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REFLECTIVE INFRARED ELLIPSOMETRY OF PLASTIC FILMS

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Abstract

High resolution reflective ellipsometry is used to study freely suspended plastic films. We determine room temperature optical constants in the infrared for a variety of plastics using ellipsometry. The films are typically 6 to 100 μm thick and measurements are performed from near infrared to long wave-IR. The setup includes modeling software to fit the ellipsometric data to a generalized oscillator model. The films studied include acrylics, fluoropolymers, and variations of polyethylene, polystyrene, and polyvinyl chloride (PVC) among others. We are able to determine in-plane and out-of-plane optical constants. Transmission spectra from FTIR measurements are plotted and compared with ellipsometry results.

Keywords: Infrared ellipsometry, FTIR, optical constants, plastics, polymer, frequency selective surface

1. Introduction

a.) General

The selection of suitable low cost organic polymers along with identification of characterization methods that can be performed with a minimal amount of sample preparation is of importance in infrared technology for military and industrial applications. In this article, we study the optical properties of polymers for potential application to infrared Frequency Selective Surfaces (FSS).[1] In a FSS device, a dielectric standoff layer is situated between a ground plane and the periodic structure, and is designed with a specific thickness to provide phase matching. The advantage of a polymer over a more traditional oxide material like ZrO_2 or SiO_2 is that it can deposited in a low-cost process such as be spin-coating rather than by RF sputtering, electron beam, or thermal evaporation. In this article, we report on infrared spectroscopic ellipsometry (SE) applied to freely suspended single layer polymer films. This technique has the advantage that there is no substrate requiring preparation or sample deposition.

Films ranging in thickness from 6 to 100 μm are suspended onto a support fixture for ellipsometric measurement. Such films are thin enough that the backside reflection is still coherent with the front-side reflection, even for thermal source illumination. This gives rise to oscillations in the data mainly due to the sample thickness. As the sample thickness increases, the interference fringes are more closely spaced in wavelength, and hence exhibit sharper features. The finite bandwidth of the instrument as well the thickness non-uniformity of the sample introduces depolarization of the signal. Depolarization effects become more significant as oscillation features become sharper due to the increase in sample thickness. It is possible to model the depolarization; however, it desirable to minimize the non-idealities in the measured data to simplify modeling. If thickness of the sample is not of interest or can be measured independently, then the back surface reflection can be removed to suppress the high frequency oscillations (and depolarization).

Since these films were thin enough to exhibit coherence reflection between the top and bottom surfaces, and because we were interested in only probing the first few hundred nanometers below the top surface, we roughened the back surface homogeneously prior to measurement. Any light reaching the back surface is then scattered. We also tried applying adhesive tape with a matte finish to the back of the sample; this actually worked quite well to scatter the back surface reflection for some of the samples. By measuring only the front-side reflection, extraction of the optical constants is simplified as the sample may be modeled as a bulk material. If the sample is uniaxially anisotropic, suppressing the back surface reflections will also help suppress anisotropic effects on the data.

We studied the optical properties of a variety of plastic films in this manner using both *reflection* spectroscopic ellipsometry (*refl*-SE) and Infrared Fourier Transform Spectroscopy (FTIR).[2] The results of the two techniques compliment each other. Use of *refl*-SE provides values of n and κ, whereas FTIR provides percent transmission and hence absorption coefficient, α, and extinction coefficient, κ, through the relation, $\alpha = 4\pi\kappa/\lambda$ (where λ is wavelength and n and κ are the unitless components of the complex index of refraction, $\tilde{n} = n + i\kappa$). In theory one could obtain n as a function of λ from FTIR data through the familiar Kramers-Kronig relation,[3,4,5] but in practice, any such transformation assumes perfect specular reflectance. In transmission spectra one has both diffuse and specular components because of scattering due to surface roughness and other non-idealities of the sample, resulting in an offset of calculated values of n. Thus, commercial instruments marketed to determine n from a Kramers-Kronig transformation of data typically rely on data spectra, which are mostly specular (e.g. no diffuse component). Transforming FTIR absorption data yields a spectrum similar to that obtained from ellipsometry in correctly identifying peak positions in n, but the curve is often skewed.

