Characterization of High-OD Ultrathin Infrared Neutral Density Filters

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ABSTRACT

We have performed transmittance measurements of metal-film neutral density filters on ultrathin polymer substrates using both Fourier-transform infrared spectrometer and laser-based (3.39 μm and 10.6 μm) systems. The use of ultrathin substrates, free of etaloning effects over the 2 μm to 20 μm spectral range, allows the FT-IR and laser measurements to be directly compared. We discuss the evaluation of the uncertainties in the transmittance values in both types of systems.

Keywords: neutral-density filter, infrared, transmittance

1. INTRODUCTION

High optical-density ($OD = -\log_{10}(T)$, where T is transmittance) filters for the infrared are useful for expanding the dynamic range of detector systems or for attenuating the output flux of lasers in known steps. Absorptive filters are widely used for specific wavelength lasers. Examples include doped glass filters for the near infrared, or BaF_2 crystalline filters for the 9 μm to 11 μm CO_2-laser band. They have the advantage that the OD can be made nearly linear in thickness over a given range, and often are available to cover a fairly wide dynamic range of OD 0 to 6. The filter materials are usually robust and able to withstand modest temperature excursions due to the absorption of several watts of laser power. However, the absorption coefficients are temperature dependent and thus may change under laser irradiation. Also, the absorption mechanism is usually fairly narrow-band (e. g., low-lying electronic excitations in dopants in the glass for the short-wave infrared, or the multi-phonon sum band absorption edge in crystals for the mid or long-wave infrared). Thus, a different set of calibrated filters is needed for each laser or detector system.

Reflective filters, on the other hand, typically consisting of thin metallic coatings on dielectric substrates, can have very neutral attenuation versus wavelength over a wide range of infrared wavelengths.[1] Filters with nominal ODs of 0 to 4 are commercially available. However, since the substrates are thin (~1 mm) parallel-sided dielectrics, these filters show large amplitude Fabry-Perot interference fringes in their transmittance spectra. While these fringes often are not an issue in low-resolution broadband applications, they can cause considerable difficulty when using these filters with narrow-band coherent radiation, as from a laser. In principle, the spectrum can be measured at high resolution with nearly plane-wave radiation, and then averaged appropriately over wavelength and angle of incidence to match a given optical configuration. However, in practice the precise beam geometry and bandwidth of the source are usually not known with sufficient accuracy for this approach to work well. In addition, non-uniformity in the thickness of the substrate makes the visibility and location of the fringes very sensitive to the size and position of the irradiated area on the filter.

An alternative approach to making filters, which retains the neutral attenuation versus wavelength, but eliminates the Fabry-Perot fringing effects, is to deposit thin metallic coatings on ultra-thin (~100 nm) substrates.[2] Such substrates are produced commercially by Luxel Corporation. Because of the lack of interference fringes, these filters can be measured and used in a wide variety of systems, both broad-band and narrow-band, and easily achieve uncertainties of 1% or less. An associated paper discusses in detail the design and production of these unique filters.[3] In this paper, we discuss transmittance measurements on these filters with both laser and Fourier-transform infrared (FT-IR) spectrometer systems, along with an evaluation of the uncertainties in these measurements.

2. EXPERIMENTAL DETAILS

Several spectrometer systems have been used to measure the transmittance of the high-OD ultra-thin neutral density filters. Two different FT-IR spectrometers, a Bio-Rad[4] FTS-60A and a Bomem DA3, can be used to make transmittance measurements on filters with OD up to 6 in the near-infrared, and OD 4 to 5 in the long-wave infrared. FT-IR transmittance

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measurements are subject to a wide variety of of errors, which often necessitate reducing the flux in the interferometer system in order to eliminate them. This limits the range over which optical density measurements may be accurately performed. Figure 1 shows a schematic optical layout for FT-IR transmittance measurements on the ultrathin filters.

