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 sub-

Transmission [–]

0 0.2 0.4 0.6 0.8

Frequency [THz]

0 100 200 300

Measured Model Residual

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 measurements were performed in vacuum with a residual pressure less than 100 Pa.

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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
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 [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:

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

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 subdominate process. The method requires an 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 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 $3\sigma = 0.023$ 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
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 \cite{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. 2.

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 \cite{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 \( \hat{\varepsilon}_r = 7.6 + i 0.08 \) over the range of 2–3 THz and \( \hat{\varepsilon}_r = 7.6 + i 0.04 \) 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 \cite{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 \cite{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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<th>( j ) [–]</th>
<th>( \varepsilon_r' ) [–]</th>
<th>( \varepsilon_i' ) [–]</th>
<th>( \omega_{\text{rj}}/2\pi ) [THz]</th>
<th>( \Gamma_{\text{rj}}/2\pi ) [THz]</th>
<th>( a_j ) [–]</th>
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<td>0.0124</td>
<td></td>
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</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 Ω cm).