Transmittance measurements for filters of optical density between one and ten

Z. M. Zhang, T. R. Gentile, A. L. Migdall, and R. U. Datla

We have developed a facility for measuring the transmittance of optical filters at a wavelength of 1064 nm, using a Nd:YAG laser, a power stabilizer, and linear photodiode detectors. A direct measurement method was used for filters with optical densities (OD's) less than or equal to 4, and a reference substitution technique was used for filters with OD's as great as 10. The apparatus and data-acquisition system are described. Measurement results for a set of filters are presented. The expanded uncertainties for the measured OD and deduced absorption coefficient are determined through a detailed analysis of all the uncertainty components. © 1997 Optical Society of America

Key words: Infrared filter, optical density, radiometry, transmittance, uncertainty.

1. Introduction

Accurate spectral transmittance measurements are important for calibration of spectrometers and for determination of the optical properties of materials.[1,2] High-accuracy spectrophotometers are commonly used for these measurements.[2–4] Recent advances in stable laser sources and highly sensitive detectors have allowed accurate determination of the spectral transmittance of filters with extremely low transmittance.[5–8] As reviewed by Gentile *et al.*,[9] both direct and heterodyne detection methods have been employed for measuring transmittance with a dynamic range as large as 10 decades at wavelengths of 633 nm (He–Ne line), 1064 nm (Nd:YAG line), and 10.2 and 10.6 μm (CO_2 lines).

A facility has been developed at the National Institute of Standards and Technology (NIST) for transmittance measurements at a wavelength of 1064 nm. Its primary components are a Nd:YAG laser, a laser power stabilizer, and linear photodiode detectors. A direct measurement method is used for filters of optical densities (OD's) from 1 to 4, and a reference substitution technique is used to measure higher OD filters. Ionically colored glass filters with OD's from 1 to 6 have been characterized and certified as Standard Reference Materials. These filters can be used to calibrate transmittance measurements made with lasers or spectrophotometers to accurately attenuate optical power or to characterize detector nonlinearity.[10] In the present paper we discuss the measurements of filters with OD's from 1 to 10, especially high OD measurements and absorption coefficient determination.

2. Theory and Instrumentation

A. Transmittance and Optical Density

For a filter plate made of a homogeneous and isotropic material with smooth and parallel surfaces, the transmittance depends on the thickness, optical constants of the material (which are wavelength and temperature dependent), the angle of incidence and polarization state of the incident electromagnetic radiation, and the degree of coherence between multiple reflected waves.[2,11] At normal incidence the reflectance at the first interface is $\rho = [(n-1)^2 + k^2]/[(n+1)^2 + k^2]$, where n and k are the real and the imaginary parts of the refractive index (both depend on wavelength). If $k \ll (n-1)$, the reflectance may be calculated from $\rho = (n-1)^2/(n+1)^2$. The internal transmittance τ for radiation propagating in the direction perpendicular to the surface is $\exp(-ad)$, where d is the thickness of the plate and $a = 4\pi k/\lambda$ (where λ is the wavelength in vacuum) is the absorption coefficient. The fraction of radiation transmitted through the plate is reduced by reflections at the two interfaces and the internal absorption. Therefore the external transmittance T is

$$T = (1-\rho)^2\tau. \quad (1)$$

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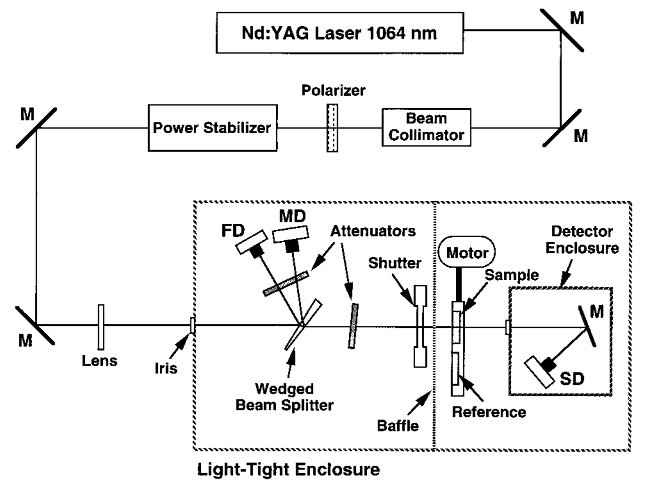

Fig. 1. Optical setup for transmittance measurements at 1064-nm wavelength: M, mirror; FD, feedback detector; MD, monitor detector; SD, signal detector.