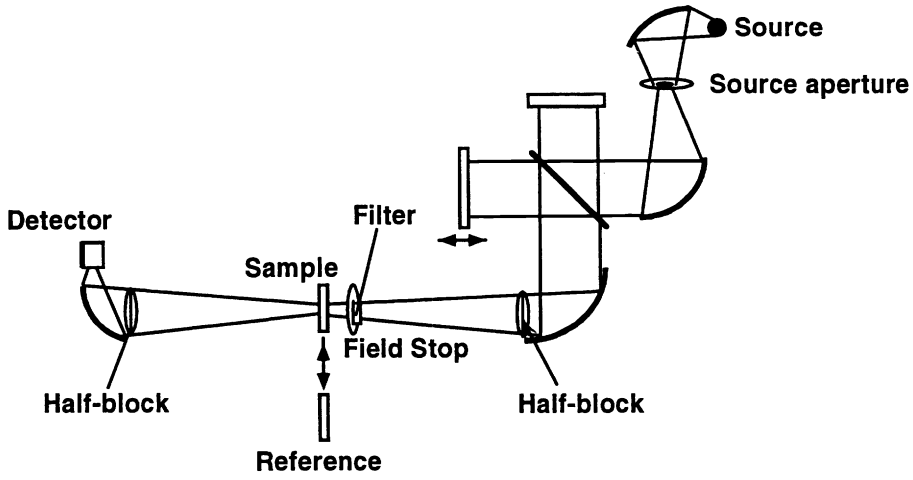


Figure 1. Optical layout of FT-IR spectrometer system for transmittance measurements of thin neutral-density filters.

For mid-infrared (2 μm to 25 μm wavelength) measurements, the FT-IR spectrometer is configured with a ceramic globar source, a KBr:Ge beamsplitter, and either a room temperature pyroelectric or liquid-nitrogen cooled mercury-cadmium-telluride (MCT) photon detector. Half beam-blocks made of infrared absorbing felt are placed as shown in Figure 1 to prevent inter-reflections among the detector, sample, and interferometer. A field stop is placed just in front of the sample position in order to limit diffuse thermal radiation from the source aperture, which heats up from the source irradiance. A filter can be placed over the field stop, or exchanged with the filter under test in order to keep the detector response linear and equivalent for both the sample and reference measurements. Typically the sample and reference are measured and exchanged several times each in order to reduce the effects of instrumental drift, and to evaluate the statistical (type A) components of

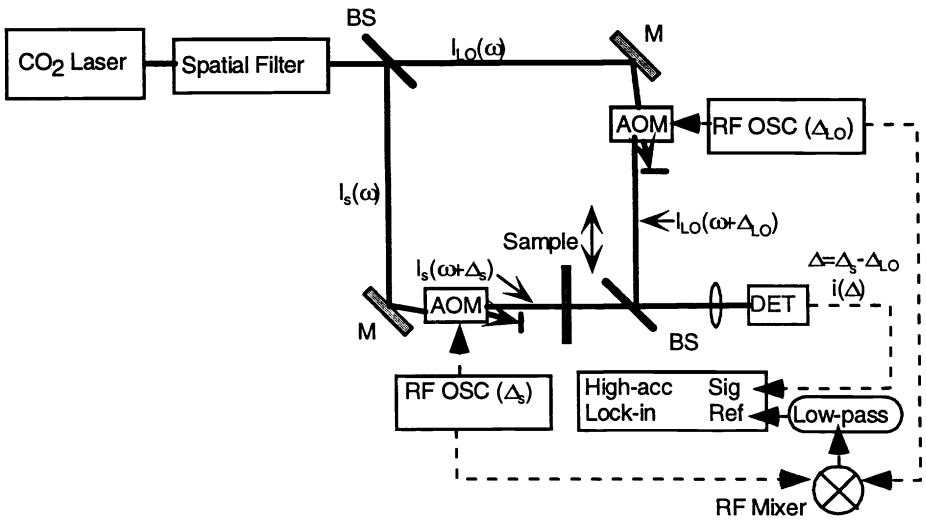


Figure 2. CO_2 laser heterodyne system for measuring transmittance of high-OD filters. BS, beamsplitter; M, mirror; AOM, acousto-optic modulator.

