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Evaluation of thickness, density and interface width of thin films by X-ray reflectometry — Instrumental requirements, alignment and positioning, data collection, data analysis and reporting

Évaluation de l'épaisseur, de la densité et de la largeur de l'interface des films fins par réflectrométrie de rayons X — Exigences instrumentales, alignement et positionnement, rassemblement des données, analyse des données et rapport

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis.*

This second edition cancels and replaces the first edition (ISO 16413:2013), of which it constitutes a minor revision. The changes compared to the previous edition are as follows:

— editorial changes, mainly for a more precise description, e.g. ‘incidence angle’ has been replaced by ‘grazing incidence angle’, ‘intensity’ has been replaced in the appropriate diagrams by ‘reflectivity’ etc.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

X-Ray Reflectometry (XRR) is widely applicable to the measurement of thickness, density and interface width of single layer and multi-layered thin films which have thicknesses between approximately 1 nm and 1 µm, on flat substrates, provided that the layer, equipment and X-ray wavelength are appropriate. Interface width is a general term; it is typically composed of interface or surface roughness and/or density grading across an interface. The specimen needs to be laterally uniform under the footprint of the X-ray beam. In contrast with typical surface chemical analysis methods which provide information of the amount of substance and need conversion to estimate thicknesses, XRR provides thicknesses directly traceable to the unit of length. XRR is very powerful method to measure the thickness of thin film with SI traceability.

The key requirements for equipment suitable for collecting specular X-ray reflectivity data of high quality, and the requirements for specimen alignment and positioning so that useful, accurate measurements may be obtained are described in Clause 3.

The key issues for data collection to obtain specular X-ray reflectivity data of high quality, suitable for data treatment and modelling are described in Clause 4. The collection of the data is traditionally conducted by running single measurements under direct operator data input. However, recently data are often collected by instructing the instrument to operate in multiple runs. In addition to the operator mode, data can be collected making use of automated scripts, when available in the software program controlling the instrument.

The principles for analysing specular XRR data in order to obtain physically meaningful material information about the specimen are described in Clause 5. While specular XRR fitting can be a complex process, it is possible to simplify the implementation for quality assurance applications to the extent where it can be transparent to the user. There are many software packages, both proprietary and non-proprietary available for simulation and fitting of XRR data. It is beyond the scope of this document to describe details of theories and algorithms. Where appropriate, references are given for the interested reader.

The information required when reporting on XRR experiments is listed in Clause 6. A brief review of the possible ways to present XRR data and results is given and, when more than one option is available, the preferred one is indicated.

This document is not a textbook; it is a standard for performing XRR measurements and analysis. For a full explanation of the technique, please consult appropriate references [e.g. D. Keith Bowen and Brian K. Tanner, "X-Ray Metrology in Semiconductor Manufacturing", Taylor and Francis, London (2006); M. Tolan, "X-ray Reflectivity from Soft Matter Thin Films", Springer Tracts in Modern Physics vol. 148 (1999); U. Pietsch, V. Holy and T. Baumbach, "High Resolution X-Ray Scattering from Thin Films to Lateral Nanostructures", Springer (2004); J. Daillant and A. Gibaud, "X-ray and Neutron Reflectivity: Principles and Applications", Springer (2009)].

Safety aspects related to the use of X-ray equipment are not considered in this document. During the measurements, the adherence to relevant safety procedures as imposed by law are the responsibilities of the user.

Evaluation of thickness, density and interface width of thin films by X-ray reflectometry — Instrumental requirements, alignment and positioning, data collection, data analysis and reporting

1 Scope

This document specifies a method for the evaluation of thickness, density and interface width of single layer and multi-layered thin films which have thicknesses between approximately 1 nm and 1 µm, on flat substrates, by means of X-Ray Reflectometry (XRR).