The effects of multiple reflections are negligible for filters of OD ≥ 3 because of the low reflectance ($\rho \approx 0.04$) and strong absorption ($\tau \ll 1$) of the filter samples used in the present study. For filters of OD ≤ 2, however, expressions that include multiple reflections or interference effects should be used.[12] The relative error in transmittance caused by neglecting interference effects is discussed in Subsection 3.D.

The optical density is defined as

$$\mathrm{OD} = -\log_{10} T. \tag{2}$$

For example, OD = 1 corresponds to a transmittance value of 0.1, and OD = 8 corresponds to a transmittance value of 10^{-8}. Based on Eqs. (1) and (2), the absorption coefficient is related to the OD, the reflectance, and the thickness as

$$a = \frac{\mathrm{OD} + 2 \log_{10}(1 - \rho)}{d \log_{10} e}. \tag{3}$$

B. Measurement Techniques

The transmittance measurement setup is shown in Fig. 1. The optical source is a continuous-wave arc-lamp-pumped Nd:YAG laser with an output power of 3 W at 1064 nm. The laser beam is directed through a collimator, a polarizer and a power stabilizer, a weakly focusing lens, and then to a light-tight enclosure. A wedged quartz beam splitter inside the enclosure reflects a small portion (≈4%) of the laser power to a feedback detector (FD), which controls the stabilizer. The laser power is stabilized to better than 0.3% root-mean-square fluctuation over several hours of operation. The reflection off the second surface of the wedge is sent to a monitor detector (MD) to normalize the laser power, which reduces the effect of residual power fluctuation. The stability of the optical power and the significance of the monitor detector on the measurement are discussed in detail by Zhang *et al.*[10] The transmitted beam passes through a shutter and a sample (or a reference) and is then reflected by a mirror to the signal detector (SD). Attenuators are used to vary the laser power and to ensure that the detectors are operated in their linear ranges. A baffle at the middle of the enclosure prevents stray light from reaching the signal detector. A detector enclosure further reduces the stray light, which is necessary to measure filters of OD > 9.

The signal detector is a Hamamatsu S1337 series silicon photodiode,[13] with an active area of 10 mm × 10 mm. The detector responsivity is ≈0.15 A/W at a wavelength of 1064 nm. The signal detector is coupled to a built-in transimpedance amplifier that has nine feedback-resistor settings from 2 kΩ to 100 GΩ. Therefore the voltage responsivity after the amplifier is between 300 V/W and 1.5×10^{10} V/W. A 6½ digit digital voltmeter (DVM) measures the dc voltage from the amplifier. The electronic design and test results of the amplifiers used with photodiode detectors to achieve a linear dynamic range of 14 decades are discussed in Refs. 5 and 6. Another DVM simultaneously measures the output voltage from the monitor detector–amplifier.

The filter is mounted on a copper holder. A thermistor on the holder monitors the filter temperature during the measurement. Computer-controlled motors move the filter holder horizontally and vertically to position the sample or reference (the reference is either blank or another filter). An automatic data-acquisition program controls the motion of the shutter and the motors, controls the DVM's (one of which also measures the resistance of the filter thermistor), and calculates the transmittance and optical density for each measurement.

Six measured values determine the relative transmittance of the sample filter at a single position, namely,

$$T_{\mathrm{relative}} = \frac{[(V_{s1} - V_{s0})/V_m]_{\mathrm{sample}}}{[(V_{s1} - V_{s0})/V_m]_{\mathrm{reference}}}, \tag{4}$$

where V is the output voltage, subscripts s and m indicate the signal detector and the monitor detector, respectively, and subscripts 0 and 1 indicate shutter closed and open, respectively. The signal when the shutter is closed (V_{s0}) is subtracted from the output signal (V_{s1}) to eliminate background. The DVM integration time is approximately 5 s (bandwidth 0.2 Hz).

