Microsymposium

MCX@Elettra: Powder diffraction in ambient and non ambient conditions

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The beamline Material Characterization by X-ray diffraction (MCX), is the general purpose powder diffraction beamline at the Elettra synchrotron in Trieste, one of currently four diffraction beamlines at Elettra . The beamline is designed to host a wide range of experiments, that cover many scientific fields with standard applications such as phase identification, structure determination, residual stress measurements and line profile analysis.

The experimental station houses a four circle Huber diffractometer. The two-theta arm can be equipped with a scintillator detector either with slits or with a Si 111 analyzer crystal. A multi- channel analyzer is also available, which can be used to eliminate the background signal resulting from the fluorescence of the sample in a diffraction experiment, or can be used to perform chemical analysis on samples. Alternatively, a ccd detector can be installed on the arm, which is especially useful in the case of in operando studies on for example batteries, where changes in the diffraction pattern in a range of 2-theta can be followed instantaneously while the battery is operating. A hot air blower and a cryo stream are available to measure powder samples in capillaries from 100 to 1273 K.

As a second experimental setup a furnace has been installed, which has been designed for powder diffraction measurements at high temperature (up to 1373 K at the present state). Around the measurement region the geometry of the radiative heating element assures a negligible temperature gradient along the capillary and can accommodate either powder samples in capillary or small flat samples. A double capillary holder allows a flow of gas in the inner sample capillary while the outer one serves as the reaction chamber. The furnace is coupled to a translating curved imaging-plate detector, allowing the collection of diffraction patterns up to 130 degrees two-theta in order to follow chemical reactions and phase transformations.

Here the current status of the beamline will be presented illustrated by recent experiments using the different setups available at MCX. A room temperature and high temperature structural studies have been performed through the whole compositional range of the (Ce1-xMx)O2-x/2 for M = Sm, Gd and Lu revealing a complicated hybrid structural behavior. The relation between microstructure and thermoluminiscence of LiF2 has been investigated using both the funcace and the hot air blower. Finally, the phase diagram at high temperature Cu-TiS2 has been studied using the furnace in an inert atmosphere.

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