IR-VASE® analysis of IR-optical materials

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The accuracy of the IR-VASE® ellipsometer makes it an ideal instrument for measuring optical constants and thicknesses of mid-infrared optical materials and devices over a spectral range of 2 to 33 μm (300 – 5000 cm^{-1}). The rotating compensator design allows precision measurements of transparent and opaque substrates, as well as single and multilayer films. Since the IR wavelengths are 10x longer than visible wavelengths, the instrument is capable of measuring films beyond 100 μm thick. Data types include standard and generalized ellipsometry, %Depolarization, Mueller Matrix quantities, transmittance and reflectance.

When used in combination with WVASE32™ software, users can perform very sophisticated analyses of many different film/substrate combinations, including: single or multilayer, transparent or opaque, isotropic or anisotropic; as well as samples with graded-index layers, non-uniform thicknesses and more. The IR-VASE can also determine optical constants of various bulk crystals, glasses, liquids and other substrate materials for which reliable mid-infrared optical constants are incomplete or unavailable.

MgF_2-Al_2O_3 film on Silicon

This IR-VASE® study determined the thickness and mid-IR optical functions, *n*(λ) and *k*(λ), for thin films of ZnS, MgF_2-Al_2O_3, GeO_2 and Al_2O_3. The samples consisted of various single layers on standard silicon wafers.

All of these films are substantially transparent between 2 μm – 10 μm wavelengths (1000 – 5000 cm^{-1}), as indicated by the full range coverage of the interference spectra (Figure 1). We exploit this transparent region to determine film thickness and refractive index. We created a two-term Sellmeier (pole) dispersion model in the GenOsc layer to account for dispersion on both ends of the 2 μm – 10 μm spectral region.

After fitting thickness and refractive index in the transparent spectral range, the entire measured range of 2 μm to 33 μm (300 – 5000 cm^{-1}) is selected, thickness and the other model parameters are fixed, and a point-by-point fit is performed to obtain *n*(λ) and *k*(λ). Although the resulting optical functions are not necessarily Kramers-Kronig consistent, they usually provide good starting values for the development of multi-oscillator functional GenOsc model. The *n*(λ) and *k*(λ) values are saved as a tabulated *.mat* file, then loaded as a reference spectra in the GenOsc layer and fit with a series of oscillators.

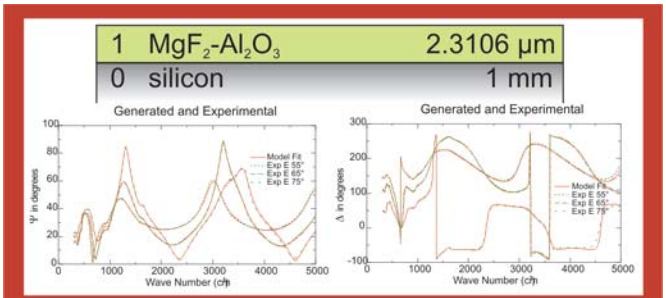

Figure 1. Model, experimental data and fits for MgF_2/Al_2O_3 combined layer.

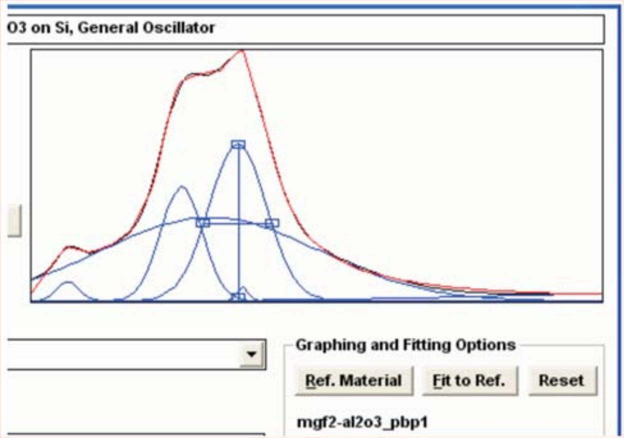

Figure 2. Dielectric function (ε_2) after fitting a portion of MgF_2-Al_2O_3 point-by-point reference spectrum. Red curve is reference spectra and black is model. Blue curves are the individual Gaussian Oscillators.

Mid-IR vibrational absorptions of amorphous materials are generally best fit with Gaussian oscillators. Each Gaussian represents the summation of a large population of narrow molecular resonances with a broader normal distribution of center energies created by slightly different bond lengths and bond angles. The fit to a portion of the ε_2 spectrum for the MgF_2-Al_2O_3 point-by-point fit is shown in Figure 2. Both ε_2 and ε_1 reference spectra are fit using procedures described in the addendum of the WVASE32 software manual.

All the parameters of the GenOsc layer, as well as layer thickness, are then fit to the ellipsometric data. The resulting fits are shown in Figure 1, with *n*(λ) and *k*(λ) shown in Figures 3a and 3b.

Beyond the determination of *n* and *k* at specific wavelengths, reproducible, accurate data from the IR-

VASE can produce additional information about a film. In Figure 3b, subtle details in $n(\lambda)$ and $k(\lambda)$ often yield useful information about the chemical make-up of the films. Vibrational absorptions in the 2 to 10 µm region can indicate the presence of contaminants that are incorporated in the film either during or after deposition. For example, the MgF_2 layer appears to have incorporated some water, as indicated by the O-H stretch absorption at ~3 µm combined with what appears to be the O-H-O scissor vibration at ~ 6 µm.

