a rotating compensator ellipsometer to provide accurate ellipsometric measurements. Wide-band 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 and polarization 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 exclusive 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 non-ideal measurement conditions including sample non-uniformity, patterned regions, incomplete film coverage, and double-side polished substrates.

Accessories

Sample Translation

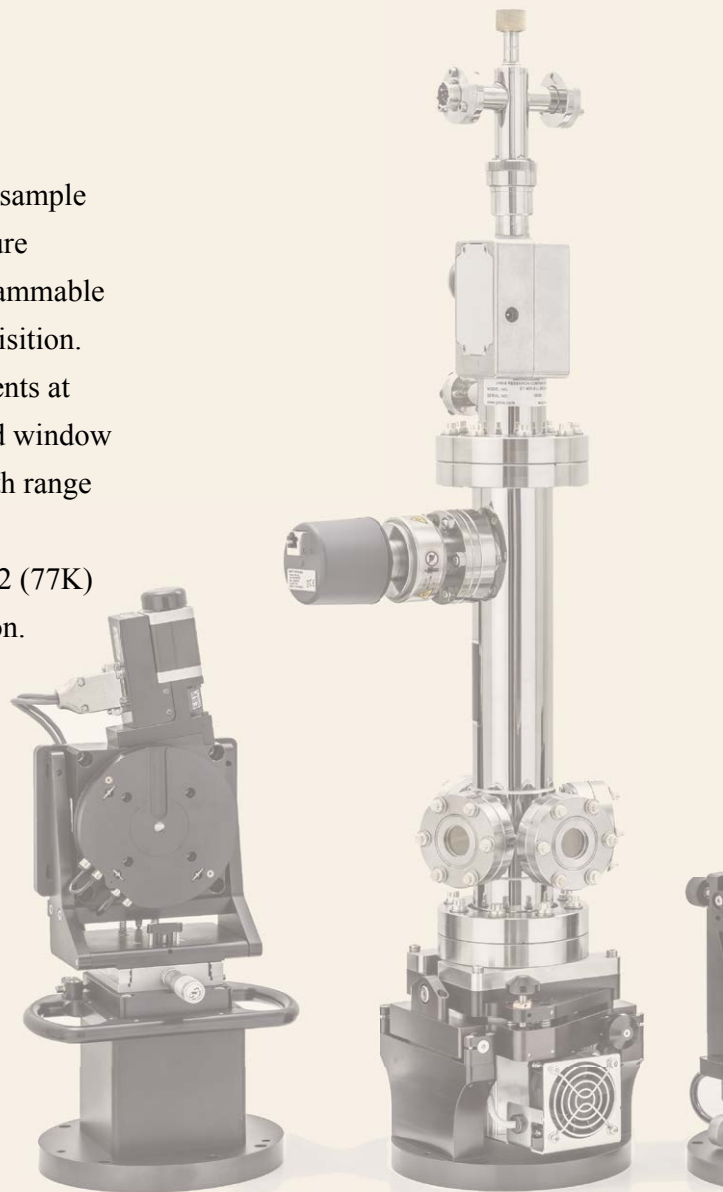
- 45mm by 45mm XY (Manual)

Standard Heat Stage Option

- Temperature range: Room Temp to 300°C
- Sample size: up to 50 mm diameter and 7.6 mm thick.
- Includes programmable temperature controller and thermocouple built into the sample chuck to monitor sample temperature.
- Enclosed cover allows measurements at 70° angle of incidence, and full variable angle capability without cover.
- No active cooling needed.
- Enclosed cover with optical windows allows sample purge. Standard window material is ZnSe, which limits upper wavelength range to about 16-22 microns when enclosed cover is used.

Cryostat Option

- Temperature range:
 - o Standard: 4.2-500K
 - o Optional: 4.2-800K
- Sample size: up to 25 x 25 mm
- Includes specially designed UHV chamber and sample mount, cryostat, turbo vacuum pump, temperature controller, custom table, and software for programmable temperature control and ellipsometric data acquisition.
- Windows in cryostat chamber allow measurements at 70° in reflection or 0° in transmission. Standard window material is ZnSe, which limits upper wavelength range to about 16-22 microns.
- Customer supplies liquid He (4.2K) or liquid N₂ (77K) and storage dewar for low temperature operation.