The reference is left blank for transmittance measurements for filters with OD's 1 to 4. Hence the sample transmittance is the same as the relative transmittance calculated from Eq. (4). The gain setting cannot be changed during each measurement. The signal-to-noise ratio is lower with higher-OD filters, since the minimum measurable voltage of the DVM is 1 μV. For filters of OD ≥ 5, a reference substitution method was used, similar to the step-down method employed by Eckerle *et al.*[2] to measure the spectral transmittance down to OD = 4 with a spectrophotometer. The transmittance of the reference filter was measured at a fixed position by use of a lower gain setting. A higher gain setting was used to measure the transmittance of a high OD filter relative to that of the reference filter. The transmittance of the sample filter is calculated by

$$T_{\text{sample}} = T_{\text{relative}} \times T_{\text{reference}}\,, \tag{5}$$

where $T_{\text{reference}}$ is the transmittance of the reference filter at the fixed position. An OD 3 filter was used as the reference for filters of OD 5, 6, and 7; an OD 4 filter was used as the reference for filters of OD 8; and an OD 8 filter was used as the reference for filters of OD 9 and 10.

3. Results and Discussion

The filters used in this study were made of an ionically colored glass material (NG-9) manufactured by Schott of Mainz,[14,15] Germany. The filters are uncoated and optically polished. Each surface is flat to within one-tenth of the He–Ne wavelength (633 nm), and the wedge angle is less than 5 μrad (1 arc sec). The filters are 51 mm × 51 mm, with thicknesses between 1 and 10.5 mm. The OD is determined by the thickness of the filter.

The laser beam incident on the filter was perpendicular to the surface (angle of incidence, $<2°$). This was achieved by observation of the reflected beam with an infrared sensing card. The slight translation of the beam through the filter (maximum 0.12 mm) has little effect on the measurement because of the high degree of spatial uniformity of the detector. The beam diameter at the filter was ≈3-mm full width at half-maximum, and the beam divergence is less than 2 mrad. The detector was tilted ≈1° to eliminate interreflections between the detector and the filter. The temperature of the laboratory was between 22 and 24 °C, except during the study of the temperature effect as discussed in Subsection 3.B.

A. Detector Linearity and Noise Equivalent Power

The linearity of the detector was tested by measuring the OD at different power levels. Attenuation filters were used to change the incident power. The amplifier gain was optimized to yield the best signal-to-noise ratio without saturating the DVM. The OD of two filters measured with different laser powers is shown in Fig. 2. It can be seen that the detector is extremely linear at optical powers of less than 1 mW. With an input optical power of 1 nW, the power reaching the detector after the OD 4 filter is only 10^{-13} W, resulting in a low signal-to-noise ratio and hence a large standard uncertainty. Measurements of zero signal (i.e., with the detector blocked) indicated that the noise power is of the order of 10^{-14} W for a 5-s measurement time (i.e., bandwidth of 0.2 Hz), which agrees with the manufacturer-specified noise equivalent power.[13] The laser power at the filter position was limited to 20 mW to avoid excessive heating of the filter. Therefore the dynamic range of the measurement is ~12 decades by the reference substitution method.

