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

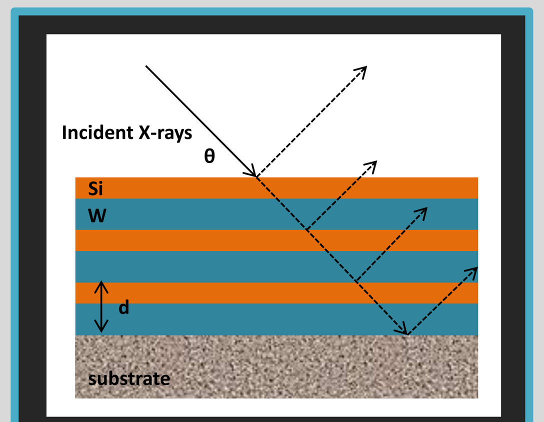


Figure 1. Cartoon representation Si/W multilayer. The X-rays reflected from each bi-layer, denoted by d, add constructively.

➢ 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

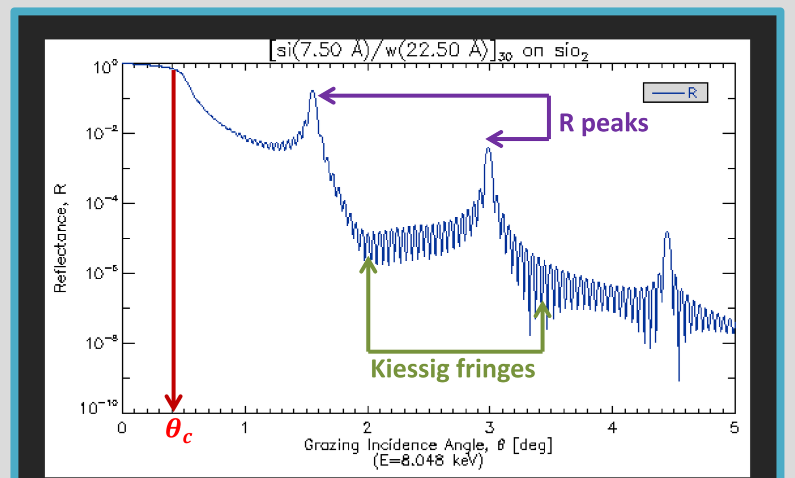


Figure 2. Theoretical R curve for Si/W small d-spacing multilayer. Surface roughness 4Å, interface roughness 4Å [2].

