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Determination of content of nonmetallic inclusions in steel - Micrographic method using standard diagrams

钢中非金属夹杂物含量的测定 标准评级图显微检验法

(ISO 4967:2013, Steel - Determination of content of nonmetallic inclusion - Micrographic method using standard diagrams, MOD)

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Determination of content of nonmetallic inclusions in steel - Micrographic method using standard diagrams

1 Scope

This document gives information on the principles, sampling, specimen preparation, determination, result expression, test reports for the evaluation of non-metallic inclusions in steel, using standard rating charts; provides an image analysis method for the determination of non-metallic inclusions (see appendix B).

This document is suitable for evaluating the content of non-metallic inclusions, in rolled or forged steel, which has a compression ratio greater than or equal to 3.

Note: The charts in this document are not suitable for evaluating certain types of steel (for example: free-cutting steel).

2 Normative references

The contents of the following documents constitute essential provisions of this document through normative references in the text. Among them, for dated reference documents, only the version corresponding to the date applies to this document; for undated reference documents, the latest version (including all amendments) applies to this document.

GB/T 13298 Inspection methods of microstructure for metals

GB/T 30067 Standard terminology relating to metallography

3 Terms and definitions

The terms and definitions, which are defined in GB/T 30067, as well as the following terms and definitions, apply to this document.

3.1

Nonmetallic inclusions

A type of composition in steel.

Note: They are formed during smelting, solidification, cooling; undergo morphological changes during subsequent cold and hot processing. Usually have non-metallic properties,

- Category B (alumina): Most have no deformation, are angular, have a small morphology ratio (generally < 3), black or bluish particles, arranged in a line along the rolling direction (at least 3 particles);

- Category C (silicate): A single black or dark gray inclusion with high ductility, smooth boundaries, a wide range of aspect ratios (generally ≥ 3); generally the ends are at sharp angles;

- Category D (spherical oxides): Non-deformable, angular or rounded, small morphology ratio (generally < 3), black or bluish, irregularly distributed particles;

- Category DS (large particle spherical oxide type): Single particle category D inclusions with a diameter > 13 μm.

4.2 Appendix C gives the standard charts of the above five categories of inclusions: Category A, category B, category C, category D, category DS. These rating images are equivalent to a square field of view, which has an area of 0.50 mm^2, on the longitudinal polished plane under 100×. Compare the observed field of view with the standard chart in Appendix C; rate each category of inclusion, separately.

4.3 The morphology of inclusions depends to a large extent on the degree of compressive deformation of the steel. Therefore, comparison of measurement results is only possible, on sections of sample blanks, that have undergone similar degrees of deformation.

4.4 Non-traditional types of inclusions can be evaluated by comparing their morphology with category A, category B, category C, category D, and category DS inclusions, indicating their chemical characteristics. For example: Spherical sulfide can be evaluated as a category D inclusion; however, a subscript shall be added in the test report (for example: D_{CaS} means spherical calcium sulfide; D_{RES} means spherical rare earth sulfide; $D_{CaS+Al2O3}$ means composite inclusion calcium sulfide wrapped alumina).

Note: Non-traditional inclusions usually refer to calcium-containing (or rare-earth-containing) inclusions, which are formed by calcium treatment or adding rare earth elements or other special treatments, during the steelmaking process, as well as composite inclusions, etc.

4.5 For the evaluation of precipitated phases, such as titanium nitride, titanium carbonitride, boride, carbide, other carbonitride compounds or nitrides, they can also be compared with the category B, category C, category D, category DS inclusions, based on their morphology. Indicate their chemical characteristics with subscripts below.

Note: Before conducting the test, non-traditional category of inclusions and precipitates are generally inspected, at a magnification greater than 100×, to determine their chemical characteristics.

4.6 Table 1 gives the rating limits (minimum values), for the total length (or quantity or diameter) of each category of inclusions, from grade 0.5 ~ grade 5.0. Category A,

steel to the center. When the thickness, diameter or wall thickness of the product is small, a sufficient number of specimens shall be taken from the same blank, to ensure that the total polishing area is 200 mm^2; these specimens shall be regarded as one specimen. When 100 mm of the continuously sampled total length, in the main deformation direction, is taken as a specimen, the polishing area less than 200 mm^2 is allowed.