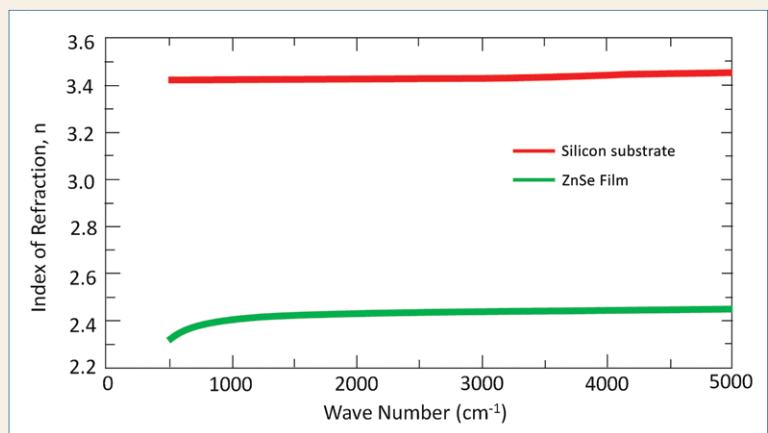
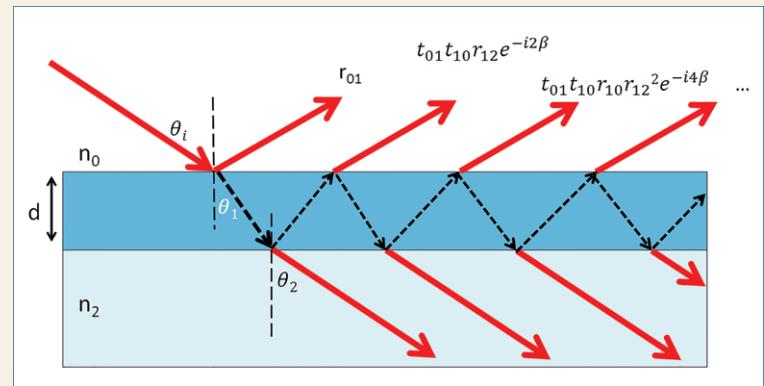
Basic IR Ellipsometry: Thickness & Index

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 index of the substrate is already known.

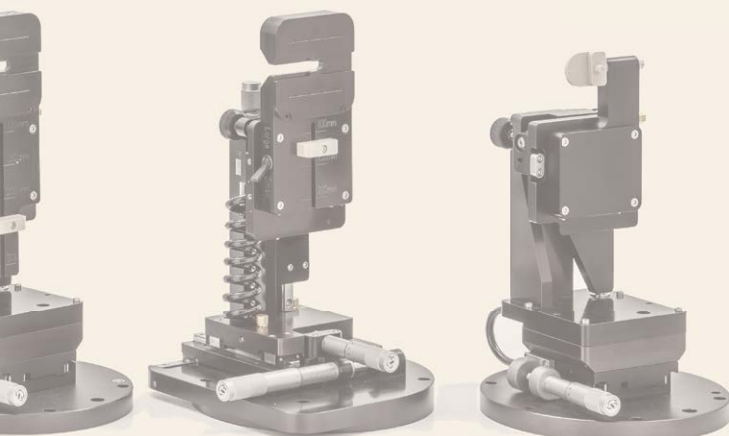
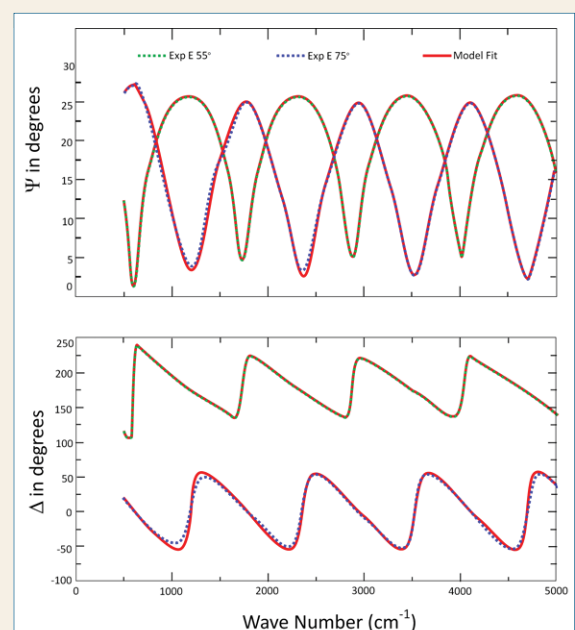
During measurement, the incident light separates into multiple rays that reflect from the top and bottom interfaces of the film. Each of the rays travels with different path lengths, causing them to have different phases. The phase differences produce constructive or destructive interference, depending 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 ratio of two indices. The strength and phase of interface reflection also depends upon angle of incidence and polarization (electric field direction).

This interference effect is responsible for oscillations seen in the ellipsometric Ψ and Δ vs. wavelength (or wave-number). The amplitude of the oscillations depend upon the index contrast between the film and substrate materials. In general, a large index contrast results in large oscillation amplitudes. The period of the oscillation depends upon film thickness and the film index.



During analysis, the software automatically adjusts the film index and thickness in the model until the model's oscillation amplitudes and periods match the experimental Ψ and Δ spectra.



