TECHNICAL REPORT

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Sintered metal materials, excluding hardmetals — Metallographic preparation and examination

Matériaux métalliques frittés, à l'exclusion des métaux-durs — Préparation métallographique et examen

Reference number
ISO/TR 14321:1997(E)

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The main task of technical committees is to prepare International Standards, but in exceptional circumstances a technical committee may propose the publication of a Technical Report of one of the following types:

— type 1, when the required support cannot be obtained for the publication of an International Standard, despite repeated efforts;

— type 2, when the subject is still under technical development or where for any other reason there is the future but not immediate possibility of an agreement on an International Standard;

— type 3, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example).

Technical Reports of types 1 and 2 are subject to review within three years of publication, to decide whether they can be transformed into International Standards. Technical Reports of type 3 do not necessarily have to be reviewed until the data they provide are considered to be no longer valid or useful.

ISO/TR 14321, which is a Technical Report of type 2, was prepared by Technical Committee ISO/TC 119, *Powder metallurgy,* Subcommittee SC 3, *Sampling and testing methods for sintered metal materials (excluding hardmetals).*

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland
Internet central@iso.ch
X.400 c=ch; a=400net; p=iso; o=isocs; s=central

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Introduction

Examining a micrographic cross-section of a sintered metal using an optical microscope can be useful in evaluating the porosity and microstructure. The techniques for sampling and surface preparation by polishing and etching are similar to those used for the examination of solid material. Because of the existence of porosity in most sintered materials, special precautions must be taken during these operations. These precautions are intended to :

- avoid interference with pore size and shape, i.e. smearing of porosity with metal or abrasives, rounding or break-out of pore edges,

- ensure that the surface observed with the microscope is truly representative of the real texture and microstructure of the material.

This technical report is therefore more particularly concerned with the method recommended for correctly preparing the surface under examination. The methods for etching and observation which are conventional, are only indicated as a reminder.

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WARNING : This technical report does not purport to adress safety problems, if any, associated with its use. It is the responsibility of the user of this technical report to establish appropriate health and safety practices and determine the applicability of regulatory limitations prior to use.

1 Scope

The purpose of this technical report is to describe the optimum methods for sample taking, polishing and etching, with a view to preparing a micrographic surface which accurately represents the sintered metal part, when viewed using an optical microscope.

This technical report applies to all sintered metals including those submitted to core or surface heat treatment. It does not apply to hard metals, for which reference shall be made to ISO 4505 and ISO 4499.

It can be applied to metal materials containing a substantial amount of non-metallic components (e.g. friction materials and cermets). The special procedures required for these materials are given in 4.6.3.

NOTE — The methods described may also be used for the micrographic examination of unsintered powder compacts. In such a case, the procedures should be adapted to the material, e.g. : cleaning (removing pressing lubricant), impregnation, sectioning or cut-off. It is also possible to examine the morphology or the structure of loose compact powder (see 4.4).

2 Normative references

The following standards contain provisions, which through reference in the text, constitute provisions of this Technical Report. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this Technical Report are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 2738:—[1], *Permeable sintered metal materials — Determination of density, oil content and open porosity.*

ISO 4499:1978, *Hardmetals — Metallographic determination of porosity and uncombined carbon.*

ISO 4505:1978, *Hardmetals — Metallographic determination of porosity and uncombined carbon.*

ISO 13944:1997, *Lubricated metallic powder mixes — Determination of lubricant content — Modified Soxhlet extraction method.*

1) To be published. (Revision of ISO 2738:1987)

3 Principle

The methods recommended for obtaining representative micrographic surfaces are based on two main requirements :

- they shall not alter the pore morphology near the surface under examination, i.e. the area fraction of observed pores shall be the same as the true volume percent of porosity and the pores shall not be filled with smeared metal;

- they shall prevent the pores from interfering with the metallographic procedure, for example by trapping etchant, abrasives or the lubricants.

Among the various precautions to be taken to satisfy those requirements, the most important is the impregnation of the sample with a thermosetting polymer. Impregnation is usually essential, if micrographic examination is intended for the detection of imperfections, e.g. cracks, loss of cohesion, and degree of sintering, as judged by the number of original particle boundaries still present. It will not be effective when the pores are isolated from the surface of the sample.

4 Procedure

4.1 General

The variety of means and facilities for preparing metallographic surfaces, and the variety of requirements and the influence of the operator, make it impossible to specify strict rules. The rules described in this test method shall be considered as recommendations and practical guidelines ensuring that a representative surface is obtained.

4.2 Taking of samples

The number of samples and places on the surfaces to be examined are generally defined on the drawings, or by agreement between the parties concerned.

