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DIFFRACTION EFFICIENCY AND RELATIVE INTENSITY OF VARIOUS X-RAY ANALYZING CRYSTALS AT $\text{Cu}_{K\alpha}$ AND $\text{Sn}_{K\alpha}$ WAVELENGTHS AND WITH TWO SURFACE CONDITIONS

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CONTENTS

Abstract	1
Introduction	1
Experiment Results	1
Experiment Conditions and Equipment	3
Measurement Conditions	3
References	4

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ABSTRACT

Crystals used for x-ray analysis come in many useful planes and 2d spacings with great variation in diffraction efficiency. This report compares the diffraction efficiency of 13 different crystals at two wavelengths, $\text{Cu}_{K\alpha}$ and $\text{Sn}_{K\alpha}$, and with two surface conditions, first with a cleaved or polished surface and second with a ground or sandblasted surface for enhanced diffraction. It is not obvious from published information how to select a crystal for best detection efficiency for a given plane or 2d spacing. The information in this report should help users select a crystal for a specific application.

INTRODUCTION

This report compares some commonly used x-ray analyzing crystals under a set of standard conditions at 8 and 25 keV. It shows that surface condition is a large factor in efficiency and relative intensity. ASTM or JCPDS card files give the relative intensity between various diffracting planes of a crystal and (usually) $\text{Cu}_{K\alpha}$ x-rays, but they do not

relate crystals to other crystals or compare them at different wavelengths of x-ray energy.

Crystals selected for this report have average rather than superior characteristics. They were randomly selected from crystals as purchased from various vendors or as cleaved from our stock.

EXPERIMENT RESULTS

This report confirms the results of an earlier study, which showed that treating the surfaces of lithium fluoride crystals enhances their efficiency.¹ Information given here shows that uniform roughening of the crystal surface by lapping or light sandblasting does indeed enhance the diffraction efficiency of all crystals used (see Table 1 and Fig. 1). The one crystal not shown in the reflection mode is quartz, which we use in transmission.

In all cases where one surface is cleaved or polished and the other side is roughened by sawing, grinding, spark discharge, lapping, or sandblasting, the rough side is more efficient.² This has two benefits:

- Where the crystal lattice planes lie parallel to a polished front surface and the rear ground surface, as ascertained by x-ray analysis giving 2d spacing and proper omega and two-theta angles, the front polished surface may be used for optical-laser alignment to the x-ray system.

- Then, in cases of a low-intensity signal, the more efficient rough side may be used by reversing the crystal in its holder without loss of alignment.

The greatest intensity gain by surface treatment applies to flat diffracting crystals. This enhancement is greatly reduced when the flat lapped or sandblasted crystal is either plastically or elastically deformed into a convex curved radius for spectrum dispersion.³

Fig. 1. Diffracted x-ray efficiency and relative intensity of various x-ray analyzing crystals at Cu_{K α} and Sr_{K α} .

Table 1. Diffracted x-ray efficiency and relative intensity of various x-ray analyzing crystals at $\text{Cu}_{K\alpha}$ and $\text{Sn}_{K\alpha}$.

