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

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Contents

Page

Foreword

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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 (seewww.iso.org/directives).

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For an explanation on 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: www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 202, *Microbeam analysis*, Subcommittee SC 2, *Electron probe microanalysis*.

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 using electron probe microanalysis (EPMA).

This document is mainly applied in continuously cast product with Mn content more than 0.01 % by mass. This method can also be used in steel ingots and steel products.

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 22309, *Microbeam analysis — Quantitative analysis using energy-dispersive spectrometry (EDS) for elements with an atomic number of 11 (Na) or above*

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
The 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
A method of analysing element in EPMA, through which element distribution in an area can be obtained

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

3.4
ratio of dendritic segregation (SR)
The 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 statistic 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 0,18 %, 0,25 %, 0,32 %, 0,53 %, 0,85 %, 1,76 %, 2,34 %, 3,45 % 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.

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. The stability of the instrument should be measured over a time period similar to that used for the mapping analysis. The check of the instrument stability could be performed according to ISO 11938

8 Procedure

8.1 Prepare the instrument

According to ISO 14594 and ISO 22489, prepare the instrument to be ready for quantitative analysis.

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 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~5s
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 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.

According to ISO 22489, Use the reference materials to establish Mn calibration curve. Then use the calibration curve to convert the measured raw data into quantitative ratios. Export all the Mn concentration data and saved as an excel file. Import the excel data to Matlab or other software to proceed further statistic analysis.

8.4 Process the data

Data processing shall be performed as follows:

a) Delete zero and negative number.

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 X*N data. C_{high} is the arithmetic average of the maximal X*N data. The value of X should be determined by agreement between the parties concerned. Unless otherwise specified, X is recommended to take 15 %.

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

$$SR=C_{low}/C_{high} \quad (1)$$

9 Uncertainty

Specimen preparation, calibration of the instrument, analytical conditions, selection of reference materials, correction method, and other factors offer information on the uncertainties in such measurements. For a list of factors, see ISO 22309:2006, Annex C.

10 Report

Records of the instrument and individual investigations shall be kept so that, if required, a test report conforming to ISO/IEC 17025:2005, 5.10, may be issued. Reports shall present at least the following information:

a) a reference to this standard and its year of publication;

b) identification of the specimen, heat, lot, etc.;

c) the number and the location of the samples taken from the test piece;

d) test conditions;

f) reference materials;

g) ratio of dendritic segregation;

f) any deviations from the procedure and any unusual features observed;

g) the date of the test.

