Research on Electron Probe Micro Analysis

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Introduction

A significant challenge in material science and engineering is understanding and controlling the precise chemical composition of materials at the microscale, especially in complex and heterogeneous samples. This is crucial in fields such as materials development, geology, and microelectronics, where the properties and performance of materials are directly linked to their elemental composition. However, the lack of detailed compositional data at small scales often leads to limitations in material optimisation and quality control. Electron Probe Microanalysis (EPMA) is essential for addressing these challenges by providing high-precision, non-destructive elemental analysis and mapping at the microscale. Despite its capabilities, there is still a need for further research to improve the accuracy, resolution, and applicability of EPMA.

Preparation of Samples for EPMA

The technique used for preparing thin films is called sputtering. Sputtering occurs when high-energy ions collide with a target material, transferring momentum and causing a series of collisions. If an ion has enough energy when it reaches the surface, it can dislodge atoms from the target material, resulting in a deposition of material.
This technique is used within a vacuum chamber shown in Figure 1, where the chamber is connected to a series of vacuumed deposition techniques. The layers made for the EPMA are shown in Table 1.

Test No.	Layers	Substrate
1a	Ta	Sapphire
2a	Nb	Sapphire
3a	Nb/Ta	Sapphire
4a	Nb/Co/Nb	Sapphire
4b	Nb/Co/Nb	Silicon
4c	Nb/Co/Nb	Silicon & Oxide layer
5a	Co/Nb/Ta	Sapphire

Table 1: Test Specimen Specification

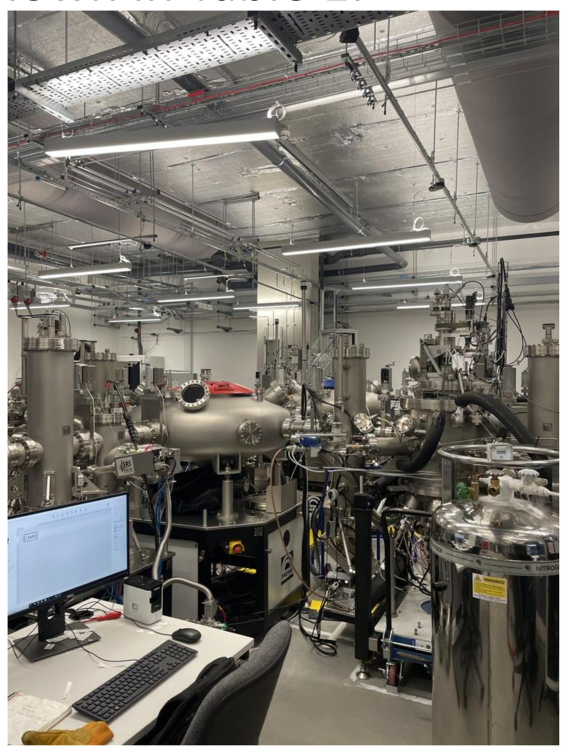

Figure 1: Lab Equipment

EPMA Research

An Electron Probe Microanalyser (EPMA)(shown in Figure 3) works by focusing a high-energy electron beam onto a sample, where the electrons interact with the atoms, causing the emission of characteristic X-rays (shown in Figure 2). These X-rays, unique to each element, are then detected and measured by the machine's spectrometers. By analysing the intensity and energy of the emitted X-rays, the EPMA determines the elemental composition of the sample with high precision.

To enhance accuracy, multiple accelerating electron voltages are used, each generating different specimen volumes based on the depth of electron penetration. By combining the specimen volume data from each voltage, a more accurate chemical composition can be determined. This combination of specimen volume data is achieved using a software called Badgerfilm.

Unfortunately, due to time constraints and laboratory equipment failure, the prepared specimens could not be fully analysed to produce a valid result. Despite this, the progress made toward achieving a validated result shows promise and looks to be a valuable insight into chemical composition in the near future.

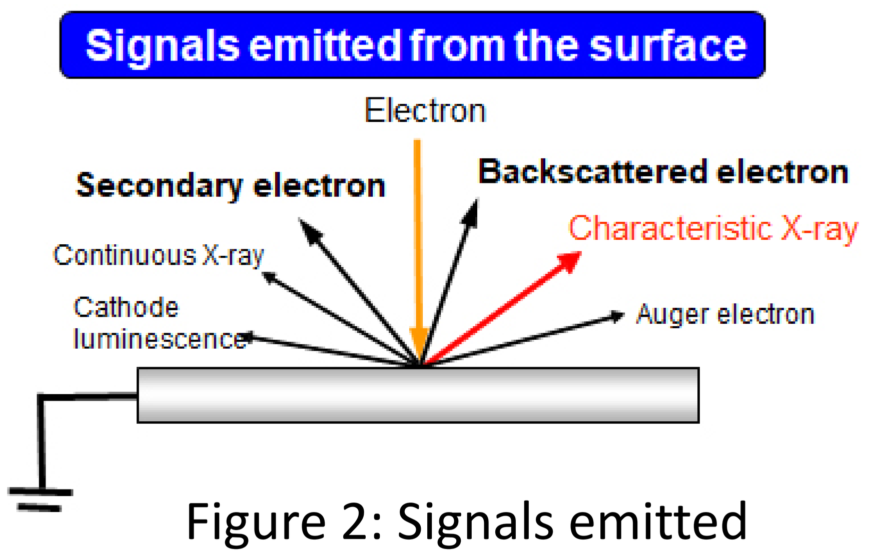

Figure 2: Signals emitted from interaction(1)

