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GB/T 13298-2015

Replacing GB/T 13298-1991

Inspection Methods of Microstructure for Metals

金属显微组织检验方法

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Foreword

This Standard was drafted as per the rules specified in GB/T 1.1-2009.

This Standard replaces GB/T 13298-1991 *Inspection Methods of Microstructure for Metals*; compared with GB/T 13298-1991, this Standard mainly has the following technical changes:

- Modify specimen preparation into “specimen preparedness” (see Chapter 3);
- Increase two situations for specimen selection (see 3.1);
- Add the schematic diagram for inspection surface (see 3.2.1);
- Add “band structure rating” for the inspection of longitudinal section parallel to the rolling direction (see 3.2.3);
- Add “specimen mark” (see 3.5);
- Adopt clear “mechanical setting fixture diagram” (see 3.7.2);
- Add the vibration polishing (see 5.5);
- Modify “specimen erosion” into “microstructure display”; add the general (see 6.1), optical method (see 6.2), thermal etching display method (see 6.3.5), anodic film method (see 6.4.2); delete the preparation and precautions of chemical etchants and electrolytic etchants (see 4.1.3 of 1991 Edition);
- Delete “metallographic microscope is divided into desktop-type, vertical-type, horizontal-type” (see 5.2 of 1991 Edition);
- Delete “precautions for protecting lenses when using a microscope” (see 5.5, 1991 Edition);
- Delete “making the shadow photograph on the glass plate clear; if necessary, use focusing magnifier to observe on the ground-glass plate” (see 6.6 of 1991 Edition);
- Microstructure inspection part (see Chapter 7), delete photographing with black-and-white negative film and colour negative film, “black-and-white negative film and photographic paper processing” (see 6.10 of 1991 Edition), and “color negative film and color photographic paper processing” (see 6.11 of 1991 Edition); add microscope illumination mode (see 7.2.1); add image acquisition (see 7.3) and image analysis part (see 7.4).
- Add “on-site metallographic inspection” (see Chapter 8);

Inspection Methods of Microstructure for Metals

1 Scope

This Standard specifies the specimen preparedness, specimen grinding, specimen polishing, microstructure display, microstructure test, on-site metallographic and test records for the metal microstructure inspection.

This Standard is applicable to the operation method of using metallographic microscope to inspect the metal structure.

2 Normative References

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this document.

YB/T 4377 Electrolytic Polishing Method of Metallographic Specimen

3 Specimen Preparedness

3.1 Specimen selection

3.1.1 General

To ensure the effectiveness of inspection, the selected metallographic specimen shall represent the researched materials objectively and comprehensively as much as possible. The specimen cutting direction, location, quantity shall be determined by the metal manufacturing method, inspection purpose, relevant standards, and provisions of both parties' agreement.

3.1.2 Routine inspection

In addition to the special provisions in the product standard, it is recommended to select from the position that can represent the characteristics of the materials; the specimen shall contain the complete processing and impact area. For instance, the specimen of steel strip or wire is suitable to cut from the end of the coil; cast specimen shall contain the maximum and minimum segregation zones; heat treatment specimen shall contain complete heat treatment layer; surface treatment specimen shall contain all surface treatment layers; welding specimen shall contain weld seam, heat affected zone, and base metal.

during the cutting period, so that prevent the structure changes.

3.5 Specimen mark

In order to avoid confusion during the specimen preparedness period, the specimen registration and marking shall be done well. Once the specimen is cut, print and engrave mark beyond the inspection surface of the specimen; ensure the mark shall not be worn, shielded during the specimen cleaning and heat treatment processes. If the specimen requires follow-up embedding, then make marks again after embedding.

3.6 Specimen cleaning

If the specimen surface is stained with grease, dirt, or residue, proper solvent (such as alcohol, acetone, and etc.) can be used to clean; cleaning can be conducted in the ultrasonic wave. Any metal coating that hinders the corrosion of the base metal shall be removed prior to grinding.

3.7 Specimen embedding

3.7.1 General

The following conditions of specimens need embedding such as small size specimen (such as sheet, ribbon material, thin tube, and etc.), too soft and brittle specimen, irregular specimen, inspecting the specimen edge structure, using automatic grinding and polishing machine for the specimen preparation. The applied embedding method shall not change the original structure; the inspection surface of the specimen shall be downward during the embedding period. According to the actual needs, the mechanical embedding (see 3.7.2) or resin embedding (see 3.7.3) methods shall be selected.

