# Designing a High Throughput Characterization Device using X-ray Diffraction and X-ray Fluorescence

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## Introduction

X-ray fluorescence (XRF) and X-ray diffraction (XRD) are powerful materials characterization techniques that provide information about chemical composition and crystal structure. While usually done separately, combinatorial high throughput (CHT) characterization methods have demonstrated that techniques such as XRF and XRD can be combined into one system to rapidly obtain high volumes of data about material properties. Since both characterization methods utilize a high energy photon source, combining these techniques would allow their results to be obtained simultaneously, substantially decreasing data acquisition time compared to when they are conducted separately. When used in conjunction with an automated data analysis program, structural materials with incrementally varied compositions can be studied to understand how their structural properties are influenced by changes in composition[1]. Characterization techniques beyond XRF and XRD can be added to this system to generate robust materials data to make the development and testing of alloys more efficient.

XRF is the focus of this study as the resolution and analysis of data is crucial for CHT systems, which can be modeled computationally and analyzed experimentally.

## Objectives

The primary objective of this project is to design a CHT system using XRF and XRD to perform rapid characterization and development of materials by:

1. Computationally determining the resolution of data that can be obtained from XRF and generating accurate spectra for metal alloys.
2. Comparing computational results to experimental results to understand how rapidly and accurately materials can be characterized in our system.
3. Using these results to determine the optimal orientation of parts for this system, including the source, sample and detectors for XRF and XRD.

## Methods

The following equation was derived to computationally model fluorescence intensity, with the mass attenuation determined by extrapolating available data to include all energy levels for each element. This initial code output the photon count rate at the fluorescence detector for a single element[2].

$$I_i = \left[(I_o J_K g_{K\alpha} \omega_K)\left(\frac{\Omega}{4\pi}\right)\right]\left[\frac{\mu_s}{\mu_s + \mu_{sE_i}}\right]$$

- Fluorescence intensity at detector ($I_i$)
- Intensity from source ($I_o$)
- Absorption jump ratio ($J_K$)
- Transition probability ($g_{K\alpha}$)
- Fluorescence yield ($\omega_K$)
- Mass attenuation coefficient of incident X-rays ($\mu_s$)
- Mass att. coeff. of fluorescence X-rays ($\mu_{sE_i}$)

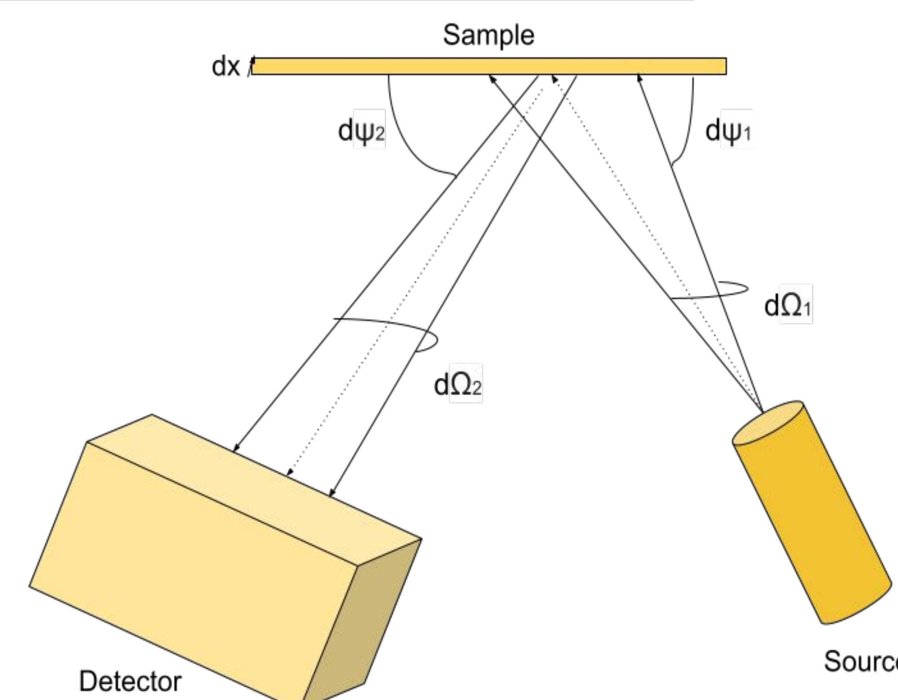


**Figure 1.** XRF setup with X-rays directed from the source to the sample and fluorescence leaving the sample in all directions with only a portion reaching the detector

A graph was generated from the photon count rate to output a spectrum showing the distribution of photons measured at the detector based on its resolution. Finally, the code was updated to generate a spectrum for an alloy based on the composition and the mass attenuation coefficients of the elements (Figure 2).