Crystal	$2d, \text{\AA}$	Surface condition	Efficiency at $\text{Cu}_{K\alpha_1}$	$2\theta^\circ$	Efficiency at $\text{Sn}_{K\alpha_1}$	$2\theta^\circ$	Mode ^a
KAP (010)	26.632	Cleaved	3.0×10^{-3}	6.6	9.505×10^{-4}	2.0	R
		Sandblasted	5.736×10^{-3}	6.6	1.818×10^{-3}	2.0	R
RAP (010)	26.121	Cleaved	4.606×10^{-3}	6.8	7.108×10^{-4}	2.2	R
		Sandblasted	9.414×10^{-3}	6.8	1.779×10^{-3}	2.2	R
Mica Fr	19.884	Cleaved	1.322×10^{-3}	8.8	6.663×10^{-4}	2.8	R
(002) US	19.884	Cleaved	1.573×10^{-3}	8.8	7.479×10^{-4}	2.8	R
US	19.884	Sandblasted	2.631×10^{-3}	8.8	1.062×10^{-3}	2.8	R
ADP (101)	10.659	Polished	3.303×10^{-3}	16.6	9.483×10^{-4}	5.3	R
		Ground	1.034×10^{-2}	16.6	5.528×10^{-3}	5.3	R
EDdT(020)	8.808	Polished	2.393×10^{-3}	20.1	4.851×10^{-3}	6.4	R
		Ground	1.633×10^{-2}	20.1	1.129×10^{-2}	6.4	R
PET (002)	8.726	Cleaved	2.012×10^{-2}	20.3	1.159×10^{-2}	6.5	R
		Ground	3.976×10^{-2}	20.3	1.954×10^{-2}	6.5	R
Quartz (10 $\bar{1}1$)	6.686	Polished	5.631×10^{-4}	26.6	3.030×10^{-4}	24.0	T
Ge (111)	6.532	Polished	1.324×10^{-2}	27.3	4.873×10^{-3}	8.6	R
		Ground	1.923×10^{-2}	27.3	1.104×10^{-2}	8.6	R
LiF (200)	4.027	Cleaved	4.152×10^{-3}	45.0	6.270×10^{-3}	14.0	R
		Sandblasted	1.766×10^{-2}	45.0	2.377×10^{-2}	14.0	R
LiF (220)	2.848	Sawed ground	5.513×10^{-3}	65.5	1.834×10^{-2}	19.8	R
Topaz (303)	2.712	Ground	7.948×10^{-5}	69.23	9.744×10^{-3}	20.8	R
Quartz (31 $\bar{4}0$)	2.3604	Polished	5.513×10^{-5}	81.5	1.208×10^{-3}	8.4	T
LiF (420)	1.801	Sawed/ground		109.0	5.407×10^{-3}	31.6	R

^aT = Transmission

R = Reflection

EXPERIMENT CONDITIONS AND EQUIPMENT

● Various flat x-ray analyzing crystals randomly selected.

● X-ray generator 50 kV power at 10 mA tungsten tube, 2.5-mm spot focus.

● $\text{Cu}_{K\alpha}$ fluorescer target 8.047 keV (1.540 $\text{\AA}$).

● $\text{Sn}_{K\alpha}$ fluorescer target 25.27 keV (0.4906 $\text{\AA}$).

● In air 0-90° 2θ angle.

● Detector 2-in.-diam sodium iodide photomultiplier (PM) tube 1 kV DC+.

● Run No. 1, surface polished as cleaved or supplied.

● Run No. 2, surface roughened (sawed, lapped, sandblasted).

MEASUREMENT CONDITIONS

Average of two 100-s counting periods.

Zero order (I_0):

1-mm-diam pinhole in lead mask over PM tube (corrected in efficiency formula). Shutter open, crystal out, pinhole in.

Zero-order background (I_0B):

Shutter closed.

Diffracted beam (I_d):

Pinhole out, 1-cm $\times$ 2.5-cm collimator mask over PM crystal in place.

Diffracted-beam background ($I_d B$):

Crystal out of beam, shutter open, collimation
and machine power constant.

Efficiency formula:

$$\epsilon = \left[\frac{(I_d - I_d B)(PH \text{ diam})}{I_0 - I_0 B} \right] \left[\text{Geometry factor } \frac{d}{d+D} \right]$$

where

d = distance from source to crystal (see Fig. 2),

D = distance from source to detector.

Fig. 2. Filter-fluorescer calibration unit used for obtaining crystal diffraction efficiency. The fluorescer foils are ellipses $1.27 \text{ cm} \times 45^\circ$. a = tungsten anode x-ray tube (50 kVP-10mA), b = fluorescer wheel (10 channels), c = collimator, d = filter wheel (filter diam 2.54 cm), e = shutter, f = photomultiplier tube, g = shield, h = detector, i = main beam pinhole (0.99 cm diam), zero order only, (D) = distance from source to detector, (d) = distance from source to crystal.

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