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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

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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Foreword

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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).

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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*.

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

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 µm, 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 µm to 0,5 µm, 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 µm to 0,8 µm, 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^2)

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)

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

l_x arithmetic mean linear intercept in phase x, in micrometres (µm)

N number of grain boundaries traversed in or between specified phases

n number of WC, carbonitride or cubic carbide grains intercepted

m magnification

m_{max} maximum magnification

m_{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).[3][4][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 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.

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 to 0,2 µm. For materials with ever smaller grain sizes, it can 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 Reference [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.

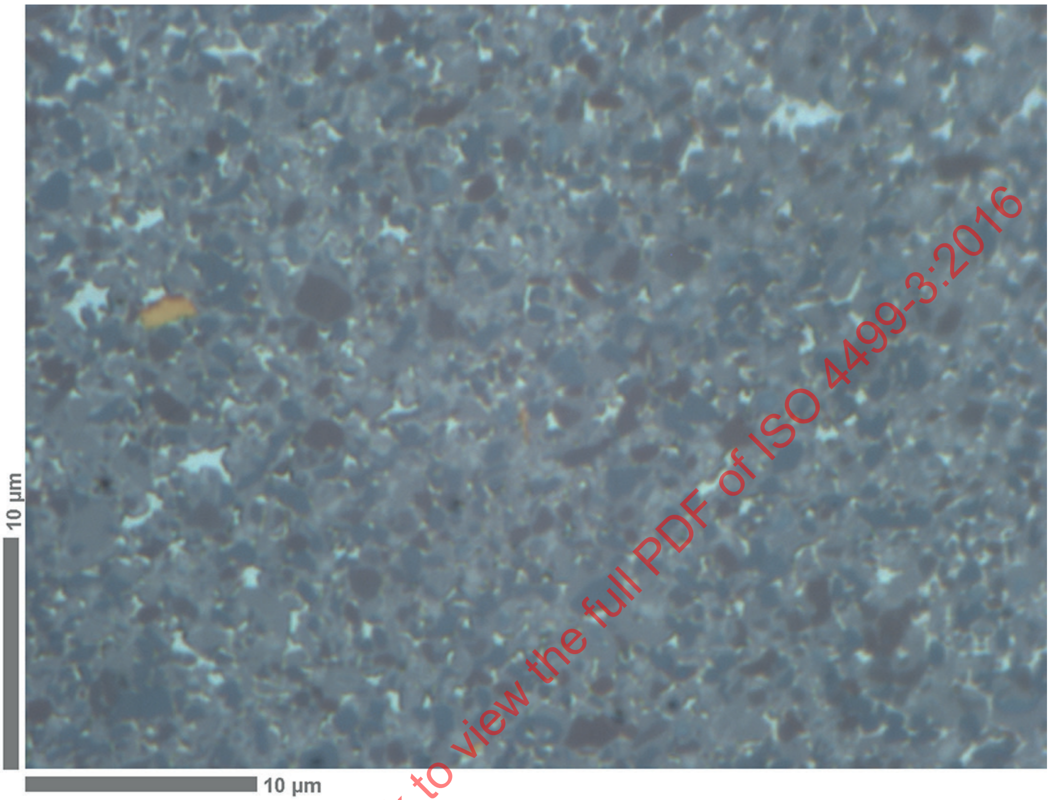
8 Preparation of test samples

8.1 Metallographic preparation

The basic steps for good metallographic sections of hard materials are given in detail in ISO 4499-1:2008, 6.1 for sectioning, mounting, grinding, lapping, polishing and cleaning, except for the final polishing stage for these materials, which was performed using colloidal suspension of silica (at 40 nm particle size) on a napless silk cloth. Appropriate etching methods for cermets and hardmetals containing cubic carbides are outlined in 8.2 and 8.3, respectively.

