

BS EN ISO 4499-4:2016



BSI Standards Publication

# Hardmetals — Metallographic determination of microstructure

Part 4: Characterisation of porosity, carbon defects and eta-phase content

**bsi.**

...making excellence a habit.™

**National foreword**

This British Standard is the UK implementation of EN ISO 4499-4:2016. It supersedes BS EN 24505:1993 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee ISE/65, Sintered metal components.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

© The British Standards Institution 2016.  
Published by BSI Standards Limited 2016

ISBN 978 0 580 81470 9

ICS 77.040.99; 77.160

**Compliance with a British Standard cannot confer immunity from legal obligations.**

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 March 2016.

**Amendments/corrigenda issued since publication**

Date	Text affected
------	---------------

---

EUROPEAN STANDARD

EN ISO 4499-4

NORME EUROPÉENNE

EUROPÄISCHE NORM

March 2016

ICS 77.040.99; 77.160

Supersedes EN 24505:1993

English Version

## Hardmetals - Metallographic determination of microstructure - Part 4: Characterisation of porosity, carbon defects and eta-phase content (ISO 4499-4:2016)

Métaux-durs - Détermination métallographique de la  
microstructure - Partie 4: Caractérisation de la  
porosité, des défauts carbone et de la teneur en phase  
êta (ISO 4499-4:2016)

Hartmetalle - Metallographische Bestimmung der  
Mikrostruktur - Teil 4: Charakterisierung von  
Porosität, Kohlenstofffehlern und Anteil an Eta-Phase  
(ISO 4499-4:2016)

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**

## European foreword

This document (EN ISO 4499-4:2016) 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.

This document supersedes EN 24505:1993.

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 4499-4:2016 has been approved by CEN as EN ISO 4499-4:2016 without any modification.

# Contents

Page

<b>Foreword</b> .....	<b>iv</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>1</b>
<b>5 Principle</b> .....	<b>2</b>
<b>6 Apparatus</b> .....	<b>2</b>
<b>7 Calibration of measurement apparatus</b> .....	<b>2</b>
<b>8 Preparation of test samples</b> .....	<b>2</b>
8.1 Methods of preparation.....	2
8.2 Characterisation of porosity and carbon (graphite) defects.....	3
8.3 Characterisation of eta-phase.....	3
<b>9 Procedure</b> .....	<b>3</b>
9.1 General.....	3
9.2 Determination of porosity.....	3
9.3 Determination of carbon defects.....	4
9.4 Determination of the $\eta$ -phase (eta-phase).....	7
<b>10 Uncertainty of measurement</b> .....	<b>9</b>
<b>11 Test report</b> .....	<b>9</b>
<b>Bibliography</b> .....	<b>10</b>

## 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](http://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](http://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 meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#).

The committee responsible for this document is ISO/TC 119, *Powder metallurgy*, Subcommittee SC 4, *Sampling and testing methods for hardmetals*.

This first edition of ISO 4499-4 cancels and replaces ISO 4505:1978, which has been technically revised.

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*

## Introduction

In standard WC/Co hardmetals, the chemistry, magnetic properties and density are generally controlled so that only two phases WC and Co are present.<sup>[1][2][3]</sup> The Co phase is an alloy and contains some W and C in solid solution. The WC phase is stoichiometric. If the composition is either high or low in total carbon content, then it is possible to see a third phase in the structure. For high C, this is graphite; for low C, it is eta phase ( $\eta$ ); typically, an  $M_6C$  or  $M_{12}C$  carbide where M is  $(Co_xW_y)$ . This part of ISO 4499 is concerned with the detection and measurement of these microstructural features together with the measurement of porosity levels. Porosity is important since these materials are manufactured by a powder metallurgical route and although the technique of liquid phase sintering is used to consolidate the multiphase structure, low levels of porosity can arise in some instances and affect properties such as density and strength.





# Hardmetals — Metallographic determination of microstructure —

## Part 4: Characterisation of porosity, carbon defects and eta-phase content

### 1 Scope

This part of ISO 4499 specifies methods for the metallographic determination of the presence, type, and distribution of porosity, uncombined carbon and eta-phase in hardmetals.

### 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 4499-2:2008, *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

##### carbon defects

macroscopic precipitates of carbon (graphite) which can be in the form of large angular rosettes or small flakes

#### 3.2

##### eta-phase

##### $\eta$ -phase

cubic carbide based on M<sub>6</sub>C or M<sub>12</sub>C structure where M is a mixture of Co and W usually in equal proportions; and which can be present as large (up to 100  $\mu\text{m}$  diameter) rosettes or small micrometre-sized particles

### 4 Symbols and Units

ECD Equivalent Circle Diameter of a specified phase, in micrometres ( $\mu\text{m}$ )

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

$l_i$  measured length of individual intercepts in a specified phase, in micrometres ( $\mu\text{m}$ )

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

$l_x$  arithmetic mean linear intercept in phase  $x$ , in micrometres ( $\mu\text{m}$ )