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Radiation Damage Studies Using The Stony Brook Cryo-STXM

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Beamline(s): X1A

Introduction: Radiation damage is one limiting factor in imaging at high resolution. Cryogenic sample preparation has been shown to effectively halt radiation-induced mass loss, thus making high resolution imaging applications such as tomography possible. Our goal in this study has been to understand the protection that cryo methods provide for x-ray absorption spectroscopy of dry organic materials.

Methods and Materials: We are using the imaging mode of the Stony Brook cryo-STXM to deliver a known dose to the specimen. This is done by scanning the specimen through the focal spot provided by a Fresnel zone plate. The spectroscopy mode of the cryo-STXM is used to record a XANES spectrum across the oxygen K edge. During the spectrum scan, the specimen is slightly defocused to minimize the dose. This procedure is repeated many times to get XANES spectra at different radiation doses. We chose a poly-methyl-methacrylate (PMMA) film as our sample. The PMMA film was cooled down to liquid nitrogen temperature before the exposure. A control experiment was conducted with the PMMA film at room temperature.

Results: Figure 1a) shows an image taken after the PMMA film was irradiated at liquid nitrogen temperature. The second image shows the same area after warm-up to room temperature; mass loss within the square-shaped high-dose area is clearly visible. The third image shows the control experiment at room temperature, again showing mass loss. Figure 2) shows the recorded XANES spectra taken at different doses at room temperature and liquid nitrogen temperature. The peak height of the C=O bond is used to quantify one type of bond loss under irradiation (see Figure 3b). The loss rate of the C=O bond is approximately the same at both temperatures. To determine the mass loss, each spectrum was fitted with an average value at the beginning (528eV-529.5eV) and at the end (538.5eV-540.0eV) of the spectrum. The ratio of the two values was used to determine that the mass loss independently from the dose (see Figure 3a).

Conclusions: While cryogenic sample preparation significantly reduces the mass loss under radiation, the C=O bonds are broken at a similar rate at both temperatures. The g-factor is of importance when studying different materials under irradiation. Therefore, a study of an hydrated organic material is in preparation.

Acknowledgments: J. Mannik - PMMA sample, M. Feser, B. Winn, S. Wirick.

References: X. Zhang, et al., *Journal of Vacuum Science and Technology*, vol. B 13, no.4, 1995.

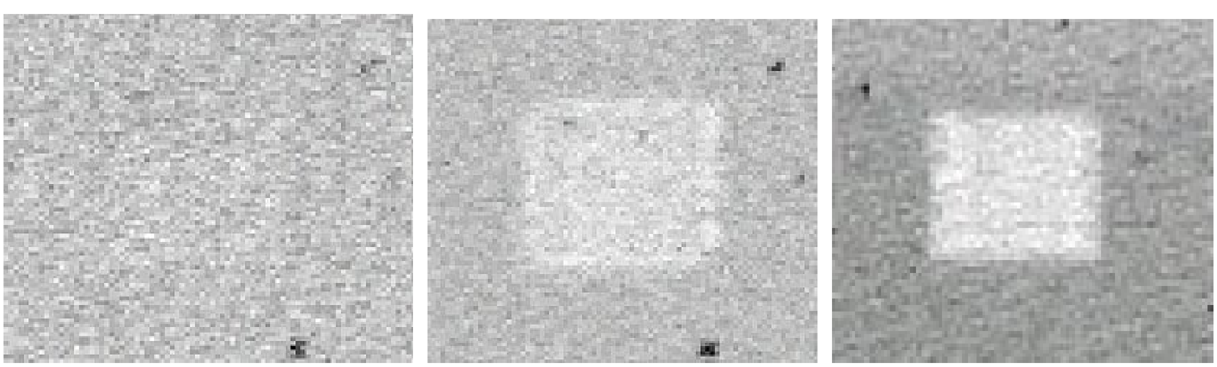

Figure 1) Area scan of the irradiated region. a) liquid nitrogen temperature, b) after warm-up, c) control experiment at room temperature