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

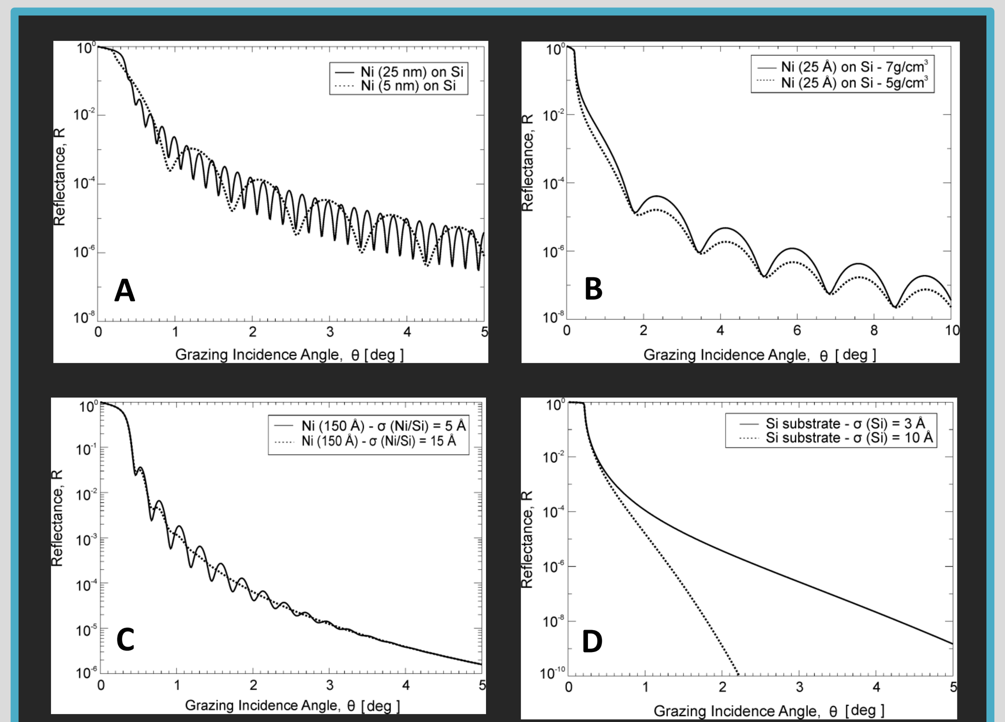


Figure 3. Reflectivity curves of four theoretical samples each with a coating parameter value varied to show the change in reflectivity response [2].

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

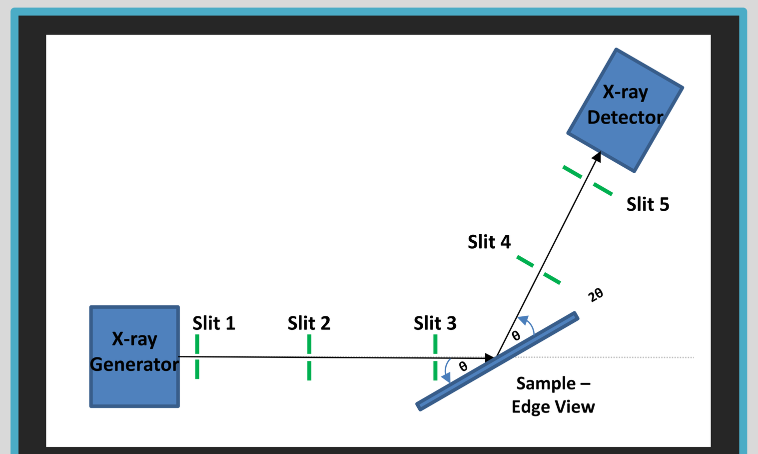


Figure 4. Schematic of XRR system with main components labeled. Note: Not to scale.

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

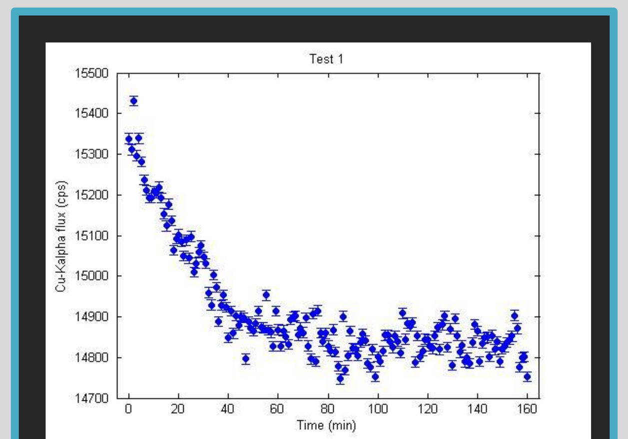


Figure 6. Plot of X-ray flux as a function of time staring just after source is powered up, Test 1. Error bars show counting error as described by Poisson statistics.