3.7.2 Mechanical embedding method

3.7.2.1 Fasten the specimen in a suitable fixture with bolts and screws (see Figure 2). The fixture hardness shall be close to the specimen hardness; so as to reduce the rounding role against the edges when grinding and polishing the specimen; the fixture composition shall be similar to the specimen to avoid forming the galvanic interaction influencing the corrosion effect.

NOTE: pay attention that the specimen shall closely contact with the fixture; be careful when fixing the specimen; if the clamping force is too large, it may damage the soft material specimen.

3.7.2.2 To reduce the penetration of polishing agent and etchant, the sheet made of soft materials can be used to fill the gaps of specimen; which shall ensure no electrolytic reaction shall occur between filling materials and specimen during the corrosion period. The typical filling materials include thin plastic sheet, lead or copper. In order to reduce the absorption of gaps against the polishing agent and etchant, the epoxide resin layer can paint to the specimen before holding; or immerse the specimen

5 Specimen Polishing

5.1 General

Polish the grinding mark on the specimen to reach the mirror finish, and there are no grinding defects. The polishing method can take mechanical polishing, electrolytic polishing, chemical polishing, vibration polishing, micro-grinding and so on.

5.2 Mechanical polishing

5.2.1 Rough polishing

After the burnishing of sand paper, the specimen can be transferred to the polishing machine equipped with nylon, woolen cloth or fine canvas to conduct rough polishing; the polishing agent can use the fine-grained diamond, alumina, magnesia, chromic oxide, iron oxide, emery, and the like; the type of polishing agent includes polishing suspension, spray polishing agent, polishing paste, and etc.. The polishing time shall be 2min ~ 5min. After polishing, use water to clean and dry.

5.2.2 Precise polishing

5.2.2.1 After rough polishing, the specimen can be transferred to the polishing disk equipped with nylon silk, velvet, or other fiber-uniform velour to conduct the precise polishing. According to the specimen hardness, different grain size of fine polishing paste, spray polishing agent, oxide suspension, and etc.. Pay attention to the polishing time and force, so as to avoid the specimen corner rounding or embossing. Generally, the polishing shall be continued till the grinding mark on the specimen is totally removed, and the surface becomes the mirror. After polishing, use water to wash; use absolute alcohol to clean and dry; so that the specimen surface has no water stains or dirt residue any more.

5.2.2.2 Precise polishing can select the manual or automatic methods. When manual polishing, gently press the specimen on the polishing disk; and polish back and forth along the disk diameter direction. Control the flannel humidity, to avoid influencing the polishing quality (too large humidity generates smear; too small humidity generates dark spots); flannel humidity shall be subject to the following condition when taking the specimen off the disk and observe, and the surface water film completely disappears and evaporates within 2s ~ 3s. The automatic polishing equipment fixes the specimen onto the fixture, the specimen is driven by the fixture to move within the polishing disk along certain trace; the force, rotation speed, rotation direction, and the like of fixture and polishing disk requires to be adjusted, so that improve the polishing efficiency.

5.3 Electrolytic polishing

Electrolytic polishing indicates that take the metal as the anode inserted in the electrolytic cell; its surface generates selective corrosion due to the electrolytic reaction;

6.4.1 Film forming method by chemical etching

A method of forming a thin film on the metal specimen surface by using the chemical reagent. Different phases in the metal may form different-thickness thin film due to the different potential, so that make the various phases, orientations, different grains, sub-grains, dendrites generate different interference color and display the structure difference due to the multiple reflections and interference phenomena. It is commonly used for phase identification, grain phase observation, and segregation structure.

6.4.2 Anode film-coating method

Anode film-coating method is the result of anodizing or anodizing treatment. In the anode region, the electrochemical anodic metal generates ionization reaction; and the metal ions in the solution of anode region react with pure chemical deposition of some anions; then form a thin film on the specimen surface that exhibits anisotropy to light. Under the polarized light, use differential interference or sensitive plate, grains with different orientations generate different colorful colours. Generally, pure aluminum, high-purity aluminum, soft aluminum alloy, and cast aluminum requires to take anode film-coating method, which shows clean grains under polarized light.

6.4.3 Potentiostatic anodizing and anodic precipitation

The anode film-coating is carried out under the condition of constant anode electrode potential; since each phase in the alloy is in different stages on the polarization curve at a selective potential, the oxidation and film-forming speed of which is different, the interference result may appear different colors, which can be commonly used for the identification of non-ferrous metal phases.