Other than in exceptional cases, examination will be made on a cross section of the part under examination. Sections taken parallel to the pressing direction are helpful in revealing pressing and sintering defects (neutral axis).

The first operation consists of cutting the part under examination with a wheel, so as to avoid modifying the metal structure by heating, oxidation or hardening. Velocity shall be about 40 m/s (about 50 s^{-1}) and the part shall be liberally sprayed with a liquid containing wetting agent and anti-oxidant.

For the selection of wheels, reference should be made to the recommendations of wheel suppliers.

As a guide, the following types of wheel may be used :

- for soft sintered materials, medium grain SiC, rubber or resinoid bonded wheels ;

- for medium hard or very hard sintered metals, fine grained alumina, resinoid bonded wheels ;

- low speed saws including diamond wafering saws.

Diamond wire cutters can be used in special cases (microparts).

NOTES

1 For some materials, which may react with the cooling liquid, dry cut-off or the use of an another more appropriate liquid is recommended.

2 If a zone near the surface is to be examined or the structure of a coating on the part under examination, the hardness of which may be very different from that of the core, is under scrutiny it is recommended that the cut off be done only after impregnation with plastic, (see 4.4.1). It is also possible to examine a coating layer sandwiched between the electrolytic deposit and the core material.

4.3 Cleaning of samples before impregnation

After cut-off under water or liquid, the part is usually saturated with water and additives. The part may also contain oil and other organic materials from prior processing. The organic matter is removed next. The cleaning of the sample shall be carried out by extraction using a Soxhlet apparatus and an appropriate solvent (acetone, toluol, or chlorinated hydrocarbon) in accordance with ISO 13944. Solvents must not affect any component of the microstructure. The sample is then dried.

Extraction shall be continued over a period of time long enough to remove all organic matter on the surface and in the pores. This may require 24 h. Alternatively, following the procedures in ISO 2738 may require less time. The sample is repeatedly extracted and dried until the weight loss between subsequent drying is less than 0,06 %.

4.4 Impregnation and mounting

Complete impregnation of the sample's (or part's) pores with a thermosetting plastic material is carried out in order to :

1 fill the pores completely, so as to prevent further penetration of liquid during polishing and etching;

2 strengthen the walls of those pores which are close to the surface, so as to prevent their deformation under the action of polishing abrasives. This deformation also results in progressive closing of the surface pores and hence an under-estimation of the apparent porosity as compared to the real volume fraction of porosity;

3 encapsulate the sample, which makes the handling, polishing and etching operations more practical.

Impregnation shall preferably be carried out under primary vacuum conditions with a fluid plastic material which is stable at the impregnation temperature, but may subsequently be polymerized into a relatively hard solid by appropriate curing. The pressure in the apparatus should not be so low as to cause boiling of the impregnant. For guidance, impregnation can be carried out using the apparatus shown in Figure 1. It is important that the mould be precoated with a mould release agent. The sample should be placed in a cylindrical plastic or silicon rubber mould and covered with liquid resin. A two-piece mould will facilitate later removal of the sample.

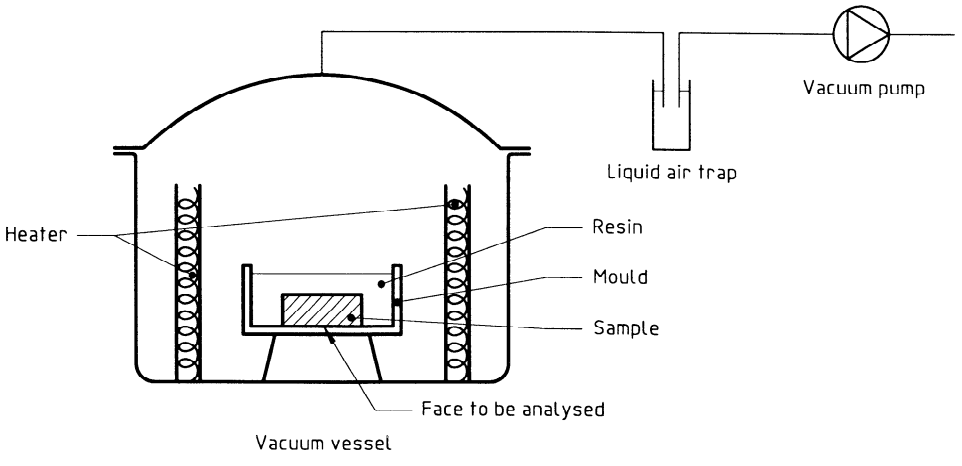


Figure 1 — An example of an apparatus for vacuum impregnation and mounting

The sample and holder are then placed in a vacuum chamber. Degassing and impregnation shall proceed for three hours at 80 °C, or at a temperature appropriate for the particular thermosetting resin. It is desired that the resin be of low viscosity, and not harden during the initial three-hour period. Examples of an appropriate thermosetting resin are phenolic resin or epoxy resin which may be impregnated at room temperature. The epoxy resin should not shrink excessively. Once returned to atmospheric pressure, the mould shall be cured in a forced air oven at 120 °C for 12 h. These conditions depend on the nature of the impregnation product. After removal from the mould the sample is ready for polishing.

