HARRICK SCIENTIFIC PRODUCTS

No. 21162

IMPROVING S:N FOR IN-SITU INFRARED DIFFUSE REFLECTION STUDIES

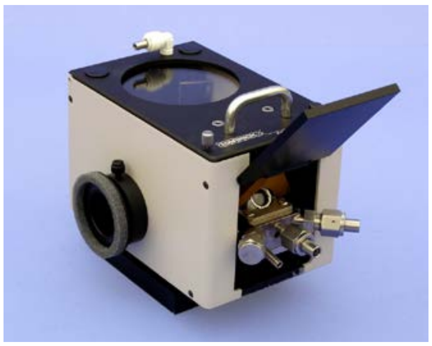

Figure 1. The Praying Mantis with its High Temperature Chamber.

INTRODUCTION

Diffuse reflection is the radiation scattered off a sample in all directions except the specular angle. Diffuse reflectance equipment that accommodate environmental chambers generally collect some fraction of the total diffuse reflection and then the reaction chambers further reduce that fraction. This is particularly problematic in the infrared where the sources and detectors are weak, and chemically inert, optically transparent, low refractive index window materials are not available. Higher sensitivity MCT detectors are sometimes used, but their utility is limited for reactions studied at elevated temperatures due to detector saturation from thermal emission.

Here we explore an approach to improving the S:N for this type of measurement using BBAR coated ZnSe windows. ZnSe windows are frequently chosen for this type of study due to their chemical inertness but their high refractive index reduces the transmission through the windows. The BBAR coatings are typically comparable to ZnSe in terms of chemical reactivity but increase the transmission through the window.

EXPERIMENTAL

Infrared measurements were acquired using the Praying Mantis diffuse reflectance accessory with its HVC high temperature reaction chamber installed in a commercial FTIR. All spectra were recorded at 4 cm^{-1} resolution, 32 scans with a gain of 8 using a DTGS detector.

Two standard domes (window assemblies) were used, one with two ZnSe infrared windows installed and the other with two BBAR-coated ZnSe infrared windows. Two samples were used: KBr powder and PTFE diluted in KBr.

The HVC sample cup was filled with KBr, packed and the top surface leveled. Then three single beam spectra were collected: one with no dome installed and one with each of the two domes installed. The single beam spectra were saved and then used to calculate the throughput of the two domes relative to KBr in the cup with no dome installed.

Next, a dilute mixture was made of PTFE powder and KBr. The ratio was selected to give weak PTFE bands. The HVC sample cup was filled with this mixture, packed and the top surface leveled. Then two single beam spectra were collected, one with each dome installed. The spectra were then calculated using the corresponding KBr single beam background. The

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noise was then calculated using the spectrometer software for comparison to the expected theoretical prediction and the difference in throughput.

RESULTS AND DISCUSSION

Assuming the BBAR coating improves the transmittance of the windows from 70% to 90%, the throughput for two windows would be 81% to 49%. In principal, this would give roughly a 60% improvement using the BBAR coated ZnSe windows.

From the throughput spectra shown in Figure 2, the throughput of the ZnSe dome is roughly 9%, whereas that of the BBAR coated configuration is roughly 17%. This gives a 53% improvement, fairly close to the theoretical approximation.

The question now becomes one of whether or not that improvement in throughput is reflected in performance. If so, it should reduce the noise in the spectra and ultimately make it easier to see weak bands. The reduction in noise should be directly proportional to the improvement in throughput.

To check the noise level, two spectra were collected of diulte PTFE in KBr with the two types of windows. The resutilng spectra are shown in Figure 3. The noise level was then measured in a non-absorbing region using the noise function of the spectrometer, as shown in Table 1. Note that the software uses transmission data in percentages rather than the more commonly used fraction. With the use of the BBAR windows, there is roughly a 54% improvement in the peak-to-peak noise and a roughly 66% improvement in the RMS noise, consistent with the improvement in throughput.

CONCLUSION

There is undeniably a reduction in noise with the use of BBAR coated ZnSe windows instead of standard ZnSe window. Thus using the BBAR coated windows in place of uncoated ZnSe windows will increase sensitivity of the in-situ measurements made with the Praying Mantis and its reaction chambers.

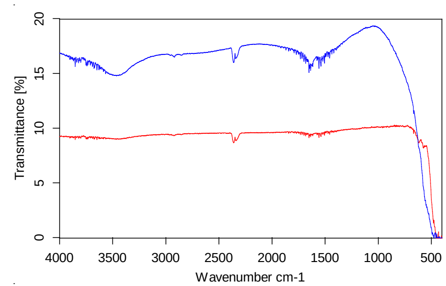

Figure 2. The throughput of ZnSe windows (red) and BBAR coated ZnSe windows (blue) installed measured with KBr in the HVC relative to KBr in the sample cup.

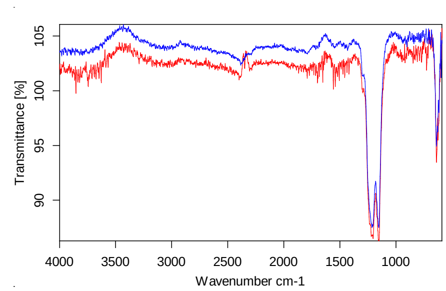

Figure 3. The spectra of PTFE in KBr powder measured with ZnSe windows (red) and BBAR coated ZnSe windows (blue) installed in the HVC.

Window	Noise at 2830-2390 cm^{-1}	
	Peak to Peak	RMS
ZnSe	1.26530	0.19544
BBAR coated ZnSe	0.68992	0.13089

Table 1: Noise comparison with and without the BBAR coating.

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