International Standard 4968

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Steel — Macrographic examination by sulphur print (Baumann method)

Acier — Examen macrographique par empreinte au soufre (méthode Baumann)

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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.

International Standard ISO 4968 was developed by Technical Committee ISO/TC 17, *Steel*, and was circulated to the member bodies in April 1978.

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

Australia	Hungary	Poland
Austria	India	Portugal
Belgium	Iran	Romania
Bulgaria	Ireland	South Africa, Rep. of
Canada	Italy	Spain
Chile	Japan	Sweden
Czechoslovakia	Korea, Dem. P. Rep. of	Switzerland
Denmark	Korea, Rep. of	Turkey
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Germany, F. R.	Norway	

No member body expressed disapproval of the document.

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Steel — Macrographic examination by sulphur print (Baumann method)

1 Scope and field of application

This International Standard specifies a method (Baumann) for the macrographic examination of steel by means of contact printing using silver salts and sulphuric acid.

The method is applicable to non-alloy and alloy steels[1] of which the sulphur content is less than 0,1 %.[2]

2 General

2.1 The sulphur print test is essentially a qualitative test. It is inadvisable to assess the sulphur content of a given steel merely on the basis of its sulphur print.

2.2 Experience shows that the degree of darkening of the photo-sensitive emulsion is not always in proportion to the quantity of sulphides present in the metal. Certain factors may influence the macrographic attack to a greater or lesser extent; as examples, the following may be quoted :

- the chemical composition of the steel : the presence of certain elements modifies the type and shape of the sulphides and consequently the appearance of the image obtained, for example concentrations of titanium greater than 0,1 % give prints which do not reveal sulphides;
- the surface condition of the sample : the presence of surface cold working may alter the image obtained;
- the sensitivity of the photographic paper.

2.3 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.

3 Principle and aim of the test

3.1 The aim of macrographic examination by sulphur printing is to detect, by printing on photo-sensitive paper[3] previously soaked in sulphuric acid solution, the position of areas containing sulphur inclusions found in the metal in various chemical forms and with various shapes : iron sulphide, manganese sulphides, mixed sulphides, oxy-sulphides etc.

3.2 The distribution of the sulphur-rich areas is revealed by the local release of hydrogen sulphide, causing darkening of the sensitive emulsion due to the chemical conversion of the silver halides to silver sulphide.

3.3 By examining the distribution and size of the sulphur inclusions detected by this process it is possible to make some asessment of the degree of uniformity of the metal from the section examined. Thus sulphur printing reveals chemical irregularities (segregations : for example those of a non-rimming free-cutting steel) and may reveal certain physical irregularities (for example cracks and porosity). Furthermore, sulphur printing may be used sometimes to distinguish rimming steel from killed steel and may also draw attention to certain areas where tests (for example, mechanical tests) or sampling for analysis may need to be carried out.

1) This method may also be applied to cast irons.

2) In the case of sulphur steels (sulphur content > 0,1 %) testing may be carried out, but with a very dilute solution of sulphuric acid.

3) The photographic paper may be replaced by a flat film. The positive and transparent prints obtained from the flat film may be used directly to produce negative proofs.

4 Products and reagents

4.1 Photographic paper

The sulphur print is made on the sensitive side of a sheet of photographic paper (or of a flat film) cut to suitable size.

In general, the paper used is thin matt paper with a thin layer of gelatine, for example bromide enlarging papers. The clear advantage of this type of paper is that there is less tendency to slip when it is applied.

4.2 Reagents

Sulphuric acid, commercial, dilute solution, having the following volumetric composition[1] :

H_2SO_4 (ϱ_{20} 1,84 g/ml) : 3 volumes
H_2O : 97 volumes

4.3 Fixing solution

A commercial fixing solution or a 15 to 20 % solution of sodium thiosulphate in water.

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

In the 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 Preparation of the test-piece surface is of prime importance in obtaining a correct sulphur print.

While rough machining, resulting in relatively coarse surfaces, may be sufficient in certain cases (routine inspection to reveal shrinkage holes, for example), it is generally required that the machining should be carried out as carefully as possible.

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 example

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

— to the use of unsuitable grinding wheels.

5.2.2 The main types of machining, generally used and leading to accurate prints which are more or less identical, are

— grinding, with or without preliminary machining;

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

5.2.3 A too smooth finish (mirror-type finish) makes it easier for the paper to slip on the test piece. In general, it is recommended that a surface finish with an R_a of at least 3,2 µm be obtained after machining.

6 Procedure

6.1 Immerse the photographic paper (4.1) for approximately 5 min in a sufficient volume of the sulphuric acid (4.2) at ambient temperature.

1) Other concentrations may be used if necessary.

6.2 After removing excess acid reagent, for example by draining, apply the sensitive side of the paper, still damp, to the surface to be examined, which should be clean and free from grease.

As an alternative to this procedure, if the piece is small, it may be applied to the paper which has been impregnated beforehand. Ensure that there is firm contact between the piece and the paper, without any slipping, throughout the test. If necessary, weigh the piece down in order to aid contact.

6.3 To ensure good contact, eliminate air bubbles and drops of liquid between the surface of the test piece and the sheet of paper, for example by means of a rubber roller.

6.4 Determine the time of application in advance from the available data concerning the metal to be examined (chemical composition, for example) and also by the type of irregularities to be detected. It may vary from a few seconds to a few minutes.

6.5 Remove the print and wash it in running water for approximately 10 min, after lightly rubbing it with a wad of wet cotton-wool.

Immerse the print for at least 10 min in the fixing solution (4.3), then wash in running water for at least 30 min, and dry.

7 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 result of the test.

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