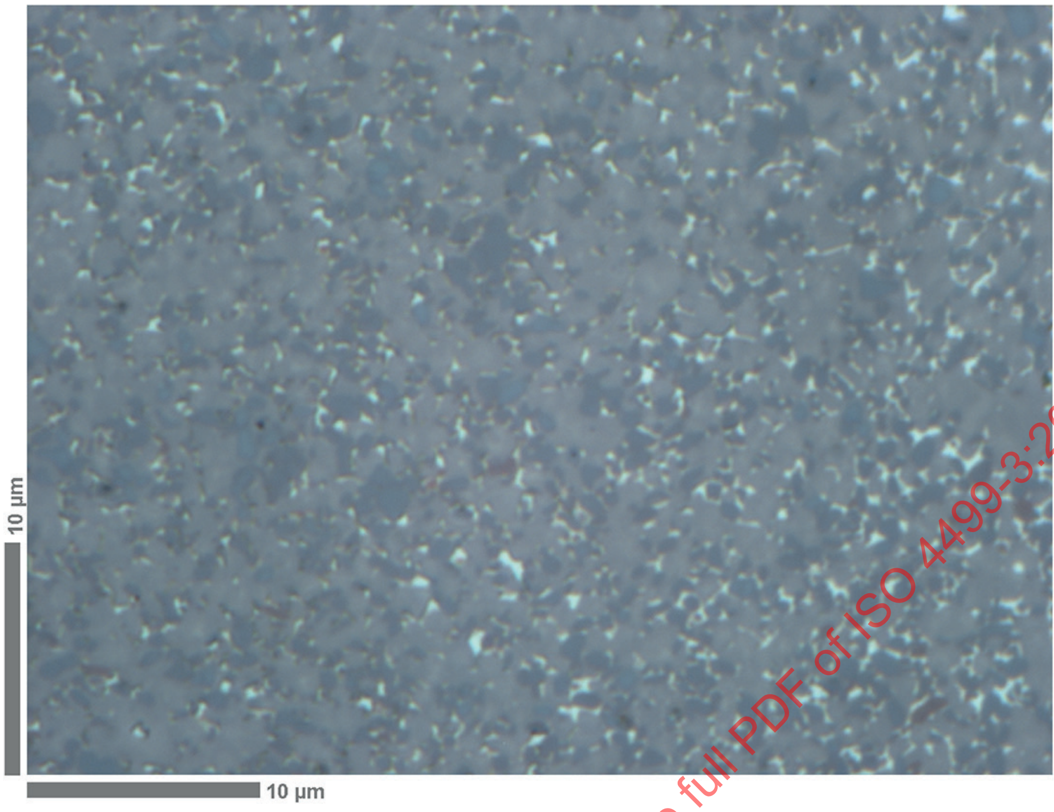
8.2 Ti(C, N) based hardmetals – cermets

The preparation of the test samples is recommended to be according to ISO 4499-1:2008, 6.2.1 by using etching technique 1. The conditions of etching in mixture A should be changed to approximately 20 °C for 30 s to 60 s. Representative images of typical cermets are shown in Figure 1 to Figure 5 for both optical and electron microscopical techniques. The SEM images show that many of the hard phase particle have a core/rim structure, where the dark “cores” are undissolved Ti(C,N) from the original powder mix and the grey “mass” are (Ti,W,X)(C,N) structures, of the same orientation as the “core” formed during liquid phase sintering at high temperature.