The freely suspended film method is useful when deposition on a substrate is inconvenient and modeling becomes simplified because there is only a single layer to deal with. Ellipsometry (either *refl*-SE or *trans*-SE) provides high resolution, detailed spectra of n, and absolute values of κ. In contrast, FTIR is faster to perform and can provide better detail of relative values in absorbance spectra. Additional parameters which may be explored using SE with the suspended film technique are reflected phase shift, depolarization, stokes parameters, birefringence, and surface roughness.

b.) Spectroscopic Ellipsometry

In spectroscopic ellipsometry[6,7,8] one is interested in measuring the change in polarization state of light after being reflected from a sample surface. The two key measurement parameters Ψ and Δ are related to amplitude and phase shift upon reflection, and are wavelength dependent. Ellipsometry is usually performed near the Brewster angle to maximize phase shift upon reflection. The goal is then to fit the data to the general equation of ellipsometry:

$$\rho = \tan\Psi \boldsymbol{e}^{i\Delta} \tag{1}$$

$$\tan\Psi = \frac{|\boldsymbol{R}_P|}{|\boldsymbol{R}_S|} \tag{2}$$

$$\Delta = \delta_1 - \delta_2 \tag{3}$$

where Δ is the phase difference between S and P polarizations before and after reflection. We then fit Ψ and Δ to our model simultaneously.

For these plastics our model consists of a linear superposition of Gaussian and Lorentzian oscillators Σf_i; where each oscillator represents an absorption peak in the spectrum. Optical constants n and κ, or permittivity values ε' and ε'' may then be extracted.[3, 9, 10]

An advantage of reflective ellipsometry is that we can obtain optical constants for the material, even in regions where the sample is highly absorbing. The same information could be obtained with transmission ellipsometry (*trans*-SE), but for thick samples in absorbing spectral regions there would either be no data (transmission = 0), or such a low signal-to-noise ratio as to make the fit difficult. Ideally, one wishes to have as broad a spectrum as possible with no gaps and, in addition, the range of acceptable fit often depends on the model.

2. Materials

For this paper, we are interested in the optical constants of polymers in the spectral range from near-IR to long wave-IR.[11] Table 1 lists the samples studied in this work. Table 2 lists chemical structure and references. Below is a brief summary of material properties.

ACRYLICS - The commercially supplied proprietary "acrylics", are based on modification of polyacrylates. These are not the familiar *Plexiglas*, *Lucite*, or *Acrylite* used for casting solid, rigid materials, which would be PMMA poly(methylmethacrylate) which has a branched methyl group attached directly on the C-C chain, adding stiffness and higher glass-transition temperatures to the otherwise flexible, low T_g poly(acrylate) polymer. The commercial suppliers of our candidate polymers modify their acrylates with epoxy, urethane, siloxy or other substituents that improve the desired adhesive or other physical property. Their proprietary formulations also have cross-linking oligomers that bind the acrylate chains together, rendering them insoluble and offering more chemical permanence. Finally, these formulations contain radical initiators that are activated by UV, so that the final material is a three dimensional product yielding a durable coating, film or adhesive layer.

FLUOROPOLYMERS[15] – These materials have C-F replacing C-H bonds in polyethylene-like polymers. We initially felt that fewer C-H stretching vibrations in the 3-4 μm region would amount to fewer absorption lines, thus ‘cleaning up’ the region. However, the C-F stretching/bending modes tend to obscure the IR region above 7 μm, likely rendering them poor candidates for transparency in the LWIR 8-10 μm region, but suitable for the 3-5 μm MWIR range.

PFA – 'PFA' stands for Perfluoroalkoxy and is a fluorocarbon. These are polymers which have all of their carbon atoms bonded only to fluorine atoms, whereas fluoropolymers have a significant portion of their fluorine atoms replaced with another atom such as hydrogen or a halogen. These replacement bonds affect the molecule's modes of excitation and hence optical properties.

PEN – This is the acronym for polyethylene naphthalate analogous to PET – polyethylene terephthalate (Mylar). These are known as polyesters. PEN has a higher heat-distortion temperature than PET, so is valuable in applications where temperature resistance is important.

PE – Abbreviation for Polyethylene. This is a low molecular weight grade polyethylene, sometimes abbreviated in the literature as LDPE for "low density polyethylene". Sample "ZP" listed in table 1 is of this variety, sample "SWS" is a LDPE with ethylene vinyl acetate coating.

SAMPLE CODE	COMPOSITION	FILM THICKNESS (μm)
APHAN	Polyacrylate	101.6
CHCF04	$CHCFO_4$	101.6
CHCF05	$CHCFO_5$	101.6
DMOP54, H10443	Acrylic	76.2
M48525P, N75	Acrylic	76.2
P432A, WB1C	Acrylic	76.2
PC0G46GL	Fluoropolymer	76.2
PEN #Q71	Polyethylene Naphthalate	6.4
PEN M6	Polyethylene Naphthalate	6.4
PFA 200LP	Fluoropolymer	50.8
POLYSTYRENE	Polystyrene	76.2
PV3F	Fluorinated PVC	101.6
PVC #B0027	PVC	68.6
SPALTAN	Polyethylene Terephthalate	6.4
ZP, SWS	Polyethylene	76.2
TPEKS	Poly-4-Methyl-Pentene-1	76.2

Table 1: Sample codes, composition, and film thickness.