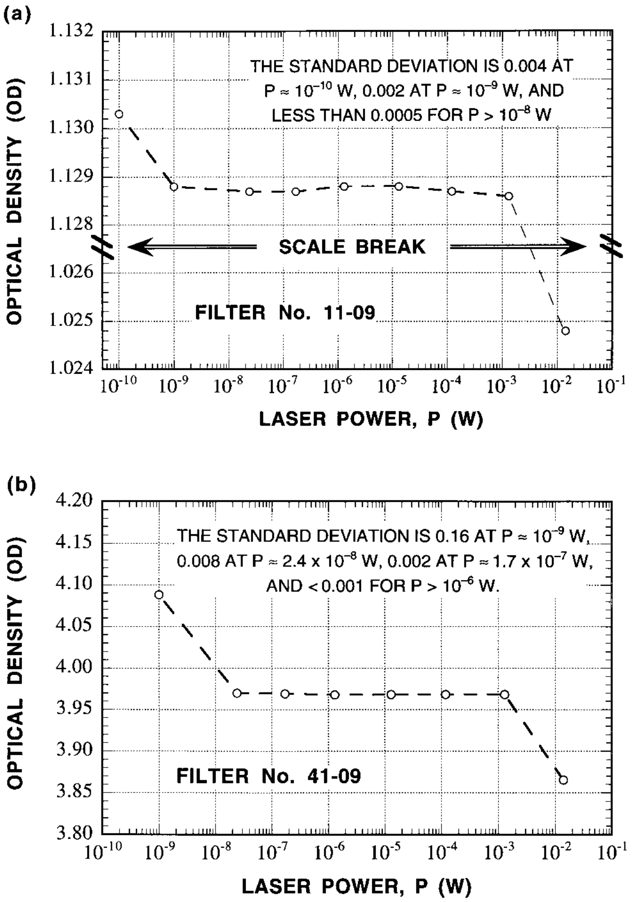

Fig. 2. Optical density OD of two filters measured at different laser powers: (a) nominal OD = 1; (b) nominal OD = 4.

B. Temperature Dependence of the Absorption Coefficient

The temperature of the filter was varied to allow us to investigate the effect on the optical density. The central air conditioner in the building was used to change the temperature in the laboratory from 23 to 27 °C. The filter temperature was assumed to be the same as that of the copper holder. The filter temperature was ~1 °C higher than room temperature owing to the heating of the positioning motors. To reduce the filter temperature below 24 °C, cold N_2 gas from a liquid-nitrogen tank was passed through the enclosure. The enclosure was closed after the cooling. The OD and the filter temperature were measured as the filter temperature increased. The change of the filter temperature (1 to 2 °C per hour) was slow enough to obtain a correlation between the OD and temperature. The measurements were made at the same position of the filter to eliminate the effect of spatial nonuniformity. A linear fit shows that a 1 °C temperature rise increases the OD by 0.00052 for an OD 2 filter and by 0.0008 for an OD 3 filter. The change in OD is almost proportional to the filter thickness, indicating that it is not caused by a change in the reflectance (which depends on the refractive index n) but is caused by a change in the internal absorption. The thickness change is negli-

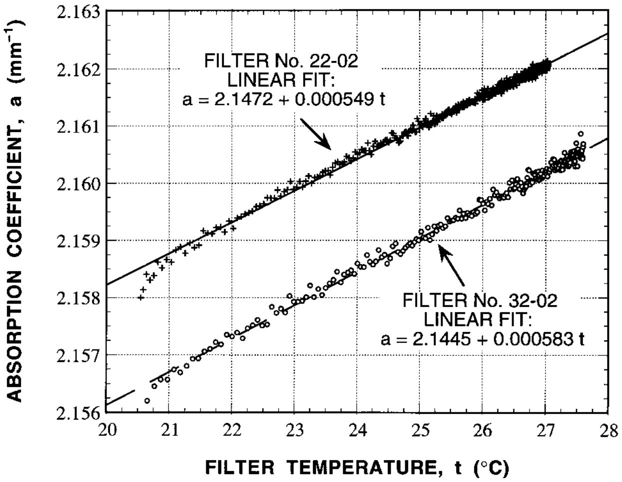

Fig. 3. Temperature dependence of the absorption coefficient.

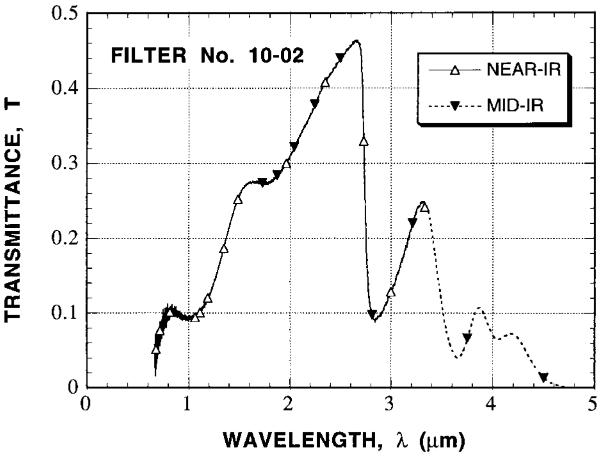

Fig. 4. Transmittance spectra measured with a Fourier transform infrared spectrometer.

