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THE UNIVERSITY OF ALABAMA IN HUNTSVILLE

Design and Implementation of an X-ray Reflectometer System for Testing X-ray Optic Coatings

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MOTIVATION

Development of Multilayer Coatings for X-ray Optics at MSFC

Applications for high energy astrophysics

- Pushing observations of X-ray sources up to several hundred keV
- Focus on broadband coatings (10 – 200 keV)

How multilayers work – Figure 1:

- Bragg reflection: $n\lambda = 2d_n \sin\theta$
- Constructive interference of reflected X-rays

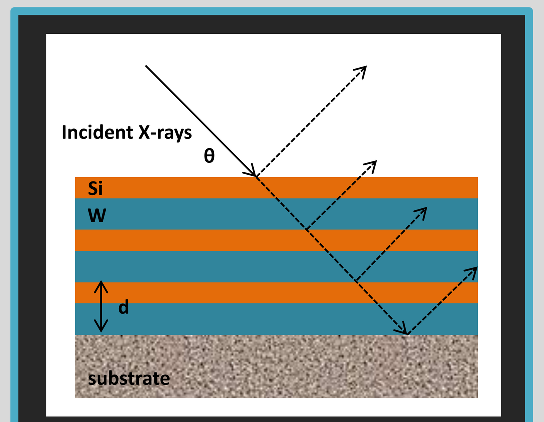


Figure 1. Cartoon representation Si/W multilayer. The X-rays reflected from each bi-layer, denoted by d, add constructively.

In-house Testing of Coatings: the X-ray Reflectometer

X-ray reflectometer: measures coating performance at X-ray energies

Purpose for this work: aid in development of hard X-ray multilayer coatings at MSFC

INTRODUCTION

What is the X-ray Reflectivity (XRR) Measurement?

Determines characteristic properties of X-ray optic coatings

Reflectivity, R, curve: sample's reflectivity response as a function of graze angle where R = reflected X-ray flux/incident X-ray flux

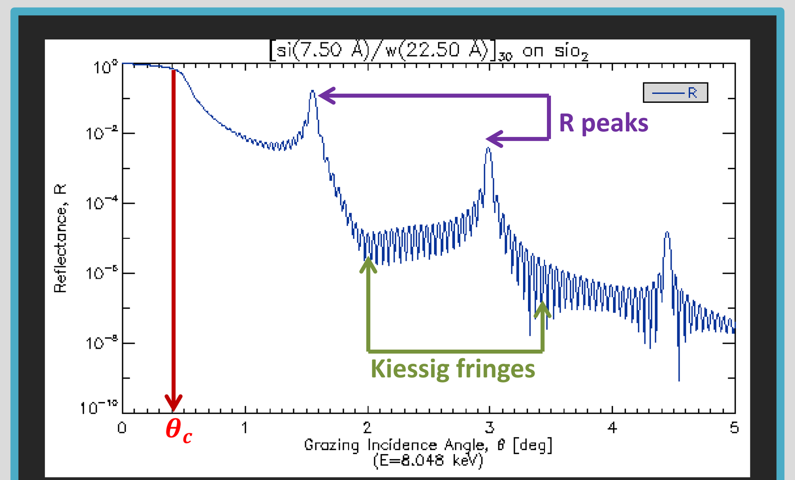


Figure 2. Theoretical R curve for Si/W small d-spacing multilayer. Surface roughness 4Å, interface roughness 4Å [2].

Features of the R curve :

- Critical angle: θ_c
- Kiessig fringes –oscillatory features at graze angles > critical angle, result of interference of reflected X-rays
- Higher order reflectance peaks (multilayer coatings)

Extracting coating properties from the R curve

Film thickness, density, and **interface/surface roughness** values can be extracted from the XRR measurement