This method uses a monochromatic, collimated beam, scanning either an angle or a scattering vector. Similar considerations apply to the case of a convergent beam with parallel data collection using a distributed detector or to scanning wavelength, but these methods are not described here. While mention is made of diffuse XRR, and the requirements for experiments are similar, this is not covered in the present document.

Measurements may be made on equipment of various configurations, from laboratory instruments to reflectometers at synchrotron radiation beamlines or automated systems used in industry.

Attention should be paid to an eventual instability of the layers over the duration of the data collection, which would cause a reduction in the accuracy of the measurement results. Since XRR, performed at a single wavelength, does not provide chemical information about the layers, attention should be paid to possible contamination or reactions at the specimen surface. The accuracy of results for the outmost layer is strongly influenced by any changes at the surface.

NOTE 1 Proprietary techniques are not described in this document.

2 Normative references

There are no normative references in this document.

3 Terms, definitions, symbols and abbreviated terms

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
grazing incidence angle
ω (omega)
angle between the incident beam and the specimen surface

Note 1 to entry: This angle is sometimes called ‘glancing angle’.

3.2
critical angle
θ_c
angle between the incident beam and the specimen surface, below which there is total external reflection of X-rays, and above which the X-ray beam penetrates below the surface of the specimen

Note 1 to entry: The critical angle for a given specimen material or structure can be found by using simulation software, or approximated from the formula $\theta_c \approx \sqrt{2\delta}$ where $1 - \delta$ is the real part of the complex X-ray refractive index $n = 1 - \delta - i\beta$.

3.3
specimen length
dimension of the specimen in the plane of the incident and reflected X-ray beams and in the plane of the specimen

3.4
specimen width
dimension of the specimen perpendicular to the plane of the incident and reflected X-ray beams and in the plane of the specimen

3.5
specimen height
dimension (thickness) of the specimen perpendicular to the plane of the specimen

3.6
layer thickness
thickness of an individual layer on the substrate

3.7
beam footprint
area on the specimen irradiated by the X-ray

3.8
beam spill-off
effect of grazing incidence that involves the reduction of the measured reflected intensity when part of the incident beam is not intercepted by the specimen, so that the part spills off the specimen

3.9
instrument function
analytical function describing the effects of instrument and resolution on the observed scattered X-ray intensity

3.10
reciprocal space
representation of the physical specimen and X-rays where the distance plotted is proportional to the inverse of real-space distances, and angles correspond to real-space angles

3.11
wave vector
k
vector in reciprocal space describing the incident or scattered X-ray beams

3.12
scattering vector
q
vector in reciprocal space giving the difference between the scattered and incident wave vectors

3.13
dispersion plane
plane containing the source, detector, incident and specularly reflected X-ray beams

3.14
specular X-ray reflectivity
reflected X-ray signal detected at an angle with the specimen surface as the incident X-ray beam with the specimen surface: $2\theta/2 = \omega$

Note 1 to entry: The detected, scattered X-ray intensity is measured as a function of either ω or 2θ or q_z (usually presented against q_z or i).

3.15
diffuse X-ray reflectivity
X-ray scatter arising from the imperfection of the specimen

3.16
fringe
one of the repeating maxima in reflectometry data which arise from interference of the X-ray waves

Note 1 to entry: Fringe periods are related to the thickness of a layer (or layers) of contrasting electron density. Multiple layers give rise to series of superposed interfering fringes.

3.17
fringe contrast
qualitative description of the height of a *fringe* (3.16) between its minimum and its maximum

Note 1 to entry: The greater the difference between minimum and maximum, the greater the contrast is said to be.

3.18
electron density
ρ_e
electrons per unit volume

Note 1 to entry: XRR typically measures electron density in electrons per nm^3 or per $Å^3$.

Note 2 to entry: This can be calculated from mass density.

3.19
mass density
ρ
common density (mass per unit volume)

Note 1 to entry: The unit of the mass density is kg m^{-3} (or g cm^{-3}).