Experimentally, a silicon drift detector (SDD) with a beryllium screen was pointed at the sample to detect fluorescence (Figure 3). A 24 keV X-ray source was used with a power of 50 W and spot size of 80 by 20 µm. Data was collected over an acquisition time of 1 minute and calibrated using Cu, Zn, Fe, and W spectra.

## Results

### XRF Spectra

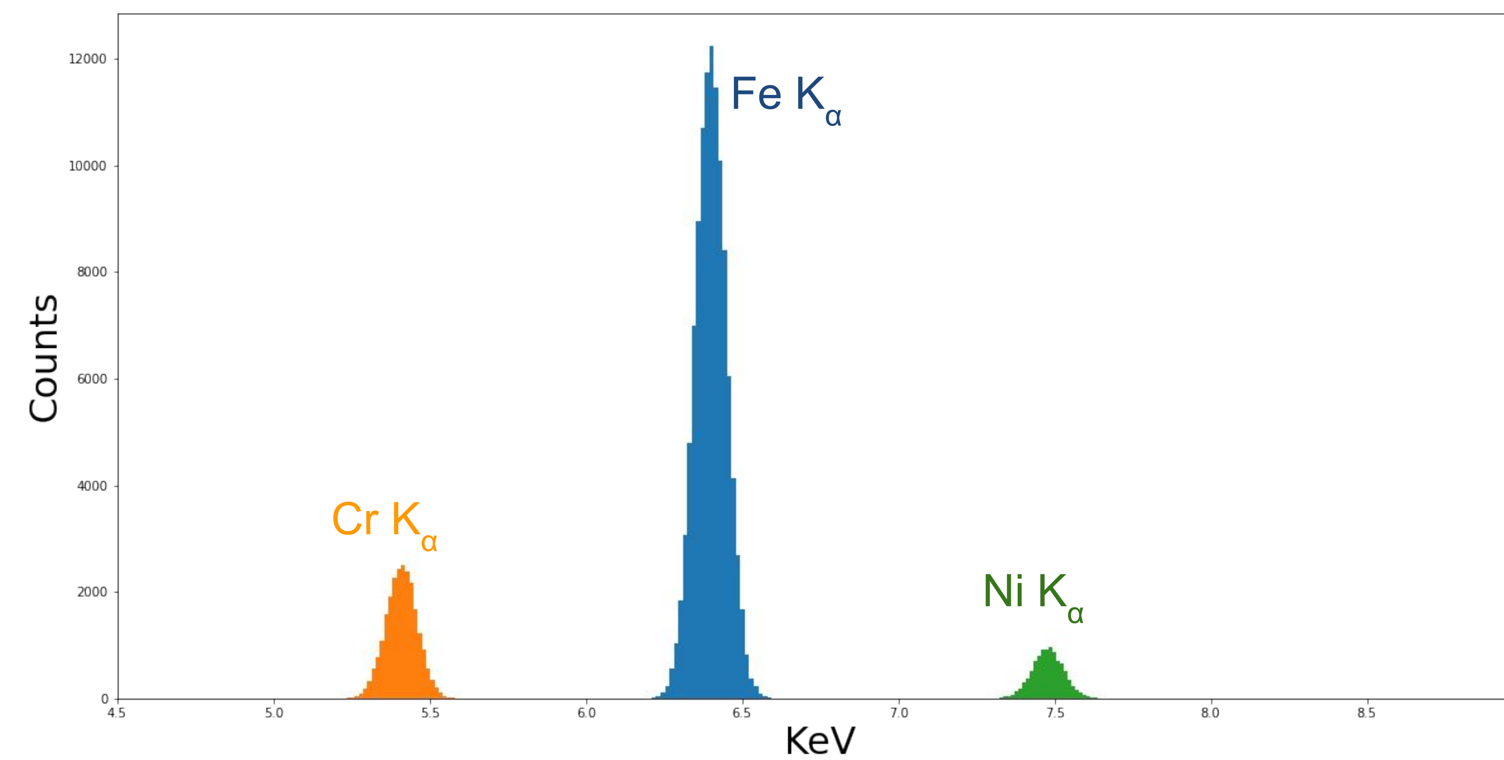