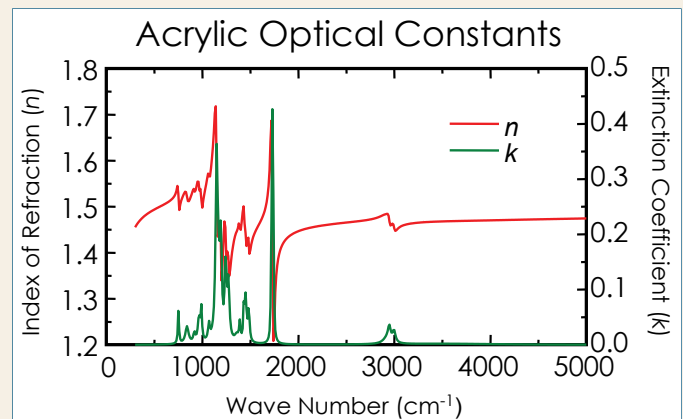
Information in the Infrared

Many of the features observed by Infrared Spectroscopic Ellipsometry are related to the following absorption mechanisms:

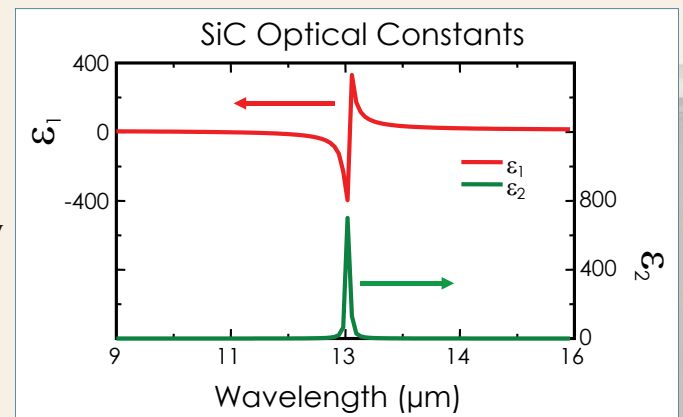
Vibrational Absorption

Vibrational absorption occurs when molecules and lattices resonate at infrared frequencies. These absorptions act as fingerprints of the materials, as the frequencies of resonance depend on the types of bonds and weight of resonating atoms or molecules. Organic materials exhibit well-known molecular vibrations, but vibrational absorptions also occur in dielectrics and semiconductors.

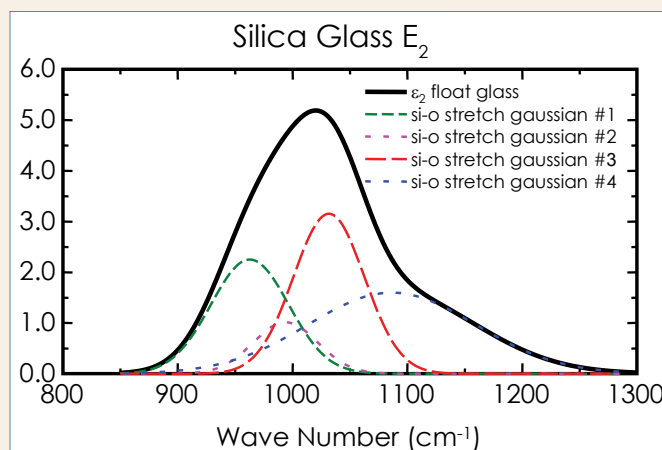
Most materials have regions of transparency that are separated by absorbing regions, with one or more resonant absorptions (see figure to the right for Acrylic film). In amorphous and glassy materials, the absorption is caused by molecular vibrational resonances. For these disordered materials, the bond lengths and angles are distributed randomly, so these absorptions tend to have a Gaussian character (see figure below for Silica). In highly-ordered materials, such as crystalline semiconductors, the vibrational resonances are no longer localized to individual molecules and they become extended lattice vibrations. These tend to have strong Lorentzian line shapes with very narrow broadening (see Figure to the right for crystalline SiC).



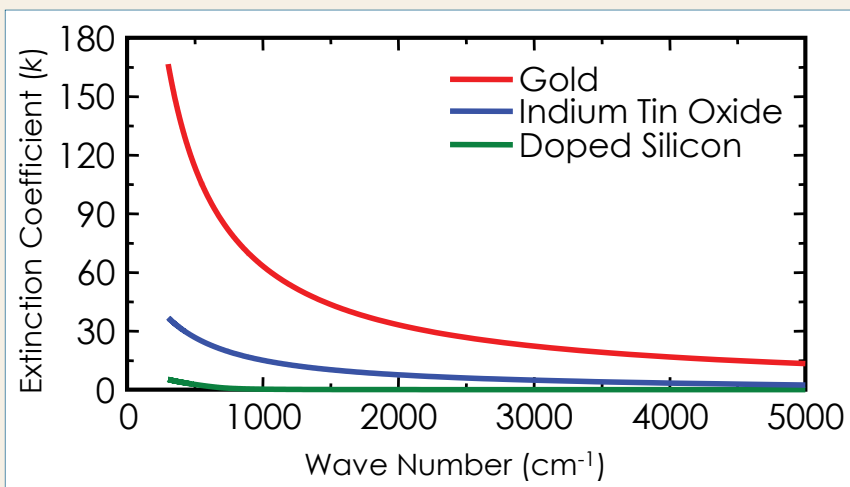
This organic material exhibits a large number of molecular vibrations related to different bonds within the material.



Strong lattice vibration absorption of crystalline silicon carbide.



