INTERNATIONAL STANDARD

ISO 11938

First edition
2012-03-01

Microbeam analysis — Electron probe microanalysis — Methods for elemental-mapping analysis using wavelength-dispersive spectroscopy

Analyse par microfaisceaux — Analyse par microsonde électronique (microsonde de Castaing) — Méthodes d'analyse par cartographie élémentaire en utilisant la spectrométrie à dispersion de longueur d'onde

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 11938:2012
https://standards.iteh.ai/catalog/standards/sist/a8ea02b0-5e77-4974-9ad0-741190f6aa65/iso-11938-2012

Reference number
ISO 11938:2012(E)

© ISO 2012

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 11938:2012
https://standards.iteh.ai/catalog/standards/sist/a8ea02b0-5e77-4974-9ad0-741190f6aa65/iso-11938-2012

COPYRIGHT PROTECTED DOCUMENT

© ISO 2012

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

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 ... iv
Introduction ... v
1 Scope ... 1
2 Normative references ... 1
3 Terms and definitions ... 1
4 Procedure of mapping analysis ... 2
4.1 General ... 2
4.2 Specimen preparation ... 2
4.3 Measurement procedure ... 3
5 Methods for displaying element maps ... 6
5.1 General ... 6
5.2 Raw X-ray intensity method ... 6
5.3 k-value method ... 6
5.4 Calibration curve method ... 6
5.5 Correlation method ... 7
5.6 Matrix correction method ... 7
6 Evaluation of uncertainty ... 7
7 Report ... 7
Annex A (normative) Comparison of absorption effects for a light element ... 9
Bibliography ... 10

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 11938:2012
https://standards.iteh.ai/catalog/standards/sist/a8ea02b0-5e77-4974-9ad0-741190f6aa65/iso-11938-2012

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 11938 was prepared by Technical Committee ISO/TC 202, *Microbeam analysis*, Subcommittee SC 2, *Electron probe microanalysis*.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 11938:2012
https://standards.iteh.ai/catalog/standards/sist/a8ea02b0-5e77-4974-9ad0-741190f6aa65/iso-11938-2012

 © ISO 2012 – All rights reserved

Introduction

Electron probe microanalysis (EPMA) has been developed over the last 50 years[1][2][3][4] and has many areas of application in science and industry. Both qualitative and accurate quantitative analyses are employed extensively in mineralogy and in metallurgical studies, for example. In recent years, with the advances in computers, digital processing techniques have been developed and, instead of X-ray dot images being used to qualitatively observe element distributions, colour mapping techniques[5] are now often employed. These enable products to be compared and evaluated for the purpose of quality control. Particle analysis and/or phase analysis using mapping requires careful selection of the experimental parameters, and it is essential that a standard be available for this purpose in order to achieve consistent and reliable results.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 11938:2012
https://standards.iteh.ai/catalog/standards/sist/a8ea02b0-5e77-4974-9ad0-741190f6aa65/iso-11938-2012

© ISO 2012 – All rights reserved

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 11938:2012
https://standards.iteh.ai/catalog/standards/sist/a8ea02b0-5e77-4974-9ad0-741190f6aa65/iso-11938-2012

Microbeam analysis — Electron probe microanalysis — Methods for elemental-mapping analysis using wavelength-dispersive spectroscopy

1 Scope

This International Standard provides procedures for electron microprobe elemental-mapping analysis using wavelength-dispersive spectrometry. The choice between mapping with the electron beam moving digitally across the specimen (electron beam mapping) and mapping with stage movement only (large-area mapping) is assessed. It describes five types of data processing: the raw X-ray intensity data method, the k-value method, the calibration method, the correlation method and the matrix correction method.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 5725-6, *Accuracy (trueness and precision) of measurement methods and results — Part 6: Use in practice of accuracy values*

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 14594, *Microbeam analysis — Electron probe microanalysis — Guidelines for the determination of experimental parameters for wavelength dispersive spectroscopy*

ISO 11938:2012
https://standards.iteh.ai/catalog/standards/sist/a8ea02b0-5e77-4974-9ad0-741190f6aa65/iso-11938-2012

ISO 16592:2006, *Microbeam analysis — Electron probe microanalysis — Guidelines for determining the carbon content of steels using a calibration curve method*

ISO/IEC 17025:2005, *General requirements for the competence of testing and calibration laboratories*

ISO 17470, *Microbeam analysis — Electron probe microanalysis — Guidelines for qualitative point analysis by wavelength dispersive X-ray spectrometry*

ISO 22489, *Microbeam analysis — Electron probe microanalysis — Quantitative point analysis for bulk specimens using wavelength-dispersive X-ray spectroscopy*

ISO 23833, *Microbeam analysis — Electron probe microanalysis (EPMA) — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 23833 and the following apply.

3.1
mapping area
orthogonal array of equally spaced pixels in the X and Y directions that define the region of the specimen being mapped

NOTE Each pixel is analysed for the same time period, and the integrity of the data from each pixel is maintained in the stored data such that the graphical display of the data exhibits the distribution for each of the elements analysed.

3.2
beam scanning
time-controlled movement of the electron beam on the specimen surface with the synchronized movement on the display screen

© ISO 2012 – All rights reserved

3.3
pseudo-colour map
false-colour map
grey-level map
element concentration display using different grey levels or colours, where each pixel grey level or colour value represents the magnitude of the measured element intensity at that pixel position

3.4
pixel
single data point in a map

3.5
stage mapping
production of an X-ray map by mechanically moving the stage under a stationary electron beam in a predefined orthogonal-array pattern

4 Procedure of mapping analysis

4.1 General

The experimental parameters to be used during the analysis should be selected using the guidelines detailed in ISO 14594.