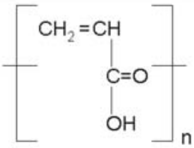
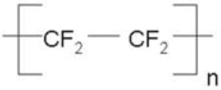
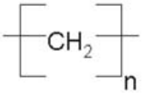
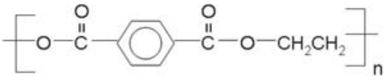
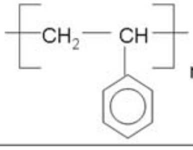
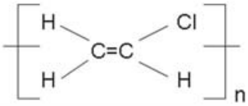
MATERIAL	FORMULA	CHEMICAL STRUCTURE	REF.
Acrylic/ Polyacrylate †	$(C_3H_4O_2)_n$		[15]
Fluoropolymer ††	$(C_2F_4)_n$		[11, 12]
Polyethylene	$(CH_2)_n$		[15]
Polyethylene Terepthalate	$(C_{10}H_8O_4)_n$		[15]
Polystyrene	$(C_8H_8)_n$		[15]
Polyvinyl-chloride	$(C_2H_3Cl)_n$		[12, 15]

Table 2: Structure for some of the polymer films and abbreviations used in the text.

- PEN = Polyethylene Naphthalate
- PET = Polyethylene Terephthalate
- PVC = Polyvinyl Chloride
- PE = Polyethylene
- Acrylic = UV-Cured Acrylic Polymer (exact formulations proprietary to vendor)

† The members of the acrylate family of polymers are made from acrylic acid and its derivatives.[15]

†† This is the base structure of fluoropolymers called PTFE (Poly-tetra-fluoroethylene). Sometimes the reader may see the base unit as (CF_2) rather than (C_2F_4) as in refs. [12, 15]. The discrepancy is simply a matter of what one calls a 'unit cell'. We choose this representation to emphasize the tetra (four) fluorine atoms in PTFE.

3. Suspended Film Ellipsometry (*refl*-SE)

In the following figures are shown optical constants obtained from ellipsometry for a variety of organic materials. Refer to table 1 for a list of samples. Measurements were done at high resolution in wave number of the spectral source, typically at 8 cm^{-1}. In most cases only three scans were performed at different detector angles about the Brewster angle. In a few we did as many as five scans. Multiple angles are not a requirement if the sample backside is roughened, but more model confidence is gained by fitting multiple angles simultaneously. FTIR results are plotted for comparison.

In figure 1 are shown n and κ values for a commercially available floropolymer in sheet form. PFA stands for perfluoroalkoxy (PFA), which is a fluorocarbon. In this model we used 14 Gaussian oscillators to fit the data to a wide range. We observed a large resonance near 8.5 μm and two smaller peaks beyond 15 μm. It is interesting to compare ellipsometric with FTIR data. The large central peak around 8 to 9 μm corresponds to the broad absorption band in the corresponding FTIR measurement. The double peak in κ is not resolved in the FTIR data, and is saturated due to high absorption of signal. Figure 1 shows no strong absorption features from 2-8 μm, but two large, broad peaks at longer wavelengths > 15 μm. Our measurements are able to pick out broad resonances in the spectrum, but do not capture the small absorption peaks in the FTIR data. At large values of absorption near 8 to 9 μm and at 19 to 20 μm κ values obtained by FTIR measurements appear to saturate. This is because the sample is absorbing too much of the signal and not enough energy is transmitted to the detector in those regions of the spectrum, i.e. the signal is buried in noise. To understand this, note that κ may be expressed in terms of α by the relation $\kappa = \alpha\lambda / 4\pi$, and α is related to percent transmission logarithmically as $\%T = 100 \cdot \exp[-\alpha x]$. A simple solution would be to use thinner samples to transmit more energy, but we did not have a variety of film thicknesses readily available from the manufacturer to choose from. In addition, because κ is an exponent the error between the two plots is actually smaller than appears. A plot in terms of percent transmission instead of κ would show the two curves almost on top of each other except for regions where the signal is comparable to the noise. The large spike in κ near 8 μm can however be resolved by reflective ellipsometry because we are probing only the first few hundred molecular layers. The two measured results follow one another quite well.

