Infrared dielectric properties of low-stress silicon nitride

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Silicon nitride thin films play an important role in the realization of sensors, filters, and high-performance circuits. Estimates of the dielectric function in the far- and mid-IR regime are derived from the observed transmittance spectra for a commonly employed low-stress silicon nitride formulation. The experimental, modeling, and numerical methods used to extract the dielectric parameters with an accuracy of approximately 4% are presented. © 2012 Optical Society of America

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The physical properties of silicon nitride thin films, namely low tensile stress, low thermal/electrical conductance, and its overall compatibility with other common materials, have facilitated its use in the microfabrication of structures requiring mechanical support, thermal isolation, and low-loss microwave signal propagation (e.g., [1–4]). Silicon nitride films are amorphous, highly absorbing in the mid-IR [5], and their general properties are functions of composition [6,7]. Here the optical properties are studied in detail for a membrane with parameters commonly employed in microfabrication.

The silicon nitride optical test films were prepared by a low-pressure chemical-vapor-deposition (LP-CVD) process optimized for tensile stress and refractive index [8]. The 5:1 SiHxCl2/NH3 gas ratio employed results in a tensile stress <100 MPa and optical index greater than ~2 [9]. The test structure is shown schematically in Fig. 1 (inset). Double-side-polished silicon (75 mm diameter, 500 μm thick) wafers [10] were used as a mechanically robust handling structure for the SiNx membranes. A 150 nm thermal oxide was grown on the silicon wafers by wet oxidation at 950°C for 31 min. This layer was subsequently used as an etch stop to protect the nitride during definition of the silicon handling wafer geometry. A low-stress SiNx layer was then deposited by LP-CVD [e.g., deposition parameters for 2 μm film are 835°C for 9.7 h with pressure 33 Pa and 12 sccm NH3, 59 sccm SiHxCl2 (SCCM denotes cubic centimeters per minute at standard temperature and pressure)]. The wafers were then patterned with a resist mask and SiNx/SiO2 windows formed by deep reactive ion etching, which removed all the silicon under the window area. The residual thermal oxide was removed with hydrogen fluoride vapor etch, leaving a set of uniform SiNx membranes each with a 10 mm diameter aperture individually suspended from the silicon handling frame.

The optical tests were performed on SiNx samples having membrane thicknesses of 0.5 and 2.3 μm with an uncertainty of 3%. Fabry–Perot resonators were made by stacking multiple samples with silicon standoff frames between adjacent samples to explore the long-wavelength response of the material in greater detail. The silicon standoffs allowed a vent path for evacuation of air between the nitride membranes. All optical measurements were performed in vacuum with a residual pressure less than 10 Pa.

The samples were characterized with a Bruker 125 high-resolution Fourier transform spectrometer (FTS) and were measured in transmission at the focal plane of an f/6 beam. A number of different sources, beam splitters, and detector configurations were used in combination to provide measurements over the reported spectral range. The single-layer SiNx sample transmission was measured over an extended range from 15 to 10,000 cm⁻¹. The mercury lamp and a multilayer Mylar beam splitter were used to access frequencies below 600 cm⁻¹. Additional mid-IR spectral data up to 2400 cm⁻¹ were acquired using a ceramic glow bar source, Ge-coated KBr beam splitter, and room-temperature deuterated tri-glycine sulfate detector. The remaining near-IR data up to 10,000 cm⁻¹ were taken with a W filament source, Si on CaF2 beam splitter, and a liquid-nitrogen-cooled InSb detector (Fig. 1). Far-IR data between 15 and 95 cm⁻¹ were taken using a mercury arc

Fig. 1. (Color online) Room-temperature transmission of a silicon nitride sample 0.5 μm thick: measured (grey), model (black dotted), and residual (red). The shaded band's width defines the estimated 3σ measurement uncertainty. A 30 GHz (1 cm⁻¹) resolution is employed for the measurement. The insert depicts the geometry of the SiNx membrane and micromachined silicon frame.
The dielectric response is represented as a function of frequency, \( \omega \), by the classical Maxwell–Helmholtz–Drude dispersion model

\[
\hat{\varepsilon}_r(\omega) = \hat{\varepsilon}_m + \sum_{j=1}^{M} \frac{\Delta \hat{\varepsilon}_j \cdot \omega_{T_j}^2}{\omega^2 - \omega_0^2 - i\omega \Gamma_j(\omega)},
\]

where \( M \) is the number of oscillators and \( \hat{\varepsilon}_r = \varepsilon'_r + i\varepsilon''_r \) is a complex function of \((5M + 2)\) degrees of freedom, which are as follows: the contribution to the relative permittivity \( \hat{\varepsilon}_m = \hat{\varepsilon}_{M+1} \) of higher lying transitions, the difference in relative complex dielectric constant between adjacent oscillators \( \Delta \hat{\varepsilon}_j = \hat{\varepsilon}_j - \hat{\varepsilon}_{j+1} \), which serves as a measure of the oscillator strength, the oscillator resonance frequency \( \omega_{T_j} \), and the effective Lorentzian damping coefficient \( \Gamma_j(\omega) \), for \( j = 1, \ldots, M \). The following functional form is used to specify the damping:

\[
\Gamma_j(\omega) = \Gamma_j \exp\left[-\alpha_j \left(\frac{\omega_j^2 - \omega_0^2}{\omega_j}\right)^2\right],
\]

where \( \alpha_j \) allows interpolation between Lorentzian \((\alpha_j = 0)\) and Gaussian wings \((\alpha_j > 0)\) similar to the approach in \([12]\). The form indicated above enables a more accurate representation of relatively strong oscillator features.