6.4.4 Film-coating method by vacuum evaporation

In vacuum chamber, use certain method (resistance heating method, as well as electron beam, laser and electric arc, and etc.) to heat, and cause the evaporation or sublimation of the coating materials, the deposition condenses on the specimen surface and forms film.

6.4.5 Sputtering film-coating method

In the vacuum chamber, use the gas glow discharge to generate ions, whereof the positive ions, under the role of electric field, bombard the cathode target surface; the bombarded target atoms and atomic groups fly to the specimen surface at certain speed and form a thin film.

6.4.6 Thermal dyeing method

Heat the polished specimen (<500°C) to form an oxide film. Since the composition structure of each phase in the metal structure is different, uneven-thickness of oxide films are formed. The interference of white light between the oxide films exhibits

7.3.5 As required, the acquired image can be saved as JPEG, BMP, TIFF, RAW, PSD, PDF and other formats.

7.4 Image analysis

The acquired microstructure can be conducted microstructure analysis and quantitative metallographic analysis as required.

7.4.1 Microstructure analysis

Usually, the following test and analysis can be carried out:

- a) Assess the steel microstructure; conduct the metallographic assessment against the free cementite, pearlite, band structure, and Widmanstatten structure and etc. within the steel;
- b) Analyze the state, distribution and grade of non-metallic inclusions within the steel;
- c) Assess the level of carbides within the steel;
- d) Determine the thickness of surface quenched layer, electroplated layer, decarburized layer, carburized layer and nitride layer.

7.4.2 Quantitative metallographic analysis

7.4.2.1 Use the principle of stereology to determine and calculate the three-dimensional spatial structure of alloy by the two-dimensional metallographic microstructure; so as to establish the quantitative relationship among the alloy composition, structure and performance; which can be used for the quantitative analysis for artificial or specialized image analyzer. Usually the following measurement and analysis can be performed:

- a) Measure the volume fraction in the microstructure of various alloys; such as the measurement of Phase-a content in the duplex stainless steels;
- b) Measure average grain size of the metal;
- c) Measure the area content of graphite carbon in the steel;
- d) Test the volume fraction, average cutting line length of particle, and other parameters of the given phase of ductile cast iron;
- e) Assess the ordinary and low-alloy cast iron structure, and the cast iron structure after thermal treatment;
- f) Measure the content of non-metallic inclusion in the metal;

		~25mm; voltage 6V; 30s ~ 90s	austenite, and then ferrite
1-5	Ferric chloride 5g Hydrochloric acid 50 mL Water 100mL	Immersing for 5s ~ 10s	Exhibit the austenitic nickel steel and stainless steel structure
1-6	Nitric acid 5mL ~ 10mL Alcohol 95mL ~ 90mL	Etching for several seconds to 1min	Exhibit the tool steel structure
1-7	Add little nitric acid into the saturated solution of reagent (1~5)	---	Exhibit stainless steel structure
1-8	Nitric acid 10mL Hydrochloric acid 20mL ~ 30mL Glycerol 30mL ~ 20mL	Preheat the specimen with warm water before etching; combine corrosion and polishing	Exhibit ferro-chrome-based alloy, high speed steel, high manganese steel, nichrome structure, and low alloy steel grain size
1-9	Nitric acid 10mL Hydrochloric acid 20mL Oxydol 10mL Glycerol 20mL	Increase or decrease the amount of hydrochloric acid slightly; increase the role of hydrochloric acid to accelerate; it is better to combine the corrosion and polishing	Exhibit ferro-chrome manganese, ferro-chrome nickel and ferro-chrome austenitic alloy steel structure
1-10	Oxalic acid 10g Water 100mL	Electrolytic etching; specimen is anode, stainless steel is cathode at the distance of 25mm; voltage is 6V; display structure etching for 1min; display carbide for 10s ~ 15s	Exhibit austenitic stainless steel and high nickel alloy structure
1-11	Copper chloride 5g Hydrochloric acid 100mL Water 100mL Alcohol 100mL	Immersion etching	Ferrite and austenitic steel; ferrite is easy to be etched; while the carbide can't be etched
1-12	Copper chloride saturated hydrochloric acid 30mL Nitric acid 10mL	After the reagent is prepared, stand for 20min ~ 30min before use; wiping erosion	Stainless alloy and high nickel high cobalt alloy
1-13	Nitric acid 30mL Acetic acid 20mL	Etching by wiping method	Stainless alloy and high nickel high cobalt alloy
1-14	Nitric acid 5mL Hydrofluoric acid (48%) 1mL Water 44mL	Etching for about 5min	Austenitic stainless steel, but not exhibiting the steel line
1-15	Hydrochloric acid 10mL Nitric acid 3mL Alcohol 100mL	Etching for about 2min ~10min	High speed steel quenching, and quenching-post grain boundaries
1-16	Hydrochloric acid 10mL Alcohol 90mL	Electrolytic etching, reagent contains no water, voltage is	Chrome steel, and nickel-chrome steel