gibly small, since the thermal expansion coefficient of the glass material is $\approx 6.3 \times 10^{-6}$ °C^{-1}.[14] Hence, the change in OD must be caused by a change in the absorption coefficient of the glass material. The absorption coefficient calculated from Eq. (3) is shown in Fig. 3 for both OD 2 and OD 3 filters, with the calculated reflectance of 0.04 used. The calculated absorption coefficient and the slope agree well between the two filters. The small difference in the absorption coefficient may be caused by the material or the uncertainty in the thickness determination. The thicknesses were measured using a micrometer with an expanded uncertainty of 2.5 μm. The absorption coefficient calculated from Eq. (3) is approximately 2.16 mm^{-1} at 25 °C and increases ≈0.026% °C^{-1} at $\lambda = 1064$ nm. The first several data points did not follow the linear curve very well, which could be explained by the thermal nonequilibrium between the filter and the holder during the first few minutes (it takes ~100 s to measure a data point).

C. Wavelength Dependence of the Transmittance

A Fourier transform infrared spectrometer was used to determine the wavelength dependence of the transmittance. A halogen source, a quartz beam splitter, and a pyroelectric detector were used for $0.85\ \mu\text{m} < \lambda < 3.3\ \mu\text{m}$. A SiC source, a Ge-coated-KBr beam splitter, and a pyroelectric detector were used for $1.8\ \mu\text{m} < \lambda < 25\ \mu\text{m}$. The beam exiting the interferometer was focused at the filter with a ≈8-mm diameter spot and a maximum divergence angle of ≈7°. The measured transmittance spectra for an OD 1 filter is shown in Fig. 4. The spectral resolution was 8 cm^{-1} (≈0.9 nm at 1064 nm), yielding nearly continuous curves. The transmittance at wavelengths greater than 5 μm was less than 10^{-5} (not shown in Fig. 4). The optical power was attenuated to improve the radiometric accuracy at the expense of a reduction of the signal-to-noise ratio.[16,17] The relative differences in the overlapping region between 1.8 and 3.3 μm were at the 1% level, i.e., within the overall uncertainty of the measurements. No absorption lines were observed, and the transmittance spectra were relatively flat near 1064 nm. The bandwidth of the Nd:YAG laser is less than 0.5 nm at 1064 nm.[18,19] The relative difference in transmittance between measurements made with the laser and those made with the spectrometer at 1064 nm is ≈1%. This demonstrates that these filters are appropriate for use in calibrating infrared spectrometers at 1064 nm.

D. Spatial Nonuniformity and Interference Effects

The spatial variation in OD depends on the individual filter. Transmittance measurements were performed either at 9 positions in a 3 × 3 matrix with 10-mm spacing or at 25 positions in a 5 × 5 matrix with a 5-mm spacing around the center of the filter. These measurements were repeated at least once for all positions. Because the surfaces of the filters are extremely flat and parallel, the spatial variation is attributed to the inhomogeneity of the material except for OD 1 filters. For OD 1 filters, interference effects of multiple reflections must be taken into consideration.

For normal incidence the transmittance of a plate with two parallel, optically smooth surfaces for completely coherent radiation is[12]

$$T = \frac{(1-\rho)^2\tau}{1 + \rho^2\tau^2 - 2\rho\tau\cos(4\pi n d/\lambda)}. \tag{6}$$

The denominator oscillates as $\xi = 2nd/\lambda$ varies. The amplitude of oscillation is estimated to be 0.8% for OD 1 filters and 0.08% for OD 2 filters. For OD 1 filters ($d \approx 1.04$ mm), a variation of either 0.18 μm in d, of 0.17 nm in λ, or of 0.016% in n would change the transmittance from a maximum to a minimum. The linewidth $\Delta\nu$ of the laser is between 1 and 5 cm^{-1}.[20] Therefore the laser radiation is not completely coherent.[11] From the formula for partially coherent radiation given by Zhang,[11] the amplitude of oscillation can be calculated from $\Delta T/T \approx 2\rho\tau$

Table 1. Uncertainty in OD for a Single Measurement at a Fixed Position[a]

Nominal OD	Repeatability σ_{repeat}	Temperature Variation σ_{temp}	Reference σ_{ref}	Combined Standard Uncertainty σ_{comb}	Expanded Uncertainty $2\sigma_{\mathrm{comb}}$
1	0.00006	0.00007	NA	0.00041	0.0008
2	0.00006	0.00015	NA	0.00043	0.0009
3	0.00020	0.00022	NA	0.00050	0.0010
4	0.00067	0.00030	NA	0.00084	0.0017
5	0.00020	0.00038	0.00050	0.00077	0.0015
6	0.00020	0.00045	0.00050	0.00081	0.0016
7	0.00100	0.00053	0.00050	0.00130	0.0026
8	0.00100	0.00060	0.00084	0.00149	0.0030
9	0.00100	0.00067	0.00149	0.00197	0.0039
10	0.00600	0.00075	0.00149	0.00624	0.0125

[a] The reproducibility and nonlinearity components are the same for all the filters.