**Figure 2.** Theoretical output of photon energy distribution for a nickel, chromium, and iron alloy

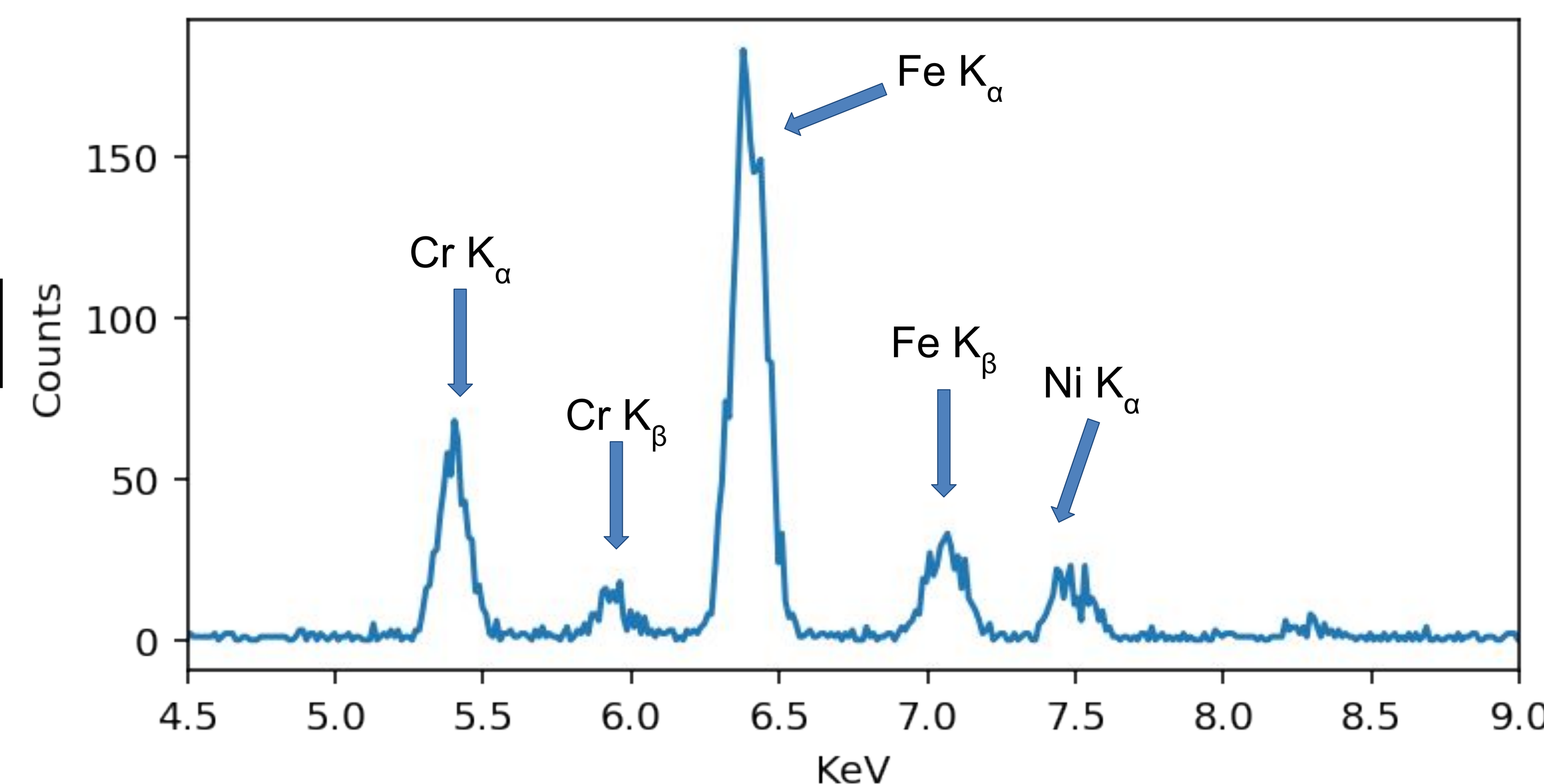


**Figure 3.** Experimental XRF spectrum of the unknown metal alloy with identified peaks

Table 1 compares the composition estimated using these spectra with the actual composition of the sample as later revealed.