3.20
absorption length
L_{abs}
distance over which the transmitted intensity falls to 1/e of the incident intensity

3.1 Symbols and abbreviated terms

2θ	2Theta, the angle of the detected X-ray beam with respect to the incident X-ray beam
ω	Omega, the angle between the incident X-ray beam and the specimen surface
ϕ	Phi, the angle of rotation about the normal to the nominal surface of the specimen
χ	Chi, the angle of tilt of specimen about an axis in the plane of the specimen and in the plane of the incident X-ray beam, X-ray source and detector
θ_c	Critical angle
λ	Wavelength of the incident X-ray beam

ρ	Mass density
ρ_e	Electron density
$\boldsymbol{k}$	Wave vector
$\boldsymbol{q}$	Scattering vector
q_z	Scalar magnitude of the component of the scattering vector in reciprocal space normal to the specimen surface (corrected or uncorrected for refraction). $q_z = 4\pi/\lambda \times \sin(\theta)$
σ	root mean square height of the scale-limited surface (according to ISO 25178-2) or interface width
L_{abs}	Absorption length in the specimen
XRR	X-Ray Reflectometry or X-Ray Reflectivity
Z	specimen height

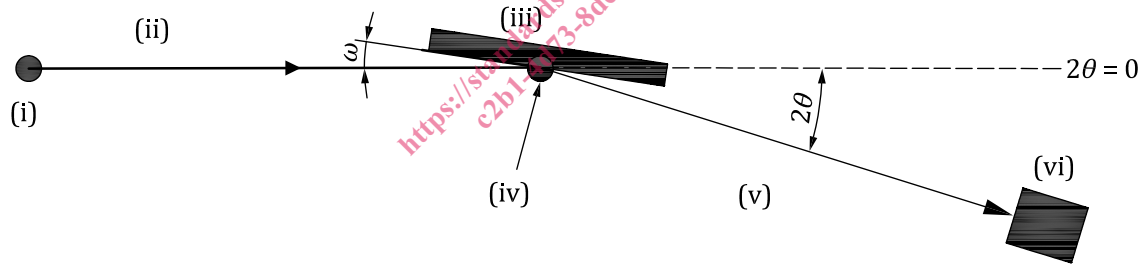
4 Instrumental requirements, alignment and positioning guidelines

4.1 Instrumental requirements for the scanning method

4.1.1 Schematic diagrams

The principal requirements are on the beam size and beam positioning over the coaxial centres of rotation of specimen (ω) and detector (2θ) axes.

Figure 1 shows a diagram of a basic collimated beam, scanning configuration for an XRR experiment. The case of a convergent beam and distributed detector is not shown.



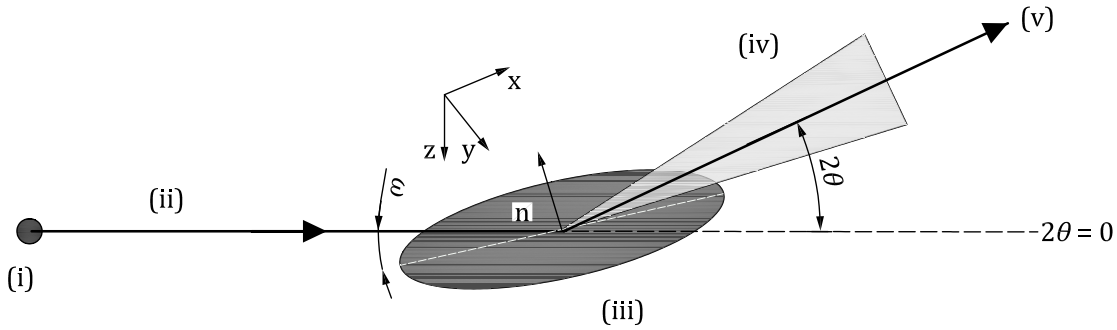
Key

ω	angle between the specimen surface and the incident X-ray beam
2θ	angle between the detected beam and $2\theta = 0$ (the extension of the incident X-ray beam)
(i)	X-ray source
(ii)	collimated incident x-ray beam
(iii)	specimen
(iv)	centre of rotation
(v)	reflected x-ray beam
(vi)	detection system

NOTE The centre of rotation, where incident and reflected beams, the specimen surface and the rotation axes of ω and 2θ coincide, is highlighted as grey disc.

