APPLICATION NOTE

Investigation of Ni on Si thin film with ARL EQUINOX 100 X-ray Diffractometer

Introduction

X-ray diffraction is one of the commonly used structural analysis techniques to characterize thin films/layers and coatings. Both in industrial and in research laboratories, demand for the analysis of thin films and coatings has been growing, thanks to the development of a large variety of applied materials. Such materials are for example used in photovoltaic collectors for green energy harvesting, vice versa as materials for generating light in LEDs and Lasers or as materials for sophisticated optical applications.

The crystallographic structure of multi-functional inorganic and hybrid organic-inorganic thin films (nanometer scale) can be characterized by GIXRD (Grazing Incidence XRD). This is important as electronic and optical properties strongly depend on the structure of compounds. In GIXRD a fixed gracing angle (~0.5° – 2° Ω) is used to exclusively measure the layer and not the substrate.

Another variable in designing thin film materials for various applications is the thickness of certain layers. One standard technique for determining the thickness of thin layers is X-ray reflectometry (XRR), which is based on the interference between X-rays reflected on different layers in the material.

Instrument

Thermo Scientific™ ARL™ EQUINOX Series represent a portfolio of XRD instruments from simple, easy to use bench-top systems for routine analysis to more advanced floor-standing, high performance, research grade systems.

Thermo Scientific™ ARL™ EQUINOX 100 employs a custom-designed 50 W Cu or 15 W Co high-brilliance micro-focus tube with mirror optics. No external water chiller is required to run this instrument. The thin film attachment provides computer controlled ω and z movement for sample alignment and measurement (cf. Figure 1). This benchtop instrument can be transported between laboratories or into the field without requiring any special infrastructure.

The ARL EQUINOX 100 (cf. Figure 2) provides very fast data collection compared to other diffractometers due to its unique curved position sensitive detector (CPS) that measures all diffraction peaks simultaneously and in real time and is therefore well suited for fast screening of thin film samples using GIXRD.

Figure 1: Thin film attachment

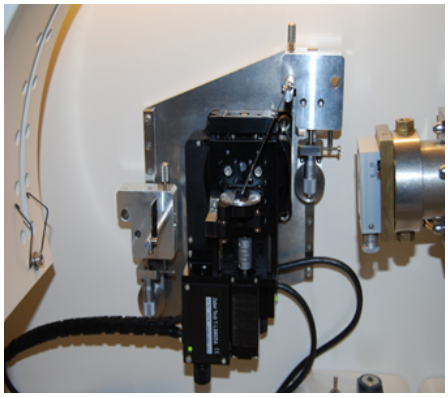

Figure 2: ARL EQUINOX 100 diffraction system

Experimental

A Ni on Si substrate sample (1 layer, 306.9 Å thickness; AXO) was measured using an ARL EQUINOX 100 (Cu-Kα radiation: 1.541874 Å) after carefully aligning z and ω. Data processing and evaluation was performed using SYMPHONIX (data processing), MATCH! equipped with the COD database (qualitative phase analysis) and MAUD (XRR data refinement).

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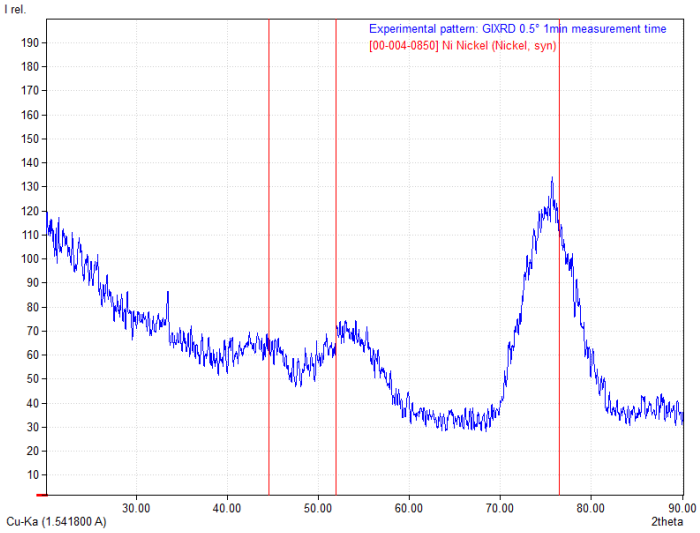
Results

The crystallographic structure of the Ni layer was investigated using GIXRD. The XRD pattern was afterwards compared with a database (COD) which clearly allows assigning the common Ni structure type (space group $Fm\bar{3}m$, a = 3.5238 Å) after 1 minute of measurement time (cf. Figure 3).

Figure 3: GIXRD pattern (20 - 90°2θ grazing angle 0.5°)



For the refinement of the XRR data a model containing the Si substrate, a buffer layer and the final Ni layer compared to air was set up and in a final step all parameters were refined independently. The model yields a sufficient fit (cf. Figure 4 and Table 1).

Figure 4: XRR pattern (black; 0 - 4°2θ) with fit (red); Intensities in logarithmic scale

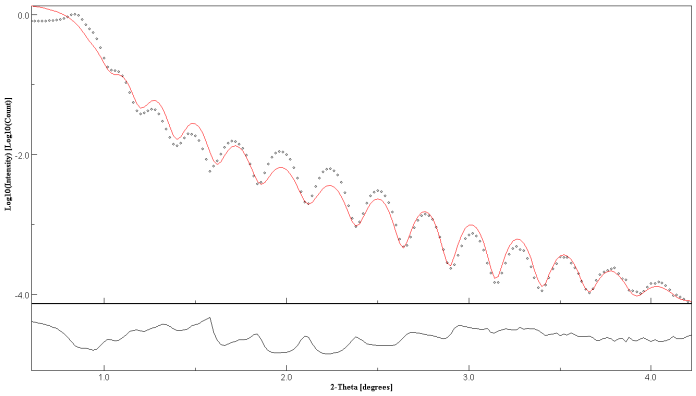


Table 1: Results of XRR refinement

Rwp	0.042
χ^2	2.47
Thickness (in Å)	320.7(11)
Roughness (in Å)	3.78(32)

The deviation between refined and given thickness is 5%, which is reasonable for a benchtop system.

Conclusion

Using the ARL EQUINOX 100 it is possible to acquire a full GIXRD scan within 1 min measurement time, which allows to distinguish between different structure types or amorphous / crystalline character of the sample. The alignment of the sample is a straight-forward procedure and requires no advanced operator.

Due to the almost parallel beam generated by the X-ray mirror optics of the ARL EQUINOX 100 even XRR measurements are possible. The evaluation of the measurement allows to determine layer thicknesses with a reasonable precision.

The ARL EQUINOX 100 combined with the Thin Layer attachment is an easy-to-use system for basic thin film / coating investigations in both industrial and academic research. It allows fast and easy QC/QA procedures, even for conventional operators.

Find out more at **www.thermofisher.com/xrd**

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