British Standard

BRITISH STANDARD

BS 6533:1984

Guide to

Macroscopic examination of steel by etching with strong mineral acids

UDC 669.14:620.183.255

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Iron and Steel Standards Committee (ISE/-) to Technical Committee ISE/72 upon which the following bodies were represented:

British Industrial Fasteners' Federation
British Railways Board
British Steel Industry
Ministry of Defence
Stainless Steel Fabricators Association of Great Britain

This British Standard, having been prepared under the direction of the Iron and Steel Standards Committee, was published under the authority of the Board of BSI and comes into effect on 28 September 1984

The following BSI references relate to the work on this standard:
Committee reference ISE/72
Draft for comment 82/71885 DC

ISBN 0 580 13995 6

Amendments issued since publication

Amd. No.	Date of issue	Comments

Contents

Foreword

This British Standard has been prepared under the direction of the Iron and Steel Standards Committee. It is based on ISO 4969:1980 "*Steel — Macroscopic examination by etching with strong mineral acids*", published by the International Organization for Standardization (ISO). Additional mineral acids to those listed in ISO 4969 have been included in this British Standard; these acids are given in **3.2**, **3.6** and **3.7**.

This standard does not purport to include all etching reagents suitable for the macroscopic etching of steel. For example, other suitable reagents widely used in industry include:

a) acid mixtures containing ferric chloride;

b) electrolytic etching reagents;

c) ammonium persulphate solutions.

Typical process solutions for the etching of steels and other metals for metallic materials and components for use in the aerospace industry are given in British Standard M 37.

WARNING. This British Standard calls for the use of substances and procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability and does not absolve the user from legal obligations relating to health and safety at any stage. This warning specifically refers to the use of strong mineral acids.

It has been assumed in the drafting of this British Standard that the execution of its provisions is entrusted to appropriately qualified and experienced people.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

This document comprises a front cover, an inside front cover, pages i and ii, pages 1 to 4, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

1 Scope

This British Standard gives guidance on and describes methods for the macroscopic examination of steel by etching with strong mineral acids.

NOTE The use of the methods, and the conditions for interpreting the results observed, depend on the particular case and details may be laid down in product standards or be subject to special agreement.

The methods given have very wide application. Selection of the type and concentration of the reagent, the temperature of the reagent and the conditions of surface preparation of the test piece make it possible to achieve an informed description of the etched surface.

NOTE The titles of the publications referred to in this standard are listed on the inside back cover.

2 Principle

2.1 Macrographic etching with strong mineral acids reveals the macrostructure of a metal sample and may indicate certain physical or chemical irregularities.

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

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

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.

2.4 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.

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

3 Etching reagents

3.1 *General*

The reagents given are used successfully for most routine examinations and for an extensive range of steel grades although other reagents are also permitted. Aqua regia is used for steels which are resistant to attack by the other acids.

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 examination, cold[1)] dilute nitric acid solutions similar to reagents used for micrographic testing may be needed.

WARNING. The acids listed in this standard **CAUSE SEVERE BURNS**. Prevent contact with skin and eyes.

In addition, hydrochloric acid and nitric acid are irritating to the respiratory system and nitric acid may cause fire in contact with combustible materials.

When making diluted acid solution, always cautiously add the acid to the water. Mix and cool if necessary, before making further additions of acid to the solution.

Attention should be paid to appropriate safety precautions, and the method should only be operated by trained personnel.

3.2 *Hydrochloric acid (concentrated solution)* (ρ 1.19 g/mL at 20 °C). This solution is used cold.

3.3 *Hydrochloric acid (dilute solution)*, having the following volumetric composition:

a) HCl (ρ 1.19 g/mL at 20 °C): 1 volume;

b) H_2O: 1 volume.

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

3.4 *Sulphuric acid (dilute solution)*, having the following volumetric composition:

a) H_2SO_4 (ρ 1.84 g/mL at 20 °C): 15 volumes;

b) H_2O: 85 volumes.

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

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

a) HCl (ρ 1.19 g/mL at 20 °C): 38 volumes;

b) H_2SO_4 (ρ 1.84 g/mL at 20 °C): 12 volumes;

c) H_2O: 50 volumes.

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

3.6 *Nitric acid solution*, having the following volumetric composition:

a) HNO_3 (ρ 1.42 g/mL at 20 °C): 1 volume;

b) H_2O: 1 volume.

This solution is used cold.

3.7 *Nitric acid (dilute solution)*, having the following volumetric composition:

a) HNO_3 (ρ 1.42 g/mL at 20 °C): 1 volume;

b) H_2O: 10 to 20 volumes.