Figure 1 — Schematic layout of a typical scanning XRR experimental configuration, projected into the plane of the source, detector, incident and specularly-reflected X-ray beams (the dispersion plane)

Figure 2 shows a schematic diagram of scanning configuration XRR in a three-dimensional view, indicating the diffuse scatter as well as the specularly reflected X-ray beam.



Key

ω angle between the specimen surface and the incident X-ray beam

2θ angle between the detected beam (at 2θ = 0) and whichever part of the reflected beam is of interest (the detected beam)

(i) X-ray source

(ii) collimated incident x-ray beam

(iii) specimen

(iv) diffusely scattered x-rays

(v) specularly reflected x-ray beam

Figure 2 — Schematic diagram showing specular and diffusely reflected X-ray beams

4.1.2 Incident beam — Requirements and recommendations

4.1.2.1 Incident beam — Requirements

The following requirements shall apply to the collimated beam, scanning method. Similar considerations apply to the convergent beam, parallel data collection method.

a) The incident beam shall be stable (or can be compensated) within the time-frame of the experiment.

b) The incident beam shall be nominally monochromatic. The wavelength dispersion dλ shall fulfil the following condition: dλ < λdθ/tan(θ_m) where dθ is the beam divergence and θ_m is typically the maximum incidence angle where fringes are still observed.

EXAMPLE If using an incident beam of Cu Kα radiation (λ = 0,154 1 nm) with an angular divergence of 50 arc seconds, and if fringes are to be observed out to an incident angle of 3,5°, then dλ needs to be less than 0,035 nm.

c) If the beam is not sufficiently collimated, the divergence of the beam limits the maximum detectable thickness. Practically, the maximum measurable thickness is less than λ/6sin(dθ) where dθ is the beam divergence for a suitable specimen. For typical laboratory equipment, the limit is a few hundred nm.

d) The incident intensity shall be such as to allow several orders of magnitude intensity range above background, since reflected intensity falls rapidly above the critical angle. Below the critical angle, there is total external reflection. Above the critical angle, reflected intensity falls at a rate proportional to q_z^{-4} for a perfectly smooth surface, and more rapidly than this for rough or/and graded surfaces.

4.1.2.2 Incident beam — Recommendations

The following recommendations concern the collimated beam, scanning method. Similar considerations concern the convergent beam, parallel data collection method.

a) The specimen should be laterally uniform under the area irradiated (the beam footprint) and observed by the detector. This may be achieved by control of incident and scattered beam slits and/or, for example, inserting a knife-edge near the specimen.

b) Beam spill-off should be minimized. This is especially important when the specimen angle is near and above the critical angle. The beam width compared to the specimen length should be such that there is no beam spill-off for a specimen angle which is above about 75 % (preferably less) of the critical angle. (See Figure 3.)

NOTE With the specimen parallel to the beam (ω = 0), the beam covers all of the specimen. The beam footprint varies with incident angle unless slits or knife-edge position are varied through the scan).

1) The maximum acceptable beam width for a given specimen size can then be found by geometry.

2) If there are very small specimens, it may not be practical to meet the recommended requirements. In this case, the accuracy and precision of densities and interface widths deduced may be compromised.

3) This is necessary so that the position of the critical angle can be ascertained with reasonable confidence, so that, if data analysis includes layer density and interface width parameters, these can be deduced with reasonable accuracy.

4) Some modelling and data fitting software allow the specimen size and beam size to be input, which allows data fitting where there is significant beam spill-off, but even so it is recommended that the specimen fill the incident beam from below the critical angle in order to have high confidence in fitting this region and obtaining good density information.