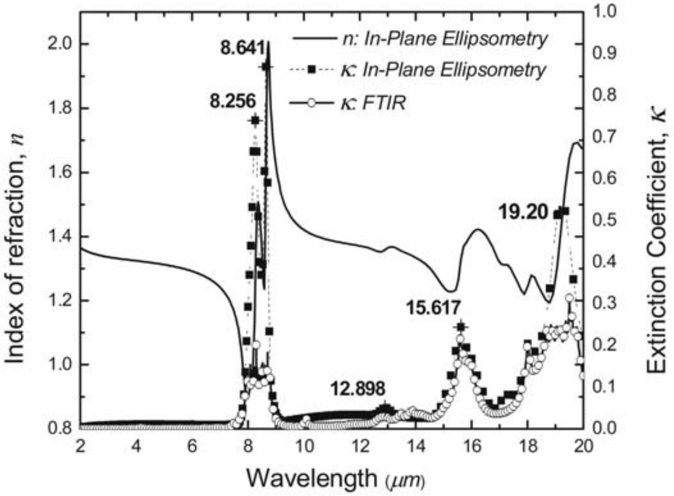


Fig. 1. In-plane values of n and κ from ellipsometry and FTIR for a fluoropolymer film, sample PFA200LP.

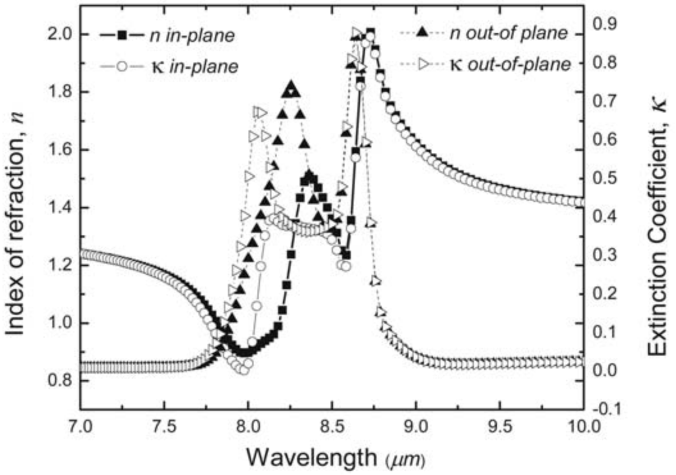


Fig. 2. Expanded view showing in-plane and out-of-plane anisotropy for the fluoropolymer sample PFA200LP.

We can observe a general trend of increasing κ from 10 to 22 μm. This film also exhibits anisotropy shown in figure 2 where the scale has been expanded around the peak near 8 μm. For a FSS stand-off layer, this material would be suitable for MWIR applications as predicted.

In figure 3 we examine a different grade of fluoropolymer based on polycarbonate. Since this material has more absorption peaks in the range of interest, we use 28 oscillators in the ellipsometric model to pick up more structure in the spectrum and focus our modeling on a shorter wavelength region, from 2-14 μm. This provides more detail in the κ spectrum and even resolves the triple splitting of the large central peak not observed in the FTIR data. This material has too many resonances and no significant region of constant κ to be of use as a stand-off layer for a MWIR or LWIR FSS. Nevertheless, the two results shown in figures 1-3 demonstrate that we are able to obtain information about features of an organic, free-standing material using ellipsometric techniques, and confirm our results with FTIR.

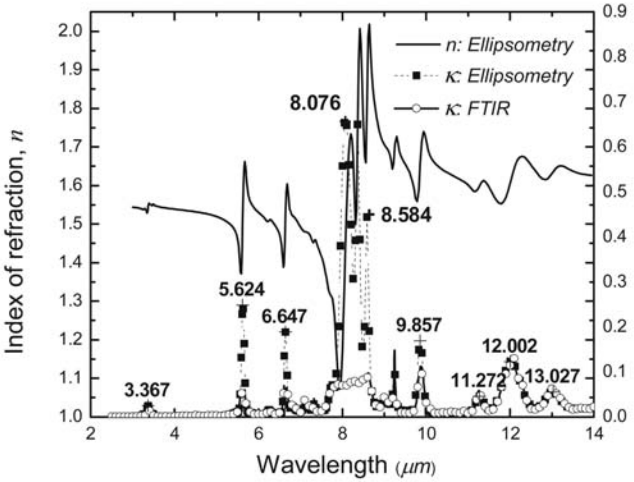


Fig. 3. Optical constants for a fluoropolymer based on a polycarbonate unit structure, sample code ‘PCOG46GL’.

