International Standard 4969

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Steel — Macroscopic examination by etching with strong mineral acids

Acier — Examen macroscopique par attaque aux acides minéraux forts

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

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It has been approved by the member bodies of the following countries :

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The member body of the following country expressed disapproval of the document on technical grounds :

United Kingdom

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Steel — Macroscopic examination by etching with strong mineral acids

1 Scope and field of application

This International Standard lays down guidelines for the macroscopic examination of steel by etching with strong mineral acids.

The method has very wide application. Selection of the type and concentration of the reagent, the temperature of the reaction and the conditions of surface preparation of the test piece make it possible to achieve the required aim.

NOTE — Depending on the conditions of surface preparation of the test piece, and on the etching process, this macroscopic examination does not always make it possible to distinguish, on the one hand porosities from segregations, and on the other hand cracks from lines of inclusions or segregations.

2 General

2.1 The use of the test, and the conditions for interpreting the results observed, depend on the particular case : details are laid down in product standards or shall be subject to special agreement.

2.2 Observation of the etched surface is carried out with the unaided eye or with a magnifying glass (magnification up to 10 ×).

3 Principle and aim of the test

3.1 Macrographic etching with strong mineral acids reveals the macrostructure of a metal test piece and indicates any physical or chemical irregularities.

3.2 The reagent acts by dissolving different parts of the metal surface at unequal rates and thus produces differences in level which make observation possible.

3.3 Macroscopic examination after etching with mineral acids reveals lack of chemical uniformity (segregation of elements) lack of physical uniformity (cracks, porosity) and any intentional or accidental structural variations such as those caused, for instance, by hardening, decarburization, and case hardening.

3.4 In addition, very great sensitivity may be achieved by altering the conditions of preparation and attack. For instance, it is possible to reveal the dendritic structure of a metal or the presence of inclusions or very small defects.

4 Reagents

The list of reagents given below is not restrictive. These reagents are used successfully for most routine examinations and for an extensive range of steel grades (hydrochloric and sulphuric etching). Aqua regia is used for special steels which are resistant to attack by the following reagents.

Furthermore, depending on the objective, the type and concentration of the acid used may be varied, as may the temperature and time of application. For very detailed examinations, cold dilute nitric solutions similar to reagents used for micrographic testing may be needed.

4.1 Hydrochloric acid, dilute solution, having the following volumetric composition :

HCl (ϱ_{20} 1,19 g/ml)	: 1 volume
H_2O	: 1 volume

This solution is used hot (60 to 80 °C).

4.2 Sulphuric acid, dilute solution, having the following volumetric composition :

H_2SO_4 (ϱ_{20} 1,84 g/ml)	: 15 volumes
H_2O	: 85 volumes

This solution may be used cold or hot (60 to 80 °C).

4.3 Sulphuric-hydrochloric acids, solution having the following volumetric composition :

HCl (ϱ_{20} 1,19 g/ml)	: 38 volumes
H_2SO_4 (ϱ_{20} 1,84 g/ml)	: 12 volumes
H_2O	: 50 volumes

This solution is used hot (60 to 80 °C).

4.4 Aqua regia, solution having the following volumetric composition :

HCl (ϱ_{20} 1,19 g/ml) : 3 volumes

HNO_3 (ϱ_{20} 1,33 g/ml) : 1 volume

This solution may be used cold or up to 40 °C.

5 Test piece

The test may be made on the product or on a test piece cut from the product. In general, this consists of a section perpendicular to the direction of rolling for products such as bars, billets and rounds, or of a surface suitably selected by agreement between the parties.

5.1 Sampling

Inothe absence of requirements in the product standards, the number and position of the surfaces examined shall be subject to agreement between the parties.

It is advisable, in particular, to locate the test surfaces away from the cut faces when cutting has been carried out

— by hot shearing, which deforms the fibres as well as the inclusions and may greatly offset the segregates;

— by flame cutting, which, in the case of hard steels, may produce local hardening, shrinkage cracks or local tempering.

5.2 Machining

5.2.1 The degree of surface preparation necessary depends on the precision required for macroscopic examination by acid etching.

While rough machining, resulting in relatively coarse surfaces, may be sufficient in certain cases (routine inspection to reveal shrinkage holes, for example), more careful machining is generally required.

The criteria to be observed when machining are as follows :

a) cutting-tool marking should not be pronounced, for example as the result of incorrect adjustment, excessively deep cuts or heavy feeds on the lathe or the shaping machine; good results are generally obtained with a feed of approximately 0,1 mm;

b) there should be as little cold working of the surface as possible, due for instance

— to a type of tool which is not suitable for the metal, or which is badly sharpened;

— to the use of unsuitable grinding wheels (less than 100 grit).

5.2.2 The main types of machining generally used are

— grinding, with or without preliminary machining;

— shaping or turning, provided that the lathe is fitted with a speed adjuster.

5.2.3 Where acid etching is used to reveal very fine defects or structural irregularities (different welding zones for instance), careful polishing is recommended; the finer the polishing, the better the definition. In general, it is recommended that a machined surface finish with an R_a of between 3,5 and 30 µm be obtained.

6 Procedure

6.1 Immerse the test piece in the acid bath, which may be heated. For large test pieces, it may be useful to pre-heat them to the temperature of the bath.

The volume of the bath shall be adequate : at least of the order of 1 litre of reagent per square decimetre of area of the test piece. In addition, the bath shall be sufficiently deep for the height of liquid above the upper face of the test piece to be at least 25 mm.

When etching several test pieces in the same bath, ensure that there is no contact between them. The establishment of galvanic couples may cause an uneven and misleading etch.

In the case of large samples or test pieces which cannot be immersed, pour the etching solution over the surface to be examined. Ensure a uniform and constant distribution of the reagent over the surface.

Renew the reagent as soon as its concentration has decreased by 10 %.

6.2 For each type of reagent, the time of application varies with the test temperature, the steel grade and even the type of examination. It is preferable that the treatment be entrusted to an experienced operator who will supervise the process and end it when he considers the etch to be adequate.

Excessive etching may lead to an exaggerated relief which is difficult to interpret.

The following approximate times of application are given for guidance :

— using hydrochloric acid (4.1), at 60 to 80 °C : about 30 min

— using a sulphuric acid (4.2),

— cold : about 10 h

— at 60 to 80 °C : about 30 min

— using sulphuric-hydrochloric acids (4.3), at 60 to 80 °C : about 30 min

— using aqua regia (4.4),

— cold : about 30 min

— up to 40 °C : a few minutes

6.3 When the etching is considered satisfactory, remove the test piece from the bath, wash it in running water, brush it carefully (with a non-metallic brush) to remove any residue from the etch, and then dry it.

7 Preservation of test pieces

In order to avoid subsequent corrosion of the surface of test pieces by sweating of the reagent , which cannot always be eliminated completely by rinsing, two techniques are recommended :

a) neutralization by immersion in a solution of 10 % ammonia in alcohol;

b) passivation by brief immersion (approximately 5 s) in concentrated nitric acid. (An additional advantage of passivation is that it whitens the etched surface and protects it to a certain extent against atmospheric corrosion.) After passivation, test pieces should be rinsed in hot water, brushed and dried.

However, these two techniques permit preservation for only a short period. If it is wished to preserve the test pieces for a long period, it is necessary to protect the etched surfaces with a plastic film or a cellulose varnish or any similar product.

8 Test report

The test report shall include the following information :

a) the steel grade examined;

b) the cast number;

c) the position of the surface examined;

d) the type of etch;

e) the result of the test (description of the etched surface, or photograph).

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