3-8	Iron chloride 5g Hydrochloric acid 16mL Alcohol 60mL	Immersing or wiping method for etching; about several seconds to several minutes	Pure copper, aluminum bronze																																
3-9	Potassium dichromate 2g Sulfuric acid (specific gravity 1.84) 8mL Saturated sodium chloride solution 4mL Water 100mL	Add hydrochloric acid to immerse for 3s ~ 60s before use	Pure copper, copper alloy, chromium copper, copper beryllium, manganese copper and other alloys, as well as silver-nickel alloy																																
3-10	Ferrous sulfate 2g Sodium hydroxide 0.4g Sulfuric acid 10mL Water 190mL	Electrolytic etching: voltage 8V ~ 10V, current 0.1A; etching time 5s ~ 15s	Blacken the Phase-β in the brass (pre-etching by oxydol), also applicable to bronze, copper alloy, and silver-nickel alloy																																
3-11	<table border="1"> <thead> <tr> <th>Ferric chloride</th><th>Hydrochloric acid</th><th>Water</th><th>Others</th></tr> </thead> <tbody> <tr> <td>5g</td><td>50ml</td><td>100ml</td><td></td></tr> <tr> <td>25g</td><td>25ml</td><td>100ml</td><td></td></tr> <tr> <td>1g</td><td>10ml</td><td>100ml</td><td></td></tr> <tr> <td>8g</td><td>25ml</td><td>100ml</td><td></td></tr> <tr> <td>20g</td><td>5ml</td><td>100ml</td><td>chromium oxide 3g</td></tr> <tr> <td></td><td></td><td></td><td>copper chloride 10g</td></tr> <tr> <td>5g</td><td>10ml</td><td>100ml</td><td>stannous chloride 0.1g</td></tr> </tbody> </table>	Ferric chloride	Hydrochloric acid	Water	Others	5g	50ml	100ml		25g	25ml	100ml		1g	10ml	100ml		8g	25ml	100ml		20g	5ml	100ml	chromium oxide 3g				copper chloride 10g	5g	10ml	100ml	stannous chloride 0.1g	Use immersing or wiping method for etching gradually, so that obtain good effect	Copper nickel, copper alloy, and pure copper
Ferric chloride	Hydrochloric acid	Water	Others																																
5g	50ml	100ml																																	
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A.4 Refer to Table A.4 for the etchants commonly used by nickel and nickel alloy

Table A.4 Nickel and Nickel Alloy

S/N	Composition	Etching Method	Applicable Range
4-1	Nitric acid 50mL Acetic acid 50mL	Immersing or wiping for 5s ~ 30s; use long time for chemical etching; sulfide boundary is easier to be etched than conventional grain boundary	Pure nickel, copper nickel, titanium nickel, and high nickel alloy
4-2	Phosphoric acid 70mL Water 30mL	Electrolysis voltage 5V ~ 10V; time 5s ~ 60s	Copper nickel, iron nickel, chromium nickel, pure nickel, and high nickel alloy
4-3	Copper sulfate 10g Hydrochloric acid 50mL Water 50mL	Immersing or wiping for 5s ~ 60s; add several drops of H ₂ SO ₄ before use to increase the activity	Copper nickel, pure nickel, high nickel alloy, iron nickel fixed-point exhibit the nickel-based high-temperature alloy grain size
4-4	Acetic acid 5mL Nitric acid 10mL Water 100mL	Electrolysis voltage 1.5V, time 20s ~ 60s; use platinum lead wire, the solution shall be prepared for immediate use	Exhibit the aluminum nickel, copper nickel, chromium nickel, iron nickel, titanium nickel, and silver nickel structure
4-5	Iron chloride 8g	Wiping and washing for 5s ~ 30s	Exhibit the copper nickel, and silver

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