
Trdine - Metalografsko določevanje mikrostrukture - 3. del: Merjenje mikrostrukturnih značilnosti v trdinah na osnovi Ti (C, N) in WC/kubičnega karbida (ISO/DIS 4499-3:2014)

Hardmetals - Metallographic determination of microstructure - Part 3: Measurement of microstructural features in Ti (C, N) and WC/Cubic Carbide Based Hardmetals (ISO/DIS 4499-3:2014)

Hartmetalle - Metallographische Bestimmung der Mikrostruktur - Teil 3: Messung von mikrostrukturellen Merkmalen in Hartmetallen auf Basis von Ti (C, N) und WC/kubischem Carbid (ISO/DIS 4499-3:2014)

Métaux-durs - Détermination métallographique de la microstructure - Partie 3: Mesure des caractéristiques des microstructures des métaux-durs à base de carbures Ti (C, N) et WC/cubiques (ISO/DIS 4499-3:2014)

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Part 3:

Measurement of microstructural features in Ti (C, N) and WC/Cubic Carbide Based Hardmetals

*Métaux-durs — Détermination métallographique de la microstructure —**Partie 3: Mesure des caractéristiques des microstructures des métaux-durs à base de carbures Ti (C, N) et WC/cubiques*

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ISO/CEN PARALLEL PROCESSING

This draft has been developed within the International Organization for Standardization (ISO), and processed under the **ISO lead** mode of collaboration as defined in the Vienna Agreement.

This draft is hereby submitted to the ISO member bodies and to the CEN member bodies for a parallel five month enquiry.

Should this draft be accepted, a final draft, established on the basis of comments received, will be submitted to a parallel two-month approval vote in ISO and formal vote in CEN.

To expedite distribution, this document is circulated as received from the committee secretariat. ISO Central Secretariat work of editing and text composition will be undertaken at publication stage.



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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 4499-3 was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*, Subcommittee SC 4, *Sampling and testing methods for hardmetals*.

ISO 4499 consists of the following parts, under the general title *Hardmetals — Metallographic determination of microstructure*:

- *Part 1: Photomicrographs and description*
- *Part 2: Measurement of WC grain size*
- *Part 3: Measurement of microstructural features in Ti(C,N) and WC/Cubic Carbide Based Hardmetals*
- *Part 4: Characterisation of Porosity, Carbon Defects and Eta-phase content*
- *Part 5: Characterisation and measurement of miscellaneous microstructural features*

Introduction

This part of ISO 4499 essentially covers the following topics:

- materials types and phases to be measured including:
 - a) Ti(C, N) cermets
 - b) WC/Cubic carbide hardmetals
- preparation methods to highlight differences between conventional WC/Co hardmetals and materials containing cubic phases;
- linear analysis techniques to acquire sufficient statistically meaningful data for phase quantification;
- analysis method to calculate representative average values;
- reporting to comply with modern quality requirements.

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Hardmetals — Metallographic determination of microstructure — Part 3: Measurement of microstructural features in Ti(C,N) and WC/Cubic Carbide Based Hardmetals

1 Scope

This part of ISO 4499 gives guidelines for the measurement of microstructural features in Ti(C,N) based hardmetals and WC/Co hardmetals that contain additional cubic carbides by metallographic techniques only using optical or electron microscopy. It is intended for sintered hardmetals (also called cemented carbides or cermets) containing primarily inorganic carbides and nitrides as the hard phase. It is also intended for measuring the phase size and distribution by the linear intercept technique.

2 Normative references

The following referenced documents are indispensable for the application 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 4499-1, *Hardmetals — Metallographic determination of microstructure — Part 1: Photomicrographs and description*

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

ASTM B 657, *Guide for Metallographic Identification of Microstructure in Cemented Carbides*

ASTM B 665, *Standard Guide for Metallographic Sample Preparation of Cemented Tungsten Carbides*

ASTM E 112, *Standard Test Methods for Determining Average Grain Size*

3 Terms and definitions

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

3.1

nano

with carbonitride or, respectively, cubic carbide phase size < 0,2 µm

Note 1 to entry: Measured by the mean-linear-intercept method described in ISO 4499-2.

3.2

ultrafine

with carbonitride or, respectively, cubic carbide phase size 0,2 µm to 0,5 µm

Note 1 to entry: Measured by the mean-linear-intercept method described in ISO 4499-2.

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3.3**submicron**

with carbonitride or, respectively, cubic carbide phase size 0,5 μm to 0,8 μm

Note 1 to entry: Measured by the mean-linear-intercept method described in ISO 4499-2.

3.4**fine**

with carbonitride or, respectively, cubic carbide phase size 0,8 μm to 1,3 μm

Note 1 to entry: Measured by the mean-linear-intercept method described in ISO 4499-2.

3.5**medium**

with carbonitride or, respectively, cubic carbide phase size 1,3 μm to 2,5 μm

Note 1 to entry: Measured by the mean-linear-intercept method described in ISO 4499-2.

3.6**coarse**

with carbonitride or, respectively, cubic carbide phase size 2,5 μm to 6,0 μm

Note 1 to entry: Measured by the mean-linear-intercept method described in ISO 4499-2.

3.7**extra coarse**

with carbonitride or, respectively, cubic carbide phase size $> 6,0 \mu\text{m}$

Note 1 to entry: Measured by the mean-linear-intercept method described in ISO 4499-2.

