

# SVENSK STANDARD

## SS-EN ISO 4499-3:2016

Fastställt/Approved: 2016-03-22  
Publicerad/Published: 2016-03-29  
Utgåva/Edition: 1  
Språk/Language: engelska/English  
ICS: 77.040.99; 77.160

---

### **Hårdmetall – Metallografisk bestämning av mikrostruktur – Del 3: Mätning av mikrostrukturella egenskaper i Ti (C, N)- och WC/kubisk karbid-baserade hårdmetaller (ISO 4499-3:2016)**

### **Hardmetals – Metallographic determination of microstructure – Part 3: Measurement of microstructural features in Ti (C, N) and WC/cubic carbide based hardmetals (ISO 4499-3:2016)**

This preview is downloaded from [www.sis.se](http://www.sis.se). Buy the entire standard via <https://www.sis.se/std-8019625>

# Standarder får världen att fungera

*SIS (Swedish Standards Institute) är en fristående ideell förening med medlemmar från både privat och offentlig sektor. Vi är en del av det europeiska och globala nätverk som utarbetar internationella standarder. Standarder är dokumenterad kunskap utvecklad av framstående aktörer inom industri, näringsliv och samhälle och befrämjar handel över gränser, bidrar till att processer och produkter blir säkrare samt effektiviserar din verksamhet.*

## Delta och påverka

Som medlem i SIS har du möjlighet att påverka framtida standarder inom ditt område på nationell, europeisk och global nivå. Du får samtidigt tillgång till tidig information om utvecklingen inom din bransch.

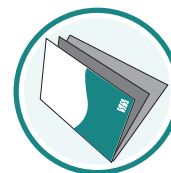
## Ta del av det färdiga arbetet

Vi erbjuder våra kunder allt som rör standarder och deras tillämpning. Hos oss kan du köpa alla publikationer du behöver – allt från enskilda standarder, tekniska rapporter och standardpaket till handböcker och onlinetjänster. Genom vår webbtjänst e-nav får du tillgång till ett lättnavigerat bibliotek där alla standarder som är aktuella för ditt företag finns tillgängliga. Standarder och handböcker är källor till kunskap. Vi säljer dem.

## Utveckla din kompetens och lyckas bättre i ditt arbete

Hos SIS kan du gå öppna eller företagsinterna utbildningar kring innehåll och tillämpning av standarder. Genom vår närhet till den internationella utvecklingen och ISO får du rätt kunskap i rätt tid, direkt från källan. Med vår kunskap om standarders möjligheter hjälper vi våra kunder att skapa verklig nytta och lönsamhet i sina verksamheter.

**Vill du veta mer om SIS eller hur standarder kan effektivisera din verksamhet är du välkommen in på [www.sis.se](http://www.sis.se) eller ta kontakt med oss på tel 08-555 523 00.**



# Standards make the world go round

*SIS (Swedish Standards Institute) is an independent non-profit organisation with members from both the private and public sectors. We are part of the European and global network that draws up international standards. Standards consist of documented knowledge developed by prominent actors within the industry, business world and society. They promote cross-border trade, they help to make processes and products safer and they streamline your organisation.*

## Take part and have influence

As a member of SIS you will have the possibility to participate in standardization activities on national, European and global level. The membership in SIS will give you the opportunity to influence future standards and gain access to early stage information about developments within your field.

## Get to know the finished work

We offer our customers everything in connection with standards and their application. You can purchase all the publications you need from us - everything from individual standards, technical reports and standard packages through to manuals and online services. Our web service e-nav gives you access to an easy-to-navigate library where all standards that are relevant to your company are available. Standards and manuals are sources of knowledge. We sell them.

## Increase understanding and improve perception

With SIS you can undergo either shared or in-house training in the content and application of standards. Thanks to our proximity to international development and ISO you receive the right knowledge at the right time, direct from the source. With our knowledge about the potential of standards, we assist our customers in creating tangible benefit and profitability in their organisations.

**If you want to know more about SIS, or how standards can streamline your organisation, please visit [www.sis.se](http://www.sis.se) or contact us on phone +46 (0)8-555 523 00**



Europastandarden EN ISO 4499-3:2016 gäller som svensk standard. Detta dokument innehåller den officiella engelska versionen av EN ISO 4499-3:2016.

The European Standard EN ISO 4499-3:2016 has the status of a Swedish Standard. This document contains the official English version of EN ISO 4499-3:2016.

**Förhållandet till övriga delar under samma huvudtitel - Utdrag ur Förord i ISO 4499-3:2016/  
Relations to other parts under the same general title - Extract from the Foreword of ISO 4499-3:2016**

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*

© Copyright/Upphovsrätten till denna produkt tillhör SIS, Swedish Standards Institute, Stockholm, Sverige. Användningen av denna produkt regleras av slutanvändarlicensen som återfinns i denna produkt, se standardens sista sidor.

© Copyright SIS, Swedish Standards Institute, Stockholm, Sweden. All rights reserved. The use of this product is governed by the end-user licence for this product. You will find the licence in the end of this document.

*Upplysningar om sakinnehållet i standarden lämnas av SIS, Swedish Standards Institute, telefon 08-555 520 00. Standarder kan beställas hos SIS Förlag AB som även lämnar allmänna upplysningar om svensk och utländsk standard.*

*Information about the content of the standard is available from the Swedish Standards Institute (SIS), telephone +46 8 555 520 00. Standards may be ordered from SIS Förlag AB, who can also provide general information about Swedish and foreign standards.*

Denna standard är framtagen av kommittén för Pulvermetallurgi, SIS/TK 133.