Figure 4 shows n & κ values for Low Density Polyethylene (LDPE), ‘sample ZP’. An expanded view is shown in the figure as an inset to illustrate the double peak occurring near 3.5 μm. This sample is a good test case for our measurement because the structure of polyethylene, $[CH_2]_n$ is simple containing only methylene groups and the spectrum has a small number of absorption peaks.[12, 17] Figure 4 shows that the spectrum is dominated by a narrow double peak close to 3.5 μm and smaller features around 6 to 7.5 μm. The double peak at 3.425 μm (2920 cm^{-1}) and 3.505 μm (2853 cm^{-1}) are the C-H bond asymmetric and symmetric stretch respectively. Note the asymmetric peak is slightly larger the symmetric peak.[17] The small peak seen at 6.822 μm (1466 cm^{-1}) is the methylene scissoring vibration and the umbrella mode near 7.26 μm (1377 cm^{-1}) is visible just barely in the FTIR data. There is a very small peak in κ from ellipsometry at 6.04 μm not seen in FTIR or but is clearly seen in the n-spectrum. Values of κ are nearly flat from 7 to 12 μm and, as a simpler organic structure than the fluoropolynmers.

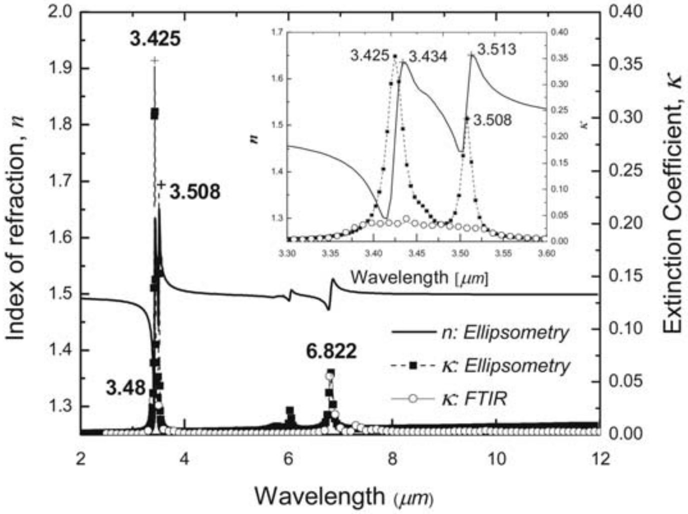


Fig. 4. Low Density Polyethylene (LDPE), sample 'ZP', a proprietary sample formulation: n and κ from ellipsometry and comparison of κ with FTIR.

Index n is almost constant from 4 to 6 μm and in the range beyond 7 μm the material provides good transmission and the index of refraction is almost constant starting at $n = 1.50$ at 7 μm and falling off to $n = 1.49$ at 12 μm. (Note the large y-axis range plotted in figure 4 from $n = 1.25$ to 2.0 and compare with the expanded scale in the inset). This is an excellent candidate material in the LWIR and portions of the mid-wave IR from ~ 4 to 6.5 μm. Low density polyethylene below in figure 4 and polystyrene shown in figure 5 are good calibration samples for our ellipsometry measurements with FTIR data since their properties are well known and gives us confidence in our (*ref*-SE) suspended film measurement technique when investigating more complex materials. Polystyrene[16] shown in figure 5 below has fairly good transmission in LWIR and absorption peaks in κ near 4, 6.5, 7.0, and 12.5 μm. Absorption peaks are relatively narrow. We see excellent agreement between FTIR and ellipsometry for principle peaks. Unresolved CH_2 symmetric and asymmetric stretch modes are observed near 3.420 μm (2924.0 cm^{-1}), aromatic ring modes at 6.25 and 6.70 μm (1600.8 and 1492.8 cm^{-1}), and an aromatic out-of-plane C-H bend observed at 13.23 μm (755.9 cm^{-1}).

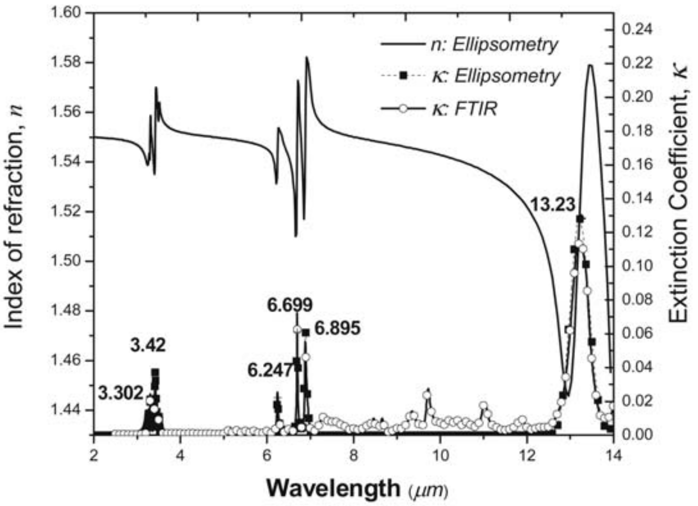


Fig. 5. Optical constants for polystyrene.[17]

Smaller features in κ between $\lambda = 8$ to 12 μm are neglected in our ellipsometry model because we are mainly interested in the broad question of a materials' suitability for FSS rather than spectroscopy. Index *n* varies significantly for Polystyrene over the spectrum but changes by less than 2% in LWIR. Small peaks in κ in LWIR are undesirable for a FSS application. For these four materials studied so far, *n* varies the most for the fluoropolymers, is almost constant for polyethylene, and a fair candidate for polystyrene, however the small peaks in κ mean we do not have constant transmission in LWIR.

