ATR Spectroscopy of Carbon Black Filled Materials

Figure 1. The DiaMaxATR™ ATR accessory

INTRODUCTION

Carbon black is used as filler in many materials to add strength and wear resistance. It also finds uses as a thermal conductor, an electrical conductor, an anti-static agent and a colorant. The addition of carbon black also helps protect resins and plastics from UV damage.

However carbon black has a high refractive index, n, and n changes in the mid-IR more than many materials. This makes studying carbon-filled materials challenging by ATR.

In this note, we investigate carbon-black filled materials using a 45° single-reflection Ge ATR.

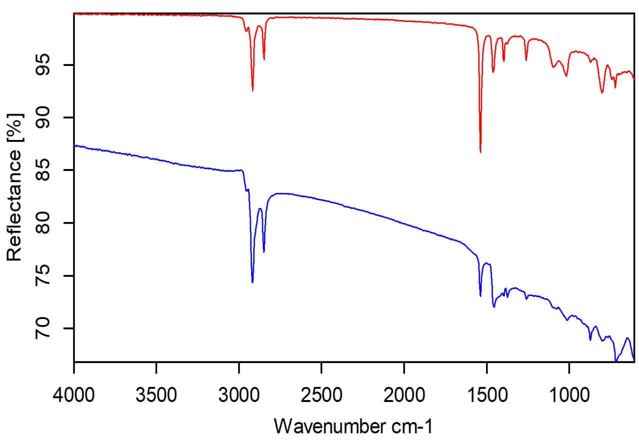

Figure 2. Spectra of an EDPM o-ring. The exterior is shown in red and the interior in blue.

EXPERIMENTAL

Harrick's DiaMaxATR™ ATR accessory (Figure 1) was equipped with its Ge ATR crystal and 24 in-oz slip-clutch. This accessory was used in an FTIR spectrometer under purge, to minimize water vapor and carbon dioxide spectral interference. All spectra were measured at an 8 cm^{-1} resolution and signal averaged over 32 scans. The background was collected from the clean Ge ATR crystal and the sample was collected by pressing the carbon-filled sample against the ATR crystal with sufficient force to observe the spectrum.

Several carbon-black filled materials were analyzed including EDPM o-rings, FKM o-rings, adhesive-backed black rubber and rubber trekking pole tips.

RESULTS AND DISCUSSION

The resulting spectra from the measurements are shown in Figures 2 through 5.

Figure 2 shows the difference between the exterior and the interior of a black EDPM o-ring. It also points to some of the problems with analyzing high refractive index materials. The most striking difference between the two spectra is the shape of the baseline. The spectrum of the interior of the o-ring has a lower overall reflectance which slopes at lower wavenumbers and has a minor inversion at 3000 cm^{-1}, indicating that the critical angle criteria was not locally met. The fact that the inversion is not seen on the exterior surface indicates that the o-ring must have a layer with a lower refractive index (n). But even the ATR spectrum of the surface shows some impact from the carbon-filled material, as the baseline slopes slightly at lower wavenumbers. It is also clear from the spectrum that the composition of the surface differs from that of the bulk.

Figure 3 compares two different o-ring bulk materials. The FKM o-ring has distinct peaks which are not present in the EPDM material. Both show C-H stretches in the 3000-2900 cm^{-1} region, C-H bends at 1460 and possibly a N-O stretch at 1540 cm^{-1}. However the FKM also has bands at 1736 cm^{-1} likely due to C=O stretch, 1190 cm^{-1} from a C-O stretch and 970 cm^{-1} (C=C bend) which are not present in the EPDM.

Figure 4 shows the ATR spectrum from the upper portion of the rubber tip used on a trekking pole. The spectrometer spectral search clearly identifies the primary infrared-active component as some form of polypropylene. Again, the baseline slopes slightly due to the high carbon content, but not enough to prevent identification.

Figure 5 shows another carbon-black filled material with a coating. In this case, the sample is an adhesive-backed rubber. There is clearly an organic component in the bulk material but the adhesive-coated side differs spectroscopically. A spectral search reveals that the adhesive is likely to be a triacontane or polyethylene based material.

SUMMARY

Carbon-black filled materials and coatings thereon can be a challenge to study by diamond or ZnSe ATR because of their high refractive index.

As shown here, single reflection Ge ATR spectroscopy at a 45° incident angle can effectively be used to examine many of these carbon-black filled materials.

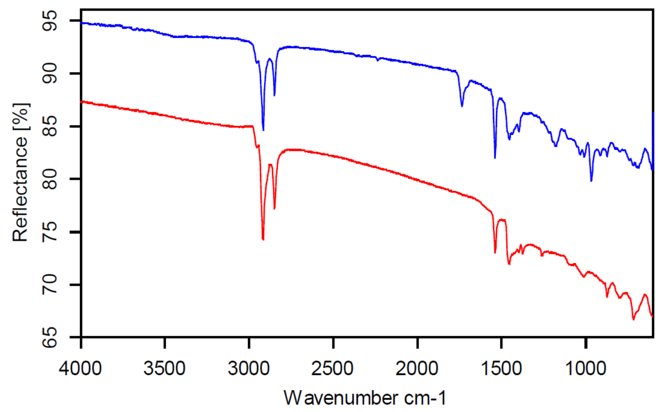

Figure 3. Spectral comparison between the insides of an EPDM o-ring (red) and an FKM o-ring (blue).

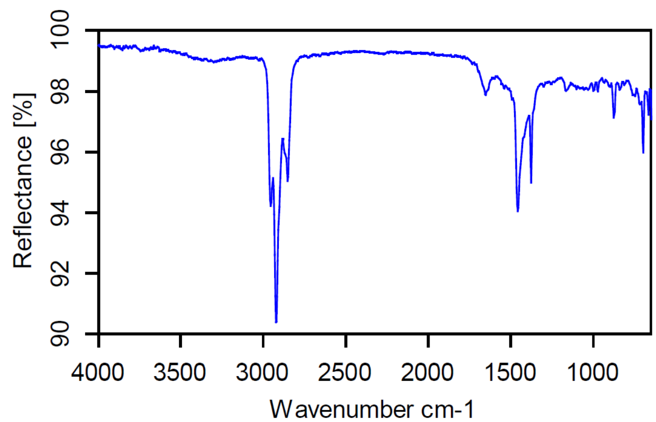

Figure 4. The ATR spectrum of a rubber tip from a trekking pole.

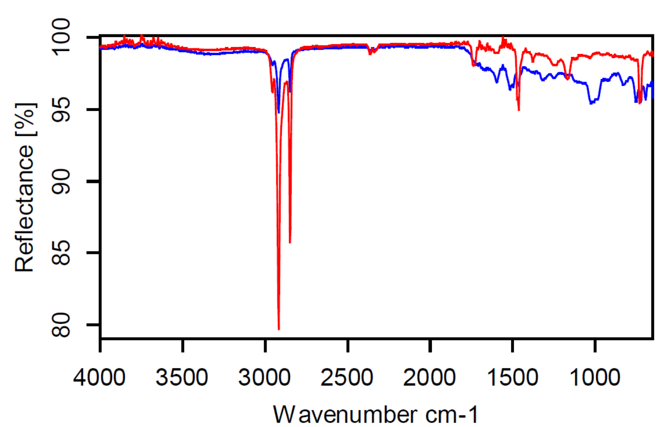

Figure 5. The ATR spectra of an adhesive backed rubber, showing the adhesive side (red) and the non-adhesive side (blue).

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