### System Design

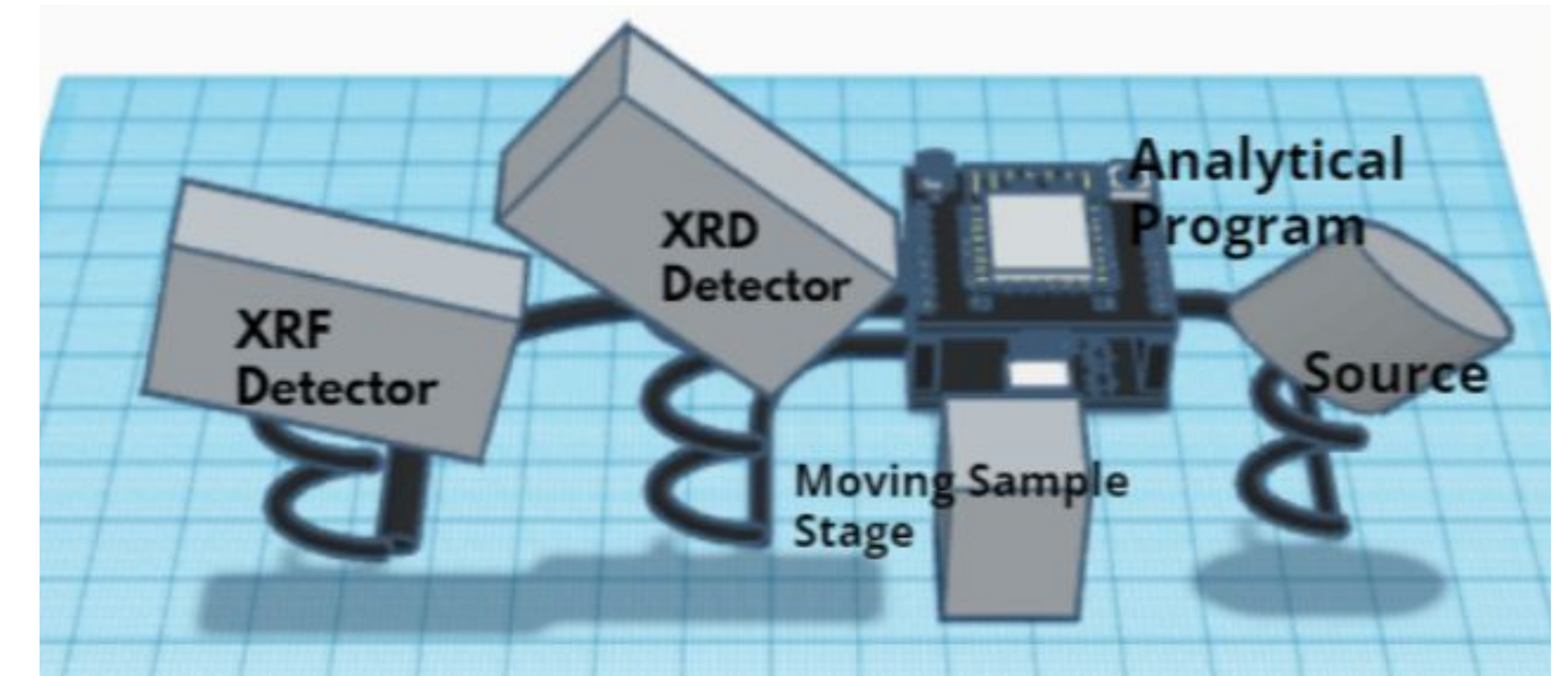


**Figure 4.** Orientation of components for CHT XRF and XRD system

As depicted in Figure 4, a single X-ray source and two detectors are required to collect XRF and XRD data via an analytical program. The movable sample stage is of paramount importance so that different sections and therefore compositions can be tested consecutively. The distance between the sample and detector should be 159 mm with an angle of 90°. A balance between data acquisition time and accuracy was weighed since one of the greatest benefits of this CHT methods is its speed. An acquisition time of 60 seconds is required to obtain suitable data for analysis with the desired accuracy.

**Table 1.** Elemental composition of unknown metal sample

| | Estimated (%) | Actual (%) |
|---|---|---|
| Nickel | 6 | 10 |
| Chromium | 40 | 18 |
| Iron | 54 | 72 |

## Conclusions and Future Work

Although we were unable to accurately estimate the composition of an unknown sample computationally, the necessary acquisition time and resolution of the data indicates that this XRF system is suitable for high throughput applications.

Future work will focus on similar analysis for the XRD system to determine how well the two methods align. Development of a computational program to sort and analyze data is necessary due to the large volume collected. Combining this system with other characterization techniques will create a comprehensive system for rapid and total materials characterization.

## Acknowledgements

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1. Miracle, D.B., et al. (2021) Annu. Rev. Mater. Res. 51, 131-64. https://doi.org/10.1146/annurev-matsci-080619- 022100
2. Thomsen, V. (2007) Spectroscopy. 22(5), 46.