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



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

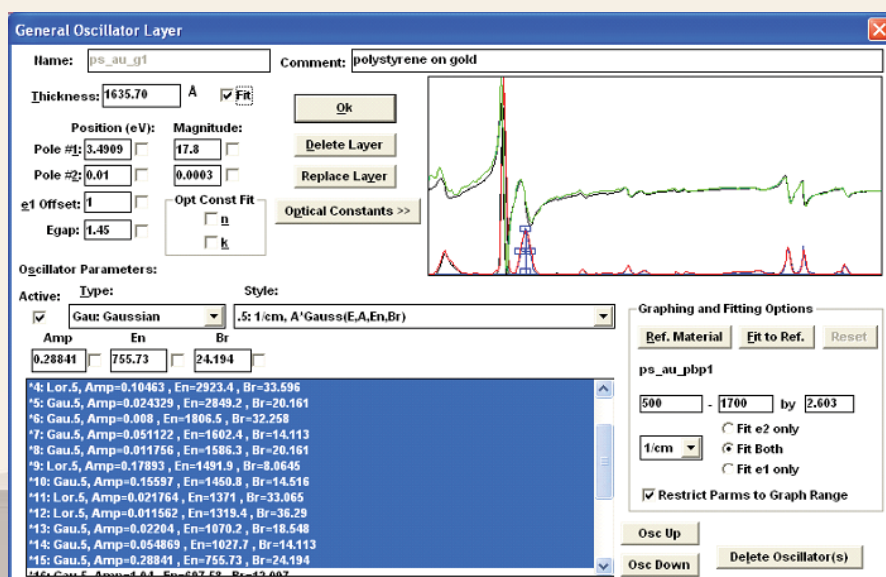
Free Carrier Absorption

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, which provides details of the material conductivity.

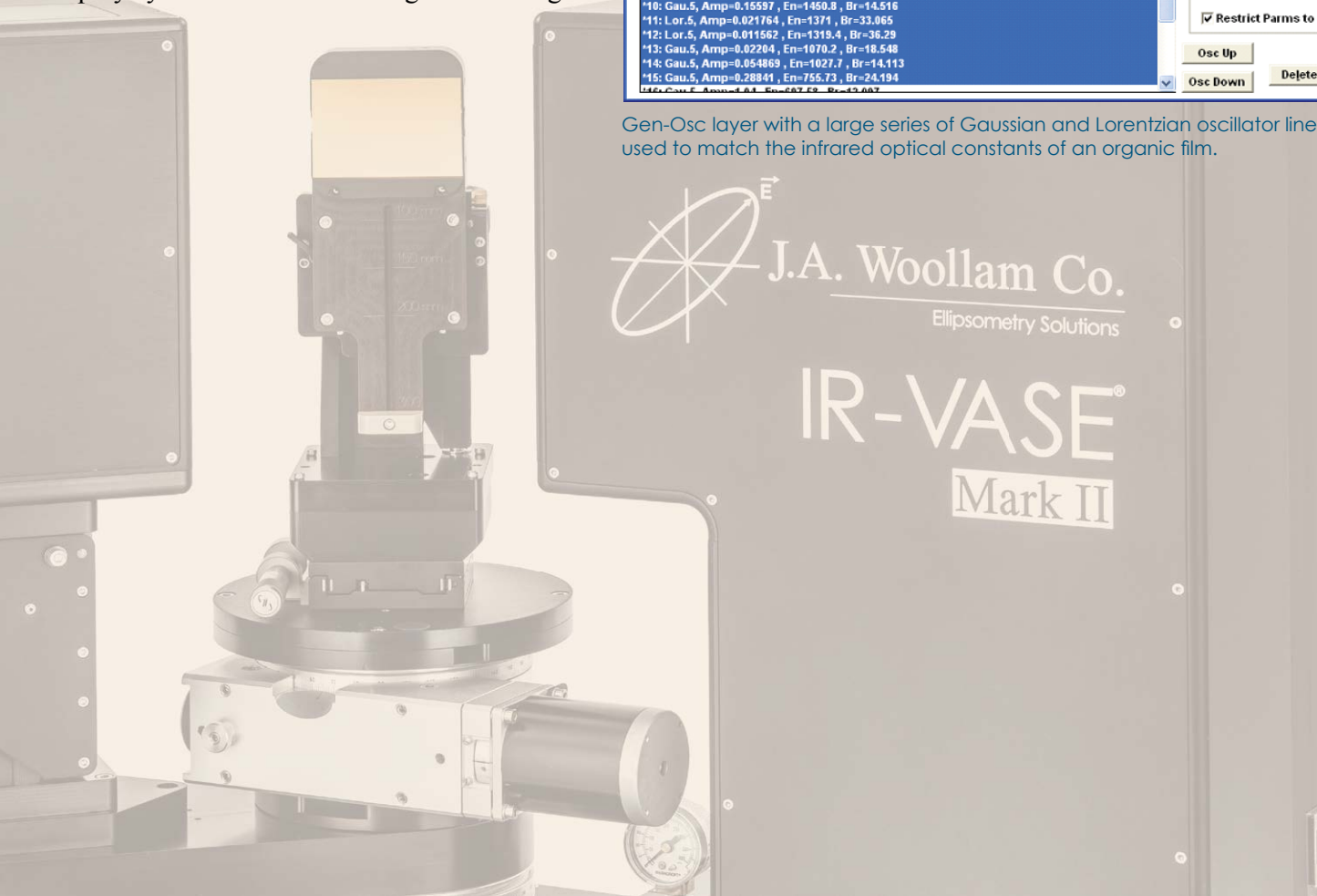
Electronic Transitions

There are a few semiconductors (such as HgCdTe) with bandgap energies in the infrared. Metals can also have interband transitions in the infrared. The line-shape for these absorptions depend on the electronic states in the material.

