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# Microbeam analysis — Electron probe microanalysis — Quantitative analysis of Mn dendritic segregation in continuously cast steel product

*Analyse par microfaisceaux — Analyse par microsonde de Castaing — Analyse quantitative de la ségrégation dendritique du manganèse dans un produit en acier coulé en continu*



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# Foreword

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

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

Electron probe X-ray microanalysis (EPMA) is a modern technique used to qualitatively determine and quantitatively measure the elemental composition of solid materials, including metal alloys, ceramics, glasses, minerals, polymers, powders, etc., on a spatial scale of approximately one micrometer laterally and in depth. EPMA is based on the physical mechanism of electron-stimulated X-ray emission and X-ray spectrometry (see ISO 23833).

The dendritic segregation rich in Mn is unavoidable during steel solidification, which is the direct cause of banded structure in steel product formed in the hot rolling process. The toughness and strength of steel are greatly affected by the banded structure. The quantification of the dendritic segregation can facilitate steel maker to optimize steel-making process parameters, and therefore control the banded structure in the final product.

It is a new method to quantify the dendritic segregation through EPMA by measuring inhomogeneity of Mn with EPMA. In order to obtain comparative and repeatable results it is necessary to standardize this method. These enable products to be compared and evaluated for the purpose of quality control.

# Microbeam analysis — Electron probe microanalysis — Quantitative analysis of Mn dendritic segregation in continuously cast steel product

## 1 Scope

This document specifies procedures for quantitative analysis of Mn dendritic segregation in steel billets, blooms, slabs using electron probe microanalysis (EPMA).

This document is mainly applicable to continuously cast products with Mn content more than 0,01 % by mass. It can also be used for steel ingots and steel products, such as cast iron and cast steel.

The minimum size of analysable dendrites is totally dependent on the resolution of microscope of EPMA and beam size of filament used for quantitative analysis.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 11938, *Microbeam analysis — Electron probe microanalysis — Methods for elemental-mapping analysis using wavelength-dispersive spectroscopy*

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

ISO 14595, *Microbeam analysis — Electron probe microanalysis — Guidelines for the specification of certified reference materials (CRMs)*

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.

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

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

— IEC Electropedia: available at www.electropedia.org

**3.1**
**dendritic segregation**
phenomenon that element content differs in the first-crystallized dendrite arms and post-crystallized inter-dendritic spaces during alloy solidification in a dendritic pattern

**3.2**
**mapping analysis**
method of analysing element in EPMA, through which element distribution in an area can be obtained

**3.3**
**line analysis**
method of analysing element in EPMA, through which element distribution in a specified direction can be obtained

**3.4**
**ratio of dendritic segregation**
**SR**
ratio of the maximum content in the inter-dendritic spaces and the minimum content in the dendrite arms of the selected element in the dendritic zone of a specimen

## 4 Principle

Mapping analysis or line analysis is firstly performed to display the dendritic segregation of Mn element in a certain area of steel billet. The map or line of X-ray counts can thus be obtained from the above analysis. The calibration curve generated from a series of Mn reference materials is then used to quantify the map or line to convert X-ray counts to concentration of Mn. All concentration values are exported to precede further statistical analysis. Finally, the ratio of dendritic segregation of Mn is calculated.

## 5 Selection of reference material

The reference materials selected for establishing the calibration curve shall meet the specifications of ISO 14595 and ISO 11938. A series of reference materials with the same matrix as the specimen and encompassing the range of compositions in the specimen is used to generate a correction curve. Example of reference material is as follows: Fe-Mn alloy, which contain 1,8g/kg, 2,5 g/kg, 3,2 g/kg, 5,3 g/kg, 8,5 g/kg, 17,6 g/kg, 23,4 g/kg, 34,5 g/kg Mn and residual Fe, respectively.



## 6 Sampling and specimen preparation

### 6.1 Sampling



If the order, or the International Standard defining the product, does not specify the number of specimens and the point at which they are to be taken from the product, these are left to the manufacturer. It is recommended that two or more sections be assessed. Care shall be taken to ensure that the specimens are representative of the bulk of the product.

### 6.2 Specimen preparation

Unless otherwise stated by the product standard or by agreement with the customer, the tested plane of the specimen shall be transversal.

