
**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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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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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 www.iso.org/directives).

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For an explanation of 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 www.iso.org/iso/foreword.html.

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.

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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 1,8 g/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 _α 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_{α} 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 file¹⁾. 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:

- Delete zero and negative numbers.
- Remove the abnormal data produced by MnS inclusions.
- 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.
- 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.
- 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.

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

Example of quantitative mapping analysis is given in [Annex A](#). Example of quantitative line analysis of is given in [Annex B](#).

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 22309:2011, 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 17025:2017, 7.8, may be issued. Reports shall present at least the following information:

- a) a reference to this document i.e. ISO 23692:2021;
- 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;
- e) reference materials;
- f) ratio of dendritic segregation;
- g) any deviations from the procedure and any unusual features observed;
- h) the date of the test.

Annex A

(informative)

Example of quantitative mapping analysis of dendritic segregation

A.1 Prepare the instrument ready for quantitative analysis.

A.2 Open the analysis software, choose Mn as the analysis element, set experiment parameters: accelerating voltage 20 kV, beam current 300 nA, beam size 6 µm, analysis area 2,4 × 1,8 mm.

A.3 Acquire the mapping of Mn by stage scanning.

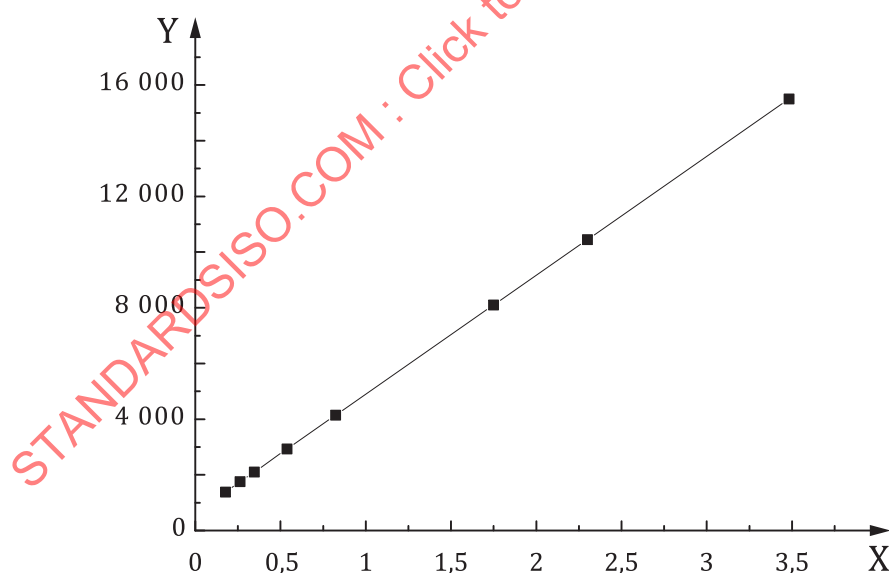
A.4 Generate the calibration curve from 8 Fe-Mn alloy serial reference materials with the identical conditions in A.2. The calibration curve (Figure A.1) is given by Formula (A.1):

$$I_i = 620 + 4274C_i \quad (\text{A.1})$$

where

I_i is the X-ray intensity of Mn K_α measured on the reference material;

C_i is the mass fraction of Mn in reference material.



Key

X Mn, mass %

Y intensity, cps

Figure A.1 — Calibration curve of Mn

A.5 Use the calibration curve to quantify the Mn element map to convert X-ray counts map into Mn concentration map (Figure A.2).

A.6 Export all data out and save as an MS-Excel file.

A.7 Import the data to Matlab to perform statistical analysis.

A.7.1 Delete zero and negative number.

A.7.2 Remove the abnormal data produced by MnS inclusions.

A.7.3 Sort all the Mn concentration data in an increased order. The data with same Mn concentration value shall be arrayed in succession. Each Mn concentration data has a sequence number, which ranges from 1 to 76800. Calculate the cumulative frequency. Draw the variation curve of Mn mass fraction ([Figure A.3](#)).