A. **Film thickness** - period of oscillations

B. **Film density** - oscillation amplitude

C. **Interface roughness** - oscillation suppression

D. **Surface roughness** - drastic loss of R

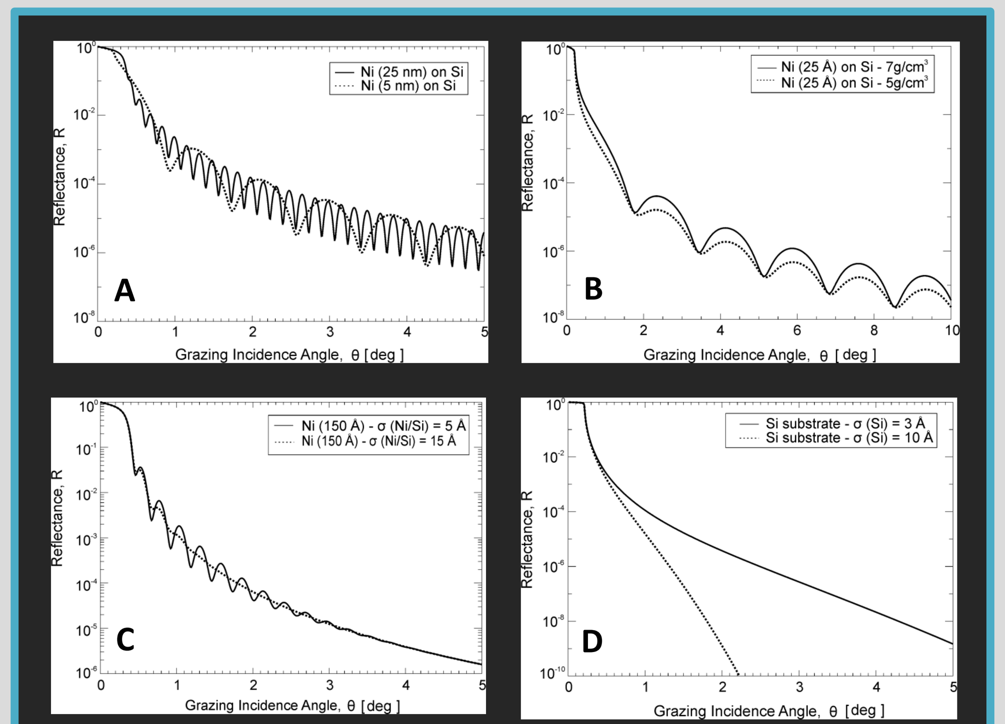


Figure 3. Reflectivity curves of four theoretical samples each with a coating parameter value varied to show the change in reflectivity response [2].

XRR DESIGN AND TESTING

Designing the XRR: Key System Components

X-ray generator: Rigaku RAS

- Cu target, Cu-Kα line: 8.048 keV
- Voltage: 5 – 35 kV, Current: 10-150 mA

X-ray detector: Amptek Fast SDD

- Good throughput at high count rates
- Cu-Kα line resolvable

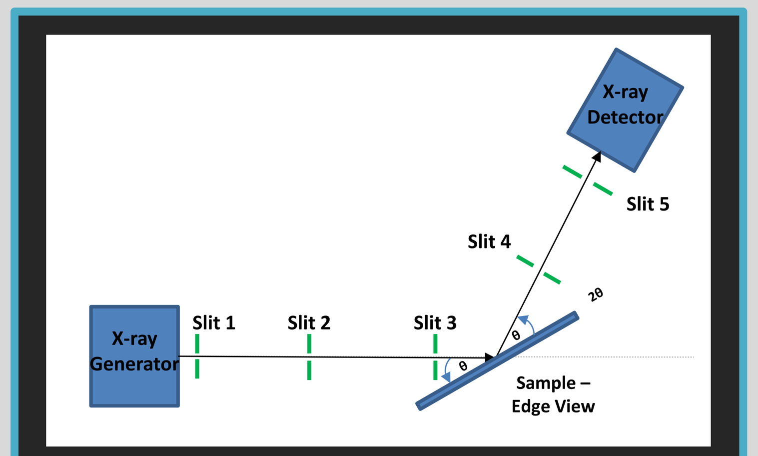


Figure 4. Schematic of XRR system with main components labeled. Note: Not to scale.

