SYSTEMATIC ERRORS IN LINEAR PSD BASED HTXRD SYSTEMS

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ABSTRACT

We present an experimental determination of the effects of systematic errors inherent in an mBraun linear PSD-based HTXRD system, and strategies for minimizing these errors. It is noted that dead time counting losses are extremely important in this system, and these can be reduced through a combination of hardware and software solutions. There is also a significant loss in diffraction peak Cu K$_{\alpha_1}$:K$_{\alpha_2}$ resolution at higher counting rates. It is also essential for non-ambient temperature diffractometer systems that the field of view of the detector be restricted to no more than the irradiated sample area to reduce the background and avoid collection of unwanted reflections from beryllium windows and/or other furnace or cryostat hardware.

INTRODUCTION

The hundred-fold faster rate of data collection possible with a linear position sensitive detector (LPSD) compared to a conventional scanning detector in laboratory XRD systems permits in-situ time-resolved studies of phase transformations, order-disorder transformations, and crystallization and grain growth. Crystal structure changes can often be tracked in-situ with a time resolution on the order of seconds, making LPSD systems a valuable tool for understanding these phenomena. LPSD systems are also used for room temperature diffraction studies because high quality patterns can be obtained relatively quickly for phase identification, determination of texture, or lattice parameters. A detailed analysis of systematic errors, such as axial divergence and defocusing, which are inherent in LPSD-based systems has been reported by Cheary and Coelho [1,2]. In this paper, we present an experimental analysis of additional sources of error and pitfalls inherent in using a linear PSD-based system, and discuss strategies for minimizing these errors.

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PROCEDURE

Experiments were conducted using an mBraun linear position sensitive detector (LPSD) on a Scintag PAD X theta-theta vertical goniometer. The tube to sample and sample to detector distances were set at 220mm. A 2 kW Cu X-ray tube was used as the source, with a diffracted-beam nickel foil for a beta radiation filter. Tests were done both with a standard ambient sample mount, and with a Buehler HDK2.3 high temperature furnace attachment. The output of the PSD was processed using an mBraun system with pulse height analysis set to eliminate unwanted signal as per the manufacturer’s manual. The data were collected in a multichannel analyser (MCA) with the active area of the detector defined by about 500 channels at a resolution of 0.02°/channel, corresponding to an angular range of 10 °2θ. The time to download the MCA data to the controlling PC computer was just under 2 seconds, which permits rapid data acquisition with this system. Software was written using Borland C++ Application Builder to run the experiments and record the data, which were imported as ASCII files into Jade 5.0 [Materials Data, Inc.] for further analysis.

RESULTS AND DISCUSSION

PSD scans were taken at a fixed angle using Ni filtered Cu radiation at an excitation voltage of 45kV at currents from 2mA to 40mA. It is expected that the counts reaching the detector should vary linearly with the tube current, and this relationship was first confirmed using a point detector [Scintag Peltier-cooled Si(Li) solid state detector]. Using the PSD at a fixed data acquisition time the total counts received at the multichannel analyser were summed and plotted versus tube current in Fig.1. The non-linearity is characteristic of detector dead time losses, but it was surprising that these losses were observed even at very low count rates. Based on this result, it is clearly useful to avoid strong beam intensities, and to apply a separate dead-time correction to the intensities for every such scan.

What was even more serious, in our opinion, was the loss of resolution at higher count rates, as illustrated in Fig.2 for the scans of the (113) reflection of Al2O3 taken at fixed angle, tube voltage, and counting time at tube currents over the range from 2mA to 40mA. It is clear from the figure that the resolution of the doublet degrades at higher count rates. To see this effect in more detail, in Fig.3a, a Pearson VII function with a 2:1 ratio of Kα1 to Kα2 is seen to fit very well to the experimental data taken in 5 minutes at 45kV and 4mA. However, in Fig.3b, the same function is a very poor fit to data taken at 45kV and 30mA. This effect is entirely a PSD issue, since no similar loss in resolution was found using a solid-state Si(Li) point detector.

In order to reduce detector dead time losses it is clearly helpful to reduce overall count rate on the detector. Note that it is the total count rate, not the peak height, which must be considered in this analysis. This may be accomplished by reducing unwanted radiation through adding a beta filter or better, by using an incident beam monochromator or Goebel mirror. Another option would be to reduce the power of the X-ray source, but this is a poor choice as it reduces the data collection rate and sensitivity to minor phases. Remaining deadtime losses should be characterized and compensated in software.
Figure 1. Plot showing the effect of detector dead time losses at higher incident beam fluxes.