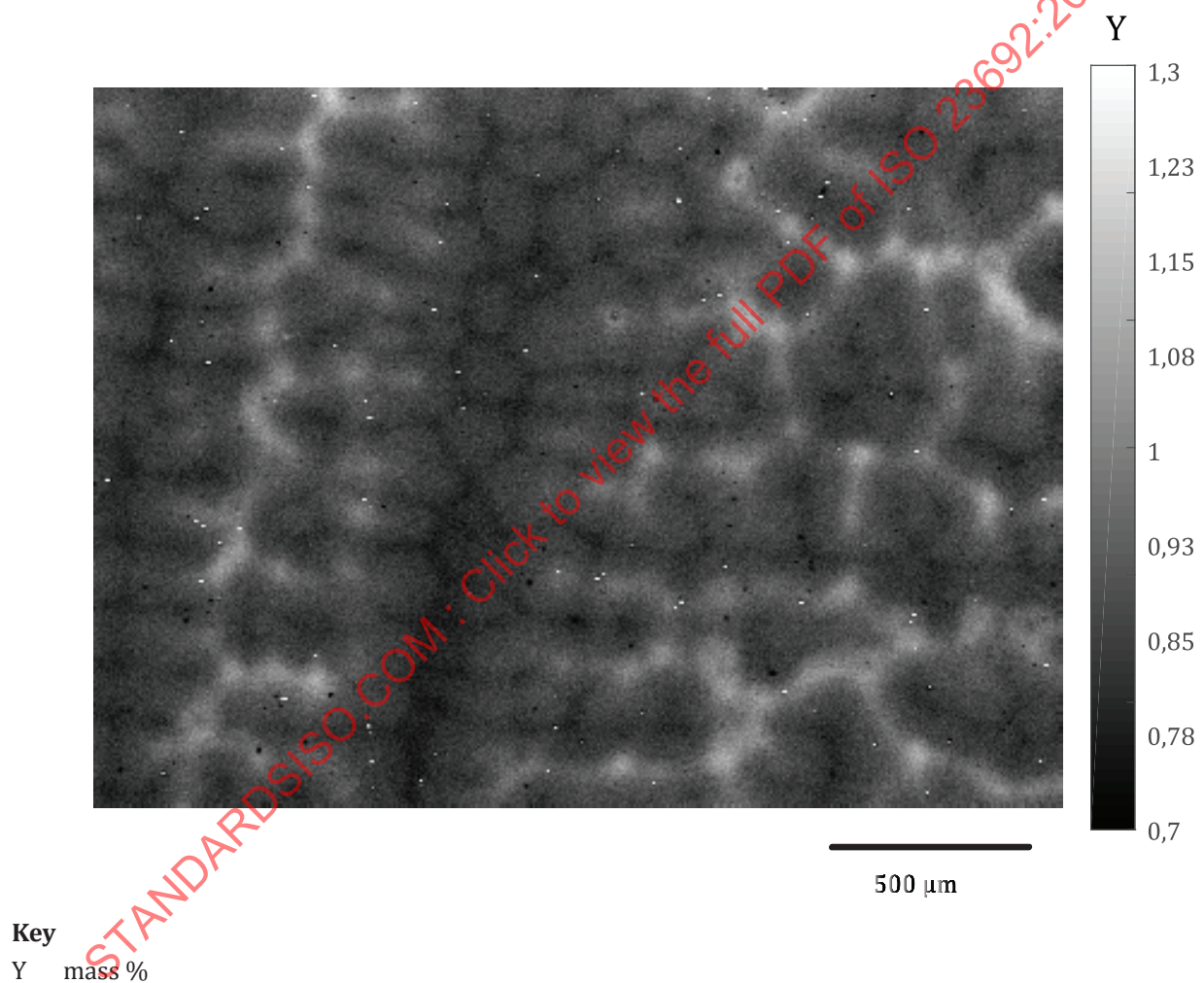
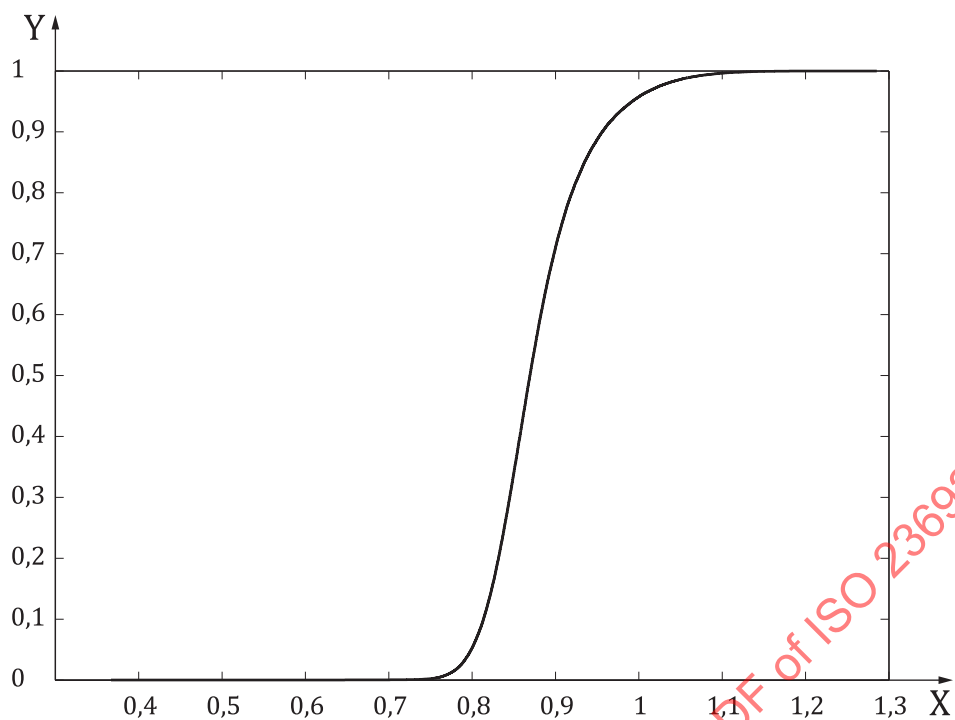


Figure A.2 — Mapping of Mn concentration



Key

X Mn, mass %

Y cumulative frequency

Figure A.3 — Variation curve of Mn mass fraction

A.7.4 Calculate C_{low} and C_{high} . P is set as 0,15. C_{low} is 0,80 and C_{high} is 0,99.

