RETRACTABLE KNIFE-EDGE FOR XRD COMBINATORIAL SCREENING

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ABSTRACT

A motorized retractable knife-edge (patent pending) for two-dimensional X-ray diffraction is introduced for combinatorial screening at low Bragg angles. The knife-edge can control the size of the viewed diffracting sample surface, so as to improve diffraction resolution and eliminate cross contamination between adjacent cells in the materials library. It also reduces the background by blocking off the primary beam air scatter. The retractable knife-edge can be driven into two positions, retracted position for automatic sample alignment and sample viewing, and extended position for data collection.

INTRODUCTION

Combinatorial chemistry refers to techniques to fabricate, test, and store the resulting data for a material library containing tens, hundreds or even thousands of different materials or compounds. Combinatorial investigations require rapid screening techniques to test and evaluate variations of composition, structure and properties within a material library. X-ray diffraction is one of the most suitable screening techniques because it is fast and non-destructive [1-2] and an abundant amount of information can be revealed from the diffraction pattern. X-ray diffraction, especially two-dimensional X-ray diffraction (XRD²), can be used to measure the structural information of a material library with high speed and high accuracy [3-5].

In many combinatorial screening applications, such as polymorphism studies in pharmaceutical chemistry and catalysis development in the oil industry, a typical 2θ measuring range is from 2° to 60°. With an XRD² system in reflection mode, the diffracted X-rays are measured simultaneously in a two-dimensional range so that it is necessary to use an incident angle lower than the minimum 2θ. However, the defocusing effect associated with low incident angle reduces the 2θ resolution and has the possibility of producing cross-cell contamination. Figure 1 shows the geometry of two-dimensional diffraction screening in reflection mode. A defocusing effect is observed with low incident angle over a flat sample surface. If one looks at the cross section on the diffractometer plane, the defocusing effect with reflection mode diffraction can be expressed as:

\[ \frac{B}{b} = \frac{\sin \theta_2}{\sin \theta_1} = \frac{\sin(2\theta - \omega)}{\sin \omega} \]

where \( \theta_1 \) is the incident angle, \( b \) is the incident beam size and \( B \) is diffracted beam size. The ratio of \( B \) to \( b \) is the defocusing factor. The diffracted beam is focused to the detector when \( \theta_2 < \theta_1 \). The defocusing effect increases with increasing \( \theta_2 \) or decreasing \( \theta_1 \). The maximum defocusing appears at \( \theta_2 = 90^\circ \). For \( \theta/2\theta \) configuration, the incident angle \( \omega \) is used in the equation.
This document was presented at the Denver X-ray Conference (DXC) on Applications of X-ray Analysis.

Sponsored by the International Centre for Diffraction Data (ICDD).

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Due to the defocusing effect, the incident X-ray beam is spread over the sample surface into an area much larger than the size of the original X-ray beam. In combinatorial screening applications, sample cells are located close each other. Therefore, the defocusing may produce cross contamination in the collected diffraction data. The measured diffraction pattern includes not only the diffraction from the intended cell but also diffraction from the adjacent cells. A motorized retractable knife-edge is used to improve resolution and eliminate cross contamination for combinatorial screening using an XRD$^2$ system.

**SYSTEM DESCRIPTION**

Figure 2 shows the front view of the retractable knife-edge in a two-dimensional X-ray diffraction system. The diffraction system is in a θ-θ vertical configuration. In a conventional diffraction system, a knife-edge is typically mounted on the sample stage. While combinatorial screening requires that the knife-edge to be mounted on a separate stationary base so that the knife-edge does not move with the sample. Instead, each cell is moved to the set instrument center and measured with the same knife-edge setting. Figure 3 is a magnified view of A-A cross section of Figure 2. It shows the space relationship between the knife-edge, the material library and the laser-video automated sample alignment system. The knife-edge has two operation positions: retracted position (solid lines) and extended position (broken lines). The laser beam and optical axis of the microscope intercept at the instrument center so the sample position can be determined by the laser spot position on the sample image. The distance between the surface of the library cell and the instrument center is calculated from the laser spot position. And the sample height (Z) is automatically adjusted to locate the sample surface into the instrument center. It is necessary to expose each cell to the laser and video camera during alignment. The motorized retractable knife-edge allows the automated alignment in retracted position. The diffraction patterns are measured at the extended knife-edge position. The knife-edge tilt angle is adjusted with an adjusting knob to form a parallel gap between the knife-edge and the sample surface. The size of the gap is adjusted through a micrometer. The knife-edge moves with a 3° take-off angle relative to the sample surface so that the gap between the knife-edge and sample surface gradually increases during the retracting operation. This is to reduce the risk of knife-edge striking adjacent sample cells. Figure 4 shows a picture of the motorized retractable knife-edge designed for the Bruker D8 DISCOVER with GADDS for Combinatorial Screening.
Figure 2. The front view of the retractable knife-edge mounted on a two-dimensional X-ray diffraction system for combinatorial screening.