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the uncertainty in transmittance. An investigation of the uncertainties in these measurements has been presented earlier.[5]

Two laser systems have also been used for measurements on the high-OD neutral density filters. Figure 2 shows a schematic layout for a CO_2 laser system which employs an optical heterodyne detection system[6] to enable transmittance measurements up to OD 12. The beam is split at the first beamsplitter, and the two resultant beams are sent through acousto-optic modulators (AOMs) tuned to 70 MHz and 40 MHz, respectively. The frequency-shifted beams are recombined at the second beamsplitter, and the heterodyne signal on the room-temperature photo-electromagnetic MCT detector is recorded with a high-accuracy lock-in amplifier. The sample can be placed after the second beamsplitter, for a conventional "direct" transmittance measurement, which can be done up to OD 6. Or, if the sample is placed in one of the arms of the modified Mach-Zender interferometer, the heterodyne signal is proportional to the square root of the sample transmittance, effectively doubling the dynamic range of the measurement. The heterodyne configuration also has the advantages of restricting the change in flux level at the detector to only ~ 50% between a perfectly transparent and perfectly opaque sample, and greatly reducing the optical bandwidth, effectively eliminating the room temperature thermal background radiation.

This system has been characterized extensively and the general factors governing the uncertainty in transmittance that can be achieved with it have been evaluated. We consider below several factors specific to the thin filter measurements, such as possible inter-reflection, polarization, angle-of-incidence, and non-uniformity effects. For these tests, the filters can be mounted on a translation/rotation stage, and a half-wave plate placed in the beam path before the sample in order to vary between s and p polarization at the sample surface. For these tests, the sample was placed after the interferometer.

Figure 3 shows the layout of a 3.39 μm HeNe laser system for filter transmittance measurements. In this system, the beam is split, with one half going to a reference detector and the other half going to the sample. The beam is chopped and the two lock-in amplifier readings are ratioed to reduce the effects of laser drift on the transmittance measurement. The sample and reference are mounted on a translation stage and moved in and out of the beam under computer control to calculate the transmittance and relative standard uncertainty in the mean. Glass filters are used to attenuate the beam in steps to keep the detector (either a room-temperature pyroelectric or liquid-nitrogen cooled InSb) response linear.

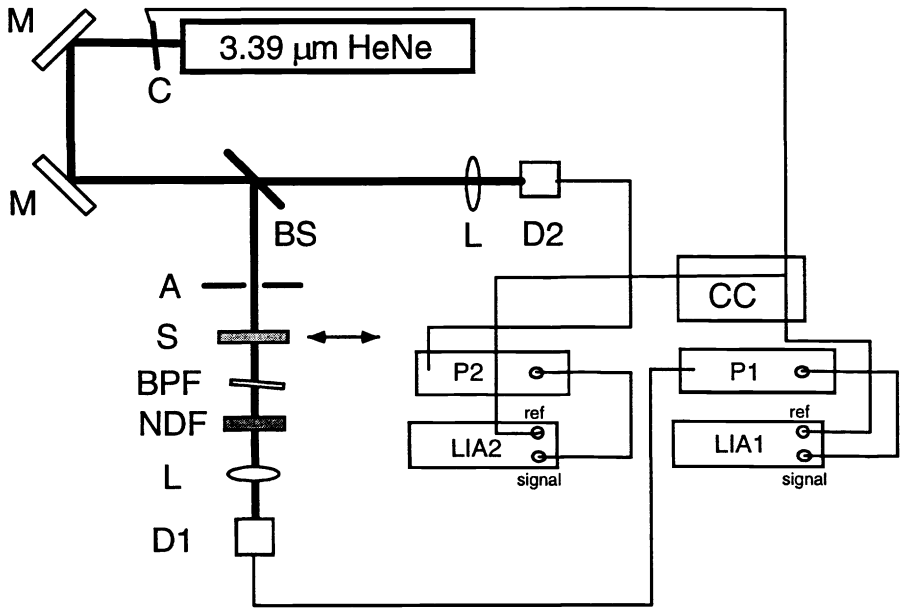


Figure 3. Schematic layout of 3.39 μm HeNe laser transmittance system. C, chopper; M, mirror; BS, beamsplitter; L, lens; D, detector; A, aperture; S, sample; BPF, band-pass filter; NDF, glass filter; CC, chopper controller; P, pre-amp; LIA, lock-in amplifier.

