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Rating and classifying of inclusions in steel - Scanning electron microscope method

钢中非金属夹杂物的评定和统计 扫描电镜法

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Rating and classifying of inclusions in steel - Scanning electron microscope method

1 Scope

This document specifies the equipment for evaluating and classifying non-metallic inclusions in steel by scanning electron microscope (SEM), calibration and verification of equipment, sampling, specimen preparation, test procedures, inclusion classification, rating and statistics, test reports, precision and bias.

This document recommends three inspection methods. Method 1 mainly classifies inclusions according to their morphology. Method 2 mainly classifies inclusions according to their chemical composition. Method 1 and Method 2 are suitable for microscopic evaluation of non-metallic inclusions larger than 2μm in rolled or forged steel with a compression ratio greater than or equal to 3. Method 3 is used to determine the specific details of a certain type of inclusion such as volume fraction, number fraction and other stereological parameters. It is suitable for the statistical classification of inclusions of all sizes (including below 2μm) in various slabs or steel products.

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 10561, *Steel - Determination of content of nonmetallic inclusions - Micrographic method using standards diagrams*

GB/T 13298, *Inspection methods of microstructure for metals*

GB/T 17359, *Microbeam analysis - Quantitative analysis using energy dispersive spectrometry*

GB/T 18876.1, *Standard practice for determining the metallographic constituent and inclusion content of steels and other metals by automatic image analysis - Part 1: Determining the inclusion or second-phase constituent content of steels and other metals by automatic*

GB/T 18876.2, *Standard practice for determining the metallographic constituent and inclusion content of steels and other metals by automatic image analysis - Part 2: Determining the inclusion ratings of steels by automatic image analysis and*

stereology

GB/T 27788, *Microbeam analysis - Scanning electron microscopy - Guidelines for calibrating image magnification*

GB/T 30067, *Standard terminology relating to metallography*

3 Terms and definitions

For the purposes of this document, the terms and definitions defined in GB/T 30067 as well as the followings apply.

3.1 acquisition analysis rules

The conditions for terminating X-ray acquisition (count, time or both), the list of elements to be analyzed, the number of fields or particles to be analyzed, the shape of particles to be analyzed, and so on.

NOTE: Typical acquisition conditions for SEM (see Annex A).

3.2 post-acquisition analysis rules

The elemental composition that identifies the chemical classification of the inclusions.

NOTE: For Method 1 and Method 2, in addition to setting the elemental composition of the chemical classification of inclusions, it is also necessary to set the type of inclusion (class A, class B or class C) to which each chemical classification belongs.

3.3 chemical classification

Set the category to which inclusions belong based on elemental composition.

NOTE: It can be broadly classified, such as sulfide, alumina, and silicates. It can also be finely classified, such as calcium sulfide, calcium silicate, and anorthite.

3.4 aspect ratio; AR

The ratio of the longitudinal and transverse directions of the extension of the inclusions.

3.5 stringer

A single inclusion that is highly extended in the direction of deformation. Two or more type A or type C inclusions, three or more type B inclusions, and these two or more inclusions must be arranged in a row, parallel to the direction of the thermal processing axis. The center line of offset stringer inclusions is less than or equal to 15μm. The distance between any two adjacent inclusions is less than or equal to 40μm.

3.6 Feret's diameter

chemical classification should be indicated in the inspection report.

4.4 Method 1 and Method 2 provide a quantitative evaluation method for each type of inclusion and each width series of inclusion levels (level 0~5) in half-level increments.

4.5 Method 3 specifies procedures for analyzing and counting inclusions by size and chemical classification. Method 3 is used to determine and record some basic stereological parameters of inclusions, such as the area fraction of sulfides and oxides, the number of sulfides or oxides per square millimeter. Method 3 is not used for rating.

5 Equipment

5.1 The scanning electron microscope should be equipped with the following accessories:

- Image acquisition software: It can acquire images.

- Sample stage: Computer control, X-Y direction motor drive. Automatic sample stage is recommended. Manual operation is also possible. Pay attention to the continuity of the field of view when operating manually.

- Energy spectrometer: Its resolution should meet the requirements of GB/T 17359. According to the set parameters, the electron beam and sample stage can be controlled, and images and spectra can be collected. The chemical composition analysis of inclusions can be carried out.

- Backscattered electron detector: One or more thresholds can be set to distinguish between matrix and inclusions.

5.2 The requirements for automatic feature analysis software are as follows:

- It should be possible to set analysis conditions for chemical classification. It should be possible to characterize by elemental composition, size and morphology.