The function of the knife-edge in the extended position is shown in Figure 5. $\theta_1$ and $\theta_2$ is the incident and diffracting angles respectively, and $\delta$ is the gap between the knife-edge and the sample surface. A part of the primary X-rays are blocked by the knife-edge so they will not reach the adjacent cells in the right side of the knife-edge. The diffracted X-rays from the adjacent cells in the left side of the knife-edge are also blocked by the knife-edge. Therefore, only the diffraction from the defined area can reach the detector. The knife-edge can also prevent the direct beam from hitting the detector.
Figure 4. Motorized retractable knife-edge designed for the Bruker D8 DISCOVER with GADDS for Combinatorial Screening.

Figure 5. The knife-edge defines the area of diffraction.

The triangle in Figure 5 shows the relationship between the size of the diffracted area $S$ and the incident angle $\theta_1$, diffracting angle $\theta_2$ and the gap $\delta$.

$$S = \delta (\cot \theta_1 + \cot \theta_2) \quad (2)$$

For a given cell size or distance between the center of adjacent cells. The required knife-edge gap $\delta$ is given as:

$$\delta = \frac{S}{\cot \theta_1 + \cot \theta_2} \quad (3)$$

If a range of $\theta_1$ and $\theta_2$ angles are used, use the lowest possible angles for this calculation.
EXPERIMENTAL RESULTS

The performance of the retractable knife-edge was tested on a Bruker D8 DISCOVER with GADDS for Combinatorial Screening system with corundum powder sample. The sample-to-detector distance was 15 cm. The primary X-ray source and optics included a sealed Cu tube run at 40kV/50mA with spot focus, a graphite monochromator and a 0.5 mm pinhole collimator. Both frames with or without knife-edge were collected at the incident angle $\theta_1 = 1^\circ$ and detector at $\theta_2 = 29^\circ$. Figure 6 shows the diffraction profiles of corundum generated by integration of the 2D frames. One frame was collected with knife-edge and the other without knife-edge. The profile with knife-edge is scaled up by a factor of 1.5 for easy comparison. The gap of knife-edge was approximately 0.05 mm. The tabulated FWHM values show that diffraction peaks without knife-edge are much broader than those with knife-edge. The knife-edge dramatically improves the $2\theta$ resolution of the instrument for this experiment. The profile with knife-edge also has significantly lower background in this experiment.

![Figure 6. Diffraction profiles from corundum power with and without Knife-edge.](image)

The cross contamination was tested with a proprietary fixture designed for microdiffraction alignment and screening system verification. The fixture has an area with three Cu samples of 1 mm in diameter and 5 mm separation. The minimum incident angle without cross contamination for a 0.5 mm beam and 5 mm separation is about $\theta_1 = 6^\circ$. Figure 7 shows two frames collected at the incident angle $\theta_1 = 4^\circ$ and detector at $\theta_2 = 41^\circ$. The first frame in the figure is collected without the knife-edge where both (111) and (200) peaks appears as three peaks. The center peak is from the Cu sample aligned to the instrument center and the two peaks on both sides are from the adjacent Cu samples. The second frame is collected with knife-edge in which no cross contamination is observed.
CONCLUSION

The motorized retractable knife-edge for combinatorial screening has the following functions:
1. It allows one gap setting ($\delta$) for all cells in a material library.
2. The motorized positioning allows automatic sample alignment and sample viewing from the laser video microscope.
3. It improves the diffraction resolution for low angle measurements.
4. It lowers background by reducing air scatter.
5. It eliminates cross-cell contamination.

REFERENCES