APPLICATION NOTE

Characterization of low-emissivity glass coatings using XPS

The Thermo Scientific K-Alpha X-ray Photoelectron Spectrometer (XPS) System was used to investigate the overall structure of a multilayered low-emissivity (low-E) coating by high-quality sputter depth profiling.

Low-E coatings are thin metal or metal oxide layers deposited onto a glass substrate in order to control radiative heat flow. These coatings are typically applied to glazing units installed in buildings to assist in controlling room temperatures, reducing the heat loss from the interior in winter months or controlling heat entering through the glass in the summer. The principle of operation is the same in both cases: the coating reflects longer wavelength infrared radiation while allowing shorter wavelength light through (Figure 1).

Generally, the coatings are 100–200 nm thick. The composition and integrity of the coating are crucial to both the efficiency of the glass and its visual appearance. Therefore, the use of a technique to perform both routine analyses and failure investigation is vital.

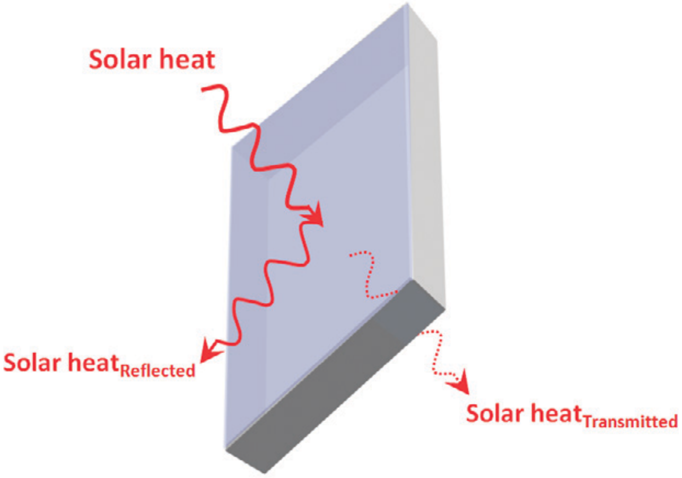

Figure 1: An example of a use of low-E glass to control room temperature by controlling transmission of infrared radiation from the sun.

By using the Thermo Scientific™ K-Alpha™ X-ray Photoelectron Spectrometer (XPS) System, the real layer structure of a coated sample can be checked against the expected layer structure. The surface sensitivity of the technique enables profiles with excellent depth resolution to be obtained. The chemical state of the individual elements present can also be detected, allowing you to identify, for example, if unwanted oxidation of one of the metal components is occurring. In the case of failure analysis, the layer where delamination has occurred can be identified by using the same depth profiling process.

Experiment

A sample of low-E glass was depth profiled using the K-Alpha XPS System. 500 eV argon ions with >1 μA beam current were used for profile. At each level in the depth profile, rapidly acquired snapshot spectra were collected, minimizing spectral acquisition time between ion etches. The integrated argon ion source is fully computer aligned and controlled and offers excellent ion flux even at low energies. The K-Alpha XPS System is also equipped with a simple turn-key charge compensation system, which makes the analysis of insulating samples simple and maintains stable analysis conditions throughout the profile.

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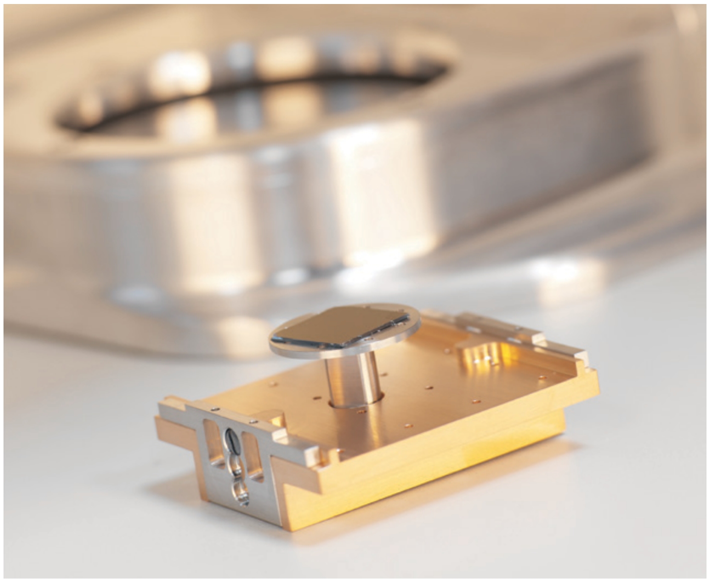

