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X-ray diffraction 2D-imaging for polycrystalline specimens

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Introduction

So far, X-ray diffraction (XRD) imaging for polycrystalline materials has been performed by both a 2D scan of the irradiated area of the specimen and an angular scan of the goniometer for finding diffraction peaks [1]. Developing methods, which do not require scans, is significant to make such experiments much more feasible [2]. This report describes an extremely efficient way of obtaining XRD images by simply repeating digital shots by an X-ray camera that has a completely fixed geometry [3].

Experimental

The instrument used is essentially the same as a projection-type X-ray fluorescence (XRF) microscope, the details of which are described elsewhere [4]. A wide beam is used for illuminating the whole area of the specimen, and X-ray images are recorded by a CCD camera equipped with a collimator inside. The system can be applied to X-ray absorption fine structure (XAFS) imaging by combining it with an incident X-ray energy scan around the absorption edge [4]. The procedure for XRD imaging also uses a combination of exposure and incident X-ray energy scan, and the difference here is simply the scan range of the X-ray energy.

Results and Discussion

As shown in Fig.1, the present experiment employs a fixed small-angle incidence of around 1 deg and also a fixed diffraction angle of around 90 deg. The diffraction plane here is inclined at about 45 deg from the surface of the specimen. By scanning the energy of the incident X-rays, one obtains a diffraction peak at E', which corresponds to the lattice spacing d' as the following simple relation;

$$E' = \frac{12.398}{\sin(\sim 45\,\mathrm{deg})} \cdot \frac{1}{2d'}$$

Note that XRD imaging is still possible even when neither the 2D scan of the sample nor the angular scan of the goniometer are employed. In the present case, the XRD images are of quite a wide area of the specimen (8mm × 8mm) and were obtained both simultaneously and rapidly. Typical exposure time for one image is 1 sec.

Figure 2 shows an XRD image of zinc die-casting (hcp, a=2.665Å, c=4.947Å), obtained at 7480eV. The image represents the inhomogeneous intensity distribution of the Zn(11-22) reflection. As can be clearly seen, many big bright spots are found in the upper half, suggesting that the seam-line represents the border of the difference in temperature during cooling. That said, the spots are found in almost the whole area. This indicates that the orientation of the crystal grains is almost random. Similar spotty XRD images are obtained for all the reflection peaks during energy scans, and changes in spot size at the seam-line were reproduced for each image.

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References

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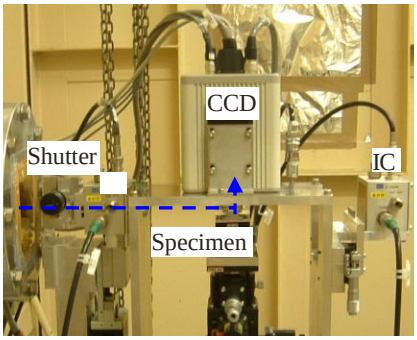

Figure 1 (top) Experimental arrangement for XRD imaging. An arrow shows incident and diffracted X-rays, with a Bragg angle of ~90 deg. The specimen is placed almost horizontally, but the surface is inclined at ca. 1 deg to the incident beam.

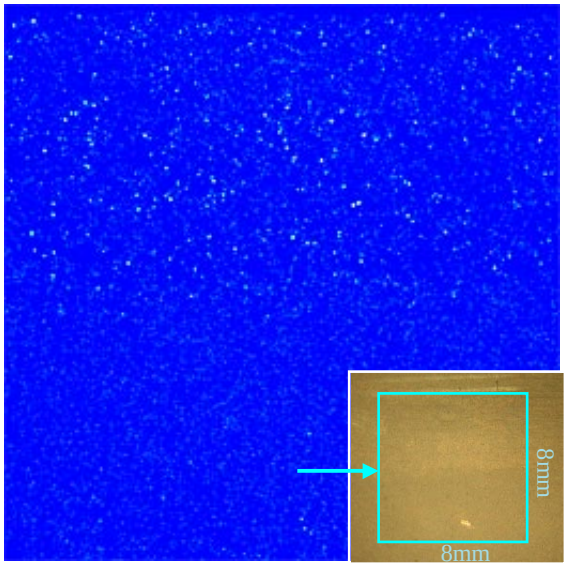

Figure 2 (right) XRD image of the cross section of zinc die-casting, used for the furniture hardware. The image corresponds to the hcp-Zn(11-22) reflection, of which the 2d value is 2.346Å. The inset shows the observed area as an optical microscope image. An arrow indicates the seam line. The change in grain size is fairly distinct at this line.