Processes for Characterization of X-Radiation of Diffraction Anodes*

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X-Ray Diffraction has been a fundamental technique in many areas of science for generations. Essential to the technique is characterization of anode available to the microscopist. This involves developing a spectra of emitted x-radiation such that reconstruction of a lattice or determination of chemical properties is possible. In this work, we demonstrate a simple and effective technique to extract the spectra from a molybdenum anode in Bragg configuration and discuss key resolution considerations to properly resolve high order diffraction.

I. INTRODUCTION

In early 1913, proposals were put forth that it may be possible to observe diffraction due to the light wave, spin-orbit interaction in crystalline lattices in an effect which is known well known as Bragg Diffraction. In the century since, application of such physics has been applied to pioneer crystallography and metrology, and in this work we aim characterize the x-radiation of molybdenum by imaging the bragg reflection upon a calibration substrate.

The emission spectra of molybdenum observed in this work arises from the fine structure of the L shell, and subsequently the spin-orbit interaction of the electrons with x-radiation [1]. Furthermore, there exist three subshells, L_I, L_{II}, L_{III}, which are subject to emission rules:

$$\Delta I = \pm 1, \qquad \Delta j = 0, \pm 1$$

Where I is the orbital angular momentum, and j the total angular momentum, therefore, two transitions from the L-shell to the K-shell are permitted.

We aim to directly measure these by first directing electrons from a source to the molybdenum anode, bombarding it with electrons and ionizing electrons in the inner K shell. This prompts the formation of a hole which is immediately relaxed by the electrons in the upper L orbital, emitting x-radiation. Bragg's diffraction relation models these constructive interference produced by emitted x-radiation via equation I. For information on the derivation, see [2].

$$n\lambda = 2d\sin\theta \qquad (I)$$

Our machine operates using Bragg-Brentano geometry, such that the substrate is flat relative to the incident x-rays which are fired into a detector (see figure 1). A photon sensitive detector is equipped to orbit the sample in increments of 0.1°. The detector sees a powder diffraction pattern, appearing as a series of rings, where brightness peaks will occur at characteristic $K_\alpha, K_\beta, K_\gamma$ lines [3].

II. CHARACTERIZATION OF BRAGG PEAKS

In general, a pristine calibration sample is needed to determine characteristic peaks in bragg diffraction spectra. We use NaCl, due to its relative commonality and uniform cubic structure. Handling the structure by the short axis is necessary to avoid strain. For more discussion, see Appendix A. We begin by choosing a range of angles to search. In our case, we wish to observe the first and fifth diffraction order to characterize K_α, K_β, and K_γ peaks. Estimation of these ranges can be determined using the known lattice spacing of NaCl of 564 nm [4].

We accelerate electrons using a 35 kV source across the anode. X-rays are collected and passed through a narrow 1 mm collimator, where they are reflected across the substrate and collected by the photosensitive detector (PSD). We maintain a 2:1 ratio between collimator angle and detector angle, as shown in Figure 1.
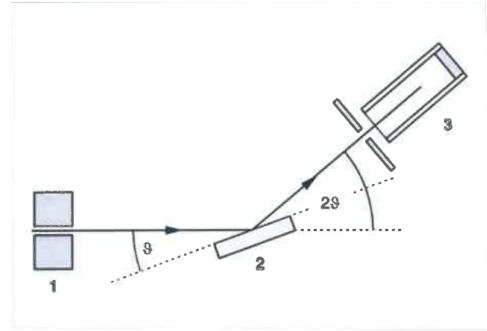

FIG. 1. Bragg-Brentano reflection geometry, where x-rays are emitted from a collimator (1), reflect off the sample (2) and are detected by the PSD (3).

It is essential to accurately characterize the peaks in the resulting powder diffraction spectra. We accomplish this by fitting a standard gaussian function in the domain of candidate peaks. This alone is surprisingly challenging for the 5th order diffraction peaks where there is large signal-to-noise-ratio in the region neighboring the K_β and K_γ peaks. Several options exist to reduce noise, namely using wider slit sizes to increase flux (results in lower resolution), and using a longer counting time (re-

sults in lower signal to noise ratio). With a similar motivation to the latter, we choose to take several scans of our sample. Because background noise is approximately white, the average between scans will result in curves that overlap about the peaks but have no discernible trend. This allows us to easily fit our peaks without the need for sophisticated techniques to isolate the background [5].