- It should be able to distinguish stringer and single spherical oxide. Then distinguish stringer oxides according to different forms (type B or type C). It can measure the length of various stringer inclusions in each field of view. Classify all spherical oxides separated from type B or type C stringer inclusions into type D or type DS oxides. After separation of various types of inclusions (type A, type B, type C, type D), the automatic feature analysis software should also be able to measure the diameter of a single inclusion or the width of a stringer inclusion. Classify each type of inclusion as fine or coarse.

- It can connect the stringer inclusions across the boundary of the field of view.

6 Calibration and verification of equipment

6.1 The magnification of the scanning electron microscope should be regularly calibrated and verified according to GB/T 27788.

6.2 Calibration of energy spectrometer should be carried out according to the provisions of GB/T 17359.

6.3 The energy resolution of the spectrometer should be regularly verified in accordance with GB/T 17359.

7 Sampling

7.1 The sampling methods of Method 1 and Method 2 should be carried out according to the provisions of GB/T 10561. The sampling method of Method 3 should be carried out according to the agreement between the supplier and the purchaser. In any case, the polished surface should be parallel to the axis of thermal processing.

NOTE: If the angle between the polished surface and the thermal processing axis is greater than 6°, it will have a great influence on the length measurement of non-metallic inclusions.

7.2 If the product standard does not specify, if the non-metallic inclusions in rolled or forged steel are classified and rated according to Method 1 and Method 2, the polished surface is generally cut at 200mm^2. At least 160mm^2 test area should be guaranteed. If the inclusions in various slabs or steels are classified and counted according to Method 3, the test area should be determined according to the analysis requirements. It is recommended that the specimen area be relatively larger so that the measurement can be performed within a specified area away from the edge of the specimen.

8 Specimen preparation

8.1 Specimen preparation should be carried out in accordance with the provisions of GB/T 10561 and GB/T 13298.

8.2 The surface of the specimen after polishing should not be disturbed by man-made pollution, foreign matter, scratches. During polishing, the true shape of the inclusions cannot be changed by excessive protrusions, pits or tearing.

8.3 In general, inclusions are more likely to remain in the quenched specimen than the annealed specimen. For treatable steels, if sufficient inclusions do not remain in the annealed specimen, the specimen should be subjected to standard heat treatment. The tempering temperature should be relatively low. After heat treatment, the oxide layer should be removed. Longitudinal specimens should be reground to below the decarburized layer.

lead, it is recommended to set multiple thresholds. It is recommended to set image grayscale compensation during operation, for example, the software automatically adjusts the image grayscale to be consistent with the initial contrast at intervals of 5min, thereby ensuring the consistency of identification of inclusions.

NOTE: Use the same contrast setting. Different analysts will get the same or similar results when testing the same specimen. The reproducibility of test results can be guaranteed. Use appropriate threshold setting to ensure the accuracy of inclusion identification.

9.6 Control the sample stage according to the procedure recommended by the manufacturer. Set the analysis area of the specimen to be tested. For specimens with irregular shapes and it is difficult to ensure that the detection surface is flat, if a special horizontal specimen seat is not used to fix the specimen, it is recommended to use multiple Z-axis position points to locate the analysis area. For the testing and analysis of multiple specimens of thin plates, it is recommended to use the setting of multiple regions for one analysis (interface regions where multiple specimens are superimposed are not included in the analysis).

9.7 Set the post-acquisition analysis rules:

- Set chemical classification. For example, in Method 1, at least two chemical classifications should be set: sulfide and oxide. In Method 2, at least three chemical classifications should be set: sulfide, alumina, silicate. Additional categories can also be set according to needs, for example, calcium silicate inclusions are similar to class B inclusions in appearance and have similar hazards, so they can be classified as class B inclusions. The chemical classification and the main inclusion types to which it belongs should be given in the report.

- Specify the peak intensity that the X-ray spectrum should satisfy. Peak intensity includes one or more of the following parameters: spectral peak intensity range, peak-to-background ratio, peak intensity ratio, element content percentage, or other chemical parameters that characterize certain types of inclusions.

9.8 Set image and energy spectrum analysis parameters, such as minimum/maximum size of inclusions, critical aspect ratio, spectrum range, X-ray acquisition mode. Annex A lists typical acquisition conditions for SEMs. The parameters are set as follows:

a) Under the selected magnification, select the appropriate image resolution. It should be ensured that the pixel size is no larger than the size of the inclusion of interest.

b) According to GB/T 10561, the critical aspect ratio AR of Method 1 and Method 2 adopts 3.

c) In Method 3, it is recommended to use the two conditions of the number of inclusions and the detection area to terminate the analysis. When any one of these conditions is met, the analysis is automatically terminated. Use two conditions to

terminate the analysis. When the inclusion content in the steel is low, it can ensure that the detection area is large enough to be representative. When the content of inclusions in the steel is high, the testing time can be ensured to improve the testing efficiency.

