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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 17360-2020

Replacing GB/T 17360-2008

Microbeam analysis - Method of quantitative determination for low contents of silicon and manganese in steels using electron probe microanalyzer

微束分析 钢中低含量硅、锰的电子探针定量分析方法

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1 Scope

GB/T 17360-2020 is the Chinese national standard on microbeam analysis - method of quantitative determination for low contents of silicon and manganese in steels using electron probe microanalyzer, in the field of chemical technology. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 2 June 2020 by the State Administration for Market Regulation; Standardization Administration of China, and has been in force since 1 May 2021. Classification: ICS 71.040.99, CCS N33. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This Standard specifies the calibration curve method that uses electron probe to determine the contents of silicon and manganese in carbon steel and low alloy steel (iron mass fraction is greater than 95%). This Standard is applicable to electron probe spectrometers, not to energy spectrometers. Scanning electron microscope with spectrometer can refer to this Standard as reference.

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.

GB/T 4930, Microbeam analysis - Electron probe microanalysis - Guidelines for the specification of certified reference materials (CRMs)

GB/T 13298, Inspection methods of microstructure for metals

GB/T 15074, General guide of quantitative analysis by EPMA

GB/T 15247-2008, Microbeam analysis - Electron probe microanalysis - Guidelines for determining the carbon content of steels using calibration curve method

GB/T 20725, Electron probe microanalysis guidelines for qualitative point analysis by wavelength dispersive X-ray spectrometry

GB/T 21636, Microbeam analysis - Electron Probe Micro Analysis (EPMA) - Vocabulary manganese) Kalpha line intensity ratio k and the silicon (or manganese) mass fraction w. As long as the intensity ratio of the silicon (or manganese) Kalpha line is measured on the test sample under the same test conditions, the mass fraction of silicon (or manganese) in the sample can be obtained from the calibration curve.

5 Instruments and auxiliary equipment

5.1 Electron probe analyzer.

5.2 Metallographic microscope and sample preparation device.

5.3 Ultrasonic cleaning device.

6 Reference material

6.1 The establishment of a calibration curve for the determination of silicon (or manganese) content REQUIRES at least 5 alloy reference substances with different silicon (or manganese) content and the content range covering the mass fraction of silicon (or manganese) element in the test sample. In addition, pure silicon (or manganese) reference materials are required.

6.2 For the selected series of alloy reference materials, in addition to meeting the requirements of GB/T 4930, the matrix composition shall be close to the chemical composition of the test sample. When the alloy elements other than silicon (or manganese) in the test sample do not have spectral interference and other factors that affect the quantitative analysis results of silicon (or manganese), Fe-Si (or Fe-Mn) solid solution can be selected as reference material. When there are other alloy elements that interfere with the measured spectrum of silicon (or manganese) in the test sample, the selected reference material shall also contain the same amount or content of these alloying

elements.

7 Sample preparation

7.1 The analysis surface of the sample shall be ground and polished. The operation method can be in accordance with GB/T 13298. Observe under a metallurgical microscope with a magnification of 200 ~ 500 times. There shall be no grinding defects such as wear marks on the surface of the sample.

7.2 Depending on the situation, the surface of the test sample and the reference material can be treated with the same degree of light corrosion, or neither is corroded. Reasonably set the pulse height analyzer parameters to eliminate the interference of high-order diffraction lines.

9 Establishment of calibration curve

9.1 According to the requirements of GB/T 20725, conduct qualitative analysis of the test sample first. According to the analysis results, select the appropriate alloy reference material combination.

9.2 Under the same test conditions, on the series of alloy reference materials with different silicon content and the pure silicon reference materials, sequentially measure the peak intensity IP and background intensity IB of silicon Kalpha. The determination of background intensity is usually shown in Figure 1. At the appropriate positions BG-, BG+ on both sides of the spectrum peak (pay attention to avoid interference lines and absorption edges), respectively measure X-ray counting. Then use linear interpolation to calculate the background intensity. For nonlinear background, special background models are required. Using linear interpolation will bring additional errors. The intensities of the alloy reference material and pure silicon reference material silicon Kalpha peak can be calculated by formula (1) and formula (2) respectively: Where, Ii(Si) -- The intensity of the ith alloy reference material silicon Kalpha line (after background correction); -- The peak intensity of the silicon Kalpha line measured on the ith alloy reference material; -- The background intensity of the ith alloy reference material silicon Kalpha line; Ipure(Si) -- The intensity of pure silicon reference material silicon Kalpha line (after background correction); -- The peak intensity of the silicon Kalpha line measured on a pure silicon reference material; Where, wi -- The mass fraction of the ith data point element; -- The arithmetic mean of data column wi; ki -- The intensity ratio of the characteristic X-ray of the ith data point; -- The arithmetic mean of data column ki; n -- The total number of data points used to build the calibration curve. The correlation coefficient R of the calibration curve shall meet: R>=0.99, otherwise all links in the test process need to be checked. Ensure that the test requirements are met. And re-determine.

10 Measurement of test sample

Use the same test conditions as when establishing the calibration curve. Measure and calculate the intensity ratio k of the silicon (or manganese) Kalpha line in the test sample. Substitute formula (7) to get the mass fraction of silicon (or manganese) in the sample.