A 3.39 μm band-pass filter is placed in the beam path to remove unwanted emission from the laser. The nominal laser output power is ~ 2 mW. Using the InSb detector, this power level is sufficient to measure up to OD 9.5. Lower OD (1 to 2)

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samples are measured directly against a blank reference, using the pyroelectric detector, while higher OD samples (3 to 9.5) are measured against a lower OD (2 to 4) reference sample using the InSb detector.

3. RESULTS AND DISCUSSION

A number of thin neutral-density filters have been characterized with the systems described in the previous section. Figure 4 shows a comparison of measurements[5] made with the two laser systems and an FT-IR system for a NiCr/Lexan filter with an OD value of ~ 2.4.

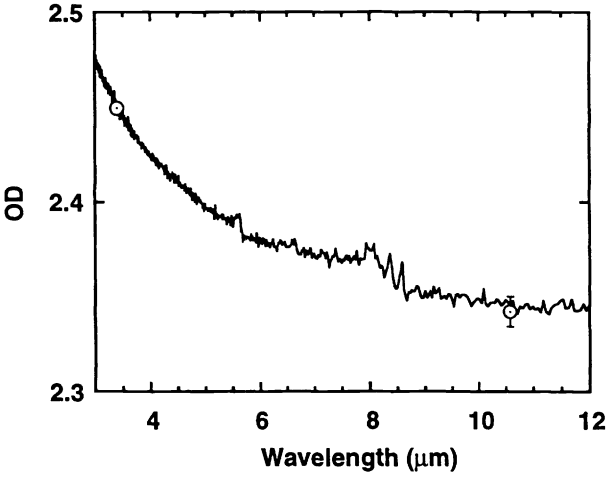


Figure 4. Comparison of OD measurements of a NiCr/Lexan film on the two laser systems (circles) and an FT-IR system (solid line).

As can be seen from this graph, the difference between the laser measurements and the FT-IR measurement is less than the estimated uncertainties (coverage factor = 2) in the laser measurements. The comparison becomes more difficult to make at higher OD levels due to the limitations of the FT-IR system, but Figure 5 shows a similar comparison on a Au/NiCr/Lexan filters with an OD level near 4. The noise in the FT-IR data, which was acquired with a pyroelectric detector, is larger than the estimated uncertainties in the laser OD values. However, the laser transmittance values are close to the average transmittance measured with the FT-IR system over a ~ 2 μm bandwidth. (This is not immediately apparent in Figure 5 because of the logarithmic scale on the vertical axis.) More sensitive FT-IR measurements using the MCT detector are

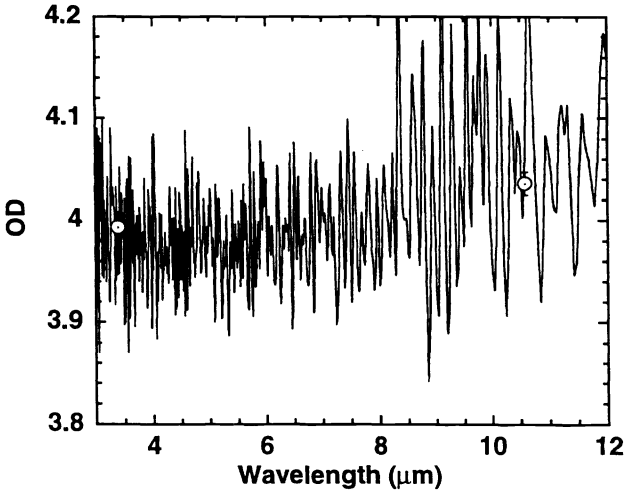


Figure 5. Comparison of OD measurements of a Au/NiCr/Lexan film on the two laser systems (circles) and an FT-IR system (solid line).