The impedance contrast between free space and the thin-film sample forms a Fabry–Perot resonator. The observed transmission can be modeled \([13]\) as a function of the dielectric response \([\text{Eq. (1)}]\), thickness, and wave-number. The dielectric parameters were solved by means of a nonlinear least-squares fit of the transmission equation to the laboratory FTS data. Specifically, a sequential quadratic programming method with computation of the Jacobian and Hessian matrices \([14,15]\) was implemented. The merit function, \( \chi^2 \), was used in a constrained minimization over frequency as follows:

\[
\min_{\text{DOF}} \chi^2 = \min_{\text{DOF}} \sum_{k=1}^{N} \left[ T(\hat{\varepsilon}_r(\omega), h) - T_{\text{FTS}} \right]^2,
\]

where \( N \) is the number of data points, \( T \) the modeled transmittance, \( T_{\text{FTS}} \) the measured transmittance data, and \( h \) the measured sample thickness. We are guided by the Kramers–Kronig relations in defining constraints for a passive material: \( |\hat{\varepsilon}_j| > |\hat{\varepsilon}_{j+1}|, \varepsilon''_j > 0 \) and \( \hat{\varepsilon}_r(0) = \hat{\varepsilon}_1 \) \([16]\). For accurate parameter determination, the sample should have uniform thickness, be adequately transparent to achieve high signal to noise, and have diffuse scattering as a subdominant process. The method requires an \textit{a posteriori} numerical verification for Kramers–Kronig consistency. In the example presented here, a numerical Hilbert transform \([17]\) of \( \varepsilon''_r(\omega) \) reproduces \( \varepsilon''_r(\omega) \) to within 2% (Fig. 3). An alternative method employing reflectivity and phase allows \textit{a priori} Kramers–Kronig consistent results \([18]\). However, given the details of the thin-film samples and available instrumentation, this approach was not implemented.

Figure 1 illustrates the measured and modeled results obtained from the analysis of a 0.5 \( \mu \)m thick sample. The peak residual in the transmittance is less than 3%, and the \( 3\sigma = 0.023 \) uncertainty band indicated corresponds to the 99.7% confidence level. The standard deviation adopted for the measured data, \( \sigma \), was estimated assuming the errors as a function of frequency were uniform and had a reduced \( \chi^2 \) equal to unity. An additional
Table 1. Fit Parameter Summary

<table>
<thead>
<tr>
<th>j [-]</th>
<th>$\varepsilon_r$ [-]</th>
<th>$\varepsilon_i$ [-]</th>
<th>$\omega_j/2\pi$ [THz]</th>
<th>$\Gamma_j/2\pi$ [THz]</th>
<th>$a_j$ [-]</th>
</tr>
</thead>
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<td>5.810</td>
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<td>15.053</td>
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<td>24.521</td>
<td>2.751</td>
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</tr>
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<td>26.440</td>
<td>3.482</td>
<td>0.0002</td>
</tr>
<tr>
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<td>0.2073</td>
<td>31.724</td>
<td>5.948</td>
<td>0.0080</td>
</tr>
<tr>
<td>6</td>
<td>4.562</td>
<td>0.0124</td>
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</table>

In Fig. 3, the values of the real and imaginary components of the dielectric function are illustrated as a function of frequency. The uncertainty in $\varepsilon_r$ was propagated and computed as described in [19]. Table 1 contains a summary of the best fit parameters for five oscillators, which can be used to reproduce the data shown in Fig. 3.

To characterize the long-wavelength portion of the dielectric function, Fabry–Perot resonators were realized from one-, two-, and three-layer samples. Representative data for the three-layer resonator stack are presented in Fig. 2. A multilayer transfer matrix analysis [13] is used to extract the dielectric function using the measured SiN$_x$ (2.3 μm) and silicon spacer (998 μm) thicknesses. The circular symbols at 1.5 and 2.5 THz indicated in Fig. 3 were computed from a composite analysis of the three Fabry–Perot measurement sets. The horizontal range indicates the data used in each fit. The best estimates are $\varepsilon_r = 7.6 + i0.08$ over the range of 2–3 THz and $\varepsilon_i = 7.6 + i0.04$ over 0.4–2 THz. The real component of the static dielectric function derived from the data is in agreement with prior reported parameters for this stoichiometry [4]. As shown in Fig. 3, the measurements are internally consistent and represent roughly a factor-of-three reduction in uncertainty relative to prior IR SiN$_x$ measurements identified by the authors [5–7]. The dielectric parameters reported here are representative of low-stress SiN$_x$ membranes encountered in our fabrication and test efforts.

References and notes

10. Addison Engineering, 150 Nortech Parkway, San Jose, California 95134 (Orientation (100), Czochralski, p-type B doped, bulk resistivity $< 0.005 \Omega \cdot cm$).