J.A. Woollam software provides the flexibility to model all different absorptions in the infrared. An example Gen-Osc layer for polystyrene is shown in the figure to the right.



Gen-Osc layer with a large series of Gaussian and Lorentzian oscillator line-shapes used to match the infrared optical constants of an organic film.



IR-VASE Applications

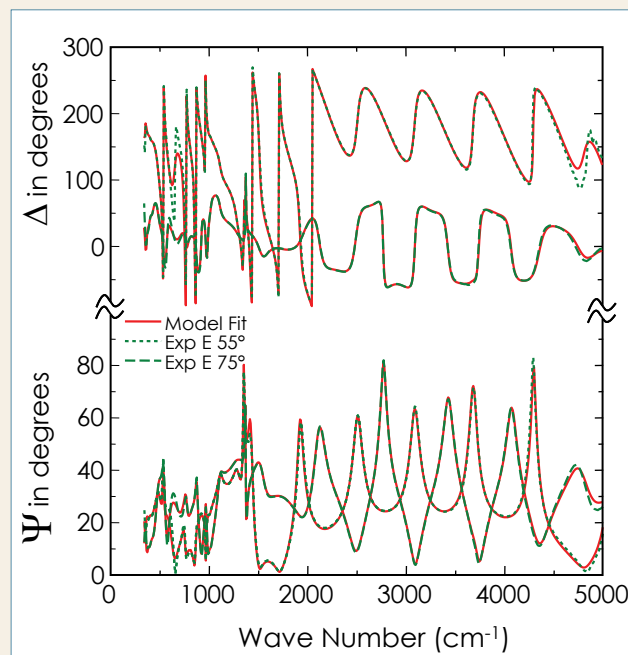
Optical Coatings

The IR-VASE allows you to characterize thickness and IR refractive index of single and multilayer films; bulk uncoated substrates; infrared optical systems; AR, HR, and single-layer and multilayer coatings of both high and low index films.

Multilayer Characterization

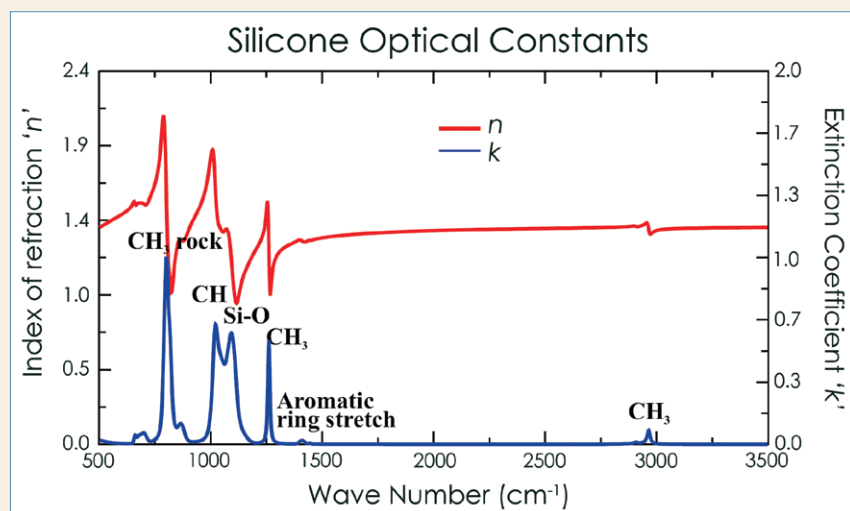
Multilayer films can be extensively studied with the wide spectral range and variable angle capability of the IR-VASE. Measurements at multiple angles provide additional information by varying the path length through each layer. The ellipsometric spectra on the right are sensitive to all 4 layers. The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