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 2[nd] order R peak
- Compare 2[nd] order peak R value, angular position and FWHM

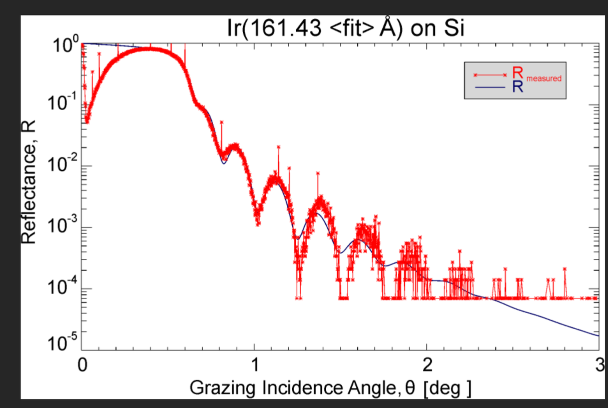


Figure 7. Measured R curve of Ir single film sample (red) fitted with theoretical curve (blue) in IMD [2]. Error bars not shown .

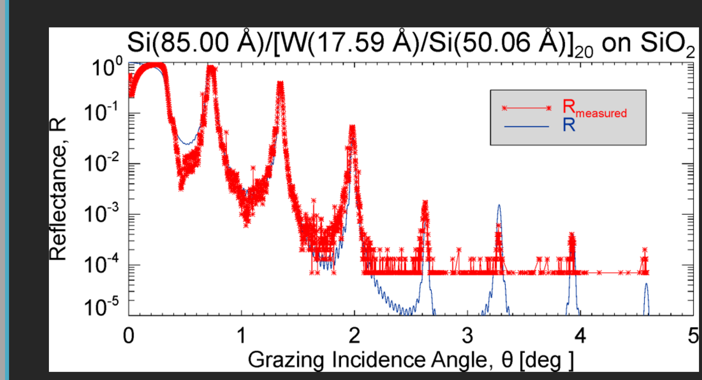


Figure 8. Measured R curve of Si/W multilayer sample (red) fitted with theoretical curve (blue) in IMD [2]. Error bars not shown.

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)

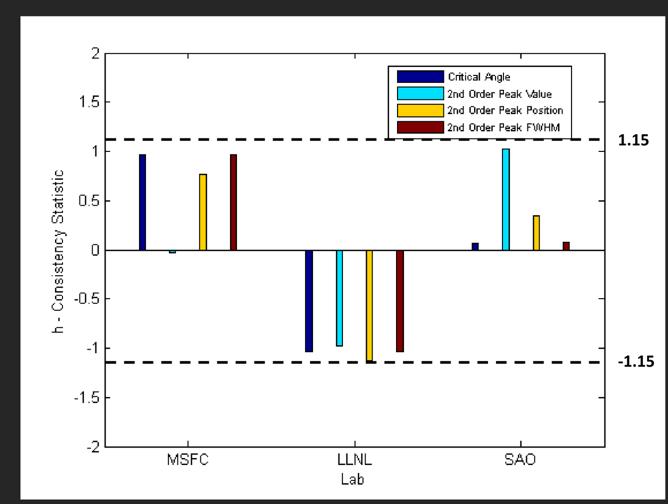
Conclusions:

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

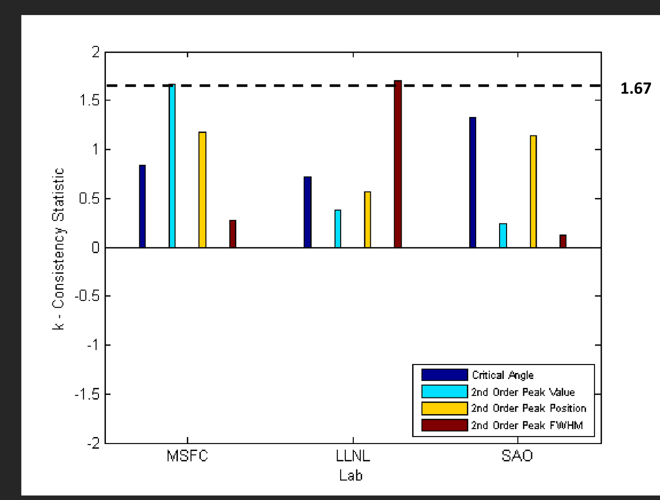
➢ Inter-laboratory Study (ILS)

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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