9.9 Run automated scan detection.

9.10 Identify inclusions by chemical composition, morphology and width according to the steps specified in 10.1 (Method 1) and 10.2 (Method 2). Calculate levels based on length, count or diameter. Then store the results and generate inspection reports. Or follow the steps specified in 10.3 (Method 3) to identify inclusions by chemical composition, shape and width. Statistical analysis is carried out based on certain conditions. Generate test reports.

9.11 Save the raw data. The raw data should include the size, shape, composition, location and other information of all inclusions.

NOTE: Raw data can be processed later.

10 Inclusion classification, rating and statistics

10.1 Method 1: Morphology classification

10.1.1 Method 1 adopts the same inclusion classification as GB/T 10561. The characteristic spectrum is used to confirm and distinguish the inclusions.

10.1.2 Method 1 first divides inclusions into two categories according to their shape, that is, their aspect ratio. Inclusions with aspect ratio greater than or equal to 3, with high ductility, are mainly divided into class A and class C. Inclusions with an aspect ratio of less than 3, with low or no ductility, are mainly divided into class B, class D, and class DS. Then, the inclusions with an aspect ratio greater than or equal to 3 are divided into class A (sulfide, containing sulfur) and class C (silicate, containing oxygen) according to their chemical composition. Then the inclusions with an aspect ratio less than 3 are divided into class B, class D and class DS according to the distribution of clusters (bars) or single particles. Discontinuous stringer inclusions consisting of 3 or more particles (single particle aspect ratio less than 3) are class B, generally alumina. Single-grain inclusion particles are class D. Among them, the oversized class D inclusions with a diameter greater than 13μm belong to class DS.

10.1.3 For class A, class B and class C inclusions, the inclusion centerlines whose transverse spacing is within 15μm and whose longitudinal spacing is within 40μm should be regarded as the same stringer. If the widths of the inclusions in the same stringer are different, the width of the inclusion whose length is dominant should be taken as the width of the stringer of inclusions.

10.1.4 After classifying various types of inclusions, they are divided into fine series and

10.2 Method 2: Chemical classification

10.2.1 Method 2 mainly classifies inclusions according to their chemical composition. Sometimes refer to form.

10.2.2 Method 2 divides the inclusions into three classes according to their chemical composition: A, B and C. Class A inclusions are sulfides (usually manganese sulfide). Class B inclusions are aluminates (usually alumina). Class C inclusions are silicates.

10.2.3 Generally, the three classifications of A, B, and C have met the requirements. If you are interested in single-grain inclusions, class D spherical oxides (aspect ratio less than 3, single particle) can also be separated from the three types A, B, and C according to the morphology (aspect ratio) and distribution [stringers or single particles] (D) and class D spherical sulfide (aspect ratio less than 3, single particle) ($D_{sulfide}$). Class D inclusions with a diameter greater than 13μm can be classified as class DS. In some applications, single particle spherical inclusions may not be classified separately. Instead, they are still classified into class A, class B or class C according to their chemical composition. The classification of single particle inclusions should be determined by agreement between supplier and purchaser. However, if the rating is required, class A, class B and class C inclusions should be divided into stringers and single particles, that is, class D, class $D_{sulfide}$ and class DS.

10.2.4 After classifying various types of inclusions, they are divided into fine series and coarse series according to width or diameter (see Table 1). For class A, class B and class C inclusions, the width of the inclusion whose length is dominant is measured as the width of the stringer inclusions. For class D and class DS inclusions, measure the diameter. Class D inclusions with a width less than 2μm (class A, B, C) or a diameter less than 2μm are not included in the subsequent grade calculations.

10.2.5 For stringer inclusions, if the length exceeds 0.710mm, or the width is greater than the maximum value of the coarse series (see Table 1), it should be evaluated as oversized (length, width) inclusions. Record separately. However, these inclusions should still be included in the rating for this field.

10.2.6 On a testing area of 0.5mm^2, measure the total length of class A, class B or class C stringer inclusions and the total number of class D inclusions. Grades are rated according to the minimum length or number of each grade given in Table 2. The measurement should be carried out on a continuous 0.5mm^2 testing area.

