FOCUS GRAZING ANGLE ACCESSORY **NO. 62804**

Grazing Angle Reflectance of Small, Coated Metallic Substrates

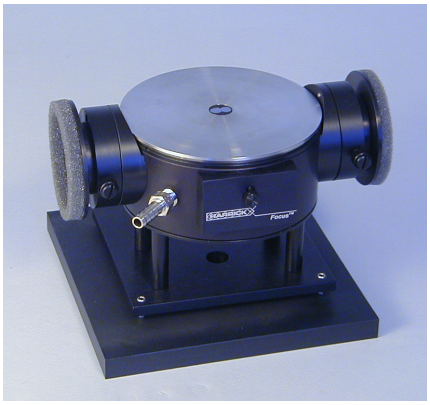

Figure 1. Focus Grazing Angle Accessory.

INTRODUCTION

Thin films on metal substrates are routinely analyzed by grazing angle specular reflection spectroscopy using accessories such as the Refractor™. The incident beam is polarized to provide radiation polarized in the plane of incidence since only p-polarized radiation exhibits sensitivity to thin films on metallic surfaces. However, in grazing angle reflectance, the incident beam has a long footprint on the sample and thus, the sample needs to be at least 32 x 15 mm in size. If the sample is smaller, the incoming beam overfills the sample and a portion of the beam is simply not reflected. Analyzing a spot on the sample that is 3 mm in diameter spot is almost impossible using the conventional grazing angle reflectance accessories.

This applications note uses the grazing angle technique to investigate small spots on coated metallic substrates with a specially configured FT-IR accessory.

EXPERIMENTAL

The samples were analyzed using the Focus™ grazing angle reflectance accessory (see Figure 1). This accessory was designed specifically to efficiently analyze small reflective samples and is equipped with a built-in polarizer plate that provides the required p-polarized light. The Focus™ also features PermaPurge™, integrating the accessory into the purge of

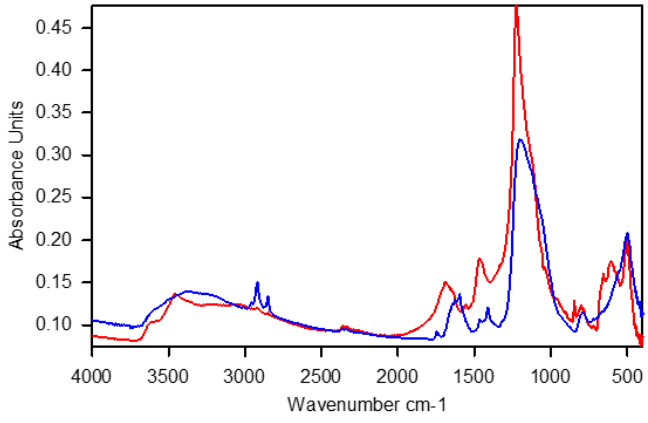

Figure 2. Grazing angle reflectance spectra of two coated steel rivets.

the FTIR spectrometer. Thus, once the purge of the system is established, the samples can be analyzed rapidly one after the other.

Two types of samples were investigated: small steel rivets and copper chips. The steel rivets were 10 mm in diameter and approximately 3 mm thick. The copper chips were roughly 10 x 10 x 1 mm. Both samples were smooth but not optically polished and were coated with thin films of silicates deposited under different processing conditions.

To examine the samples, a background was collected using a front-surface aluminum mirror placed face-down on the sample stage. Then the mirror was replaced by the sample and the spectrum was collected. All spectra were collected on a commercial FT-IR spectrometer at 4 cm^{-1} resolution with 64 scans co-added. These conditions resulted in a data collection time of just over 1 minute per sample.

RESULTS AND DISCUSSION

The spectra recorded from two of the steel rivets are shown in Figure 2. Both spectra show a band at approximate 1200 cm^{-1}. This is the Si-O band from the depositied silicates. The spectrum shown in blue also has stronger C-H bands, around 2900 cm^{-1}, indicating a higher concentration of hydrocarbons in the coating.

The spectra measured from the copper chips are presented in Figure 3. The spectra of the copper chips show a strong Si-O band around 1200 cm^{-1}, as expected. The three spectra shown indicate differing compositions, particularly with respect to the hydrocarbon and hydrocarbon and hydroxide bands.

Note that some of the copper samples were visibly twisted, as seen in Figure 4. The curvature of the sample defocuses the beam of the spectrometer on the detector. Thus, generally a baseline offset or a curved baseline results from the curvature of the sample.

CONCLUSION

Grazing angle reflectance spectroscopy is the technique to analyze thin coatings on smooth but unpolished metal substrates. Normally, the technique requires relatively large, uniform samples. However, small samples are often all that is available. For these small samples, as well as for analysis of small spots on large samples, the Focus™ represents a unique and important extension to the standard grazing angle reflectance equipment.

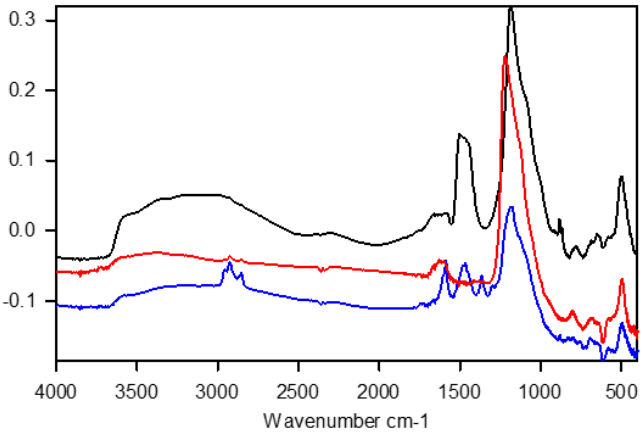

Figure 3. Grazing angle reflectance spectra of three coated copper chips.

Figure 4. The three coated copper chips.

[1] N.J. Harrick and M. Milosevic, *Appl. Spectros.*, 44 (3), 519 (1990).

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