NOTE — If the surface hardness of the sample is lower than its core hardness, there is risk of obtaining a convex examination surface after polishing. This can be avoided by including around the part under examination, some pieces of material having a hardness approximating to that of the part core. In all cases, flatness should be retained throughout the preparation sequence.

For metal powders, put about 2 cm^3 of liquid plastic and 1 cm^3 of metal powder into the mould and carefully stir the mixture to wet the metal powder. Then add the balance of the plastic to form a mould about 1,5 cm to 2 cm in thickness. Powders may be impregnated under vacuum, as with the other porous samples. Care shall be taken to avoid segregation of powder with the resin.

Figure 2 shows examples of micrographs of polished sintered bronze samples.

4.5 Prepolishing

Prepolishing shall be carried out by successive processes using abrasive papers of progressively finer particle sizes, water spraying and decreasing pressure. Any prepolishing scratches from the previous operation shall be removed at the next stage and the following prepolishing shall then be pursued over an equivalent period of time. Prepolishing shall preferably be performed using a disc rotating at a speed of 2,5 s^{-1} to 5 s^{-1} with a diameter of 200 mm to 250 mm. If hand prepolishing on a plate is used, the sample shall be rotated through 90° between each prepolishing stage so that new scratches cross the previous scratches at 90°.

After each prepolishing stage, the sample shall be carefully washed with water or a suitable solvent containing a detergent and, if possible, ultrasonically cleaned to remove abrasive grains and metal particles. After rinsing, the sample shall be dried in a clean air jet and submitted to the next prepolishing stage using a finer abrasive paper. The preferred succession of abrasive paper grain sizes is generally 180, 220, 400 and 600 (I to 0000 Emery). Metal powders are only prepolished on the 600 mesh paper. At the end of the prepolishing stage, the clean sample shall be rinsed and dried.

NOTE — Suppliers of metallographic equipment offer special lapping discs which greatly reduce the time of prepolishing.

4.6 Polishing

4.6.1 General

Final polishing is carried out using alumina for high or low hardness sintered materials and diamond for harder sintered materials. When the samples have been fully impregnated with resin, electrolytic polishing may be used. (see 4.6.4)

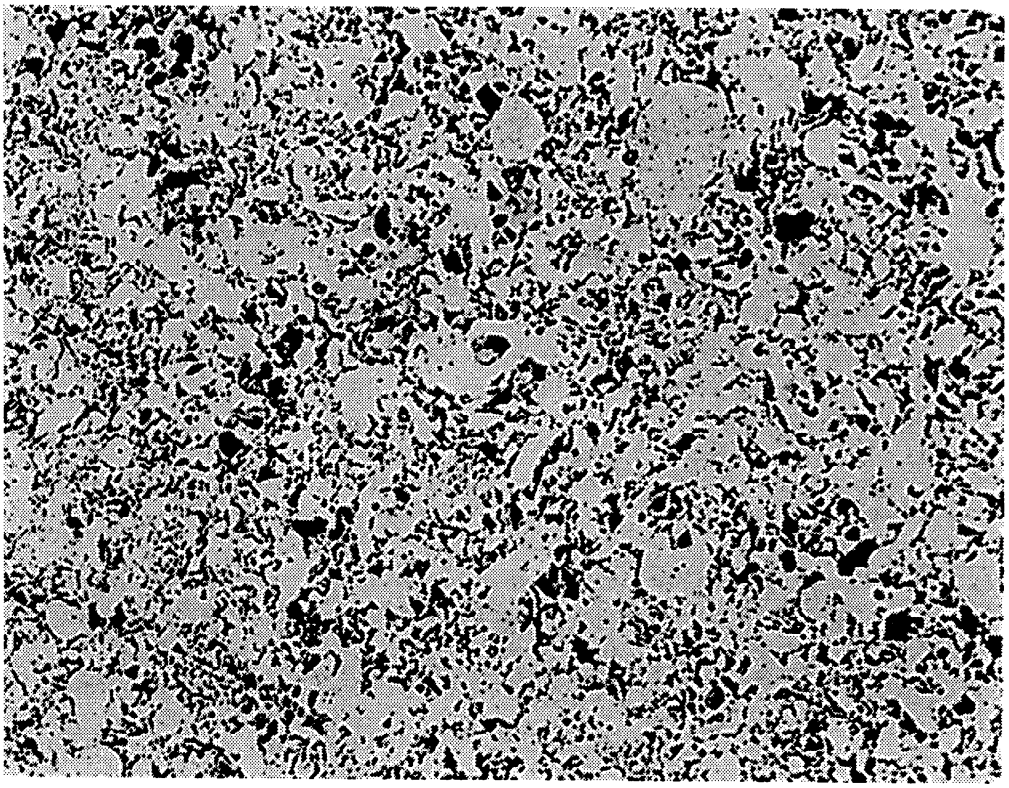
This is easily applied when material porosity is less than 10 %.