In figures 6-7 are shown results for two different grades of acrylic. Their chemistry was described in section 2. These are UV-cured acrylic polymers. We see quite a bit of detail in the spectrum of both. Because relatively poor transmission and no flat regions of the spectrum it would not be useful of as a stand-off layer for a frequency selective surface in MWIR or LWIR. We have seen similar FTIR spectra for other acrylics summarized in plot form at the end of this article. But these two graphs show nicely how we can create a generalized oscillator model to fit ellipsometric data for a suspended thin film, and we see good agreement with FTIR.

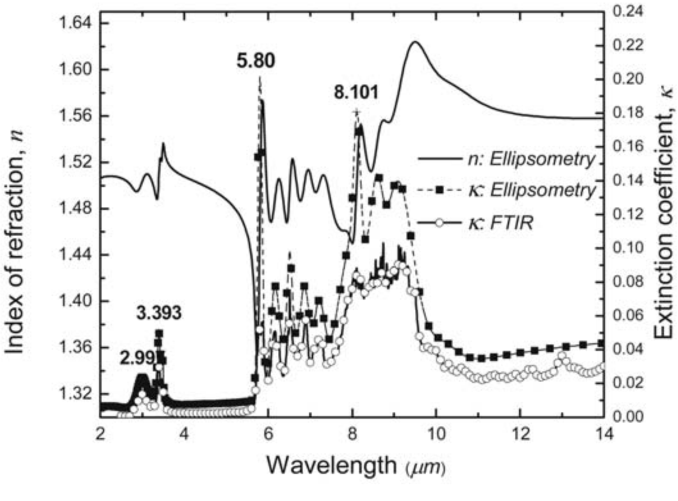


Fig. 6. A proprietary UV-cured acrylic polymer, sample code 'DMOP54'. As with most of the acrylic films, we see a lot of detail in its spectrum and no flat regions of low absorption in MWIR or LWIR.

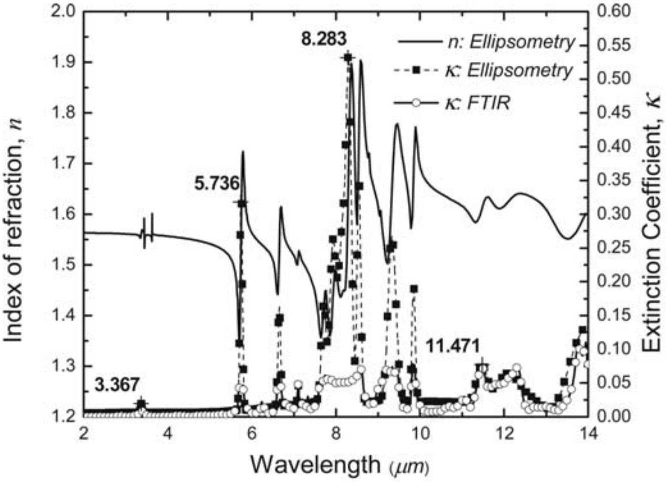


Fig. 7. Another proprietary UV-cured acrylic polymer sample code 'APHAN'. The broad absorption from λ = 6 to 10 μm has been somewhat suppressed, but many small peaks remain.

4. FTIR measurements

A few useful equations to convert between absorbance, n, α and κ, are summarized below.[17]

Absorbance (A) is defined as:

$$A \equiv \log\left(\frac{I_o}{I}\right) \equiv \log\left(\frac{1}{T}\right), \tag{4}$$

where I_o and I are the initial and final intensity of light impinging and exiting from an absorbing sample, and T is percent transmission divided by 100. This may also be written as:

$$\alpha = \frac{1}{t}\ln\left(\frac{I_o}{I}\right) = \frac{1}{t}\ln\left(\frac{1}{T}\right), \tag{5}$$

where α is the absorption coefficient and t, sample thickness. Absorbance is a unitless number, while absorption coefficient is usually expressed in units of [cm^{-1}]. Absorbance (A), absorption coefficient (α), and extinction coefficient (κ) may be related by the following expressions:

$$\alpha = \frac{\ln(10)}{t} A \tag{6}$$

$$\alpha = 4\pi \frac{\kappa}{\lambda} \tag{7}$$

where we recall that α is the exponent in Beer's law, $I = I_o e^{-\alpha t}$. The extinction coefficient (κ) is a unitless term in the imaginary part of the complex index of refraction notation: $\tilde{n} = n + i\kappa$.

