

XPS and XAS Studies Up to High Temperatures (150-1300K) Using a Cylindrical Mirror Analyzer Chamber

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We describe the construction, and successful operation, of an insert to perform high temperature measurements (300 K – 1400 K) of core-level photoemission and x-ray absorption spectroscopy inside a Cylindrical Mirror Analyzer (CMA) chamber at the Synchrotron Radiation Center (SRC). Recent interest in spintronic materials with ordering temperatures above 300K, as well as other high-temperature electronic and crystal structure phase transitions in unconventional magnetic and magnetoelectric materials, call for measurement of electronic properties at higher temperatures.

The Cylindrical Mirror Analyzer (CMA) chamber at the SRC lacks reliable means to heat such samples. An e-beam heated sample stage had previously been in use on the CMA for the purpose of annealing samples positioned at the measurement site; this could not be used for measurements while open to the beamline due to outgassing during operation, and also flooded total electron yield measurements. In the new heater insert (schematic shown below), we use a small cartridge heater encased in a molybdenum shell. This heater is then mounted on insulating standoffs within a cylindrical heat shield at the end of the manipulator arm. With proper conditioning, the new heated sample stage reached temperatures in excess of 1000°C while holding a vacuum of better than 10^{-10} Torr inside the CMA. There were no issues with either outgassing or excess heating of the sample manipulator arm. To enable measurement of total electron yield in XAS, we electrically isolated the sample and heater casing using appropriately positioned ceramic spacers.

We collected high temperature data (500-1000K) across phase transitions in magnetoelectric nanoparticles of Pb-Ti-Fe-O (20-80nm) and a large single crystal of magnetoelectric TbMnO₃ (0.5x0.5x1cm) using this heater stage. Nanoparticles in powder form were mounted directly on the end of the cartridge heater with silver paste and annealed. The single crystal of TbMnO₃, grown by us from a floating zone, was attached using silver paste and suspended against the cartridge heater using a basket of tantalum wire. Closed loop control of temperature was not found necessary, as temperature quickly reached an asymptotic value, perhaps due to resistance increasing along with temperature. In this configuration, it is also possible to perform measurements down to lower temperatures without breaking vacuum.