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planned.

Metal coatings on Lexan and polyimide substrates have been designed and tested to produce neutral density filters up to an optical density of 10 over the 2 μm to 25 μm wavelength region. These consist of various different combinations of metals to produce attenuations that vary as little as possible (less than 1 in OD) over this range of wavelengths. Table I shows OD values obtained for a set of these filters using the two laser systems.

Table I. Measured optical density values for five thin metal film/Lexan filters at two different laser wavelengths, along with expanded (coverage factor=2) uncertainties in OD.

Filter	NF111-126	NF111-127	N111-110	N111-140	N111-106
OD (3.39 μm)	3.376±0.02	5.304±0.017	7.984±0.012	7.996±0.013	9.55±0.25
OD (10.6 μm)	3.47±0.04	5.42±0.004	8.50±0.015	8.363±0.017	9.021±0.028

It can be seen from the values in this table that the metal film/Lexan filters are reasonably neutral as a function of wavelength, and with optimization of the coating compositions and thicknesses, can be tailored to the desired optical density.

4. SOURCES OF UNCERTAINTY IN LASER MEASUREMENTS

Lasers provide intense, monochromatic, and collimated light, making transmittance measurements of samples which are free of etaloning effects fairly straightforward to perform. However, there are a number of possible systematic (type B) uncertainties in these measurements. Type A uncertainties are mainly those due to drift in the laser power and noise in the detectors, which vary for different samples and laser powers. These are evaluated in each case by repeating the measurements and calculating the sample statistics. Also, there are type B uncertainties associated with the sample such as non-uniformity or temperature dependence, which also need to be evaluated for each individual type of sample. In Table II, we list estimates of both types of uncertainties for the two laser systems for an OD 8 metal film/Lexan sample.

Offset in the electronics can lead to an error in the transmittance, which can be corrected by measuring the signal with the laser beam blocked and subtracting it from both the sample and reference measurements. In the 10.6 μm system, this effect was found to be negligible for all but the highest OD (>10) samples, while in the 3.39 μm system, the effect was important at the level of OD 8 or more. In both cases, the residual error can be reduced by using sample and reference filters that are fairly close together in optical density.

Another source of uncertainty is inter-reflections between the sample and laser or other optical elements. This can lead to an increase in apparent transmittance, while reflections back into the laser can cause the laser to become unstable. Because the laser beam is highly collimated, only a small tilt in the sample is needed to eliminate this effect. One way to test for the presence of inter-reflection effects is to tilt the sample and see if the measured transmittance follows the expected form according to the Fresnel equations for s and p polarized light incident on the sample. Figure 6 shows an example of this test using the 10.6 μm system with an OD 1.65 sample.

As can be seen in Figure 6, the measured optical density versus angle follows the expected behavior fairly closely, with no evidence of a sudden change in signal at small angles that would indicate an inter-reflection effect. The calculation of the expected signal was done using an estimated complex index of refraction, and thicknesses for the NiCr and Lexan films derived from fitting the transmittance measured with the FT-IR spectrometer to a Drude form for the NiCr optical conductivity. However, the behavior shown in Figure 6 was found to be fairly insensitive to the details of this fit.

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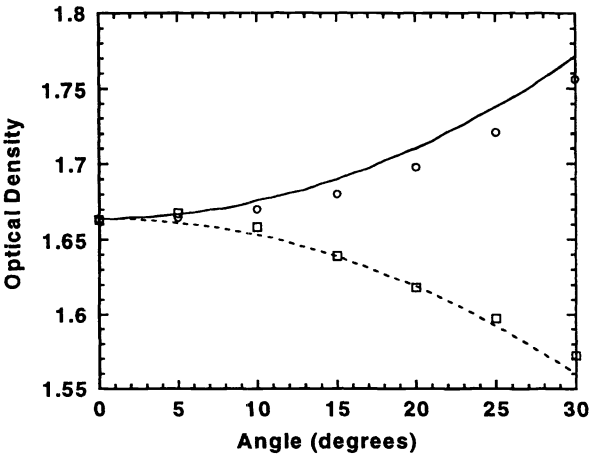


Figure 6. Measured OD value for a NiCr/Lexan sample at 10.6 μm as a function of angle of incidence for s (circles) and p (squares) polarized incident light, along with the predicted behavior (solid and dashed lines) for each mode as described in the text.