Figure 3: EPMA

Validation using X-Ray Reflectivity (XRR)

Using the prepared samples shown in Table 1, each of these were tested using X-ray reflectivity (XRR) equipment shown in Figure 4. The purpose of this test is to validate the results obtained from the EPMA equipment ensuring accurate and increased confidence in results.
X-Ray Reflectivity (XRR) works by directing a beam of X-rays at a shallow angle onto a material's surface, where the X-rays partially reflect off different layers. These reflected beams interfere with each other, creating a pattern of peaks and dips in reflectivity. By measuring the intensity of the reflected X-rays as a function of the incident angle, information about the thickness, density, and roughness of the material's layers can be obtained.
The reflected X-ray and incident data are imported into a software called GenX, which plots the intensity of X-rays against the angle of incidence. By adjusting variables such as thickness, roughness, and density of the layers to fit the XRR data, the software provides estimated values for these parameters for each sample. Examples of this is shown in Figure 5, where the blue curve represents the XRR data, and the red curve overlapping the blue represents the fitted data.

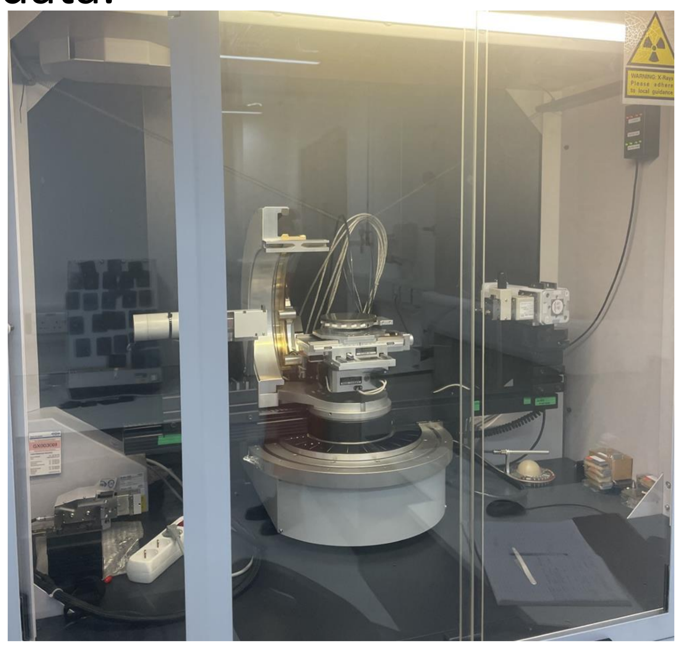

Figure 4: XRR Equipment

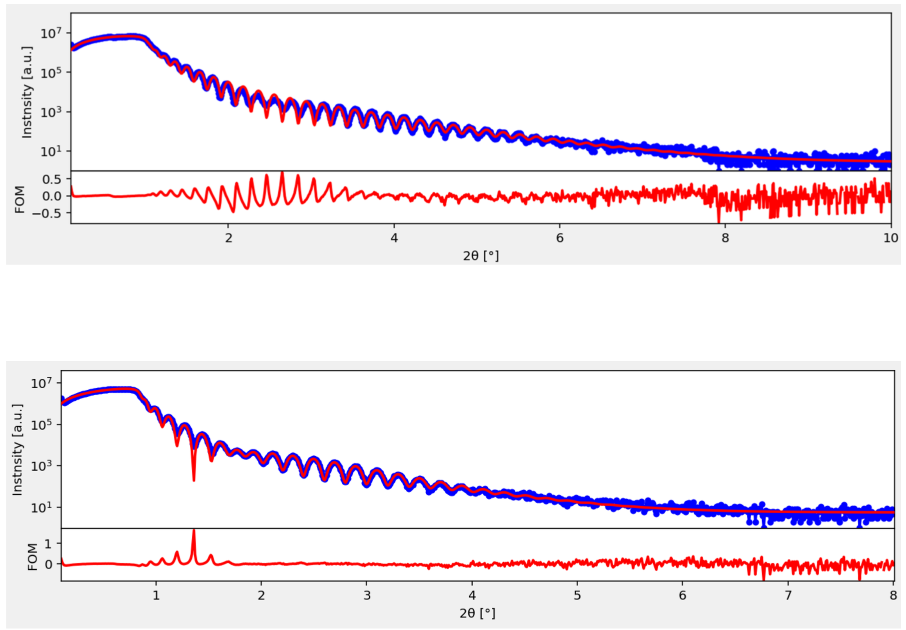

Figure 5: Examples of GenX fitting

Conclusion

This study highlights the critical role of Electron Probe Microanalysis (EPMA) in advancing the understanding of material composition at the microscale, particularly in fields where precise elemental analysis is crucial. Despite challenges such as time constraints and equipment failures, significant progress was made in preparing and analysing samples using EPMA, supplemented by validation through X-Ray Reflectivity (XRR). The integration of multiple accelerating electron voltages and advanced software tools like BadgerFilm and GenX demonstrates the potential for achieving highly accurate compositional data. Although the full analysis could not be completed, the methodologies employed show promise for future research and contribute valuable insights into the accurate determination of chemical compositions

References

1) Toray Research Centre (2024). Electron Probe Micro Analyzer : EPMA | Commissioned Analysis and Research | Technical Information | Toray Research Center. [online] Toray Research Center. Available at: https://www.toray-research.co.jp/en/technical-info/analysis/EPMA.html [Accessed 24 Aug. 2024].