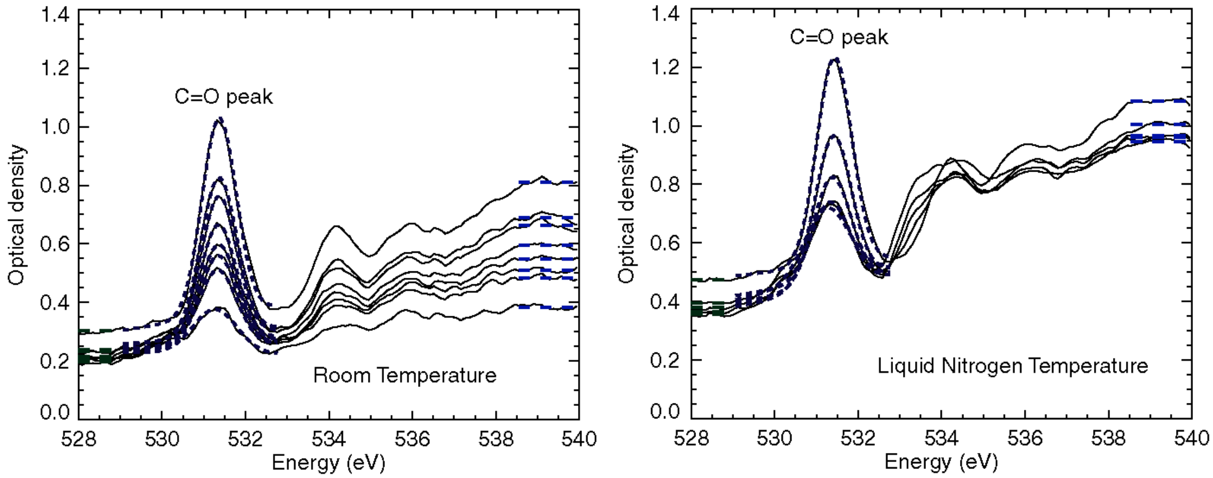

Figure 2) Oxygen XANES of PMMA at room and liquid nitrogen temperature in dependence of the radiation dose

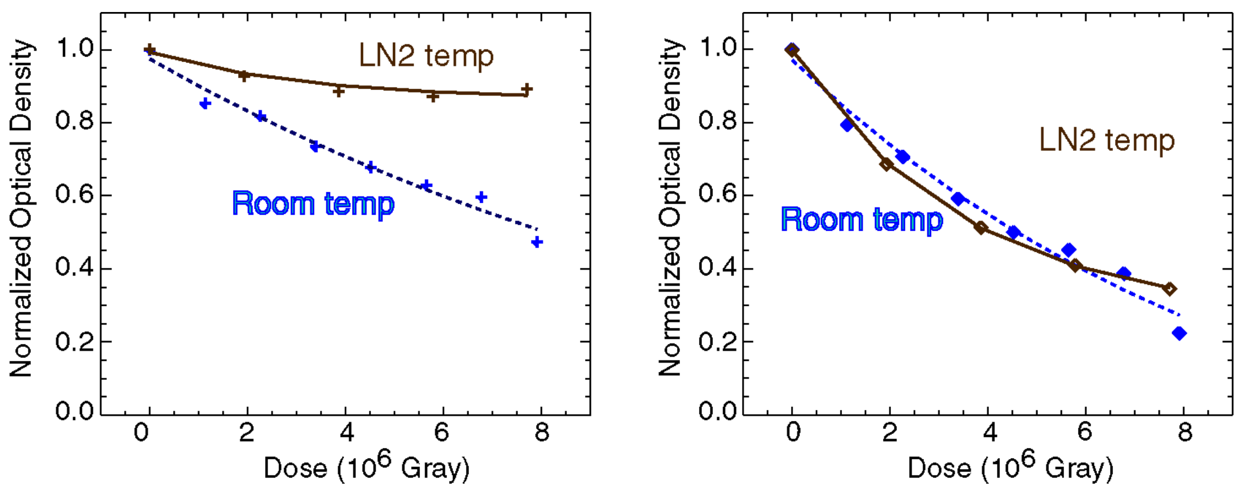

Figure 3) a) mass loss for PMMA at room and liquid nitrogen temperature in dependence of the radiation dose b) C=O bond loss for PMMA at room and liquid nitrogen temperature in dependence of the radiation dose