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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

Reference number
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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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 4.

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 5. 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 6. 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 7. 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 and definitions

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 Terms and definitions

3.1.1
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’.