SLOVENSKI STANDARD
oSIST prEN ISO 4499-1:2018

01-junij-2018

Trdine - Metalografsko določevanje mikrostrukture - 1. del: Fotomikrografi in opisi (ISO/DIS 4499-1:2018)

Hardmetals - Metallographic determination of microstructure - Part 1: Photomicrographs and description (ISO/DIS 4499-1:2018)

Hartmetalle - Metallographische Bestimmung der Mikrostruktur - Teil 1: Gefügebilder und Beschreibung (ISO/DIS 4499-1:2018)

Métaux-durs - Détermination métallographique de la microstructure - Partie 1: Prises de vue photomicrographiques et description (ISO/DIS 4499-1:2018)

Ta slovenski standard je istoveten z: **prEN ISO 4499-1**

ICS:

77.040.99	Druge metode za preskušanje kovin	Other methods of testing of metals
77.160	Metalurgija prahov	Powder metallurgy

oSIST prEN ISO 4499-1:2018 **en,fr,de**

DRAFT INTERNATIONAL STANDARD

ISO/DIS 4499-1

ISO/TC **119**/SC **4** Secretariat: **DIN**

Voting begins on: **2018-04-19**

Voting terminates on: **2018-07-12**

Hardmetals — Metallographic determination of microstructure —

Part 1: Photomicrographs and description

Métaux-durs — Détermination métallographique de la microstructure —

Partie 1: Prises de vue photomicrographiques et description

ICS: 77.160; 77.040.99

This document is circulated as received from the committee secretariat.

ISO/CEN PARALLEL PROCESSING

Reference number
ISO/DIS 4499-1:2018(E)

COPYRIGHT PROTECTED DOCUMENT

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

Contents

Page

Foreword ..... **iv**
1 Scope **1**
2 Normative references **1**
3 Terms and definitions **1**
4 Apparatus **1**
5 Preparation of testpiece section **2**
6 Procedure **2**
6.1 Metallographic preparation 2
6.1.1 General 2
6.1.2 Sectioning 2
6.1.3 Mounting 2
6.1.4 Grinding 3
6.1.5 Lapping 3
6.1.6 Polishing 3
6.1.7 Cleaning 4
6.2 Etching 4
6.2.1 General 4
7 Test report **8**
Bibliography **9**

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 procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 119 *Powder metallurgy*, Subcommittee SC 4 *Sampling and testing methods for hardmetals*.

This third edition cancels and replaces the second edition (ISO 4499-1:2008), which has been technically revised.

The main changes compared to the previous edition are as follows:

— Clause 4.3 "Electron Back Scatter Diffraction (EBSD)" inserted.

A list of all parts in the ISO 4499- series can be found on the ISO website.

Hardmetals — Metallographic determination of microstructure —

Part 1:
Photomicrographs and description

1 Scope

This document specifies the methods of metallographic determination of the microstructure of hardmetals using photomicrographs.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3878:1983, *Hardmetals — Vickers hardness test*

ISO 4499-2, *Hardmetals — Metallographic determination of microstructure — Part 2: Measurement of WC grain size*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
***α*-phase**
tungsten carbide

3.2
***β*-phase**
binder phase (for example, based on Co, Ni, Fe)

3.3
***γ*-phase**
carbide having a cubic lattice (for example, TiC, Tac) which may contain other carbides (for example WC) in solid solution

4 Apparatus

4.1 Metallographic microscope, permitting observations at magnifications up to 1 500 ×.

4.2 Scanning electron microscope for magnification over 1 500 ×.

4.3 Electron Back Scatter Diffraction (EBSD).

4.4 Equipment for preparation of testpiece sections.

5 Preparation of testpiece section

The testpiece section shall be prepared as for metallographic examination, and the surface to be examined shall be free from grinding and polishing marks. Care shall be taken to avoid tearing-out of particles, which may lead to a wrong evaluation of microstructure.

NOTE There are several methods for preparation of hardmetal surfaces for metallographic examination. Firstly, careful coarse grinding is carried out to remove sufficient material to ensure that the true structure is revealed. After grinding with fine diamond-grit wheels, polishing is effected by using diamond paste or diamond powders, of progressively finer grain size down to 1 µm, on rigidly supported laps of, for example, thin plastic, thin felt or paper. A Good Practice Guide[[1]] has been written by the UK National Physical Laboratory that gives detailed guidelines on microstructural preparation. Key points from the Good Practice Guide are included in 6.1. ASTM has also prepared a thorough standard on sample preparation that merits study (ASTM B665).[2]

6 Procedure

6.1 Metallographic preparation

6.1.1 General

Good metallographic preparation is essential to produce a plane polished sample that when etched reveals the true microstructure of the hardmetal. Bad preparation can lead to grain tear-out, uneven etching and misleading detail in the microstructure which will affect any subsequent measurement.