In order to carry out mapping analysis, the analysing instrument shall be sufficiently stable for any variation due to instrument drift over the total mapping period to be reasonably expected to be significantly less than the variation due to differences in the measured element intensities. The instrument's stability will be defined from a test of the instrument's performance. The stability should be measured over a time period similar to that used for the mapping analysis.

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 11938:2012
https://standards.iteh.ai/catalog/standards/sist/a8ea0260-5e77-4974-9ad0-741190f6aa65/iso-11938-2012

NOTE 1 An Si wafer is sufficiently homogeneous and suitable to check the stability.

NOTE 2 There are two options for carrying out the background measurement. The first method is to measure both the peak and the background intensities pixel by pixel. This minimizes the effects of stage and/or beam energy drift, but requires the spectrometer to move between the peak and background positions at every pixel, significantly increasing analysis time and introducing potential drift in the spectrometer positioning. The second method is to acquire a complete peak map and a complete background map. This significantly reduces the total analysis time and removes the risk of spectrometer drift, but is more prone to stage and/or beam-energy drift.

4.2 Specimen preparation

The specimen shall be prepared so as to minimize the effects of artefacts and errors on the mapping analysis, but without destroying the integrity of the specimen (see ISO 22489).

The specimens (reference specimen and unknown specimen) shall be clean and free of dust.

The specimen shall be as flat as possible. If necessary, the specimen shall be embedded in a mounting medium and metallographically polished.

The specimen shall have good electrical conductivity. Charging under electron beam irradiation can be avoided by coating the specimen with a conductive layer of a suitable material. The coating shall be as thin as possible whilst still providing sufficient charge dissipation. A conducting path shall be established between the specimen surface and the metallic specimen holder. Carbon coating is generally used but, in particular cases, other materials should be considered (Au, Al, etc.). Carbon to a thickness of around 10 nm to 20 nm is usually sufficient to establish good conduction. It is recommended that both the reference material and unknown specimen be coated with the same element at the same thickness.

 © ISO 2012 – All rights reserved

4.3 Measurement procedure

4.3.1 General

Mapping analysis shall be performed as follows:

a) Select the location and size of the required map area on the specimen.

b) Select the number of pixels to meet the required spatial resolution, and the method of acquisition (stage or beam mapping).

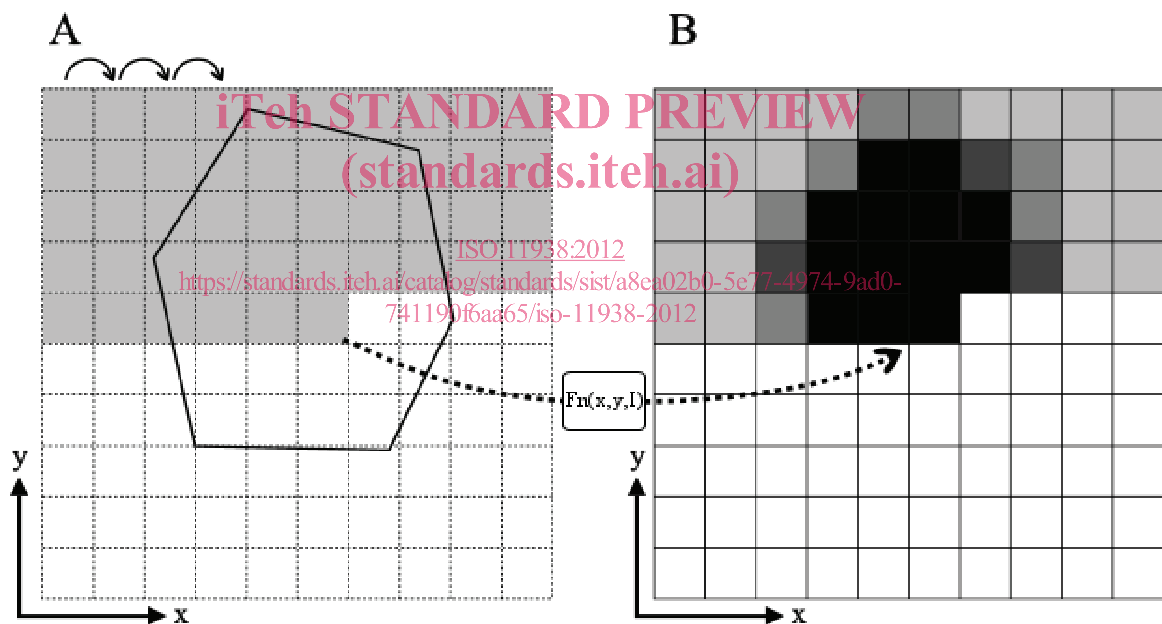
c) Select and apply the element and instrument conditions.

d) Collect characteristic peak and background X-rays and any required electron signals (e.g. backscatter, secondary electrons, absorbed current) at each pixel, and store all data in the computer memory point by point[1].

e) Apply the chosen correction method to the X-ray data (see Clause 5).

f) Finally, the data can be displayed as pseudo-colour maps, as shown in Figure 1.

g) The mapping procedure is summarized in Figure 2.



iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO 11938:2012
https://standards.iteh.ai/catalog/standards/sist/a8ea02b0-5e77-4974-9ad0-741190f6aa65/iso-11938-2012

Figure 1 — Procedure for converting mapping data into pseudo-colour maps

The mapping area and resolution are selected on the specimen (grid A) and the mapping is acquired by electron beam movement or stage movement pixel by pixel (solid arrows above grid A). At each pixel position, x,y, the measured X-ray intensity, I, is converted via the chosen correction method, Fn(x,y,I), into a false-colour level at the correlating pixel position in the result map (grid B).

© ISO 2012 – All rights reserved