SiO ₂	4.975μm
MgF ₂ -Al ₂ O ₃	1.488μm
ZnS	0.72μm
Al ₂ O ₃	0.014μm
Silicon	



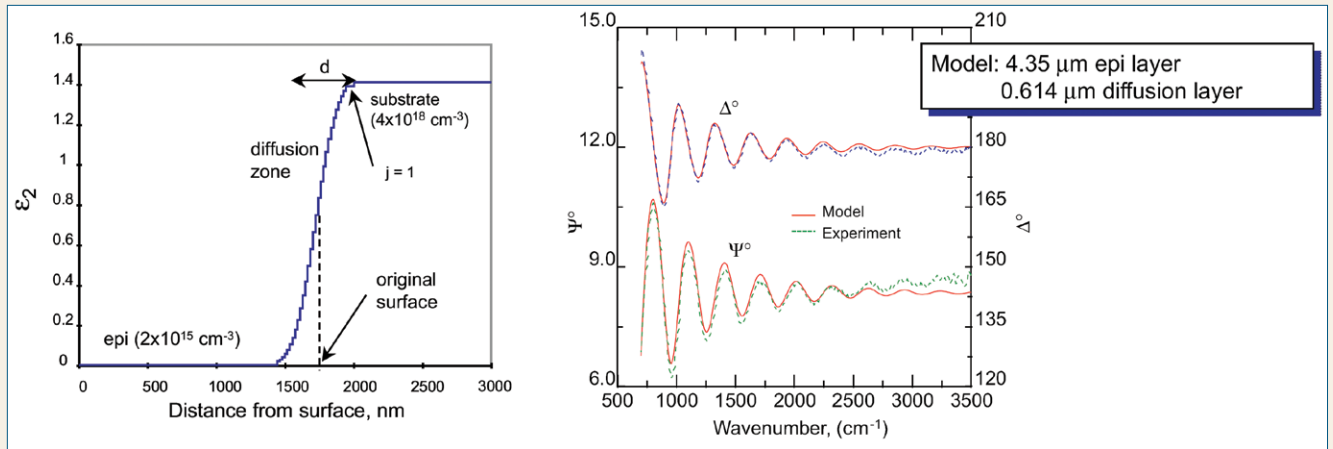
Chemical Composition via Molecular Bond Vibrational Absorptions

Like standard FTIR spectroscopy, IR ellipsometry contains information about molecular bonds via vibrational absorptions. Infrared absorption caused by these vibrations can be studied in bulk or 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 simple absorbance. The figure below shows measured optical constants of a silicone thin film with vibrational absorptions labeled.

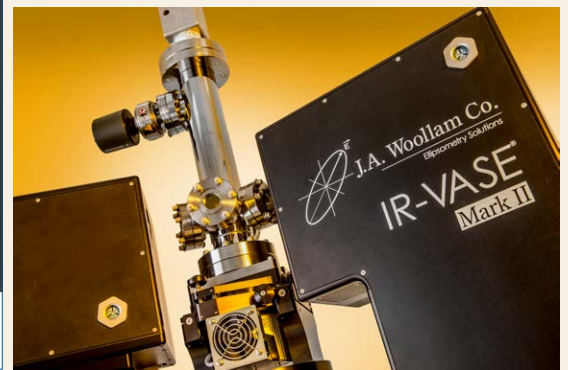
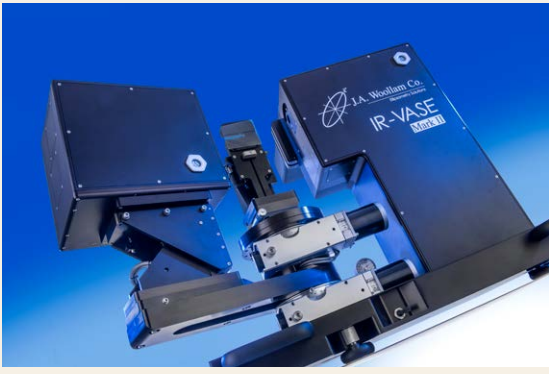


Epitaxial Layers, Doping Concentration and Doping Profiles

At infrared wavelengths, differences in free carrier density produce optical contrast between epitaxial or implanted layers and underlying substrate. This results in great sensitivity to epitaxial layer thickness and substrate doping concentration, which are not seen using UV-VIS wavelengths. The IR-VASE also provides good sensitivity to carrier gradients at interfaces. Carrier profiles versus depth are generated from non-destructive IR-VASE analysis compare well with those generated by destructive SIMS and Spreading Resistance Probe measurements.



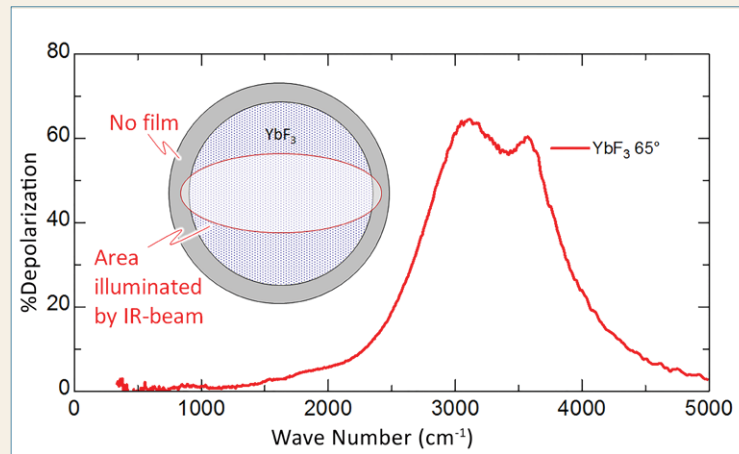
T.E. Tiwald et al., *Phys. Rev. B*, **60** (1999) 11 464.