$\mathrm{sinc}(2\pi n d \Delta\nu)$, where $\mathrm{sinc}(x) = \sin(x)/x$. Using $\Delta\nu = 1\ \mathrm{cm}^{-1}$, the standard uncertainty in OD caused by interference effects (σ_{interf}) is estimated to be 0.0016 for OD 1 filters, 0.0001 for OD 2 filters, and negligible for filters of OD ≥ 3. The large spatial variation for OD 1 filters is likely caused by interference effects, since the thickness variation is of the order of 0.2 μm. Variations in the range of ± 0.0025 OD were observed for an OD 1 filter as the filter temperature was varied from 21 to 27 °C, which could be explained by a slight temperature dependence of n. The OD variation is less than $2\sigma_{\mathrm{interf}}$, indicating that the calculated σ_{interf} on the basis of $\Delta\nu = 1\ \mathrm{cm}^{-1}$ is still a conservative estimate.

E. Measurement Uncertainty

The uncertainty for a measurement at a single position is calculated according to the guidelines given by Taylor and Kuyatt.[20] The standard uncertainty associated with the measurement repeatability (σ_{repeat}) at the same position is shown in Table 1. The repeatability is better for the OD 5 and OD 6 filters than for the OD 4 filter, because the substitution technique has improved the signal-to-noise ratio. The reproducibility (σ_{reprod}) of the experimental setup was calculated based on various sets of measurements with filters of OD 1, 2, and 3, performed during a couple of weeks when the samples had been unmounted and remounted. The reproducibility of approximately 0.00035 was obtained and used to calculate the measurement uncertainty for all filters. The standard uncertainty associated with the detector nonlinearity (σ_{nonlin}) was estimated to be 0.0002. The standard uncertainty caused by the temperature variation (σ_{temp}) of ± 0.5 °C is also given in Table 1. Since the absorption coefficient changes with temperature, σ_{temp} is proportional to the filter thickness. For filters with OD ≥ 5, the uncertainty due to the reference measurement (σ_{ref}) needs to be included. Filters of OD 5, 6, and 7 were measured with an OD 3 filter used as the reference; filters of OD 8 were measured with an OD 4 filter used as the reference; and filters of OD 9 and 10 were measured with an OD 8 filter used as the reference. The combined uncertainty for a measurement at a single location is[20]

$$\sigma_{\mathrm{comb}} = (\sigma_{\mathrm{repeat}}^2 + \sigma_{\mathrm{reprod}}^2 + \sigma_{\mathrm{nonlin}}^2 + \sigma_{\mathrm{temp}}^2 + \sigma_{\mathrm{ref}}^2)^{1/2}, \quad (7)$$

where $\sigma_{\mathrm{reprod}} = 0.00035$ and $\sigma_{\mathrm{nonlin}} = 0.0002$ for all filters. The expanded uncertainty (95% confidence) is twice the combined uncertainty (see Table 1). An absolute difference of 0.001 in OD corresponds to a relative difference $\Delta T/T$ of 0.23% in transmittance. Therefore the relative expanded uncertainty in trans-

Table 2. Measurement Results for a Set of Filters with OD's from 1 to 10[a]