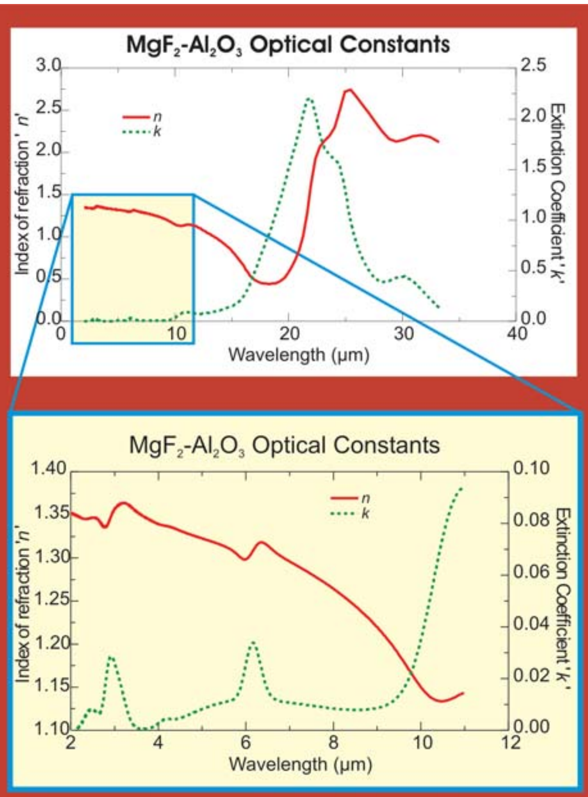

Figure 3. a) The optical functions $n(\lambda)$ and $k(\lambda)$ for MgF_2-Al_2O_3 film, for $2 \leq \lambda \leq 33$ µm. B) Close-up between 2 and 11 µm showing subtle O-H absorption bands.

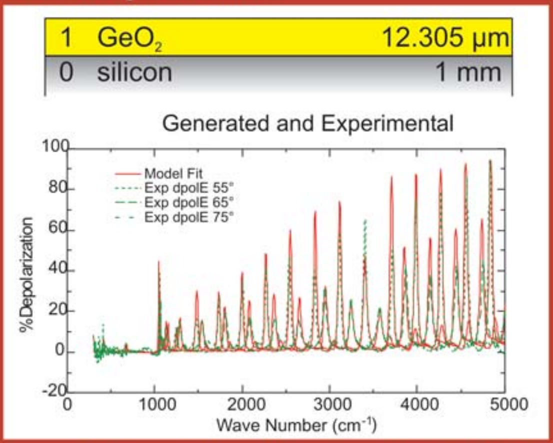

Figure 4 Percent Depolarization data for GeO_2 layer.

Thickness non-uniformity & %Depolarization

The ability to quantitatively measure percent depolarization is an important diagnostic tool for analysis of non-ideal thin films, and is a quantity that is readily measured by the IR-VASE®. In this example, "%Depolarization" gave quantitative information regarding the effects of non-uniformity in the film thickness (see Figure 4). The depolarization effects of such non-uniformities are amplified by thicker films.

MgF_2-Al_2O_3/ZnS/Al_2O_3 on Si

The MgF_2-Al_2O_3 film was part of a larger study that included a multilayer stack of different materials. During analysis, only layer thicknesses were varied (optical constants for each layer were previously determined). The fit results are shown in Figure 5. The fits are reasonably good, but there are some obvious misfits; particularly in the 700 – 1600 cm^{-1} and 2800 – 4000 cm^{-1} regions.

Because the IR-VASE yields accurate Ψ–Δ data, one can be confident that these misfits are real and not measurement artifacts. In this case, misfits indicated that optical properties of at least one of the films changed when deposited in the multilayer stack rather than as individual layers on silicon.

Despite the fact that the fits are not perfect in some regions, this model still provided a very good match to IR-Reflectance data.

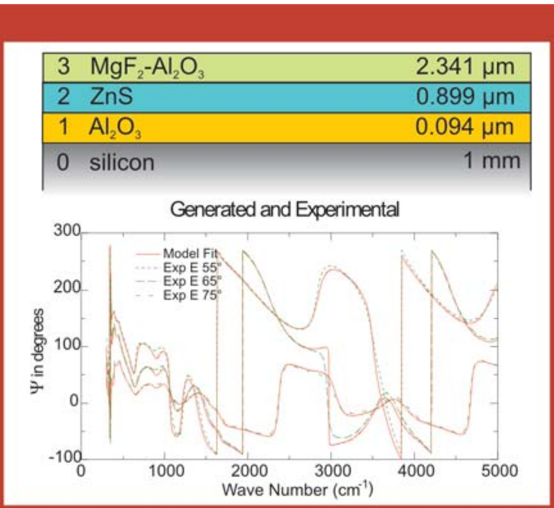

Figure 5. Model, experimental data and fits for the multilayer MgF_2-ZnS/Al_2O_3 on Si.