Small Angle X-ray Scattering (SAXS) and Near Edge X-ray Absorption Fine Structure (NEXAFS) Spectroscopy Testing for Glass, Antireflective Coatings, and PV Module Materials

Purpose: Document repeatable testing procedures for SAXS and NEXAFS characterization on industrially-relevant PV materials

SAXS Testing at SSRL:
SAXS is a technique for characterizing the elastic scattering behavior of X-rays as they interact with a material, with structural dimensions in the range of ~1-150 nm. Standard transmission-mode SAXS requires thin sample thicknesses (typically less than 100 microns) to allow sufficient scattered X-ray intensity to penetrate the sample during a measurement. Since the goal is to apply this technique to industrially-relevant PV materials (e.g., an antireflective coating on glass), the thickness limitation can be resolved by performing experiments in grazing-incidence (GI) mode at the expense of slightly more complicated scattering analysis (Fig. 1).

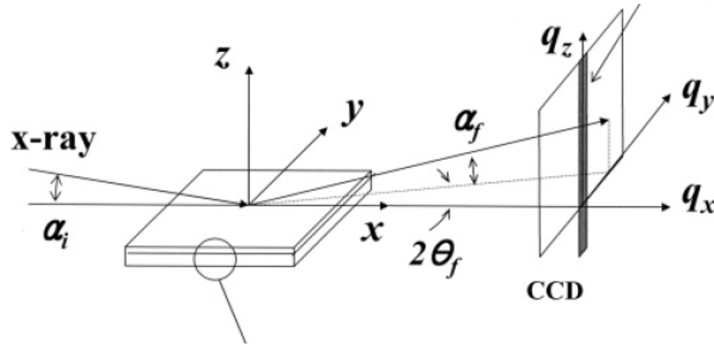

Fig. 1: Schematic of a GI-SAXS experiment geometry. The tilt angle is α_I, and q_z and q_y represent the out-of-plane and in-plane scattering vectors, respectively.

A typical experiment using beam line 1-5 at SSRL would use a half-beam alignment with a 0.2° tilt angle. A measurement on Ag behenate should be performed in order to calibrate the sample-to-detector distance for data analysis purposes. Note that for coatings on a substrate, smooth substrates are preferred. Textured substrates (e.g., solar glass) can negatively influence the scattering signal from the coating

An example plot of scattering intensity vs. in-plane scattering vector is shown in Fig. 2 for an open-pore antireflective coating on flat glass. There is an inverse relationship between the magnitude of the scattering vector and the real-space size of the scattering feature, with a larger scattering vector corresponding to a smaller feature size. Also shown graphically is the approximate location of where the scattering peak of a 25-nm sphere would be in q-space. In this plot, the main scattering peak at 0.025 $Å^{-1}$ corresponds to the scattering feature size of $\sim 2\pi/q_y = 25.1$ nm.

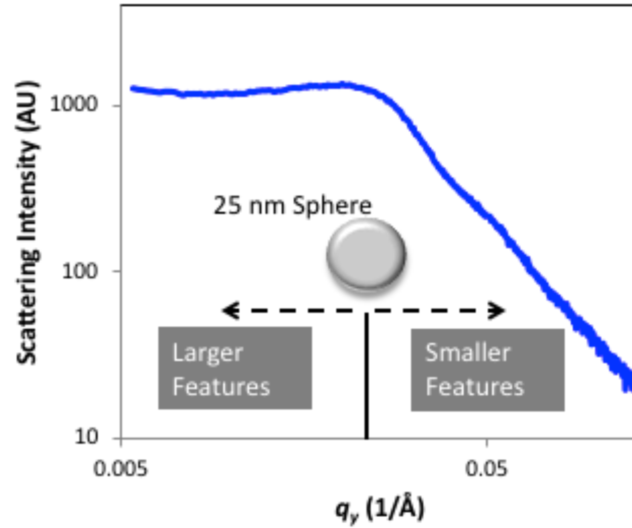

Fig. 2: Scattering intensity vs. in-plane scattering vector for an antireflective coating on glass.

