SIST

SLOVENSKI STANDARD
oSIST prEN ISO 4499-2:2018

01-september-2018

Trdine - Metalografsko določevanje mikrostrukture - 2. del: Merjenje velikosti zrn volframovega karbida (WC) (ISO/DIS 4499-2:2018)

Hardmetals - Metallographic determination of microstructure - Part 2: Measurement of WC grain size (ISO/DIS 4499-2:2018)

Hartmetalle - Metallographische Bestimmung der Mikrostruktur - Teil 2: Messung der WC Korngröße (ISO/DIS 4499-2:2018)

Métaux-durs - Détermination métallographique de la microstructure - Partie 2: Mesurage de la taille des grains de WC (ISO/DIS 4499-2:2018)

Ta slovenski standard je istoveten z: **prEN ISO 4499-2**

ICS:

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

oSIST prEN ISO 4499-2:2018 **en,fr,de**

DRAFT INTERNATIONAL STANDARD
ISO/DIS 4499-2

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

Voting begins on: **2018-06-21** Voting terminates on: **2018-09-13**

Hardmetals — Metallographic determination of microstructure —

Part 2: Measurement of WC grain size

Métaux-durs — Détermination métallographique de la microstructure —

Partie 2: Mesurage de la taille des grains de WC

ICS: 77.160; 77.040.99

This document is circulated as received from the committee secretariat.

ISO/CEN PARALLEL PROCESSING

Reference number
ISO/DIS 4499-2:2018(E)

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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.

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 4499-2:2008), which has been technically revised.

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

— Clause 5, 2nd paragraph "Electron Back Scatter Diffraction (EBSD)" inserted;

— Clause 7.2.1, list revised;

— Clause 7.3.3, Table 1, row "Electron Back Scatter Diffraction" added and in row "Scanning electron microscope" the value for the "Minimum visible intercept length" corrected from 200 nm into 400 nm.

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

Hardmetals — Metallographic determination of microstructure —

Part 2: Measurement of WC grain size

1 Scope

This document gives guidelines for the measurement of hardmetal grain size by metallographic techniques only using optical or electron microscopy. It is intended for sintered WC/Co hardmetals (also called cemented carbides or cermets) containing primarily WC (de: Wolframcarbid, en: tungsten carbide) as the hard phase. It is also intended for measuring the grain size and distribution by the linear-intercept technique.

This document essentially covers four main topics:

— calibration of microscopes, to underpin the accuracy of measurements;

— linear analysis techniques, to acquire sufficient statistically meaningful data;

— analysis methods, to calculate representative average values;

— reporting, to comply with modern quality requirements.

This document is supported by a measurement case study to illustrate the recommended techniques (see Annex A).

This document is not intended for the following.

— Measurements of size distribution.

— Recommendations on shape measurements. Further research is needed before recommendations for shape measurement can be given.

Measurements of coercivity are sometimes used for grain-size measurement, but this current guide is concerned only with a metallographic measurement method. It is also written for sintered hardmetals and not for characterising powders. However, the method could, in principle, be used for measuring the average size of powders that are suitably mounted and sectioned.

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 3369, *Impermeable sintered metal materials and hardmetals — Determination of density*

ISO 3738-1, *Hardmetals — Rockwell hardness test (scale A) — Part 1: Test method*

ISO 3738-2, *Hardmetals — Rockwell hardness test (scale A) — Part 2: Preparation and calibration of standard test blocks*

ISO 3878, *Hardmetals — Vickers hardness test*

ISO 4489:1978, *Sintered hardmetals — Sampling and testing*

ISO 4499-1, *Hardmetals — Metallographic determination of microstructure — Part 1: Photomicrographs and description*

ISO 4499-4, *Hardmetals — Metallographic determination of microstructure — Part 4: Characterisation of porosity, carbon defects and eta-phase content*

3 Terms, definitions, abbreviations, symbols and units

3.1 General

A very wide range of terms are used to describe powders or sintered hardmetals of different sizes. For example, the following have been used in a variety of publications and reports.

Extra coarse	Fine	Microfine
Coarse	Very fine	Micrograin
Coarse/Medium	Ultra fine	Nanophase
Medium	Extra fine	Nanograin
Medium/Fine	Submicron	Super fine

None of these terms have commonly agreed or well-defined size ranges among users and producers of powders or sintered products.

Consequently, following discussion in the hardmetal community, the following terms for the sizes defined in 3.2 are recommended.

The uncertainty associated with the measurement of linear-intercept grain size is about 10 %, if typically (200 to 300) grains are counted. Thus, measurements on or close to the class boundaries should be treated carefully. It is recommended that measurements that fall within 10 % of any of the class boundaries should be classed as follows:

EXAMPLE

0,19 µm as Nano/Ultrafine	0,21 µm as Ultrafine/Nano
0,75 µm as Submicron/Fine	0,85 µm as Fine/Submicron
1,29 µm as Fine/Medium	1,31 µm as Medium/Fine
2,4 µm as Medium/Coarse	2,6 µm as Coarse/Medium

3.2 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

3.2.1
nano
with WC grain size < 0,2 µm

Note 1 to entry: Measured by the mean-linear-intercept method described in this document.

