INTERNATIONAL STANDARD

ISO 16413

First edition
2013-02-15

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
ISO 16413:2013(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 16413 was prepared by Technical Committee ISO/TC 201, *Surface chemical analysis*.

Introduction

X-Ray Reflectometry (XRR) is widely applicable to the measurement of thickness, density and interface width of single layer and multilayered 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)].

Note that proprietary techniques are not described in this International Standard.

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 International Standard specifies a method for the evaluation of thickness, density and interface width of single layer and multilayered 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.

2 Terms, definitions, symbols and abbreviated terms

2.1 Terms and definitions

2.1.1
incidence angle
angle betwen the incident beam and the specimen surface

2.1.2
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 $n = 1 - \delta - i\beta$.

2.1.3
specimen length
dimension of the specimen in the plane of the incident and reflected X-ray beams and in the plane of the specimen

2.1.4
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