FINAL DRAFT

INTERNATIONAL STANDARD

ISO/FDIS 4499-4

ISO/TC **119**/SC **4**

Secretariat: DIN

Voting begins on:
2015-10-29

Voting terminates on:
2015-12-29

Hardmetals — Metallographic determination of microstructure —

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

Métaux-durs — Détermination métallographique de la microstructure —

Partie 4: Caractérisation de la porosité, des défauts carbone et de la teneur en phase êta

Please see the administrative notes on page iii

Reference number
ISO/FDIS 4499-4:2015(E)

ISO/CEN PARALLEL PROCESSING

This final 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. The final draft was established on the basis of comments received during a parallel enquiry on the draft.

This final draft is hereby submitted to the ISO member bodies and to the CEN member bodies for a parallel two-month approval vote in ISO and formal vote in CEN.

Positive votes shall not be accompanied by comments.

Negative votes shall be accompanied by the relevant technical reasons.

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Contents

Page

Foreword ..... **iv**
Introduction ..... **v**
1 Scope **1**
2 Normative references **1**
3 Terms and definitions **1**
4 Symbols and Units **1**
5 Principle **2**
6 Apparatus **2**
7 Calibration of measurement apparatus **2**
8 Preparation of test samples **2**
8.1 Methods of preparation 2
8.2 Characterisation of porosity and carbon (graphite) defects 3
8.3 Characterisation of eta-phase 3
9 Procedure **3**
9.1 General 3
9.2 Determination of porosity 3
9.3 Determination of carbon defects 4
9.4 Determination of the η-Phase (eta-phase) 7
10 Uncertainty of measurement **9**
11 Test report **9**
Bibliography **10**

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 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.[1][2][3] 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 (η); 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 precipates of carbon (graphite) which can be in the form of large angular rosettes or small flakes

3.2
eta-phase
η-phase
cubic carbide based on M6C or M12C structure where M is a mixture of Co and W usually in equal proportions; and which can be present as large (up to 100 μm diameter) rosettes or small micrometre-sized particles

4 Symbols and Units

ECD	Equivalent Circle Diameter of a specified phase, in micrometres (μm)
L	total line length in a specified phase, in millimetres (mm)
l_i	measured length of individual intercepts in a specified phase, in micrometres (μm)
$\sum l_\mathrm{i}$	sum of the measured length of each individual intercept
l_x	arithmetic mean linear intercept in phase x, in micrometres (μm)

N	number of eta-phase particles intercepted
m	magnification
m_{max}	maximum magnification
m_{min}	minimum magnification

5 Principle

This part of ISO 4499 handles the following key issues:

— description of methods for sample preparation;

— description of how to identify and measure the relevant feature.

In some cases, the approach adopted in ISO 4499-2 and ISO 4499-3 can be useful, whereby the intercept method is used on microphotographs of the structure for relevant quantification of feature size, such as eta-phase or graphite rosettes.

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 (SEM), permitting observations and measurements of features too small to be resolved with an optical microscope.

6.3 Equipment for preparation of test-piece sections.

7 Calibration of measurement apparatus

For reliable quantitative measurements, images shall be calibrated against a stage micrometre or scale traceable to a National Reference Standard. The most commonly used stage micrometres for SEMs are the SIRA grids. These are ruled lines which form a grid and are available with 19,7 lines/mm and 2 160 lines/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 Reference [4].

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.

8 Preparation of test samples

8.1 Methods of preparation

There are several methods for the preparation of hardmetal surfaces for metallographic examination. For example, a detailed description is given in ISO 4499-1. 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.

8.2 Characterisation of porosity and carbon (graphite) defects

For porosity and carbon defects, the test-piece 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 can lead to a misleading evaluation of porosity.

8.3 Characterisation of eta-phase

Etching is necessary to reveal eta-phase particles (see 9.4). Eta-phase is metal carbide (usually M6C or M12C, where M is a combination of Co and W, for example, Co3W3C), that forms when the overall carbon content of the hardmetal is relatively low. Generally, it can grow in one of two morphologies, either as large rosettes or as small particles of a similar size to the other hard phases (WC or cubic carbides) present in the hardmetal (see 9.4). The presence of the eta-phase is usually determined after light etching in 10 % Murakami's reagent for a few seconds with immediate water flush following etching (see ISO 4499-1), which works well for identifying large eta-phase rosettes. When the etas-phase is present as smaller particles (see Figure 6), it is recommended to use a 5 % Murakami's solution for 20 min followed by washing the sample with water. In both cases, the surface should be dried carefully with acetone or alcohol without wiping.

9 Procedure

9.1 General

If the porosity or uncombined carbon is not uniform over the area of the test piece section being examined, the locations on the section to which the evaluation refers shall be identified, for example, as top, bottom, edge, rim (case), core, etc.

It should to be noted that the figures given in this Clause are only representing approximately $\frac{1}{7}$ of the field of view in the microscope.

9.2 Determination of porosity

Pore size is defined as the maximum dimension of the pore in the section. Special reference shall be made to the presence of cracks or slits.

9.2.1 Pores up to 10 µm shall be assessed by scanning the surface of the test-piece section at a magnification of either ×100 or ×200. An area fully representative of the test-piece section shall be examined and compared with the range of photomicrographs shown in Figure 1 or Figure 2, according to the chosen magnification. The porosity level shall be reported by reference to the appropriate photomicrograph and designated as A02, A04, A06 or A08. If the level of A-type pores is less than 50 % of that shown in Figure 1 (A02) or Figure 2 (A02), it shall be designated as A00.

9.2.2 Pores within the range 10 µm to 25 µm shall be assessed by scanning the surface of the test-piece section at a magnification of ×100. An area fully representative of the test-piece section shall be examined and shall be compared with the range of photomicrographs shown in Figure 3. The porosity level shall be reported by reference to the appropriate photomicrograph and designated as B02, B04, B06 or B08.

If the number of B-pores appears to be less than or equal to that represented by B02, the number of B-pores in the representative area (≥0,25 cm^2) shall be counted. This number shall be divided by the area examined to obtain the number of B-pores/cm^2.

If this number is less than 70 pores/cm^2, it shall be designated as B00 - #, where # is the number of B-pores/cm^2 so obtained. If the number is greater than or equal to 70 pores/cm^2, it shall be designated as B02.