[1)] For the purposes of this standard the term 'cold' means at ambient temperature.

This solution is used cold.

NOTE 1 This solution is especially suitable for examination of weldments.
NOTE 2 Water may be replaced by alcohol when good macrographs are required.

3.8 *Aqua regia acid solution*, having the following volumetric composition:

a) HCl (ρ 1.19 g/mL at 20 °C): 3 volumes;

b) HNO_3 (ρ 1.42 g/mL at 20 °C): 1 volume.

This solution is used either cold or up to 40 °C.

4 Sample

The examination is made on the product or on a sample cut from the product.

NOTE 1 The surface or section to be examined may be perpendicular or parallel to the principal direction of working depending upon the product and application, and the surface or section should be subject to agreement between the purchaser and the supplier.
NOTE 2 In the absence of requirements in the product standards, the number and position of the surfaces examined should be subject to agreement between the purchaser and the supplier.

Locate the surfaces to be examined away from the cut faces when cutting has been carried out:

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

b) by flame cutting, which, in the case of hardenable steels, may produce local hardening, shrinkage cracks or local tempering.

5 Preparation of sample

5.1 General

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

5.2 Machining

Rough machining, resulting in relatively coarse surfaces, may be sufficient in certain cases, e.g. in routine inspection to reveal shrinkage holes. However, more careful machining is generally required.

The criteria to be observed when machining are as follows:

a) cutting-tool marking should not be pronounced, e.g. 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 final cut 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;

2) to the use of unsuitable grinding wheels.

NOTE 1 The main types of machining generally used are:
a) grinding, with or without preliminary machining;
b) shaping or turning.
NOTE 2 In general, it is recommended that a surface finish be obtained with an R_a value of:
a) less than 6.3 μm for components;
b) less than 12.5 μm for materials.
This range of surface finish can be obtained by grinding, turning, boring, milling, shaping and planing (see BS 1134-2).

5.3 Polishing

Where etching is used to reveal very fine defects or structural irregularities (different welding zones for instance) careful polishing is necessary and the finer the polishing the better the definition.

5.4 Surface cleanliness

The surface to be etched should be free of dirt and grease which might influence the result of the examination.

6 Etching procedure

6.1 Method

Immerse the sample in an acid bath, which may be heated.

NOTE For large samples, it may be useful to pre-heat them to the temperature of the bath.

The volume of the bath should be adequate, at least of the order of 1 L of reagent per square decimetre of area of the sample. In addition, the bath should be sufficiently deep for the height of the liquid above the upper face of the sample to be at least 25 mm.

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

As an alternative to immersion and in the case of large samples or sections which cannot be immersed, swab the etching reagent over the surface to be examined. Ensure a uniform and constant distribution of the reagent over the surface.

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

6.2 Time of application

For each type of etching reagent, the time of application depends upon the temperature of the etching reagent, the steel grade, the surface condition and the physical condition of the steel sample and 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.

Table 1 — Approximate times of application for etching reagents

Etching reagents	Temperature of reagent	Approximate time of application
	°C	
Hydrochloric acid (concentrated solution, see **3.2**)	Cold	4 h
Hydrochloric acid (dilute solution, see **3.3**)	60 to 80	30 min
Sulphuric acid (dilute solution, see **3.4**)	a) Cold b) 60 to 80	a) 10 h b) 30 min
Sulphuric-hydrochloric acids solution (see **3.5**)	60 to 80	30 min
Nitric acid solution[a] (see **3.6**)	Cold	Up to 3 min
Nitric acid (dilute solution, see **3.7**)	Cold	2 min
Aqua regia acid solution (see **3.8**)	a) Cold b) Up to 40	a) 30 min b) A few min

NOTE It may be necessary to give consideration to hydrogen de-embrittlement of surface layers after etching.
[a] Several applications may be necessary to obtain the desired results.

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

For guidance, the approximate times of application are given in Table 1. The times given may be adjusted as necessary to suit individual applications.

7 Guidance for preservation of samples

In order to avoid subsequent corrosion of the surface of samples by see page 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, samples 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 samples 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 Report

The report shall include the following information:

a) the steel grade examined;

b) the cast number or any other identification number;

c) the position of the surface examined;

d) the type of etch;

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

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Publications referred to

BS 1134, *Method for the assessment of surface texture.*
BS 1134-2, *General information and guidance.*
BS M 37, *Method for the etch inspection of metallic material and components*[2)].

[2)] Referred to in the foreword only.

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