3.8**Ti(C, N) cermets**

a TiCN-based cermet contains 3-30 weight % of a binder phase mainly composed of Co and/or Ni, but also may include Mo; the balance being substantially a hard phase and a few minor impurities

Note 1 to entry: The hard phase is mainly composed of titanium carbide, nitride and/or carbonitride, but may also include carbonitrides of (Ti,Ta), (Ti,W) or (Ti,Ta, W).

Note 2 to entry: These materials typically contain hard phases that can have grains with a core/rim structure.

3.9**WC/Cubic carbide hardmetals**

hexagonal WC based hardmetals containing substantial amounts of a carbide having a cubic lattice, such as, for example TiC or TaC and which can contain W in solid solution

Note 1 to entry: These materials typically contain hard phases that may have grains with a core/rim structure.

4 Symbols and units

For the purposes of this document, the following symbols, abbreviations and units apply.

A	is the area, in square millimetres (mm) ²
ECD	is the equivalent circle diameter, in micrometres (µm), of a specified phase
L	is the total line length, in millimetres (mm), in a specified phase
l_i	is the measured length of individual intercepts, in micrometres (µm), in a specified phase
$\sum l_i$	is the sum of the measured length of each individual intercept
l_x	is the arithmetic mean linear intercept in phase x, in micrometres (µm)
N	is the number of grain boundaries traversed in or between specified phases
n	is the number of WC, carbonitride or cubic carbide grains intercepted
m	is the magnification
m_{\max}	is the maximum magnification
m_{\min}	is the minimum magnification

5 Principle

This part of ISO 4499 addresses the issue of good practice for the measurement of a mean value for the hard phase and binder phase size in hardmetals other than straight WC/Co. It recommends the use of a linear intercept technique for obtaining data on feature sizes. The measurements are to be made using good practice for the preparation of suitable microstructures for examination outlined in ISO 4499-1.

Methods of metallographic preparation and etching techniques are as important as the phase size measurement method (see also ASTM B 657, ASTM B 665, [1-2]). Basic methods are described in ISO 4499-1. Further relevant information is given in clause 8. The principal types of hardmetal considered are those that contain cubic carbides as well as WC and those that are based on TiC or Ti(C,N) [3-5]. A cubic carbide phase is defined as a carbide having a cubic lattice, such as, for example, TiC or TaC, and which usually also contains W in solid solution after sintering. These materials typically contain hard phases that have grains with a core/rim structure. Guidelines to measure these internal details are included in an Appendix Case Study in this part of ISO 4499.

The most direct way to measure the phase size is to polish and etch a cross-section of the microstructure and then to use quantitative metallographic techniques to measure a mean value for the feature size, either by area counting or by linear intercept techniques.

There are three ways by which the mean size by number of the various phases can be defined:

- by length (of a line across a 2D section of a phase);
- by area (of 2D sections of phase regions);
- by volume (of individual phase regions).

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A number average is obtained by counting each measurement of the parameter of interest (length, area or volume) and dividing the total value of the parameter (length, area or volume) by the number of this parameter counted.

The values for phase size most used to date have been based on a length parameter. This can be obtained in several ways, for example, by parallel lines or circles as described in ASTM E112:

- by linear intercept, called the Heyn method, from a straight line drawn across the structure;
- by equivalent circle diameter (see ISO 4499-2), this is obtained by measuring hard phase grain areas and then taking the diameter of a circle of equivalent area.

6 Apparatus

6.1 Metallographic optical microscope, or other suitable equipment permitting observations and measurements on a screen up to the required magnification.

6.2 Scanning electron microscope, permitting observations and measurements of features too small to be resolved with an optical microscope.

6.3 Equipment for preparation of test-piece sections.

Phase size measurements are obtained from images of the microstructure. ISO 4499-1, ASTM B 657 and ASTM B 665 should be consulted for best practice in the preparation of surfaces for imaging.

Structural images are usually generated by either optical microscopy or Scanning Electron Microscopy (SEM). For accurate measurements it is better to use scanning electron microscopic images. Even in coarse grained materials the imaged surface cuts through a substantial number of the corners of grains giving a proportion of small intercepts that can only be measured accurately using the scanning electron microscope.

Measurements of intercept lengths from the acquired images can be obtained manually or semi-automatically using image analysis. Automatic image analysis can be used in some circumstances when the images are fairly coarse and good contrast can be obtained but for many materials, especially those with very fine grain sizes, good images are difficult to acquire and are generally not amenable to automatic analysis.

For the ultrafine and nano structural materials, good images are particularly difficult to acquire using conventional scanning electron microscopes with tungsten filament electron sources. It is recommended for these materials that a field emission SEM is used. These systems give significantly higher resolution images, sufficient to measure materials with mean intercept sizes of about 0,1 μm – 0,2 μm . For materials with ever smaller grain sizes it may be necessary to use Transmission Electron Microscopy (TEM). However, the problems of sampling and specimen preparation are particularly severe. Careful specimen preparation for good images is vital for these materials and often a combination of etching methods is helpful (see ISO 4499-1).

7 Calibration

To give reliable quantitative measurements images shall be calibrated against a stage micrometer or scale traceable to a National Reference Standard.

For images obtained from an optical microscope, an image of the calibration graticule shall also be obtained using the same objectives (and internal magnification step changers or zoom position) and illuminating technique. The microscope shall be set up for Köhler illumination to obtain the maximum resolution, see [6].

For images obtained from a scanning electron microscope, images of the graticule should be obtained under the same conditions (accelerating kV, working distance, illumination aperture) as those used for the hardmetal.