Goniometer: 2 rotary stages

- Newport, resolution of 0.001˚
- Moves sample through θ while moving detector through 2θ

Series of beam-defining slits

- Open along same axis
- Minimize projected area of beam on sample and scattered radiation

Sample holder and stages

- Vacuum chuck for sample placement
- Stages for sample motion: 2 linear + 1tipping (Newport), 0.0001mm and 0.001˚ resolution

Custom control software

- Full automation of alignment and data collection routines
- Developed in LabVIEW by Danielle Gurgew

Completion of the XRR System

Alignment of system components: Laser (rough) and X-ray (fine)

Figure 5. Photo of completed XRR system at MSFC's X-ray Cryogenic Facility (XRCF) source building.

How the system operates

1) X-rays produced by generator travel down a beam tube under vacuum in which slits 1 and 2 are mounted
2) X-rays leave vacuum through Be window on end of beam tube and enter region enclosed by radiation shielding
3) X-ray beam further defined by slit 3 just outside Be window
4) Beam incident on sample mounted on vacuum chuck at angle θ
5) X-rays are reflected off of sample and travel through slits 4 and 5 to reduce scattered radiation entering detector
6) Reflected radiation collected by detector at angle 2θ

Verification of the XRR System

X-ray flux variability test – Figure 6

- Monitor X-ray flux as a function of time (2 tests)
- Most variability → counting statistics, other has no significant impact on measurement
- Warm-up period of 60 min before data collection begins

X-ray beam peak position consistency test

- Monitor X-ray beam peak position as a function of time, source current and source voltage
- Small in beam position over 6 hours found to be statistical

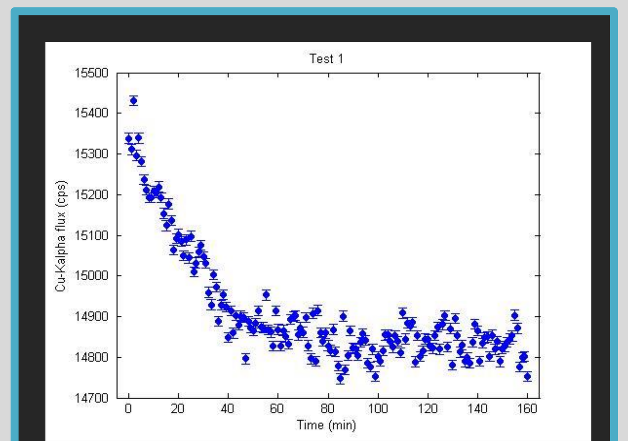


Figure 6. Plot of X-ray flux as a function of time staring just after source is powered up, Test 1. Error bars show counting error as described by Poisson statistics.

INITIAL MEASUREMENT RESULTS

XRR Measurement Repeatability

10 MSFC XRR measurements of both a single layer coating and multilayer coating

Single layer coating: Ir on Si substrate

- Data fit in IMD using genetic algorithm
- Compare best fit layer thickness, surface roughness and film density

Si/W multilayer on SiO2, Si cap layer

- Analysis of critical angle and 2nd order R peak
- Compare 2nd order peak R value, angular position and FWHM

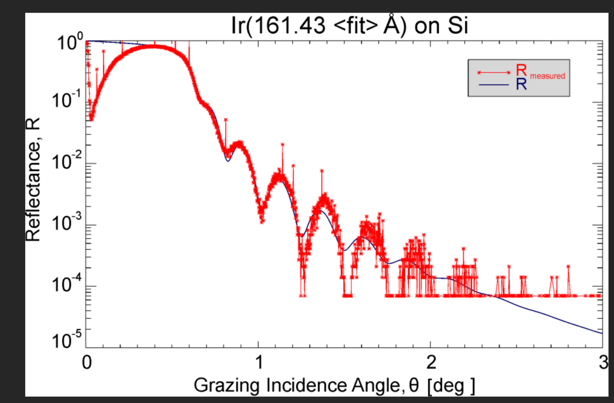


Figure 7. Measured R curve of Ir single film sample (red) fitted with theoretical curve (blue) in IMD [2]. Error bars not shown .