3.2.2
ultrafine
with WC grain size 0,2 µm to 0,5 µm

Note 1 to entry: Measured by the mean-linear-intercept method described in this document.

3.2.3
submicron
with WC grain size 0,5 µm to 0,8 µm

Note 1 to entry: Measured by the mean-linear-intercept method described in this document.

3.2.4
fine
with WC grain size 0,8 µm to 1,3 µm

Note 1 to entry: Measured by the mean-linear-intercept method described in this document.

3.2.5
medium
with WC grain size 1,3 µm to 2,5 µm

Note 1 to entry: Measured by the mean-linear-intercept method described in this document.

3.2.6
coarse
with WC grain size 2,5 µm to 6,0 µm

Note 1 to entry: Measured by the mean-linear-intercept method described in this document.

3.2.7
extra coarse
with WC grain size > 6,0 µm

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

3.3 Symbols, abbreviations and units

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

A	is the area, in square millimetres $(\text{mm})^2$
d_{wc}	is the arithmetic mean linear intercept of WC grains, in micrometres (µm)
ECD	is the equivalent circle diameter, in millimetres (mm)
L	is the line length, in millimetres (mm)
LI	is the arithmetic mean-linear-intercept distance, in micrometres (µm)
l_i	is the measured length of individual intercepts, in micrometres (µm)
$\sum l_i$	is the sum of the measured length of each individual intercept
N	is the number of grain boundaries traversed
n	is the number of WC grains intercepted
m	is the magnification

m_{max} is the maximum magnification

m_{min} is the minimum magnification

s_m is the measured size, in millimetres (mm)

s_a is the actual size, in millimetres (mm)

4 General information

This part of ISO 4499 addresses the issue of good practice for the measurement of a mean value for WC grain size. It recommends the use of a linear-intercept technique for obtaining data. The measurements shall be made using good practice for the preparation of suitable microstructures for examination outlined in ISO 4499-1.

The properties and performance of hardmetals are directly dependent on the microstructure developed during manufacture, which in turn is controlled by the character of the starting powder batch. Understanding the microstructure is the key to controlling or improving properties, and therefore the measurement of microstructural features, particularly grain size and size distribution, is of paramount importance.

Methods of metallographic preparation and etching techniques are as important as the grain-size measurement method (see [1] to [4]), and are included in ISO 4499-1. The principal type of hardmetal considered is WC with a Co binder. However, the procedure can be used for hardmetals that contain cubic carbides or which are based on TiC or Ti(C, N).

The most direct way to measure the WC grain 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 grain size, either by area counting or by linear-intercept techniques.

There are three ways by which the mean size by number of the WC grains can be defined:

— by length (of a line across a 2D section of a grain);

— by area (of 2D sections of grains);

— by volume (of individual grains).

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 value most used to date has been a length parameter. This can be obtained in several ways, for example, by parallel lines or circles as described in ASTM E112[12]:

— by linear intercept, called the Heyn method, from a straight line drawn across the structure;

— by the equivalent circle diameter; this is obtained by measuring grain areas and then taking the diameter of a circle of equivalent area. It is possible, for equiaxed grains, to convert an equivalent circle diameter (ECD) grain size to a linear intercept (LI) value using Equation (1).

$$\mathrm{LI} = \sqrt{A} = \sqrt{\pi / 4}\ \mathrm{ECD} \qquad (1)$$

Thus ECD = 1,13 LI.

This expression is discussed in References [1] and [5].

An additional method is that established by Jefferies, where the number of grains per unit area can be counted. This can, if required, be converted to an equivalent circle diameter.

It shall be noted that

— point/area counting provides no information on distribution, and

— the Jefferies method is not intended for use on multiphase materials such as hardmetals.

The recommended technique for measurement of hardmetal grain size is the linear-intercept method.

5 Apparatus

Grain-size measurements are obtained from images of the microstructure. ISO 4499-1, ASTM B657[10] and ASTM B665[11] should be consulted for best practice in the preparation of surfaces for imaging.

Hardmetal structural images are usually generated by either optical microscopy, scanning electron microscopy (SEM) or Electron Back Scatter Diffraction (EBSD). 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 semiautomatically 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 grades, good images are particularly difficult to acquire using conventional scanning electron microscopes with tungsten-filament electron sources. For these materials, it is recommended that a field emission SEM (FESEM) be used. These systems give significantly higher resolution images, sufficient to measure materials with mean intercept sizes of about 0,1 µm to 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 (see [7]). Careful specimen preparation for good images is vital for these materials, and often a combination of etching methods is helpful (see ISO 4499-1).

6 Calibration

To give reliable quantitative measurements, images shall be calibrated against a stage micrometer or scale traceable to a National Reference Standard. The most commonly used stage micrometers for SEMs are the SIRA grids. These are ruled lines which form a grid and are available with 19,7 lines per mm and 2 160 lines per mm. However, these shall also be calibrated and certified as being 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 [8]).

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.