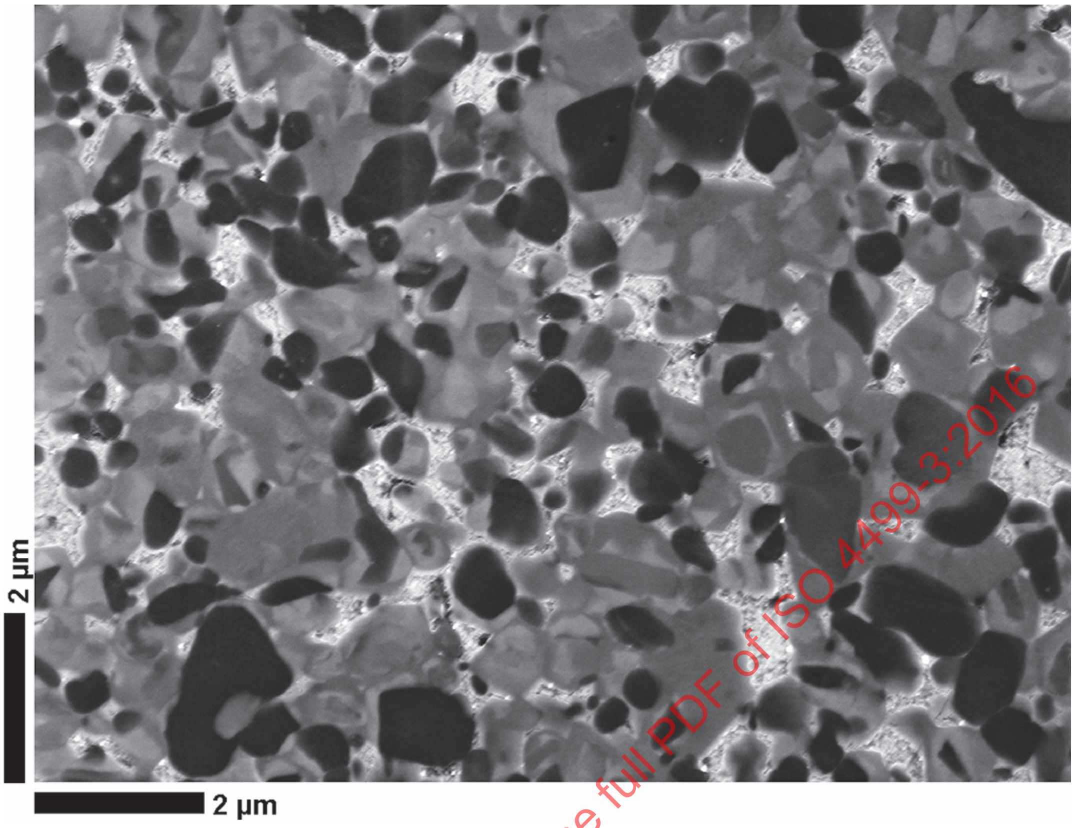
NOTE When using an optical microscope, major phases appear as binder phase (light blue), undissolved Ti(C,N) from original powder (dark blue-grey), (Ti,W,X)(C,N) (grey) and TiN impurity (gold).

Figure 1 — Low binder phase content (6 wt %) commercial cermet, optical micrograph using oil immersion objective, original magnification ×1 600



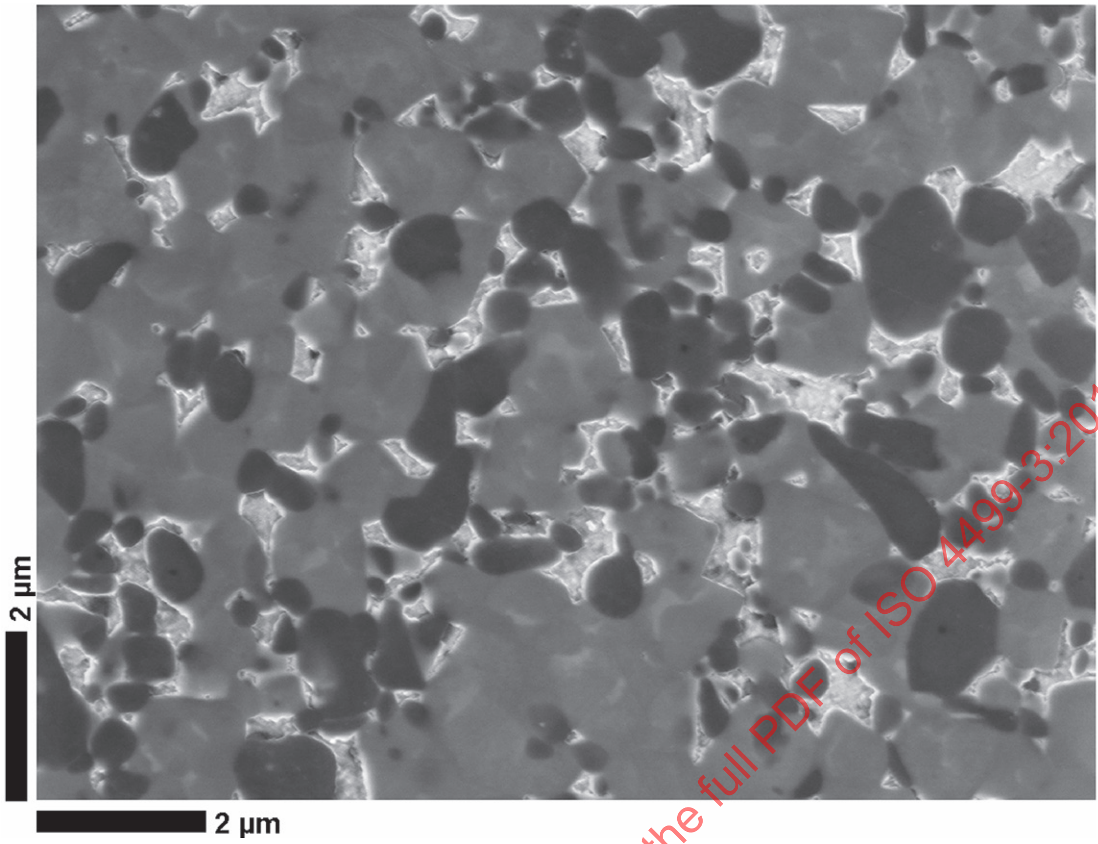
NOTE When using an optical microscope, major phases appear as binder phase (light blue), undissolved Ti(C,N) from original powder (dark blue-grey), and (Ti,W,X)(C,N) (grey).