III. RESULTS & DISCUSSION

In this paper, we directly measure the x-ray emission spectra of a molybdenum anode and demonstrate how it is possible to use this characteristic spectra to determine lattice parameters in unknown materials. Our parameters are found to be within 1.7% for first-order diffraction, and within 6.29% for fifth-order diffraction, when compared to literature values [4], shown in table I.
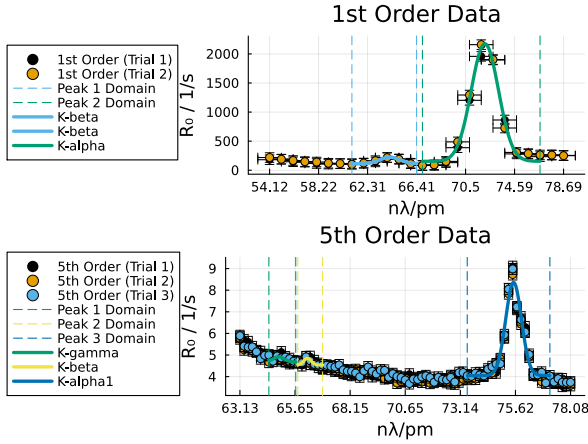

FIG. 2. Bragg spectra collected by the x-ray diffraction equipment. Gaussian fits are drawn over locations of peaks. In the first-order data, peaks are well estimated to be $K_\beta + K_\gamma$=64.16, and K_α = 72.147. At the higher diffraction order, lattice impurities become much more significant, resulting in a substantial shift in the mean, μ of the peaks.

Literature notes an additional $K_{\alpha 2}$ peak neighboring the fifth-order $K_{\alpha 1}$ peak. Due to the angular resolution limitations, we were unable to image this peak, instead we observe the smearing between $K_{\alpha 1}$ and $K_{\alpha 2}$ peak (see Appendix A for more details). Future work on the sub-

ject would benefit from sub 0.1° angular resolution, as well as a narrower collimator and longer counting times in this very narrow band.

It is apparent from our results that there is a significant shift in the peak locations when compared to literature, and we attribute this to strain and thermal contraction in our sample invoking a shift in path length.

Type	μ $(\frac{\lambda}{\text{pm}})$	μ (Literature Value)	Order
$K_\beta + K_\gamma$	64.16±0.1	63.09	1
K_α	72.147±0.031	71.08	1
K_γ	64.9 ±0.29	62.09	5
K_β	66.21 ±0.21	63.26	5
$K_{\alpha 1}$	75.553 ±0.024	70.93	5
$K_{\alpha 2}$	N/A	71.36	5

TABLE I. Location of exact emission peaks obtained from gaussian fits compared with literature values [4].

Appendix A: Discussion of Precision and Uncertainty

Throughout this work, we use linear propagation theory for the propagation of error via the excellent measurements Julia library [6].

Furthermore, fitting is done using iMinuit and underlying algorithms [7].

Instrument uncertainty is unspecified by the manufacturer, and therefore we take detector uncertainty to be negligible. Statistical uncertainty is significant, We preform several standard statistical assessments of our data, necessary for chi-square testing. Goodness of fit (chisquare) parameters can be found in table II. In general, all except K_γ are modeled well. This suggests that this is not simply unusually wide peak. Instead, this suggests a second peak which cannot be resolved with the angular resolution of our equipment.

Peak	Chi-Squared
K_β	0.008647
K_α	0.802178
$K_{\alpha-1}$	0.156140
K_β	0.141042
K_γ	1.954210

TABLE II. Chi-squared parameters for fits.

[1] LD Didactic, *X-Ray Apparatus*, LD Didactic GmbH, manufacturer's manual.
[2] W. L. Bragg, The diffraction of short electromagnetic waves by a crystal, Scientia **23**, 153 (1929).
[3] M. Taddei, Fundamentals and applications of x-ray diffraction (2015), accessed: 2025-03-29.
[4] D. V. Rao, Table of isotopes, edited by c. m. lederer and v. s. shirley, Medical Physics **6**, 540 (1979), https://aapm.onlinelibrary.wiley.com/doi/pdf/10.1118/1.594620.
[5] Wikipedia contributors, Rietveld refinement — Wikipedia, the free encyclopedia (2024), [Online; accessed 29-March-2025].

[6] M. Giordano, Uncertainty propagation with function-ally correlated quantities, ArXiv e-prints (2016), arXiv:1610.08716 [physics.data-an].

[7] F. James and M. Roos, Minuit: A System for Function Minimization and Analysis of the Parameter Errors and Correlations, Comput. Phys. Commun. **10**, 343 (1975).