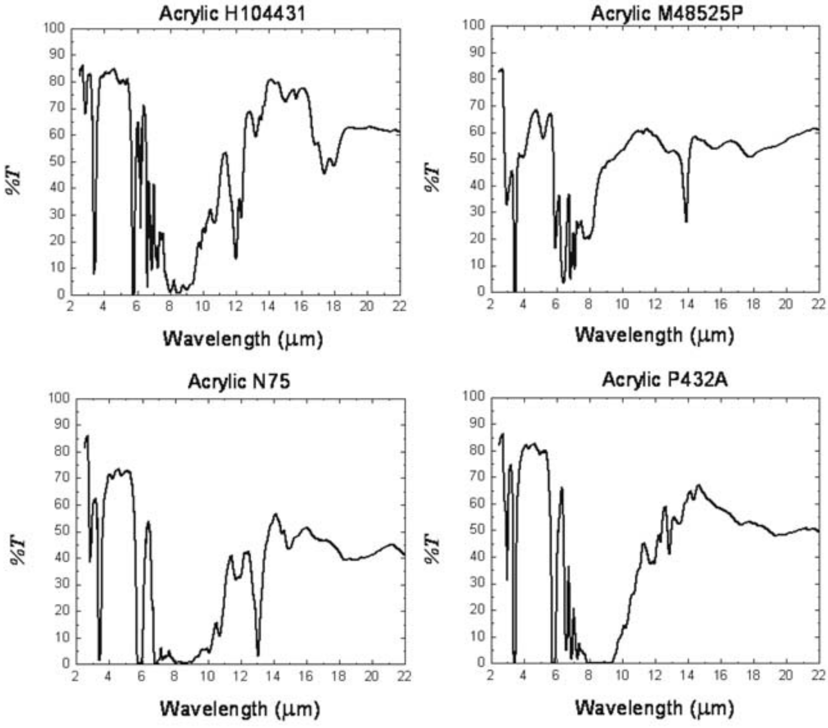


Fig. 8. FTIR scan for a set of proprietary acrylics. Scale is plotted in terms of percent transmission.

Remaining samples were characterized by FTIR and are presented in figures 8 through 10. We are looking for materials that transmit well and have relatively flat or at least monotonically increasing or decreasing spectrum, free from large spikes in the region of interest. The samples shown in figures 8 through 10 may be grouped in terms of chemical structure. In every sample we see the CH_2 symmetric and asymmetric stretch modes as would be expected in an organic. These peaks could be shifted by using deuterated compounds, replacing CH_2 by CD_2 bonds. The spectra of the acrylics are all similar, with a large trough of absorption near 8 microns. Sample M48525P could potentially be used in LWIR. Recall that these samples are quite thick, 76.2 μm, so a thin spin-coating a few hundred nanometers thick on a substrate would not absorb as much light. None of the acrylics we studied would be suitable for MWIR however since we would like to see no sharp spikes in the spectrum. Samples $CHCFO_4$ and $CHCFO_5$ are strongly absorbing over a large range from 7 to 20 μm and have similar spectra from 2 to 7 μm, note the large peak near 3.7 μm. The two spectra are almost identical except additional small features from 10 to 14 μm for $CHCFO_5$.