Figure 2: The K-Alpha XPS System sample holder fitted with the 3 cm diameter rotating stage for depth profiling.

The sample was mounted on a rotating stage as shown in Figure 2. Rotating the sample during the Ar$^+$ etch cycles helps ensure the best possible depth resolution. The Thermo Scientific Avantage Data System can perform both azimuthal and off-axis (compucentric) rotation, enabling several profiles to be carried out on the same sample without the need to remove it from the instrument. The experiment was configured to use "multi-phase" etching, whereby the etch time and individual region acquisition times can be set per level or group of levels to minimize the time required. These parameters can also be edited as the experiment is running.

Results

The results of the depth profiling of a low-E glass coating can be seen in Figure 3. The image of the etch crater was taken with the K-Alpha XPS System's live reflex optics system after the depth profile. It shows the high quality of the etch crater.

The critical components of the low-E glass are the two silver layers indicated in dark blue in the profile. The depth resolution on the second, deeper layer is not visibly degraded compared to the first silver layer, demonstrating the K-Alpha XPS System's ability to sputter complex multi-component films while maintaining excellent depth resolution throughout the profile.

Other than the silver layers, the majority of the coating is comprised of silicon nitride, tin oxide and zinc oxide. Nickel layers are deposited over the silver layers to act as oxidation protection. Low concentrations of aluminum and chromium are also found in the silicon nitride and zinc oxide layers.

One of the main features of an XPS depth profile is the ability to measure chemical state information as a function of depth. It can be seen that there are at least two chemical states of nickel in the multilayer coating. These states have been assigned as nickel oxide and nickel metal. The nickel oxide appears at the interface with the tin oxide layers.

Summary

A low-E glass coating was depth profiled using the K-Alpha XPS System. The overall structure of the coating can be easily determined using this technique, identifying both elemental components and their chemical environment as a function of depth. The excellent depth resolution of the data enables accurate interfacial characterization.

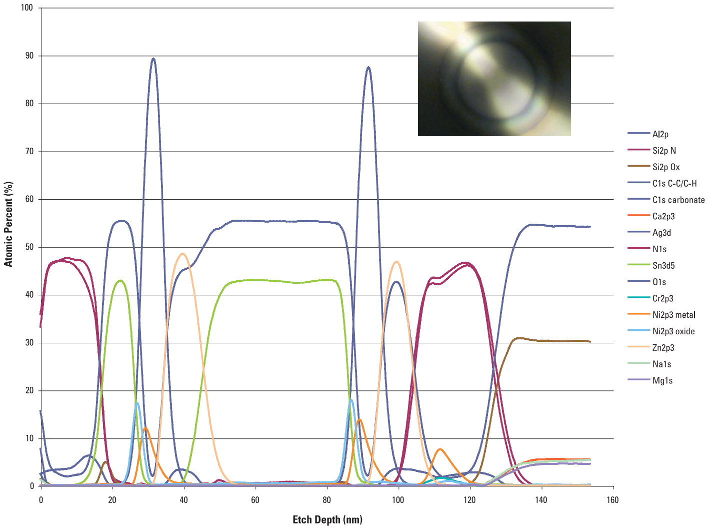

Figure 3: Depth profile of low-emissivity glass. The image of the etch crater was taken with the sample viewing system of K-Alpha XPS System. The depth scale was calibrated to a known standard.

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