INTERNATIONAL STANDARD

ISO 4969

Second edition
2015-07-01

Steel — Etching method for macroscopic examination

Acier — Méthode d'attaque pour examen macroscopique

Reference number
ISO 4969:2015(E)

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Foreword

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The committee responsible for this document is ISO/TC 17, *Steel*, Subcommittee SC 7, *Methods of testing (other than mechanical tests and chemical analysis)*.

This second edition cancels and replaces the first edition (ISO 4969:1980), which has been technically revised.

Steel — Etching method for macroscopic examination

1 Scope

This International Standard establishes guidelines for the macroscopic examination of steel by hot etching, room temperature etching and electrolytic etching.

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

NOTE It might be difficult to see fine voids and cracks and discriminate between them and determine their nature by macroetching.

2 Principle

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

2.2 Macroscopic etching reveals the macrostructure of a metal test piece and any gross physical or chemical irregularities present.

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

2.4 Macroscopic examination after etching 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 or case hardening.

2.5 In addition, enhanced sensitivity can 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.

2.6 Observation of the etched surface is carried out with the unaided eye and/or with a magnifying glass, or with a stereomicroscope.

3 Sampling

3.1 The position and number of sampling shall be determined according to the requirements of the product standard, specification, contract or order. In the absence of any special requirements, sampling must be planned according to the details of the manufacturing process and the grade being evaluated.

3.2 The macroetching test, as applied to the inspection of steel products of this method, is carried out on slices, usually 13 mm to 25 mm in thickness. Disks or specimens are usually cut to reveal a transverse surface, but the requirements of the specification, contract, or order can include the preparation and examination of a longitudinal surface. In most cases, a longitudinally-oriented macroetching disc is taken with the plane to be etched along the centre line of the wrought product and it includes both outside surfaces with a length in the longitudinal direction, usually 1,5 times greater than the thickness or diameter.

3.3 Samples may be cold cut from the source by any convenient fashion; saws and abrasive cut-off wheels are particularly effective. The use of torch cutting or hot cutting should be used only when necessary to cut a sample from a large piece. It is advisable to locate the test surfaces well, away from the hot-cut surface avoiding sampling defects, such as deformations, heat affected zone, cracks, and so on.

3.4 Large cross sections can be cut into smaller pieces to facilitate handling and to comply with safety requirements. The sectioning of the large specimens should be done so as not to disturb the central portion of the section.

4 Preparation

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

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

4.3 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

   1) to a type of tool which is not suitable for the metal or which is badly sharpened, and

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

4.4 The main types of machining generally used are the following:

a) grinding, with or without preliminary machining;

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

4.5 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 a *R*a less than 30 µm be obtained.

4.6 After surface preparation, the sample is cleaned carefully with suitable solvents. Any grease, oil, or other residue will produce uneven attack. Once cleaned, care should be taken not to touch the sample surface or contaminate it in any way.

5 Solutions

5.1 The solutions used for macroetching are given in the tables listed under each method. In most cases, a good grade of reagent should be used but need not be chemically pure or of analytical quality. The commercial quality is usually satisfactory. The solution should be clean and clear, free of suspended particles, scum, etc.

5.2 Caution must be taken in mixing. Many of the etchants are strong acids. In all cases, the various chemicals should be added slowly to the water or solvent while stirring. In the cases where hydrofluoric acid is used, the solution should be mixed and used in polyethylene vessels.

WARNING — Hydrofluoric acid should not be allowed to contact the skin since it can cause painful serious ulcers if not washed off immediately.

5.3 The most commonly used solutions for macroetching iron and steel are Solution N° 1 and 2 in Table 1.

5.4 Renew the reagent as soon as its concentration is too low to be effective on etching.

6 Procedure

6.1 Hot etching and room temperature etching

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

6.1.2 Many of the solutions are aggressive and can give off irritating and corrosive fumes. Etching should be done in a well-ventilated room, preferably under a fume hood. The solution should be mixed and placed in a corrosion resistant tray or dish and brought to the operating temperature.

6.1.3 The volume of the bath shall be adequate, at least of the order of 1 l 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.

6.1.4 The specimen or specimens should be placed on some non-reactive support. Glass rods often are placed on the bottom of the acid container and the specimens are laid directly on the rods.

6.1.5 When etching several test pieces in the same bath, ensure that there is no contact between them, avoiding an uneven and misleading etching.

6.1.6 In the case of large specimens which cannot be immersed, such as ingot sections, swabbing might be the only practical method of macroetching. Saturate a large wad of cotton held in stainless steel or nickel tongs with the etchant and sweep over the surface of the specimen. An effort should be made to wet the entire surface as soon as possible. After the initial wetting, keep the swab saturated with solution and frequently sweep over the surface of the specimen to renew the solution. Ensure a uniform and constant distribution of the reagent over the surface. When the structure has been suitably developed, rinse the specimen, either with a swab saturated with water, or better still, by pouring water over the specimen. After rinsing with water, blow the specimen dry with compressed air.