It is important that the polishing be carried out so that the area fraction of porosity viewed on the sample surface accurately reflects the true level of porosity in the sample. Improper polishing can lead to an exaggeration or under estimation of the area fraction of porosity. It is suggested that a sample of known uniform density and area fraction of porosity be used for initial trials. In this way, the approximate duration of polishing may be estimated.

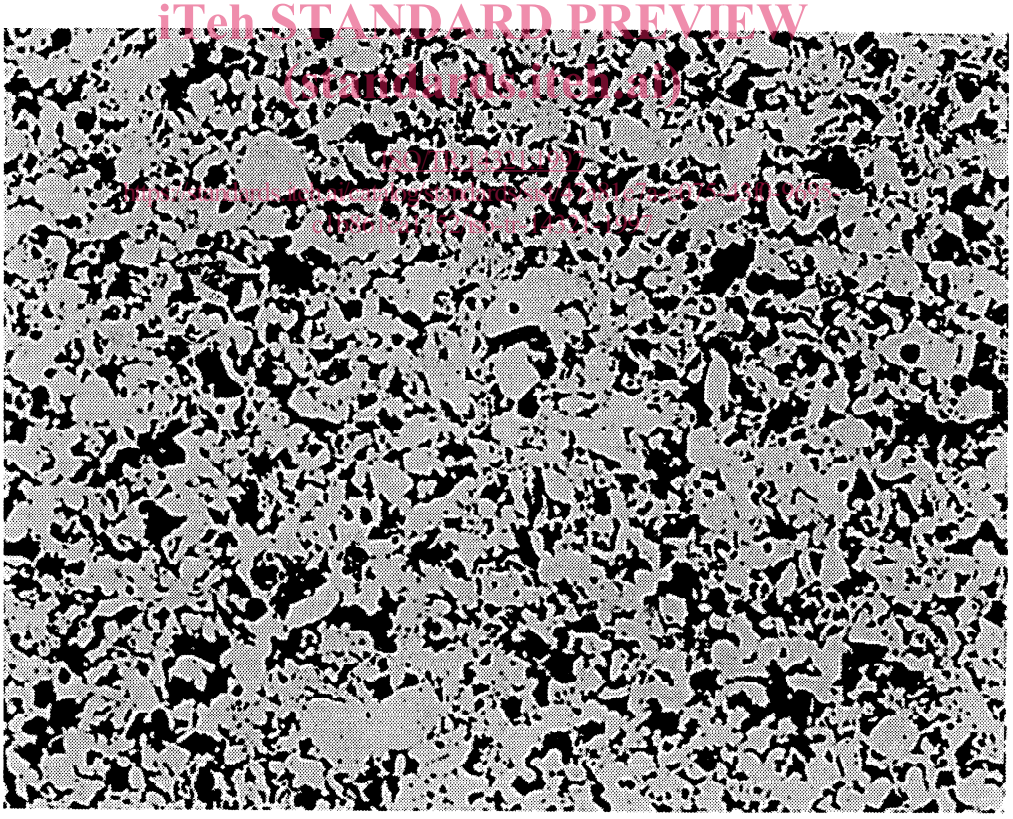
It is essential not to mix different grains of alumina or diamond on the discs. Discs shall also be reserved for given metal types and care shall be taken not to polish a metal with a disc having been used for a different metal.

Aluminium alloys may be polished on diamond or magnesia, but not on alumina, which causes deterioration of the surface of the specimen.

Friction materials and cermets may be polished on diamond.



a) Micrograph without impregnation — magnification ×140



b) Micrograph with plastic impregnation — magnification ×140

Figure 2 — Examples showing the influence of plastic impregnation on morphology and fraction area of porosity (bronze bearing — density 6,4 g/cm^3)