INTERNATIONAL STANDARD



First edition 2017-11

F Mi eJ c Microbeam analysis — Analytical electron microscopy — Method for the determination of interface position in the cross-sectional image of the **Mayered materials**

sicro, de déter, ansversale. Analyse par microfaisceaux — Microscopie électronique analytique - Méthode de détermination de la position d'interface dans l'image de coupe transversale des matériaux en couches



Reference number ISO 20263:2017(E)



© ISO 2017, Published in Switzerland

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office Ch. de Blandonnet 8 • CP 401 CH-1214 Vernier, Geneva, Switzerland Tel. +41 22 749 01 11 Fax +41 22 749 09 47 copyright@iso.org www.iso.org

Page

Contents

Fore	word		iv
Intro	oductio	n	v
1	Scop	e	
2	Norr	native references	
3	Tern 3.1 3.2	ns, definitions and abbreviated terms Terms and definitions Abbreviated terms	1 1 .4
4	Spec 4.1 4.2	imen preparation for cross-sectional imaging General Requirements for the cross-sectional specimen	4 4 5
5	Determination of an interface position		6
	5.1 5.2	General Preliminary considerations 5.2.1 Ideal model of an interface 5.2.2 More realistic model of an interface 5.2.3 Dealing with intensity fluctuations in the image	
6	Detailed procedure for determining the position of the interface		
	6.1 6.2	General Preparing cross-sectional TEM/STEM image 6.2.1 Preparing digitized Image 6.2.2 Displaying the digitized image	
	6.3	Setting the ROI 6.3.1 General 6.3.2 Classification of image	11 11 11 11
	6.4 6.5 6.6 6.7	Acquisition of the averaged intensity profile Moving-averaged processing Differential processing Final location of the interface	12 17 19 20 21
7	Unce	ertainty	22
	7.1 7.2	Uncertainty accumulating from each step of the procedure Uncertainty of measurement result on image analysis	
Anne	ex A (in imag	formative) Examples of processing the real TEM/STEM images for three ge types	
Anne	ex B (in	formative) Two main applications for this method	36
Anne	x C (in	formative) Calibration of scale unit: Pixel size calibration	43
Bibli	ograph	ly	

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 <u>www.iso.org/directives</u>).

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 <u>www.iso.org/patents</u>).

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

For an explanation on 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 the following URL: https://www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 202, Microbeam analysis, Subcommittee SC 3, Analytical electron microscopy.

SO/TC 2L.

Introduction

Multi-layered materials are widely used in the production of semiconductor devices, various kinds of sensors, coating films for optical element, new functional materials, etc. One of the factors used to determine the characteristics of multi-layered materials is the layer thickness, for evaluation of products and verification of the production process. In practice, measuring the total thickness and/or the thickness of each layer and checking the uniformity of thickness and/or flatness of the interface are often done using recorded images of the materials. Evaluations can be made from the cross-sectional TEM/STEM images by accurately determining the averaged interface position between two different layered materials.

In relation to the determination of the interface position in the HR atomic imaging, analysis by the multi-slice simulation (MSS) method can be applied for the target measurement, if the atomic structural models can be constructed. However, in real materials, there are a lot of cases when they cannot, as follows:

- the interface between amorphous layers, or layers of amorphous substance and crystal;
- the interface recorded in low-resolution image in which the atomic columns cannot be identified: 1) very thick single-layered material, 2) thick multi-layered material.

This document relates the method to determine the averaged interface position, using a differential processing of the accumulated intensity profile getting from the ROI set in the cross-sectional TEM/STEM image of the multi-layered materials. The thickness of the layer that can be applied ranges from a few nanometers to a few micrometers. Thus, this document is not intended for the determination of the simulated position of the layer interface analysed by the MSS method.

n Phu. ze analy.

this document is a preview demendence of the document is a preview demendence of the document of the document

Microbeam analysis — Analytical electron microscopy — Method for the determination of interface position in the cross-sectional image of the layered materials

1 Scope

This document specifies a procedure for the determination of averaged interface position between two different layered materials recorded in the cross-sectional image of the multi-layered materials. It is not intended to determine the simulated interface of the multi-layered materials expected through the multi-slice simulation (MSS) method. This document is applicable to the cross-sectional images of the multi-layered materials recorded by using a transmission electron microscope (TEM) or a scanning transmission electron microscope (STEM) and the cross-sectional elemental mapping images by using an energy dispersive X-ray spectrometer (EDS) or an electron energy loss spectrometer (EELS). This document is also applicable to the digitized image recorded on an image sensor built into a digital camera, a digital memory set in the PC or an imaging plate and the digitalized image converted from an analogue image recorded on the photographic film by an image scanner.

2 Normative references

There are no normative references in this document.

3 Terms, definitions and abbreviated terms

3.1 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/

3.1.1

atomic column image

TEM/STEM image recorded at atomic-resolution from a specimen along a high-symmetry crystalline orientation

Note 1 to entry: Crystalline orientation is the direction of crystal which is represented by Miller indices. During TEM imaging, it is often useful to have a crystalline specimen aligned so that a specific (low index) *zone axis* (3.1.26) is parallel, or near parallel, to the beam direction (optical axis).

3.1.2 cross-sectional image

TEM/STEM image of the multi-layered materials along a plane perpendicular to the stacking direction

3.1.3

differential processing

calculation of the difference between the values of adjacent pixel data in the intensity profile