5.3 The sampling quantity shall be specified in the product standard or agreement.

5.4 Sampling methods shall be as specified in product standards or agreement. If not specified in the product standard or agreement, the sampling method shall be as follows:

- Steel rods which have a nominal diameter or side length less than or equal to 25 mm: The inspection surface is the entire cross-section through the diameter; its length shall ensure a polished area of approximately 200 mm^2, see Figure 2a);

- For steel bars or billets which have a nominal diameter or side length greater than 25 mm and less than or equal to 40 mm: The inspection surface is half of the cross-section through the diameter from the center to the edge of the specimen, see Figure 2b);

- For steel bars or billets which have a nominal diameter or side length greater than 40 mm: The inspection surface is a partial radial section from the outer surface of the steel to the center, see Figure 2c);

- Steel plates which have a nominal thickness less than or equal to 25 mm: Full-thickness section with inspection surface located at 1/4 of the width, see Figure 2d);

- For steel plates which have a nominal thickness greater than 25 mm and less than or equal to 50 mm: The inspection surface is located at 1/4 of the width and from the surface to the center of the steel plate; the inspection surface is a section 1/2 of the thickness of the steel plate, see Figure 2e);

- For steel plates which have a nominal thickness greater than 50 mm: The inspection surface is located between 1/4 of the width and the middle position from the surface of the steel plate to the center; the inspection surface is a section 1/4 of the thickness of the steel plate, see Figure 2f);

- Steel pipes which have a nominal wall thickness less than or equal to 25 mm: The inspection surface is located at the radial section of the full wall thickness, see Figure 2g);

- Steel pipes which have a nominal wall thickness greater than 25 mm: The inspection surface is a partial radial section from the outer wall to the middle of the inner wall, see Figure 2h).

Note: Welded pipes should be sampled away from the weld.

D.3.

7.2.2.7 According to the requirements of product standards or agreements, the precipitated phase can be evaluated, according to its morphology, together with the closest category B, category C, category D, category DS inclusions. Its chemical characteristics can be expressed according to the method of 7.2.2.6. For examples of evaluation of precipitated phases, see D.4.

8 Result representation

8.1 General

8.1.1 The inclusion inspection results are usually expressed by the inclusion category code (A, B, C, D, DS), series code (fine series T or coarse series H), grade (0.5, 1.0...5.0). Unless specified in the product standard or agreement, the evaluation results are expressed, in accordance with 8.2 (Method A) or 8.3 (Method B).

8.1.2 If the length of inclusions or string (bar)-shaped inclusions, in a certain field of view, exceeds the side length of the field of view (0.710 mm) OR the width is greater than the maximum value of the coarse series (see Table 2), it shall be recorded separately in the test report. The letter s is used to indicate the presence of oversized (length or width) inclusions.

8.1.3 For non-traditional types of inclusions or precipitates appearing in a certain field of view, if they need to be recorded separately in the test report, use a subscript to indicate their chemical characteristics. Chemical characteristics are represented by molecular formulas or (and) major elements. If the composition of inclusions can be clarified through energy spectrum analysis, it can be expressed by a molecular formula, with the dominant content listed first, each molecular formula separated by a plus sign. If the composition of the inclusion cannot be clarified through energy spectrum analysis, chemical elements can be used to represent its main components. Each element is separated by commas; the one with the dominant content is written first.

Example:

$DS_{MgO+CaS}$ 2.0; $DS_{RE(S,O)}$1.5; D_{TiN}1.5.

Note: $DS_{MgO+CaS}$2.0 means single particle composite inclusion grade 2.0. The composite inclusion is composed of MgO and CaS, in which MgO is dominant.

$DS_{RE(S,O)}$1.5 indicates single particle rare earth sulfur oxide grade 1.5, in which sulfide is dominant.

D_{TiN}1.5 represents dispersedly distributed granular titanium nitride grade 1.5.

8.2 Method A

Appendix B

(Informative)

Relationship between rated image grades and measured inclusion values

B.1 Figures B.1 ~ B.5 respectively represent the relationship -- between the rated image grade i of category A, category B, category C, category D, category DS inclusions and the measured value of the inclusion. The length L or diameter d in the Figure is expressed in microns (μm), OR expressed in the number n of inclusions per field of view. The formulas listed in B.2 can be used to calculate the inclusion grade i from the measured values of the inclusions. The formula listed in B.3 can calculate the measured value of inclusions from the grade of inclusions. For example: When measuring inclusions, which have a rated image grade of 3.0 or above, it can use the formula in B.2, to calculate the grade from the inclusion measurement values.

B.2 Calculate the grade of inclusions from the measured values of the inclusions.

Category A sulfide, length (L) expressed in μm:

$lg(i) = [0.5617lg(L)] - 1.182$

Category B alumina, length (L) expressed in μm:

$lg(i) = [0.4633lg(L)] - 0.873$

Category C silicate, length (L) expressed in μm:

$lg(i) = [0.4797lg(L)] - 0.901$

Category D spherical oxide, number per field of view (n):

$lg(i) = [0.5lg(n)] - 0.301$

Category DS large particle spherical oxide, the diameter (d) expressed in μm:

$i = [3.3117lg(d)] - 3.221$

Except for category DS inclusions, the i value can be obtained from the inverse logarithm.

B.3 Calculate the measured value of inclusions from the grade of inclusions.

Category A sulfide, length (L) expressed in μm:

$lg(L) = [1.7802lg(i)] + 2.1038$

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