Advanced Applications

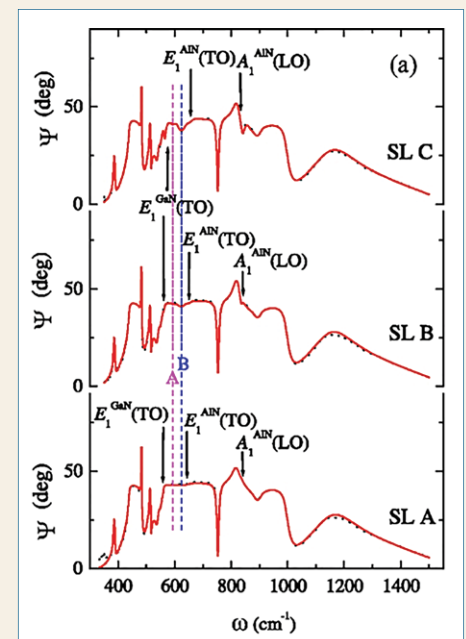
Depolarization

Beyond the standard ellipsometric Psi and Delta values, the IR-VASE rotating compensator technology allows users to measure other quantities, including depolarization. The ellipsometer illuminates the sample with light that is completely polarized. An ideal sample will simply change that polarization from one state into another (for example, linear becomes left-circular). Non-ideal samples will partially depolarize the reflected light. The causes of depolarization include variations in film thickness, incomplete film coverage, bandwidth effects, and other factors. In the figure below, the large depolarization (red curve) is the result of a small portion (5%) of the total detected light reflecting from an area of the sample surface that is not coated by the YbF_3 film.



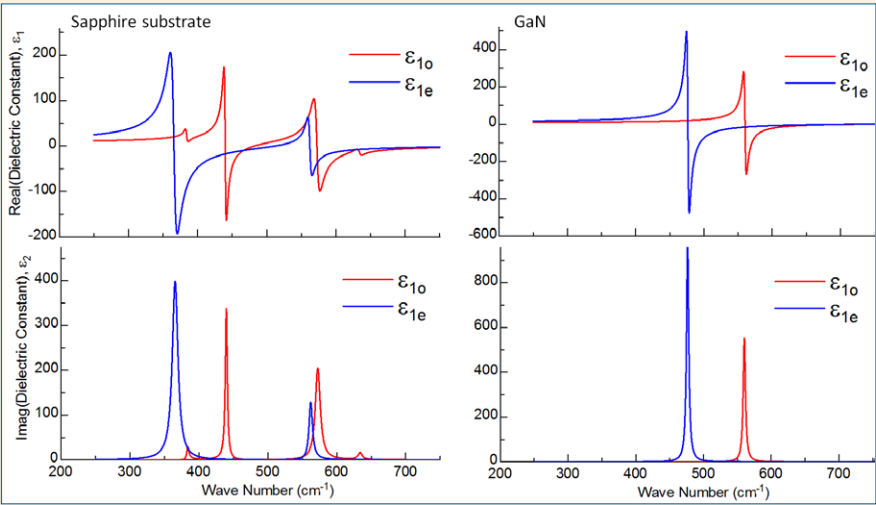
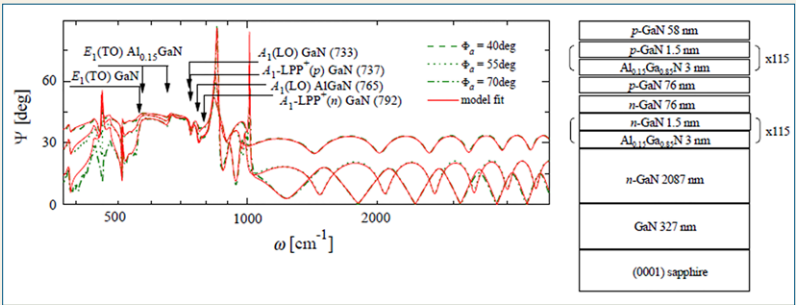
Phonon Structure (Compound Semiconductors)

The wide spectral range of the IR-VASE is important for phonon absorption studies of compound semiconductors and other crystalline solids. Data on the right show phonon modes of three 10-period AlN/GaN super lattice (SL) structures, each having a different AlN/GaN thickness ratio (from V. Darakchieva et al, Phys. Rev. B 71, 115329 (2005)). Phonon modes originating from the SL constituents are also visible, as well as two delocalized modes that are not predicted by the theory (marked A and B). The researchers were also able to correlate phonon shifts to strain and other properties.