NEXAFS Testing at SSRL:

NEXAFS is a powerful X-ray absorption spectroscopy technique for probing surface chemistry and chemical bonding of surface species. The utility of the technique is its ability to resolve absorption edge splitting, in which the main peak at the absorption edge (the "white line" region) is split into several overlapping energy signatures corresponding to different elemental bonding environments. Changes in these bonding environments after an external stimulus are characterized by energy shifts and reductions in peak intensity. An oxygen *K*-edge spectrum is shown in Fig. 3, clearly demonstrating the fine structure of the white line region due to peak splitting.

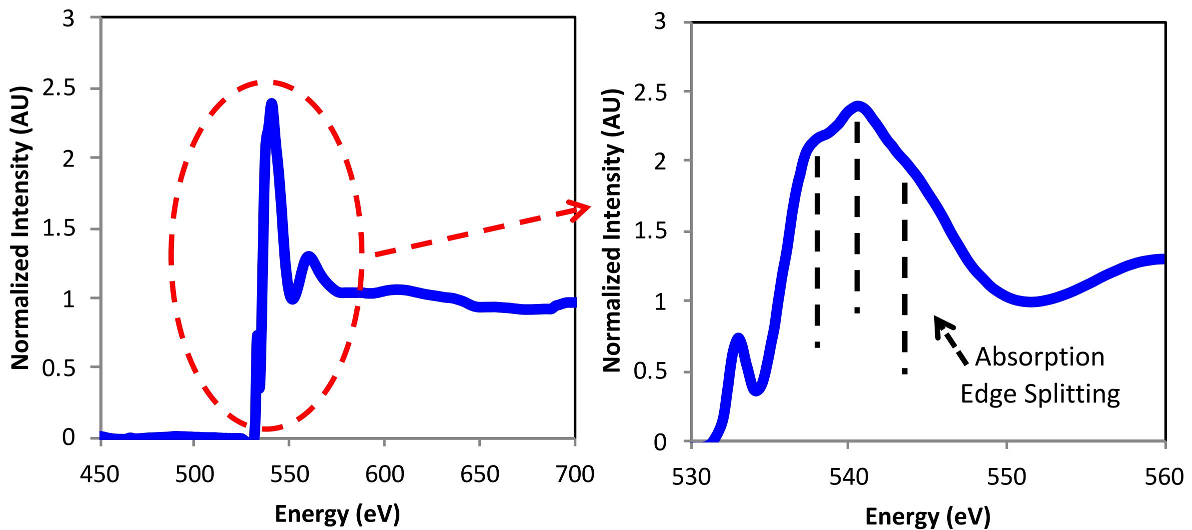

Fig. 3: Oxygen *K*-edge spectrum of an antireflective coating surface.

In a NEXAFS experiment, X-ray absorbance is quantified by one of two ancillary signals: total fluorescence from X-rays emitted from excited core electrons as they relax or total electron yield from ejected electrons. Electron yield has a smaller penetration depth compared to fluorescence, so it is more surface sensitive and a more effective technique for probing changes in surface chemistry. A substantial amount of data normalization and reduction has be applied to raw NEXAFS data prior to analysis. A helpful tutorial can be found here (hosted by xafs.org).

Since NEXAFS characterizes the elemental bonding environments, selection of the absorption edge to measure depends on the chemistry of the material in question. If the material is silica, for example, the oxygen *K*-edge may be of interest due to the different types of oxygen bonds exhibited in silica, such as SiO_2 tetrahedra, siloxane bonds, silanol surface terminations, etc. Beam line 10-1 at SSRL is capable of X-ray energies in the range of 250-1200 eV. Relevant *K*-edges that lie within in this energy range include:
C – 284.2 eV
N – 409.9 eV
O – 543.1 eV
F – 696.7 eV
Na – 1070.8 eV

Several elements also have *L*-edge transitions in this energy range that may be of interest:
Ca – 438.4, 349.7, 346.2 eV
Ti – 560, 460, 453.8 eV
Mn – 769.1, 649.9, 638.7 eV
Fe – 844.6, 719.9, 706.8 eV
Cu – 1196.2, 1044.9, 1021.8 eV
Ga – 1143.2, 1116.4 eV