Preparation and etching procedures may require toxic or dangerous chemicals. Personnel should be adequately trained, and facilities and precautions as laid down in the relevant safety guidelines in place at the laboratory concerned should be observed.

6.1.2 Sectioning

In many cases, the hardmetal sample may have to be sectioned into smaller samples for metallographic preparation. Diamond tooling is normally required to cut hardmetals into smaller samples. There is a wide variety of cut-off machines which use blades with diamond embedded on the rim of the disc. Fast cutting times are easily achieved. Wire-cutting machines, in which diamond is embedded in a wire, offer a cheaper method. Very fine slices can be obtained using this method but only at a relatively slow rate of cutting. A faster method is electro-discharge machining, but sufficiently fast machines are not generally suited to a metallographic preparation facility.

6.1.3 Mounting

Mounting of the sample into a resin has several advantages: it is more suitable for automatic preparation, the sample is easier to handle, and the specimen codes or identification can be inscribed or written onto the mount. Cold-setting and thermo-setting resins are available.

Thermo-setting powders (such as phenolic or diallyl phthalate powders) require a mounting press in which the sample is placed in a chamber, resin is then added, and is melted under pressure. The cycle time for these machines can be quite slow. The advantage is that hazardous chemicals are not used and the resin has an unlimited shelf life.

Cold-setting resins (such as epoxy, acrylic or polyester resins) do not require additional equipment apart from the moulds in which the sample is placed and the resin is poured on top. The resins normally consist of two parts, a monomer and a catalyst. Occasionally a third filler material is used; this may be to increase the hardness or to act as a conductive medium. Various types are available which set

in minutes or hours; the quicker-setting material reaches relatively high temperatures while the slow-setting resin remains cool. Mounting of samples in "quick-setting resins" is faster, whilst cold-setting resin is more economical for batches. However, a drawback is that these resins usually have an associated chemical risk, they have a limited shelf-life and they shall be stored in cool conditions.

A further consideration is that, following metallographic preparation of a flat surface, the hardmetal sample may have to be removed from the mount before etching or placing in the SEM. Cold-setting resins can be quite difficult to remove and may require grinding away from the sample.

6.1.4 Grinding

Whichever method is used to obtain a section of a hardmetal sample, it will have considerable surface and subsurface damage that shall be removed. Diamond grinding discs are available from all the main suppliers of metallographic equipment. These come in a range of diamond abrasive sizes and can produce very high rates of material removal from the surface of the sample. They should be used in order of decreasing abrasive size to both remove surface and subsurface damage and to obtain the plane section of the hardmetal sample which is to be polished. At each stage of grinding, the process should be continued until the surface damage (observed by optical examination without magnification) from the previous stage of preparation is removed, and then continued for the same amount of time to remove subsurface damage. Typically, for homogenous microstructures, at least 200 µm of material (see ISO 3878:1983) should be removed during the grinding process to obtain a section representative of the bulk microstructure. For materials with gradient structures, more care may be needed in controlling the amount removed.

The diamond grinding discs are available in several forms; metal bonded, resin bonded, wire mesh and plastic encapsulated. These vary considerably in cost and longevity of use, the resin bonded being the most durable and expensive.

6.1.5 Lapping

Occasionally, a lapping stage may be incorporated into the preparation procedure immediately after grinding. Lapping is normally carried out on a glass, metal, plastic or composite platen to which diamond abrasive is applied. The key features of lapping are the following:

— the production of a plane surface;

— an intermediate step between grinding and polishing;

— the removal of surface damage without imparting substantial further subsurface damage;

— relatively high rates of surface removal compared to the same size of diamond abrasive used on a polishing cloth.

Several suppliers provide laps of different compositions depending on the hardness of the material to be prepared. However, this stage of preparation may not be necessary if a coarser size of diamond abrasive is used on a polishing cloth as an additional stage. As with the grinding stages, the time of lapping should be sufficient to remove all the surface damage, and then continued for an equivalent length of time to remove subsurface damage.

6.1.6 Polishing

The polishing stages are normally carried out on a short nap or napless cloth[1)] with decreasing sizes of diamond abrasive. The abrasive sizes are typically 15 µm, 6 µm, 3 µm and 1 µm for each of the polishing stages. The abrasive may be applied as a suspension, aerosol, paste or other proprietary method.

1) Texmet and DP Pan are examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.