A.7.5 Calculate the segregation ratio.

$$SR = C_{\text{high}} / C_{\text{low}} = 1,23$$

Annex B (informative)

Example of quantitative line analysis of dendritic segregation

B.1 Prepare the instrument ready for quantitative analysis.

B.2 Open the analysis software, choose Mn as the analysis element, set experiment parameters: accelerating voltage 20 kV, beam current 300 nA, beam size 6 µm, 6 analysis lines including three 2,4 mm-long lines in X direction and three 1,8 mm-long lines in Y direction equally divided the analysis area (see [Figure B.1](#)).

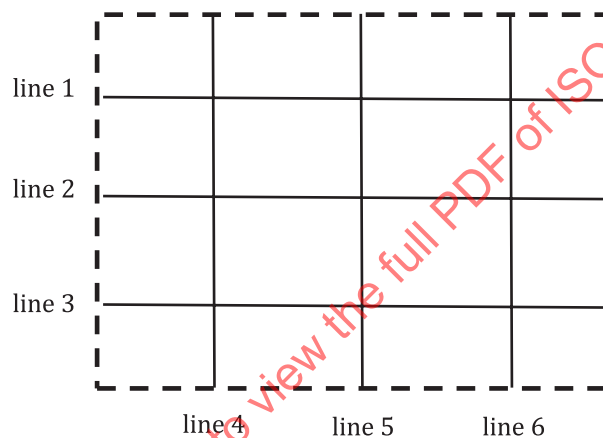


Figure B.1 — Schematic diagram of the distribution of lines in line analysis

B.3 Acquire the K_{α} intensity of Mn along each line by stage scanning.

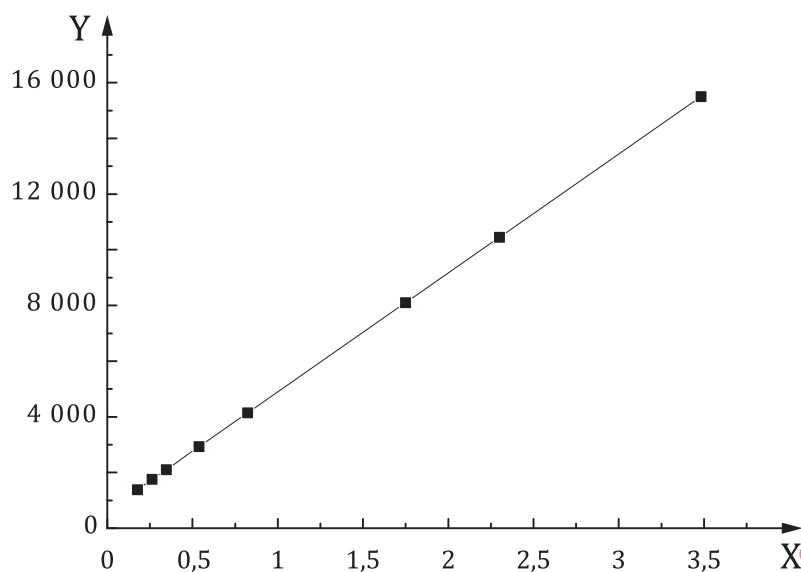
B.4 Generate the calibration curve from 8 Fe-Mn alloy serial reference materials with the identical conditions in [B.2](#). The calibration curve ([Figure B.2](#)) is given by [Formula \(B.1\)](#):

$$I_i = 620 + 4274 C_i \quad (\text{B.1})$$

where

I_i is the X-ray intensity of Mn K_{α} measured on the reference material;

C_i is the mass fraction of Mn in the reference material.



Key

X Mn, mass %

Y intensity, cps

Figure B.2 — Calibration curve of Mn

B.5 Use the calibration curve to quantify the Mn to convert X-ray counts lines into Mn concentration lines ([Figure B.3](#)).

B.6 Export all data out and save as an MS-Excel file.

B.7 Import the data to Matlab to perform statistical analysis.

B.7.1 Delete zero and negative number.

B.7.2 Remove the abnormal data produced by MnS inclusions.

B.7.3 Sort all the Mn concentration data in an increased order. The data with same Mn concentration value shall be arrayed in succession. Each Mn concentration data has a sequence number, which ranges from 1 to 2088. Calculate the cumulative frequency. Draw the variation curve of Mn mass fraction ([Figure B.4](#)).

B.7.4 Calculate C_{low} and C_{high} . P is set as 0,15. C_{low} is 0,79 and C_{high} is 0,96.

B.7.5 Calculate the segregation ratio.

$$SR = C_{high} / C_{low} = 1,22$$