Filter No.	d (mm)	Filter Temp. (°C)	$\sigma_{\mathrm{spatial}}$	σ_{interf}	Optical Density OD ± ΔOD	Absorption Coefficient $a \pm \Delta a$ (mm^{-1})
10-01	1.044	24.7	0.00129	0.0016	1.0111 ± 0.0042	2.1518 ± 0.0113
22-01	2.177	25.4	0.00065	0.0001	2.0785 ± 0.0016	2.1609 ± 0.0035
32-01	3.157	23.8	0.00021	NA	2.9931 ± 0.0011	2.1572 ± 0.0023
42-01	4.194	24.1	0.00072	NA	3.9679 ± 0.0022	2.1590 ± 0.0020
54-01	5.410	25.1	0.00026	NA	5.1140 ± 0.0016	2.1615 ± 0.0014
64-01	6.391	25.3	0.00023	NA	6.0325 ± 0.0017	2.1606 ± 0.0012
75-01	7.526	24.8	0.00160	NA	7.1015 ± 0.0041	2.1619 ± 0.0015
84-01	8.418	25.2	0.00960	NA	8.1084 ± 0.019	2.2082 ± 0.0054
94-01	9.413	24.9	0.00200	NA	9.1038 ± 0.0056	2.2183 ± 0.0016
105-01	10.549	24.7	0.00900	NA	10.1885 ± 0.022	2.2162 ± 0.0048

[a] Expanded uncertainties are given as ± values.

mittance is less than 0.25% for filters of OD 1 to OD 3, less than 0.4% for filters of OD 4 to OD 6, less than 1% for filters up to OD 9, and less than 3% for filters of OD 10.

F. Results for a Set of Filters

Table 2 shows the measurement results for a set of filters from OD 1 to 10. The filter thickness was measured with a micrometer with an expanded uncertainty $\Delta d = 2.5\ \mu\text{m}$. The average temperature during each measurement is listed (with an expanded uncertainty of 0.5 °C). The uncertainty given in Table 2 associated with the nonuniformity (σ_{spatial}) is the standard deviation of the measurements at different positions of the filter. The average OD values for 9 or 25 positions are listed together with the overall expanded uncertainty, which includes the spatial nonuniformity and interference effects:

$$\Delta\text{OD} = 2 \times (\sigma_{\text{comb}}^2 + \sigma_{\text{spatial}}^2 + \sigma_{\text{interf}}^2)^{1/2}. \qquad (8)$$

The spatial nonuniformity caused by the inhomogeneity of the material is a major source of uncertainty in the measurements for filters with $\text{OD} \geq 2$. The uncertainty caused by interference effects dominates the overall uncertainty in the measurement of OD 1 filters.

The absorption coefficient for each filter was calculated from Eq. (3). The refractive index of NG-9 glass was assumed to be 1.5,[14] which yields a reflectance $\rho = 0.04$. The expanded uncertainty of the absorption coefficient was determined from the expanded uncertainties of the optical density (ΔOD), the thickness ($\Delta d = 2.5\ \mu\text{m}$), and the refractive index (assuming a 1% relative expanded uncertainty, i.e., $\Delta n = 0.015$):

$$\Delta a = \left[\left(\frac{\partial a}{\partial \text{OD}}\,\Delta\text{OD}\right)^2 + \left(\frac{\partial a}{\partial \rho}\frac{\partial \rho}{\partial n}\,\Delta n\right)^2 + \left(\frac{\partial a}{\partial d}\,\Delta d\right)^2\right]^{1/2}. \qquad (9)$$

4. Conclusions

A Nd:YAG laser, a power stabilizer, and several linear photodiode detectors were employed to characterize the optical densities of transmission filters (51 mm × 51 mm) at a wavelength of 1064 nm for a laser beam normally incident on the filter. The optical density of these filters ranged from OD 1 to OD 10 depending on filter plate thickness. The expanded uncertainty for the OD measurements at a single position was estimated to be <0.001 for filters of OD 1 to OD 3, <0.002 for filters of OD 4 to OD 6, <0.004 for filters of OD 7 to OD 9, and <0.013 for filters of OD 10. The spatial inhomogeneity of the material is a major source of uncertainty. The standard uncertainty in OD associated with the spatial nonuniformity ranges from 0.0002 to 0.01 for the measured filters. Interference effects introduced a large uncertainty in the measurement of OD 1 filters. The absorption coefficient for each filter was obtained with a relative expanded uncertainty between 0.06% and 0.52%. The effect of temperature on the absorption coefficient was investigated at temperatures from 21 to 27 °C, which showed that the absorption coefficient at 1064 nm increases with temperature by $\approx 0.026\%\ ^{\circ}\text{C}^{-1}$.

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