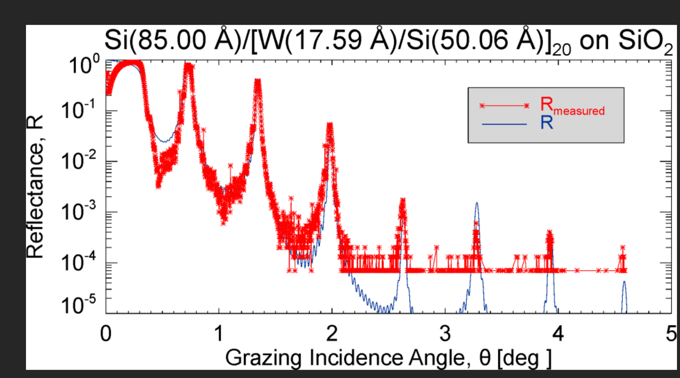


Figure 8. Measured R curve of Si/W multilayer sample (red) fitted with theoretical curve (blue) in IMD [2]. Error bars not shown.

Results:

- No significant variation in repeatability measurements for both samples
- Noise background for both samples: R approx. 10^{-4} → artifact of detector integration time (1s)

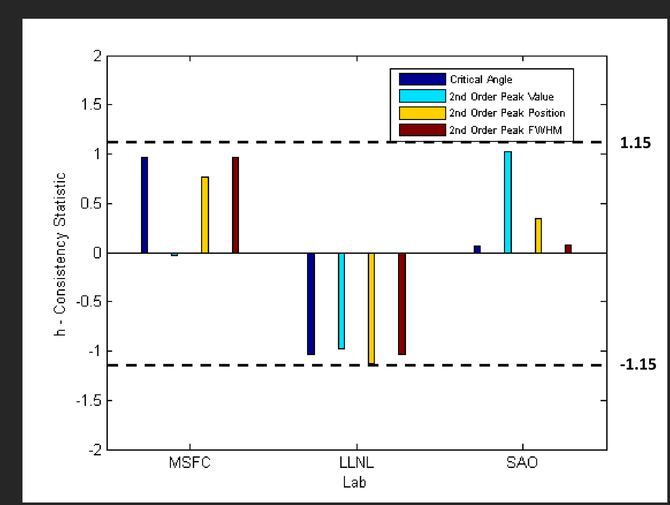
Conclusions:

- In-house XRR measurements consistent and repeatable
- Final verification of system needed

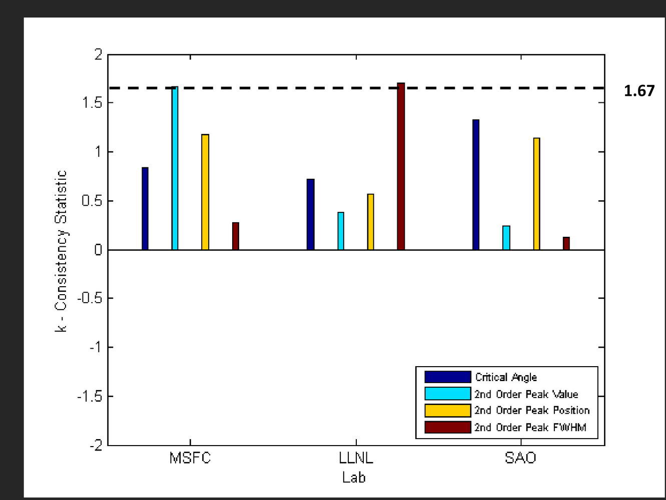
Inter-laboratory Study (ILS)

Comparing MSFC XRR measurements of the Si/W multilayer with XRR measurements made at LLNL and SAO of the sample

- Followed ILS study described in ASTM standard practice E691 - 14



h = between laboratory statistic
($h_{crit} = \pm 1.15$ at 0.5% significance level)



k = within-laboratory statistic
($k_{crit} = 1.67$ at 0.5% significance level)

Majority of measurements from labs in ILS are consistent at 99.5% confidence

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