Har du synpunkter på innehållet i den här standarden, vill du delta i ett kommande revideringsarbete eller vara med och ta fram andra standarder inom området? Gå in på [www.sis.se](http://www.sis.se) - där hittar du mer information.



EUROPEAN STANDARD

EN ISO 4499-3

NORME EUROPÉENNE

EUROPÄISCHE NORM

March 2016

ICS 77.040.99; 77.160

English Version

Hardmetals - Metallographic determination of  
microstructure - Part 3: Measurement of microstructural  
features in Ti (C, N) and WC/cubic carbide based  
hardmetals (ISO 4499-3:2016)

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 4499-3:2016)

Hartmetalle - Metallographische Bestimmung der  
Mikrostruktur - Teil 3: Messung von  
mikrostrukturellen Merkmalen in Hartmetallen auf  
Basis von Ti (C, N) und WC/kubischem Carbid (ISO  
4499-3:2016)

This European Standard was approved by CEN on 4 February 2016.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels

# Contents

Page

<b>European foreword</b> .....	<b>vi</b>
<b>Introduction</b> .....	<b>v</b>
<b>1 Scope</b> .....	<b>1</b>
<b>2 Normative references</b> .....	<b>1</b>
<b>3 Terms and definitions</b> .....	<b>1</b>
<b>4 Symbols and units</b> .....	<b>2</b>
<b>5 Principle</b> .....	<b>3</b>
<b>6 Apparatus</b> .....	<b>3</b>
<b>7 Calibration</b> .....	<b>4</b>
<b>8 Preparation of test samples</b> .....	<b>4</b>
8.1 Metallographic preparation.....	4
8.2 Ti(C, N) based hardmetals – cermets.....	4
8.3 WC/Cubic carbide based hardmetals.....	8
<b>9 Procedure for characterisation of structures</b> .....	<b>20</b>
9.1 Sampling of images of structure.....	20
9.1.1 General.....	20
9.1.2 Representative selection.....	20
9.1.3 Determination of homogeneity of hard phase sizes.....	20
9.1.4 Inhomogeneous materials.....	21
9.2 Phase size measurement.....	21
9.2.1 General.....	21
9.2.2 Phase measurement by intercepts.....	21
<b>10 Uncertainty of measurement</b> .....	<b>23</b>
<b>11 Test report</b> .....	<b>23</b>
<b>Bibliography</b> .....	<b>25</b>

## European foreword

This document (EN ISO 4499-3:2016) has been prepared by Technical Committee ISO/TC 119 "Powder metallurgy".

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by September 2016, and conflicting national standards shall be withdrawn at the latest by September 2016.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

## Endorsement notice

The text of ISO 4499-3:2016 has been approved by CEN as EN ISO 4499-3:2016 without any modification.

## Introduction

This part of ISO 4499 essentially covers the following topics:

- materials types and phases to be measured including the following:
  - Ti(C, N) cermets;
  - 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.



# 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 documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 4499-1:2008, *Hardmetals — Metallographic determination of microstructure — Part 1: Photomicrographs and description*

ISO 4499-2:2008, *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.

#### 3.1

##### **nano**

with carbonitride or cubic carbide phase size  $<0,2 \mu\text{m}$ , respectively

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

#### 3.2

##### **ultrafine**

with carbonitride or cubic carbide phase size  $0,2 \mu\text{m}$  to  $0,5 \mu\text{m}$ , respectively

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

#### 3.3

##### **submicron**

with carbonitride or cubic carbide phase size  $0,5 \mu\text{m}$  to  $0,8 \mu\text{m}$ , respectively

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

**3.4**

**fine**

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

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

**3.5**

**medium**

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

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

**3.6**

**coarse**

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

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

**3.7**

**extra coarse**

with carbonitride or cubic carbide phase size >6,0 µm, respectively

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

**3.8**

**Ti(C, N) cermets**

TiCN-based cermet contains 3 to 30 weight % of a binder phase mainly composed of Co and/or Ni, but may also include Mo

Note 1 to entry: The balance being substantially a hard phase and a few minor impurities.

Note 2 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 3 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.

Note 2 to entry: See [Table 1](#).

**3.10**

**phase region**

single constituent of the hardmetal like WC, cubic carbide or binder

**4 Symbols and units**

*A* area, in square millimetres (mm<sup>2</sup>)

ECD Equivalent Circle Diameter of a specified phase, in micrometres (µm)

*L* total line length in a specified phase, in millimetres (mm)

*l<sub>i</sub>* measured length of individual intercepts in a specified phase, in micrometres (µm)

$\sum l_i$  sum of the measured length of each individual intercept

$l_x$	arithmetic mean linear intercept in phase $x$ , in micrometres ( $\mu\text{m}$ )
$N$	number of grain boundaries traversed in or between specified phases
$n$	number of WC, carbonitride or cubic carbide grains intercepted
$m$	magnification
$m_{\text{max}}$	maximum magnification
$m_{\text{min}}$	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, Reference [1] and Reference [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).<sup>[3][4][5]</sup> 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 ISO 4499-2:2008, Annex A.

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.

The following 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).

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 the following 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.