6.1.7 When the etching is considered satisfactory, remove the test piece from the bath taking great care not to touch the etched surface, wash it in running water, brush it carefully (with a non-metallic brush) to remove any residue from the etch, and then dry it. When desmutting is required, dip the specimen into a second solution, such as 3 % to 5 % sodium carbonate (Na_2CO_3) or 10 % to 15 % nitric acid solutions.

6.1.8 The type of etchant, etching time and etching temperature are chosen according to the steel grade, as described in Table 1 and Table 2.

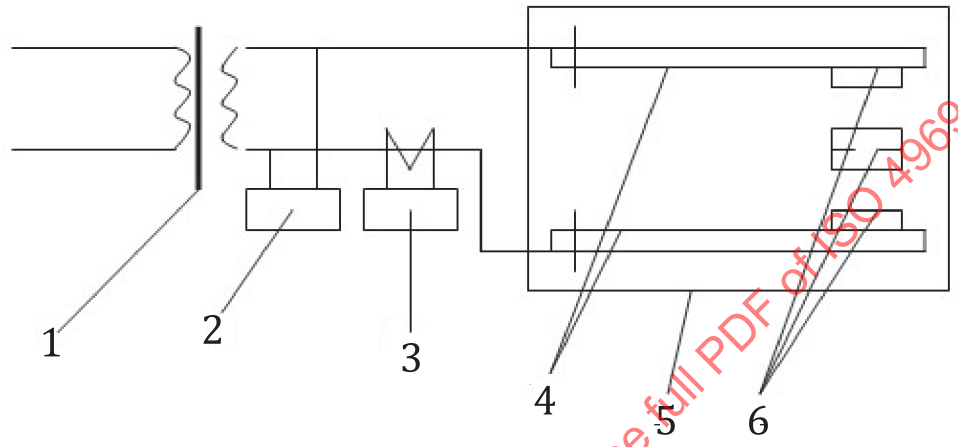
6.1.9 For each type of etchant, the time of application varies with the test temperature, the steel grade and even the type of examination. It is preferable that the treatment be entrusted to an experienced operator who will supervise the process and end it when he/she considers the etch to be adequate. Generally, over-etching can often lead to misinterpretation. The times given in individual tabulations are only intended as guides. The actual time to develop a structure properly can be quite different from the one suggested.

6.1.10 If a specimen has been over-etched, it must reground any evidence of the etched surface. This can require depth removal of 1 mm or more depending upon the extent of the excessive etch depth and possibly the flatness of the disk.

6.2 Electrolytic etching

6.2.1 AC power supply

6.2.1.1 The electrolytic etching schematic with an AC power is shown in Figure 1.



Key

1 AC transformer
2 volt meter
3 ampere meter
4 electrode
5 acid tank
6 specimen

Figure 1 — Electrolytic etching schematic with an AC power

6.2.1.2 Electrolytic etching with an AC power supply may use a solution of 15 % to 30 % concentrated HCl in water at room temperature.

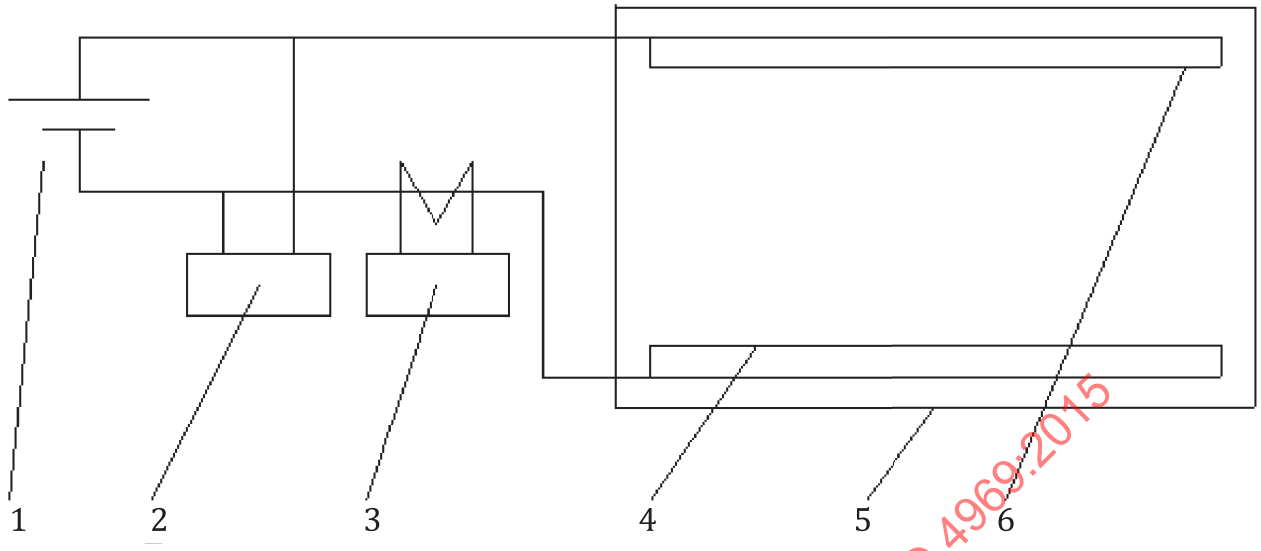
6.2.1.3 The specimen is immersed in the solution and the surface is oriented parallel to the electrode length. The power supply is AC. Generally, the working current is less than 400 A and the voltage in less than 36 V. The etching time is around 5 min to 30 min.