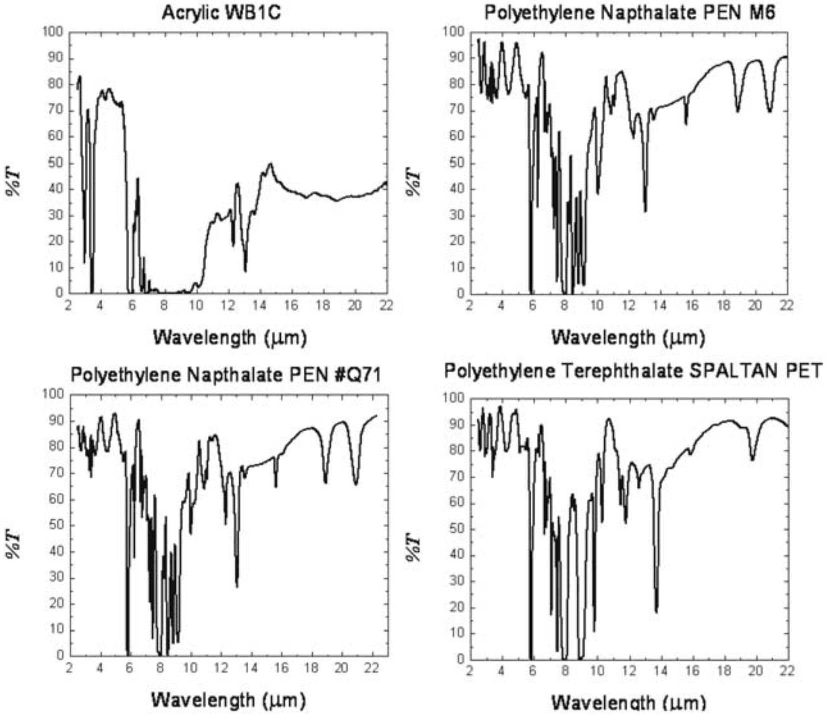
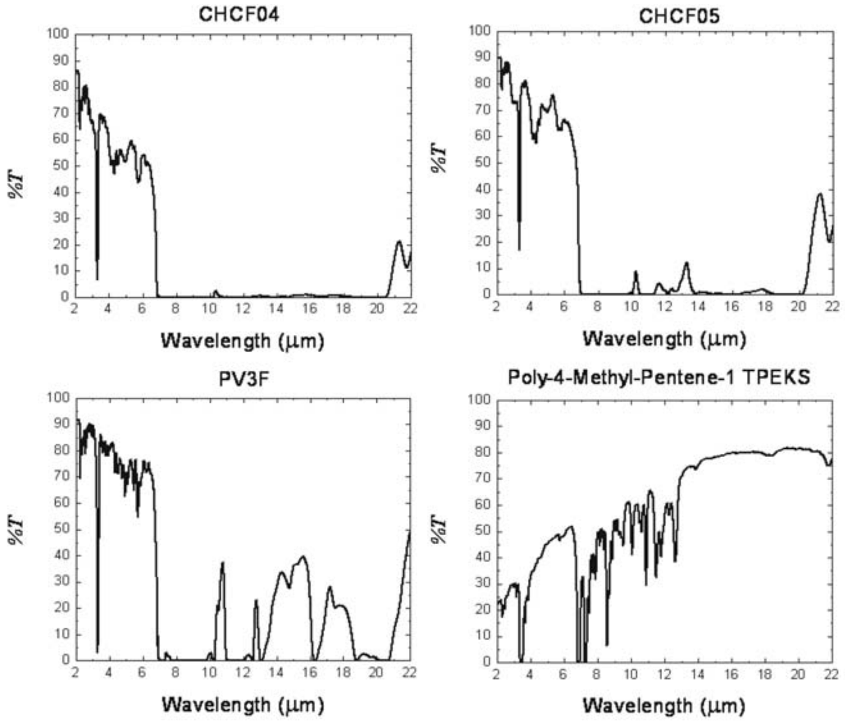


Fig. 9. FTIR scans comparing a typical acrylic blend with a set of three variations of polyethylene.


CHCF04

CHCF05

PV3F

Poly-4-Methyl-Pentene-1 TPEKS

Fig. 10. FTIR scans comparing a typical acrylic blend with a set of three variations of polyethylene.

Materials like $CHCFO_4$ and $CHCFO_5$ would not be suitable in either LWIR or MWIR for a FSS standoff layer, but could be very useful for low-pass filter. Note the shoulder of the curve near λ = 7 μm, sharpness of the cutoff, and broad trough of absorption, especially for $CHCFO_4$ which extends all the way to λ = 20 μm. A thin film of this material could make an excellent filter from λ = 3.5 to 7 μm. We also see a similar behavior for sample PV3F.

Samples PEN-Q71 and PEN-M6 are different grades of polyethylene napthalate, and have the same chemistry. Again, we see too much structure and small spikes in the spectrum to be used for our purpose. Sample SPLATAN PET is similar except for the substitution of the terapthalate group. Its spectrum is likewise similar except for small changes in features of the LWIR and beyond 16 μm. An acrylic like sample WB1C could have good transmission in the LWIR if band from

~ 8 to 10 μm could be broadened by substitution of the molecule with an appropriate chemical group.

5. Conclusion

In this work we presented optical constants and transmission spectra for a series of organic materials. We introduced the technique of ellipsometry on freely suspended organic films. We were able to detect significant features in the spectrum for these samples using this method of performing reflective ellipsometry measurement on a free-standing film. Reflective ellipsometry has the advantage over transmissive ellipsometry in that we are probing only the first few hundred molecular layers of the film, and therefore, we can measure strongly absorbing peaks without loss of signal, as would be the case transmission ellipsometry or FTIR. This technique could be extended to study other features such as temperature dependence of optical constants, anisotropy, surface quality, depolarization, and Stokes parameters.

6. Acknowledgements

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