10.2.7 Composite inclusions (such as oxysulfide or dual-phase inclusions) can be classified according to chemical composition and content. If the inclusion is mainly sulfide and its area is greater than 50%, it is classified as class A. If it is mainly non-deformable oxides (such as alumina inclusions) and its area is greater than 50%, it is classified as class B. If it is mainly deformable silicate and its area is greater than 50%, it is classified as class C. For composite inclusions with critical aspect ratio AR<3, if they exist alone, they can be classified as class D or class $D_{sulfide}$.

10.2.8 For steel treated with rare earth or calcium, the endogenous inclusions can also be treated in the same way as in 10.2.2~10.2.7. Set the chemical classification (e.g., calcium sulfide, calcium silicate, anorthite, spessartine). Assign the chemical classification to the main inclusion types.

10.2.9 For specimens containing heavy element inclusions such as RE, Pb, Bi, they generally contain traditional types of inclusions such as oxides, sulfides, or silicates and composite inclusions of heavy elements. The automatic statistical software should have the function of double or multi-threshold gray scale and screening function to ensure the identification of all inclusions and realize the accurate statistics of heavy element composite inclusions.

10.3 Method 3: Custom analysis method

10.3.1 Method 3 is recommended when neither Method 1 nor Method 2 is applicable. Method 3 allows inclusions to be analyzed individually by material and application. Allow customizing the criteria for chemical classification. The critical aspect ratio can be properly selected according to actual needs. The termination conditions of the analysis can be set as the maximum number of inclusions and the testing area. Inclusions can be further subdivided by morphology.

10.3.2 Method 3 can be used to determine the number and size distribution of inclusions of a certain chemical composition (such as titanium inclusions in stainless steel) or to analyze inclusions in as-cast steel. Method 3 can classify inclusions according to special methods, such as classifying all non-sulfide particles by width in tire cord steel. This method can also be used in some steels with special restrictions or regulations.

10.3.3 Method 3 can set the relevant chemical classification, size range and morphological classification, rather than using indirect terms such as A, B, C, and D, or fine, coarse, and wide. Chemical classification is the taxonomic designation for inclusions. The size distribution of each type of inclusion can then be determined. The minimum size for inclusion analysis is not specified in this document but is determined by the specific application. Higher magnifications can be used to determine small inclusions that cannot be seen with an optical microscope.

10.3.4 Method 3 can give the volume and number fraction of inclusions in each field of view, the maximum Feret's diameter of each inclusion and other stereological parameters. The ratio parameters such as average area and mean free path of inclusions can also be calculated according to GB/T 18876.1.

11 Inspection report

11.1 The inspection report should include the following:

a) Reference to this document;

Annex F

(informative)

X-ray counting and chemical classification statistics

F.1 X-ray counting statistics

F.1.1 The generation and measurement of X-rays is a statistical process. Electron beams are irradiated onto the specimen in the scanning electron microscope. If the electrons carry enough energy, then X-rays may be produced. A programmed beam of X-rays may then leave the specimen surface and hit the detector. Finally, it will be tested and counted. Therefore, the measured intensities of specific X-rays vary widely even for ideal specimens tested by high-quality instruments.

F.1.2 The degree of dispersion of test results is processed by Gaussian software. The true value of the measured count is determined by the mean $\sqrt{I^{\circ}}$. The standard deviation is also given by $\sqrt{I^{\circ}}$. Gaussian distribution shows that 95.4% of the measured values are distributed in the range of $\sqrt{I^{\circ}} \pm 2\sqrt{I^{\circ}}$. 99.7% of the measured values are distributed in the range of $\sqrt{I^{\circ}} \pm 3\sqrt{I^{\circ}}$. That is, any measured value has a 95% probability of falling within the interval of $\sqrt{I^{\circ}} \pm 2\sqrt{I^{\circ}}$; it has 99% probability of falling within the interval of $\sqrt{I^{\circ}} \pm 3\sqrt{I^{\circ}}$. In the actual X-ray measurement, if the number of spectral peak counts exceeds the background count by $3\sqrt{B}$, considering the statistical probability of background floating is only 0.3%, then it is considered that the spectral peak must exist. The 3σ criterion is usually used when defining the minimum testing limit. The longer the counting time and the number of counts, the closer the measured value of the spectral peak is to the real value. If the number of counts I is 10000, assuming I=I°, it is estimated that 99% of the counts will fall in the interval of 9700~10300, that is, $I \pm 3\sqrt{10\ 000}$. A variation of ±3% may not have any effect on the accuracy of inclusion classification. However, if I is only 36, then the estimated measurement will be off by 50%, leading to misclassification. However, as mentioned in F.2, if the classification is sufficiently distinct, even a small number of counts can be accurately classified.

F.2 Termination criteria for X-ray analysis of individual inclusion

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