CURVED IMAGE PLATE (CIP) DETECTOR FOR RAPID HIGH RESOLUTION POWDER X-RAY DIFFRACTION USING SYNCHROTRON RADIATION

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Introduction

A new Curved Image Plate (CIP) detector has been designed for x-ray diffraction (XRD) investigations of materials using synchrotron radiation. This detector has been developed in collaboration with scientists from the Hamburger Synchrotronstrahlungslabor (HASYLAB) at the Deutsche Elektronen-Synchrotron (DESY), Hamburg, Germany [1]. The primary objective of this study was to validate the capabilities of the CIP detector using standard materials. High temperature XRD (HTXRD) experiments were also performed to evaluate the potential of the CIP detector for kinetic investigations.

Methods and Materials

CIP detector: The CIP detector is a curved, one-dimensional detector (see Fig. 1.) which simultaneously records the entire diffraction pattern in the 2θ range from 0° to 37°. The detector is constructed as a section of a circle with a polycrystalline sample in its center. The detector is positioned at a distance of 1045 mm from the sample. The diffracted x-rays create color-centers in a photostimulable phosphor (Fujifilm BAS-IP SR 2040) glued on a cylindrically curved aluminum plate. The stored latent image is read by an onsite scanner consisting of a red laser diode and a photomultiplier tube (Hamamatsu H5784), both shielded inside a scanner-head. The scanner-head is fixed on a mobile carriage guided by a curved rail and can be positioned with an accuracy of approximately 10 μm to read the exposed image plate.

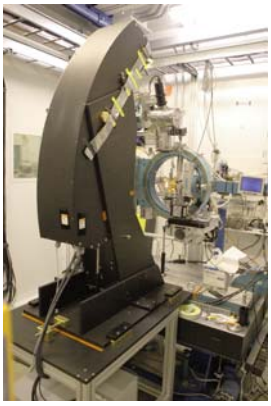

Fig.1. The CIP detector at the XOR/UNI 33BM-C beamline at APS.

Synchrotron XRD: All experiments were preformed at the bending magnet beamline at XOR/UNI 33BM-C, at the Advanced Photon Source, Argonne National Laboratory, Argonne, IL. Standard reference materials 660a (LaB_6) and 674a (TiO_2 and Cr_2O_3) were used for evaluating the detector performance. Powder samples of these materials were mounted in glass capillaries, rotated at 60 rpm, and XRD patterns were acquired in transmission geometry.

HTXRD: Sintered plate specimens of TiB_2 were prepared from commercial TiB_2 powders (Aldrich, Milwaukee, WI) by heating pressed plate samples in inert atmospheres (Ar) at 1600°C for 2 hours. In the HTXRD experiment, a lamp furnace was used to heat the TiB_2 sintered plate sample in air at 1200°C and XRD patterns were acquired *in-situ* [2].

Results

Detector Resolution: The intrinsic resolution of the CIP detector was measured as 0.007º for a wavelength of 0.700643 Å (≈ 17.7 KeV) with a 0.1 mm capillary sample of SRM 660a, LaB_6 powder. This translates into accuracy of at least 0.001° 2θ in peak position or approximately 0.0001 Å in lattice spacing.

Detector Sensitivity: A Cr_2O_3-1wt%TiO_2 mixture was used to evaluate the ability to detect minority compounds. The CIP detector provides the flexibility to grow the minor phase peaks in intensity, while saturating the higher intensity peaks from the major phase.

Kinetic Studies: Successive scans were recorded with 30 seconds of exposure, every 110 seconds to capture the oxidation of TiB_2 and formation of TiO_2.

Discussion

The design of the CIP detector has enabled rapid acquisition of high resolution x-ray diffraction patterns suitable for structural refinement. An onboard reader enables data collection, extraction, transfer, and storage of X-ray intensity information in ≤ 30 seconds. This detector overcomes the usual limitations encountered with the image plate (IP) detectors such as the requirement for an external IP scanner, or a small accessible angular range (in flat plate geometry) and low spatial resolution due to short sample-to-plate distances. The CIP detector is capable of detecting and storing X-ray intensity information proportionally over a dynamical range of about 4.5 orders of magnitude, for each exposure.

Acknowledgments

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