The effect of non-linearity in the detector or electronics was tested for by measuring the power dependence of the transmittance, using filters to attenuate the beam, as shown in Figure 7. This figure shows the incident power dependence of the measured OD at 10.6 μm of the NiCr/Lexan sample shown in Figure 6. The error bars are just the standard uncertainty in the mean evaluated from repeating the measurements several times and vary due to drift in the laser system. For measurements done with less than 5 mW incident power, the measured OD level seems to saturate around a value of 1.665, with the lowest power point at ~ 1 mW somewhat higher, but with a large error bar.

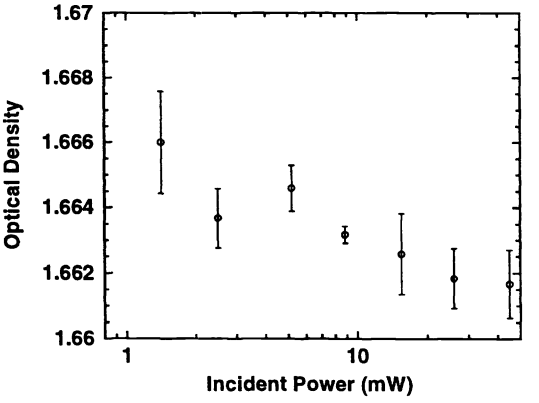


Figure 7. Incident power dependence of measured optical density for a NiCr/Lexan sample at 10.6 μm.

Emission of light from the laser or other components in the system at wavelengths other than the nominal laser wavelength is another potential source of uncertainty in the measurements. This was filtered out in the 3.39 μm system using the narrow-band filter mentioned above, which was characterized with an FT-IR spectrometer and found to have less than 10^{-4} out-of-band transmittance. In the 10.6 μm system the modulation of the two beams is very effective at eliminating signals from background thermal radiation. Effects of uncertainty in the angle of incidence (~0.5°) and beam polarization are on the order

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of 0.0003 in optical density for this type of sample. Vignetting of the laser beam by the samples was tested using a blank sample holder and found to be negligible; the beam diameter was typically less than 1/3 of the sample diameter. Also, the effects of the ultra-thin samples on the beam geometry at the detector are negligible. Table II below lists estimated standard uncertainties discussed in this section for both laser systems, with the total expanded uncertainty (coverage factor = 2) calculated for an OD 8 sample.

Table II. Estimated standard uncertainties in OD and expanded (k=2) uncertainty for an OD 8 metal film/Lexan filter.

Uncertainty Source	**3.39 μm HeNe system**	**10.6 μm CO_2 system**
Type B		
0 offset	<0.001	<0.001
Inter-reflections	<0.001	<0.001
Detector/amplifier non-linearity	0.002	0.001
Non-source emission	<0.001	<0.001
Beam geometry effects	<0.001	<0.001
Polarization/angle of incidence	0.0003	0.0003
Vignetting	<0.0003	<0.001
Sample non-uniformity	0.005	0.005
Quadrature sum	0.006	0.006
Type A		
Standard uncertainty in mean	0.002	0.005
Expanded uncertainty	0.012	0.015

5. CONCLUSIONS

Optical density measurements have been made of ultra-thin metal film on Lexan or polyimide substrates with both FT-IR and HeNe and CO_2 laser based spectrometer systems. Results of the broadband and laser measurements have been compared up to OD 4. Estimates have been made of the uncertainties in the high-OD laser measurements, which can be performed up to OD 9.5 with both laser systems. Future plans include the addition of data from a tunable CO laser for 5 -7.5 μm measurements, which is now being incorporated into the heterodyne interferometer system, as well as use of the CO_2 laser from 9.2 μm to 11.2 μm

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