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

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

6.2.2 DC power supply

6.2.2.1 The electro-etching schematic with a DC power is shown in Figure 2.



Key

1 DC transformer
2 volt meter
3 ampere meter
4 cathode
5 acid tank
6 specimen (anode)

Figure 2 — Electro-etching schematic with a DC power

6.2.2.2 Electrolytic etching with DC power supply can use a solution of 6 ml to 12 ml HCl (concentrated) in 100 ml water for specimens less than130 cm^2 area, or a solution of 6 ml HCl (concentrated) and 1 g HBO_3 in 100 ml water for specimens over 130 cm^2 area, at room temperature.

6.2.2.3 The specimen is immersed in the solution and acts as an anode. For specimens less than 130 cm^2 area, the advised working current is 8 mA to 16 mA per square mm. For specimens over 130 cm^2 area, the advised working current is 48 mA to 64 mA per square mm.

6.2.2.4 After etching, the specimen is cleaned by using a vegetable fibre brush and a 10 % sodium citrate solution. The specimen is finally dried by compressed air.

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).

Table 1 — List of acids, etching temperature and time for hot etching

Number	Solution		Temperature	Time	Scope of application	Comments
1	HCl	50 ml	60 °C to 80 °C	5 min to 40 min	Plain and alloy steels	
	H_2O	50 ml				
2	HCl	38 ml	60 °C to 80 °C	15 min to 45 min	Plain and alloy steels	
	H_2SO_4	12 ml				
	H_2O	50 ml				
3	HCl	100 ml	60 °C to 70 °C	5 min to 25 min	High alloy steels	
	HNO_3	10 ml				
	H_2O	100 ml				
4	HCl (concentrated)	50 ml	≤75 °C		Austenitic stainless steels	Marble's reagent. Light etch, good for structures. Amount of $CuSO_4$ solution may be increased to 1 + 1 ratio for difficult alloys.
	Sat soln of $CuSO_4$ in H_2O	25 ml				
5	HCl	50 ml	70 °C to 80 °C		Stainless steels, high-alloy steels	Mix HCl and water, then heat to 70 °C to 80 °C. Immerse specimen and add H_2O_2 in several parts. Do not mix. Make each subsequent addition after foaming from previous addition has stopped.
	H_2O	50 ml				
	H_2O_2 (30 %)	20 ml				
6	H_2SO_4	15 ml	60 °C to 80 °C		Plain and alloy steels	
	H_2O	85 ml				
7	HCl	75 ml	≤40 °C		Stainless steels	
	HNO_3	25 ml				

Table 2 — List of etchants and scope of its application for room temperature etching

Number	Solution		Scope of application	Comments
1	HCl H_2SO_4 $CuSO_4$	500 ml 35 ml 150 g	Plain and alloy steels	
2	$FeCl_3$ HNO_3 H_2O	200 g 300 ml 100 ml		
3	HCl $FeCl_3$ H_2O	300 ml 500 g to 1 000 ml		
4	HCl (concentrated) HNO_3 (concentrated) H_2O	50 ml 25 ml 25 ml	High-alloy steels	Ratio HCl:HNO_3 runs 2:1 to 3:1. Immerse specimen for 10 min to 15 min in solution at room temperature. Rinse in warm water and dry.
5	$(NH_4)_2S_2O_4$ H_2O	10 ml to 20 ml 90 ml to 80 ml	Plain and alloy steels	
6	HNO_3 H_2O	10 ml to 40 ml 90 ml to 60 ml		
7	HNO_3 Sat Soln $FeCl_3$ in H_2O H_2O	10 ml 500 ml		
8	$CuCl_2{\cdot}2NH_4Cl{\cdot}2H_2O$ H_2O	100 g to 350 g 1 000 ml		
9	$(NH_4)_2S_2O_8$ (ammonium persulfate) H_2O	10 g 100 ml		Swab solution at room temperature over specimen. Rinse and dry. Grain size, weldments.
10	$CuCl_2$ $MgCl_2$ HCl (concentrated) Alcohol-up to	2,5 g 10 g 5 ml 250 ml		Stead's reagent. Salts dissolved in HCl with minimum of hot water to bring out P-rich areas and P banding. Immerse in solution at room temperature until a coppery sheen appears. Rinse thoroughly and dry.
11	HCl HNO_3	75 ml 25 ml	Alloy steels	
12	$CuSO_4$ HCl H_2O	100 g 500 ml 500 ml		