

Powder diffraction imaging by a projection-type X-ray microscope

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Introduction

The powder diffraction imaging of a specimen in the order of $\text{mm}^2 \sim \text{cm}^2$ is extremely attractive in the area of materials characterization [1]. When combined with an energy-scan of the incident X-rays, a projection-type X-ray microscope can work well even if all parts, including the specimen, are completely fixed [2]. This report describes the feasibility of such a combination in distinguishing materials prepared in the same substrate.

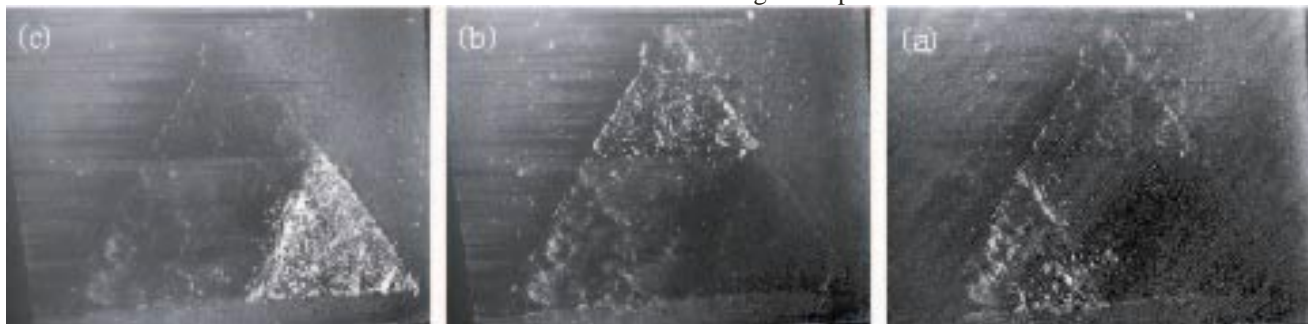
Experimental

The instrument used is essentially the same as a projection-type X-ray fluorescence microscope, the details of which are described elsewhere [2]. The experiment is simply a repetition of the exposure of a CCD camera as a function of the energy of the incident monochromatic X-rays, which satisfies the Bragg condition for a fixed scattering angle of ca. 90 deg. The beam size is 13mm(H) \times 0.2mm(V). Typical exposure time for one image was 3 sec. The sample measured consisted of three kinds of oxide powders, $\alpha\text{-Al}_2\text{O}_3$ (rhombohedral), Y_2O_3 (cubic), and HfO_2 (monoclinic), which were put at specific positions on the same glass substrate. In the present study, Y_2O_3 (311) ($2d=3.174\text{\AA}$), HfO_2 (113) ($2d=2.937\text{\AA}$), and $\alpha\text{-Al}_2\text{O}_3$ (300) ($2d=2.749\text{\AA}$) reflections were chosen in order to distinguish the three powders from each other.

Results and Discussion

From the Bragg's equation, one knows that Y_2O_3 (311), HfO_2 (113) and $\alpha\text{-Al}_2\text{O}_3$ (300) peak at 5647 eV, 6012 eV and 6427 eV, respectively, when energy-dispersive measurements are carried out at a fixed scattering angle of 89 deg. Figure 1 shows the X-ray images obtained at those three X-ray energies. It was found that the X-ray images agree well with the real positions of each oxide powder. One concern is the interference of the $\alpha\text{-Al}_2\text{O}_3$ (116) peak ($2d=3.174\text{\AA}$), because it is very close to that of Y_2O_3 (311). Actually, they overlap in an ordinary $\theta/2\theta$ scan with Cu $K\alpha$ radiation (8004 eV). Since this is an energy-dispersive measurement, the resolution is around 1~3 eV, and is mainly dependent on the monochromator. It was possible to separate them from each other in this experiment.

The selected diffraction lines are not major lines, and therefore their intensity is rather low. This is a compromise considering the available X-ray energy at the beamline. With a normal X-ray beamline, 5~8 keV X-ray photons are stronger and easier to use, compared with 2~5 keV. The present method would be feasible for identifying materials at a specific position in an inhomogeneous system. Its application in the area of combinatorial screening is one promising direction. The authors would like to thank Professors H. Sawa, Y. Wakabayashi, and Y. Uchida, for their kind cooperation during the experiment.



References

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Figure 1
XRD images obtained at 5647eV (a), 6012eV (b), and 6427eV (c), corresponding to Y_2O_3 (311), HfO_2 (113) and $\alpha\text{-Al}_2\text{O}_3$ (300), respectively. The graph shows powder diffraction patterns obtained by normal $\theta/2\theta$ scan. Diffraction lines used for imaging are shown as dotted lines. Exposure time and repetition were 3 sec and 120 times, respectively.