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 low tensile stress and refractive index [8]. The 5:1 SiH4:Cl2/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 μm 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, 9.7 h with pressure 33 Pa and 12 sccm BH3, 59 sccm SiH4:Cl2 (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 100 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 $3\sigma$ 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.](https://ntrs.nasa.gov/search.jsp?R=20150000283 2019-06-07T19:33:25+00:00Z) © 2012 Optical Society of America
Fig. 2. Measured (solid grey) and model (black dotted) transmission for a three-layer stack of silicon nitride samples 2.3 μm in thickness with 998 μm intermembrane delays that complements the data shown in Fig. 1. The sample response in the far-IR was acquired with a resolution of 3 GHz (0.1 cm⁻¹).

The dielectric response is represented as a function of frequency, \( \omega \), by the classical Maxwell–Helmholtz–Drude dispersion model [11],

\[
\hat{\epsilon}_r(\omega) = \hat{\epsilon}_\infty + \sum_{j=1}^{M} \frac{\Delta \hat{\epsilon}_j \cdot \omega_{j}^{2}}{\omega_{j}^{2} - \omega^{2} - i\omega \Gamma_{j}(\omega)}, \tag{1}
\]

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

\[
\Gamma_{j}(\omega) = \Gamma_{j} \exp \left[ -\alpha_{j} \left( \frac{\omega_{j}^{2} - \omega^{2}}{\omega_{j}^{2}} \right)^{2} \right], \tag{2}
\]

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:

\[
\min_{\text{DOF}} \chi^{2} = \min_{\text{DOF}} \sum_{k=1}^{N} \left[ T(\hat{\epsilon}_r(\omega), h) - T_{\text{FTS}}(\omega) \right]^{2}, \tag{3}
\]

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: \( |\hat{\epsilon}_j| > |\hat{\epsilon}_{j+1}|, \hat{\epsilon}_j' > 0 \) and \( \hat{\epsilon}_j(0) = \hat{\epsilon}_1 \) [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 \( \hat{\epsilon}_r'(\omega) \) reproduces \( \hat{\epsilon}_r''(\omega) \) 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 μm thick sample. The peak residual in the transmittance is less than 3%, and the 3σ = 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

![Fig. 3. (Color online) Real and imaginary parts (solid red curves) of the dielectric function as extracted from the data shown in Fig. 1. The line thickness is indicative of the propagated ~4% error band. The numerical Hilbert transform of the modeled \( \hat{\epsilon}_r''(\omega) \) is indicated in the upper panel (dashed blue line) to facilitate comparison with \( \hat{\epsilon}_r''(\omega) \). The filled symbols indicate the parameters derived from the data presented in Fig. 2.](image-url)
uncertainty in the FTS normalization influences the dielectric response function at the 1% level. In addition to the channel spectra, the observed spectrum shows two predominant features at 12 and 25 THz. Simulations with $M = 2$ oscillators lead to a peak residual on transmission of 5% and do not enable recovery of the resonance at 25 THz. Using five oscillators satisfactorily recovers the observed transmittance and computes as described in Eq. (4.6) in [12]. In these regions, the peak transmission residuals were decreased by a factor $\sim 2$ through the use of Eq. (2).

In Fig. 2, the values of the real and imaginary components of the dielectric function are illustrated as a function of frequency. The uncertainty in $\hat{\varepsilon}_r$ 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 $\hat{\varepsilon}_r = 7.6 + i0.08$ over the range of 2–3 THz and $\hat{\varepsilon}_r = 7.6 + i0.04$ 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.

Table 1. Fit Parameter Summary

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<tr>
<th>$j$ [-]</th>
<th>$\varepsilon_r$ [-]</th>
<th>$\varepsilon_i$ [-]</th>
<th>$\omega_r / 2\pi$ [THz]</th>
<th>$\Gamma_r / 2\pi$ [THz]</th>
<th>$\alpha_j$ [-]</th>
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<td>0.0124</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

References and notes

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