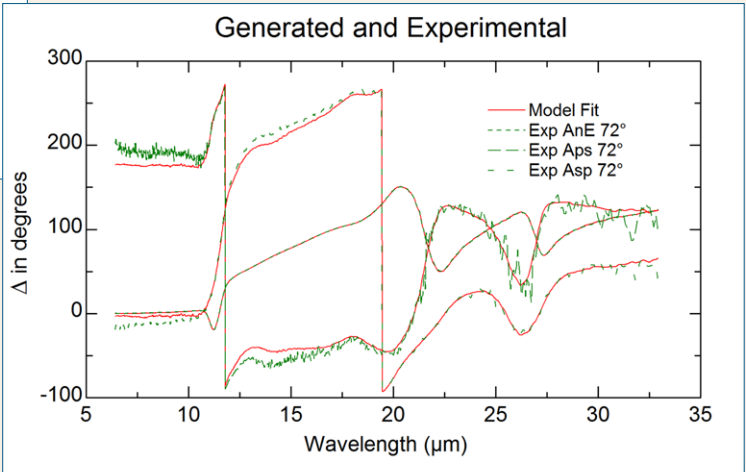
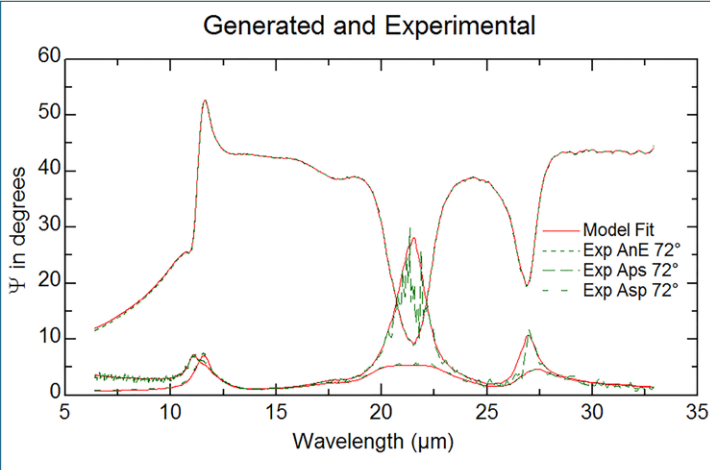


Anisotropy

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 in the Basic Measurements section are replaced by intricate, information-rich spectra. An example is the Ψ spectra for the GaN / AlGaIn laser structure (from M. Schubert et al., SPIE Vol. 4449-8 (2001)). 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 / AlGaIn laser data shown, the researchers were able to determine the phonon modes, alloy ratios, doping concentrations, and film thicknesses and quality.

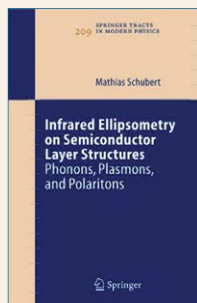


Many anisotropic samples exhibit mode conversion, meaning that some of the p-polarized light is converted to s-polarized, and vice versa. The IR-VASE can fully quantify mode conversion by measuring the three generalized-ellipsometry Psi and Delta spectra – AnE, Aps, and Asp – instead of a single Psi-Delta pair. The IR-VASE can even measure 12 of the 16 Mueller Matrix elements, allowing full characterization of anisotropic samples that exhibit both mode-conversion and depolarization.



Research

The IR-VASE has been used in hundreds of research papers. Here are a few that show the scope of measurement capabilities:



M. Schubert, "Infrared Ellipsometry of Semiconductor Layer Structures Phonons, Plasmons, and Polaritons" Springer-Verlag: Berlin (2004).

T.I. Willett-Gies, et al. "Two-phonon absorption in LiF and NiO from infrared Ellipsometry" *J. Vac Sci. Technol. A* **33** 6 (2015) 061202-1.

H.G. Dantanarayana, et al. "Refractive index dispersion of chalcogenide glasses for ultra-high numerical-aperture fiber for mid-infrared supercontinuum generation" *Optical Materials Express* **4** 7 (2014) 210165.

S. Schöche, et al. "Free-charge carrier parameters of n-type, p-type and compensated InN:Mg determined by Infrared Spectroscopic Ellipsometry" *Thin Solid Films* **571** (2014) 384.

S. Schöche, et al. "Infrared dielectric anisotropy and phonon modes of rutile TiO₂" *J. Appl. Phys.* **113** (2013) 164102-1.

E. Sachet, et al. "Mid-infrared surface plasmon resonance in zinc oxide semiconductor thin films" *J. Appl. Phys.* **102** (2013) 051111-1.

R. Lovrincic, et al. "Orientation of Nonplanar Molecules in Polycrystalline Layers from Infrared Spectra: Core-Chlorinated Naphthalene Tetracarboxylic Diimides" *J. Phys. Chem. C* **116** (2012) 5757.

V.R. D'Costa, et al. "Infrared dielectric function of p-type Ge_{0.98}Sn_{0.02} alloys" *Phys. Rev. B* **80** (2009) 125209.

M.F. Saenger, et al. "Infrared ellipsometric characterization of silicon nitride films on textured Si photovoltaic cells" *Mater. Res. Soc. Symp. Proc.* **1123** (2009) 1123-P02-02.

H. Arwin, et al. "Infrared Ellipsometry studies of thermal stability of protein monolayers and multilayers" *Phys. Stat. Sol. (c)* **5** 5 (2008) 1438.

T.E. Tiwald, D. Thompson, "Determination of the Mid-IR Optical Constants of Water and Lubricants Using IR Ellipsometry Combined with An ATR Cell" *Thin Solid Films* **313-314** (1998) 718.

T.E. Tiwald, et al. "Application of IR variable angle spectroscopic ellipsometry to the determination of free carrier concentration depth profiles" *Thin Solid Films* **313-314** (1998) 661.

J.A. Dobrowolski, et al. "Toward Perfect Antireflection Coatings. 3. Experimental Results Obtained with the Use of Reststrahlen Materials" *Applied Optics* **45** 7 (2005) 1555.