Figure 2. Plots of (113) reflection from Al₂O₃ polycrystalline plate sample showing increasing intensity accompanied by loss of $\alpha_1:\alpha_2$ resolution with increasing tube current (2-20mA in 2mA steps, 25-40mA in 5mA steps) at fixed tube voltage (45 kV).

Figure 3. Detail of (113) reflection from Al₂O₃ polycrystalline plate sample with Pearson VII profile fit with fixed $\alpha_1:\alpha_2$ ratio for 5 minute data collection at 45kV and (a) 4mA, or (b) 30mA.
As shown in Fig. 4, the wide field of view of a PSD allows stray reflections from beryllium windows, scatter from incident beam slit edges, etc., to reach the detector unless steps are taken to block this. In our case, it was found that the curved beryllium window of the HT furnace attachment [Buehler model HDK 2.3] strongly diffracted the incident beam at certain goniometer angles, given in Table 1.

### Table 1. Identification of strong reflections from beryllium window

<table>
<thead>
<tr>
<th>Line # (from Fig.4)</th>
<th>Beryllium (hkl)</th>
<th>2-theta (beryllium)</th>
<th>2-theta (goniometer)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>101</td>
<td>52.8</td>
<td>66.7</td>
</tr>
<tr>
<td>2</td>
<td>103</td>
<td>97.7</td>
<td>114.5</td>
</tr>
<tr>
<td>3</td>
<td>112</td>
<td>101.4</td>
<td>122.3</td>
</tr>
</tbody>
</table>

In Table 1, the goniometer angles are quite different from the 2-theta values expected for beryllium reflections, but this may be readily understood from the instrument geometry, as shown in Fig. 4. It is also apparent that the beam diffracted from the curved Be window does not focus at the detector, as the source to Be distance is much shorter than the Be to detector distance. Consequently, the resulting peak is much broader than diffraction peaks from the sample, as may be seen by comparing the beryllium and LaB₆ peaks in Fig. 5. An anti-scatter shield was designed and attached to the support bracket of the PSD to restrict the field of view of the detector to radiation originating from the irradiated area of the sample. The shield completely eliminated the beryllium reflection, as seen in the lower trace of Fig. 5. It is of course equally conceivable that diffracted X-rays from the sample will also be scattered by the beryllium window en route to the detector, but this effect has not been found to contribute significantly to artifacts in the diffraction spectra to date.

As shown in Fig. 6, the anti-scatter shield also reduces the number of background counts by more than a factor of 4, in this case from 85 counts/min/channel to 20 counts/min/channel. Given there were 500 active channels, the integrated background in this example was reduced by over 30,000 counts/minute. This has obvious advantages in terms of reducing detector dead time losses as discussed previously, and in increasing the peak to background ratio of the scans.

In Fig. 7, we consider the effect of the diffraction peak being offset from the nominal Bragg angle of the goniometer. That is, when the peak is not centered along the length of the detector window, assuming the center of the PSD corresponds to the 2θ angle, the incident beam angle with respect to the sample is not half of the goniometer 2θ. This non-focusing condition has potential impact on the intensity and breadth of the diffraction peak. However, the figure suggests that these effects are relatively minor for a peak located within ±3° or so of the detector midpoint.
Figure 4. Ray diagram of HTXRD system. Source is at A, scatter from Be window at B, scatter from sample at C, detector at D. Strong Be reflections occur at Bragg angles labeled 1, 2, and 3.

Figure 5. PSD scans taken without (upper trace) and with (lower trace) the antiscatter shield showing reduction of background and removal of Be window peak.

Figure 6. PSD scans taken without (upper trace) and with (lower trace) the antiscatter shield between the sample and PSD showing reduced background and improved resolution.
Figure 7. Peak intensity and FWHM of (100) peak for PSD scans on LaB$_6$ powder as the peak is stepped across the active area of the detector.

SUMMARY

Users of PSD based systems need to consider additional sources of error beyond those for conventional slit and point detector systems. We have looked specifically at the mBraun detector, but the issues are likely the same for other PSD products. In addition to the issues discussed by Cheary and Coelho [1-2], three factors were shown to be of particular importance. (1) Detector deadtime counting losses are extremely important in PSD systems, particularly if high quality data for structure refinement are required, but these can be reduced through a combination of hardware and software solutions. (2) There is a significant loss in diffraction peak Cu $K_{α1}$:$K_{α2}$ resolution at higher count rates. (3) The field of view of the detector should be restricted to no more than the sample irradiated area to reduce background and avoid collection of unwanted reflections from various sources.

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REFERENCES