X-Ray Diffraction Title*

Grant Saggars†
University of Kansas

Jose Marquez
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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 demonstrate how this may be used to reconstruct the lattice structure.

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 NaCl crystals.

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. 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 0.5°. 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 [2].

II. CHARACTERIZATION OF BRAGG PEAKS

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 5^{th} order diffraction peaks where there is severe noise 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 (results 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 [3].

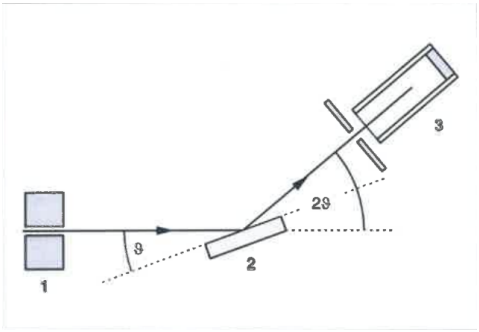

FIG. 1. Bragg-Brentano reflection geometry, where x-rays are emitted from a collimator (1), reflect off the sample (2) and are detected by the PSD (3).

III. 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.

Literature notes an additional $K_{\alpha 2}$ peak neighboring the fifth-order $K_{\alpha 1}$ peak. Due to the angular resolution

* Mr. Bragg, for the Background
† Grant-S@ku.edu

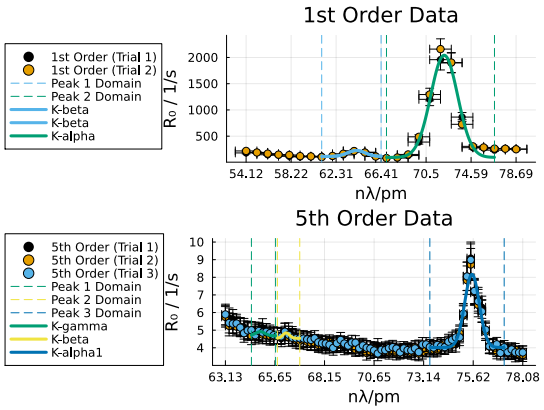

FIG. 2.

limitations, we were unable to image this peak, instead we observe a perceptual widening of the larger $K_{\alpha 1}$ peak. Future work on the subject would benefit from sub 0.5° 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.

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]. *Note, uncertainty is underestimated.*

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 [5].

Furthermore, fitting is done using iMinuit and underlying algorithms [6].

We preform several standard statistical assessments of our data, necessary for chi-square testing. *note: information related to detector uncertainty is not yet available at the time of writing.* Goodness of fit (chi-square) parameters can be found in table **??**.

Chi-square values cannot be computed accurately until full uncertainty is understood and characterized.

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[3] Wikipedia contributors, Rietveld refinement — Wikipedia, the free encyclopedia (2024), [Online; accessed 29-March-2025].

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

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