The specimens should be prepared using a well-conceived method, starting with sectioning with a device that imparts minimal damage, followed by an appropriate sequence of grinding and polishing steps, finishing with an abrasive of at least 1 µm, to yield a flat surface with minimal preparation-induced damage. After polishing, the specimen should be carefully washed with water, cleaned with alcohol and finally dried using either clean compressed air or a flow of hot air from a device similar to a hair dryer or a hand dryer.

If necessary, the specimen shall be embedded in a conducting medium and metallographically polished. In case of the use of non-conducting mounting medium, a conductive path must be established from the specimen to the ground to prevent charging.

# 7 Calibration of the instrument

The instrument shall be calibrated at regular intervals. The stability of beam current, magnification, specimen stage and X-ray intensity shall be periodically checked according to ISO 11938. The stability of the instrument should be measured over a time period similar to that used for the mapping analysis.

# 8 Procedure

## 8.1 Prepare the instrument

Prepare the instrument to be ready for quantitative analysis according to ISO 14594 and ISO 22489,

## 8.2 Set up the experiment parameters

Select the location and size of the required mapping or line area on the specimen. Choose Mn as analysis element. The experimental parameters shall be set in accordance with ISO 14594. The recommended parameters for mapping and line analysis are listed in Table 1 and Table 2, respectively.

**Table 1 — Recommended test conditions for quantitative mapping analysis**

| Item | Test conditions |
|---|---|
| WDS crystal | LIF |
| Characteristic X-Ray | $K_\alpha$ line |
| Scanning method | Stage scanning |
| Accelerating voltage | 15 kV~20 kV |
| Beam current | 100 nA~300 nA |
| Beam size | 1 μm~10 μm |
| Pixel size | Equal to beam size |
| Dwell time | 5 ms~5 s |
| Number of pixels | More than 10 000 |
| Area | More than 1 $mm^2$, including more than or equal to 10 dendrites |

**Table 2 — Recommended test conditions for quantitative line analysis**

| Item | Test conditions |
|---|---|
| WDS crystal | LIF |
| Characteristic X-Ray | $K_\alpha$ line |
| Scanning method | stage scanning |
| Accelerating voltage | 15 kV~20 kV |
| Beam current | 100 nA~300 nA |
| Beam size | 1 μm~10 μm |
| Pixel size | equal to beam size |
| Dwell time | 0,1~5 s |
| Pixel number | more than 1 000 |
| Line length | more than 1 mm |
| Line number | more than 6 |
| Line distribution | more than or equal to 3 lines parallel to X direction, evenly distributed in a more than 1 $mm^2$ area; more than or equal to 3 lines parallel to Y direction evenly distributed in a more than 1 $mm^2$ area |

## 8.3 Perform the analysis

Perform the mapping or line analysis of Mn and collect X-ray intensity data. The mapping analysis shall be in accordance with ISO 11938.

Use the reference materials to establish Mn calibration curve according to ISO 22489. Then use the calibration curve to convert the measured raw data into quantitative concentration values. Export all the Mn concentration data and save as an MS-Excel file[1)]. Import the data to Matlab or other software to proceed further statistical analysis.



## 8.4 Process the data

Data processing shall be performed as follows:

a) Delete zero and negative numbers.

b) Remove the abnormal data produced by MnS inclusions.

c) Sort all the Mn concentration data in an increased order. The data with the same Mn concentration value shall be arrayed in succession. Each Mn concentration data has a sequence number, which ranges from 1 to N. N is the number of pixels of mapping or line analysis. The cumulative frequency X can be obtained by the sequence number of the data divided by N. The variation curve of Mn concentration can be drawn with the Mn concentration value as x-axis and the accumulated frequency as y-axis.

d) Calculate $C_{low}$ (the minimum value of Mn concentration) and $C_{high}$ (the maximum value of Mn concentration). $C_{low}$ is the arithmetic average of the least P*N data. $C_{high}$ is the arithmetic average of the maximal P*N data. The value of P should be determined by agreement between the parties concerned. Unless otherwise specified, P is recommended to take 0,15.

e) Calculate the segregation ratio according to Formula (1)

1) Microsoft Excel™ is the trademark of a product supplied by Microsoft. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.