Figure 2 — Medium binder phase content (11 wt %) commercial cermet, optical micrograph using oil immersion objective. Original magnification ×1 600



NOTE Major phases are binder phase (light), undissolved Ti(C,N) from original powder (dark grey) and (Ti,W,X)(C,N) of variable composition (pale grey).

Figure 3 — Low binder phase content (6 wt %) commercial cermet, scanning electron microscope secondary electron image, original magnification ×30 000



NOTE Major phases are binder phase (light), undissolved Ti(C,N) from original powder (dark grey) and (Ti,W,X)(C,N) of variable composition (pale grey).

Figure 4 — Medium binder phase content (11 wt %) commercial cermet, scanning electron microscope secondary electron image, original magnification ×30 000

8.3 WC/Cubic carbide based hardmetals

In the former hardmetal microstructural standard ISO 4499, this type of material was illustrated with three examples of mixed WC/cubic carbide materials where the hard phase is given the Greek letter, γ, as identifier. It is recommended in this update to the International Standard that the γ name is retained but that the size is defined more precisely by linear intercept measurements. Preparation of samples in the use of etching technique 2 is as described in ISO 4499-1:2008, 6.2.1, i.e. etch in Murakami's reagent (mixture A) for about 3 min; followed by etch in concentrated HCl (mixture B) for 10 s; followed by wash in water then alcohol and a final etch in mixture A for 20 s.

Four different grades of cubic carbide hardmetals are illustrated in the standard with compositions as shown in Table 1.

Table 1 — Composition of WC/Cubic carbide based hardmetals

	Cobalt %	Tantalum %	Titanium %	Niobium %
Material 1	5,7	1,9	Trace	Trace
Material 2	6,3	3,5	2,0	1,5
Material 3	11,5	1,9	Trace	0,4
Material 4	9,5	5,5	6,0	2,5

Representative images are shown in Figure 5 to Figure 12 (optical) and Figure 13 to Figure 20 (SEM).

The optical images were obtained using an ×100 oil immersion objective with a numerical aperture of 1,3 at nominal magnifications of ×1 000 and ×1 600. When using an optical microscope, major phases appear as binder phase (white), tungsten carbide (blue/grey) and cubic carbide (pale orange).

The SEM images were taken under the same operating conditions, 9 kV accelerating voltage, 15 mm working distance, secondary electron mode. The SEM images show the major phases binder phase (black), tungsten carbide (light grey) and cubic carbide (medium grey).

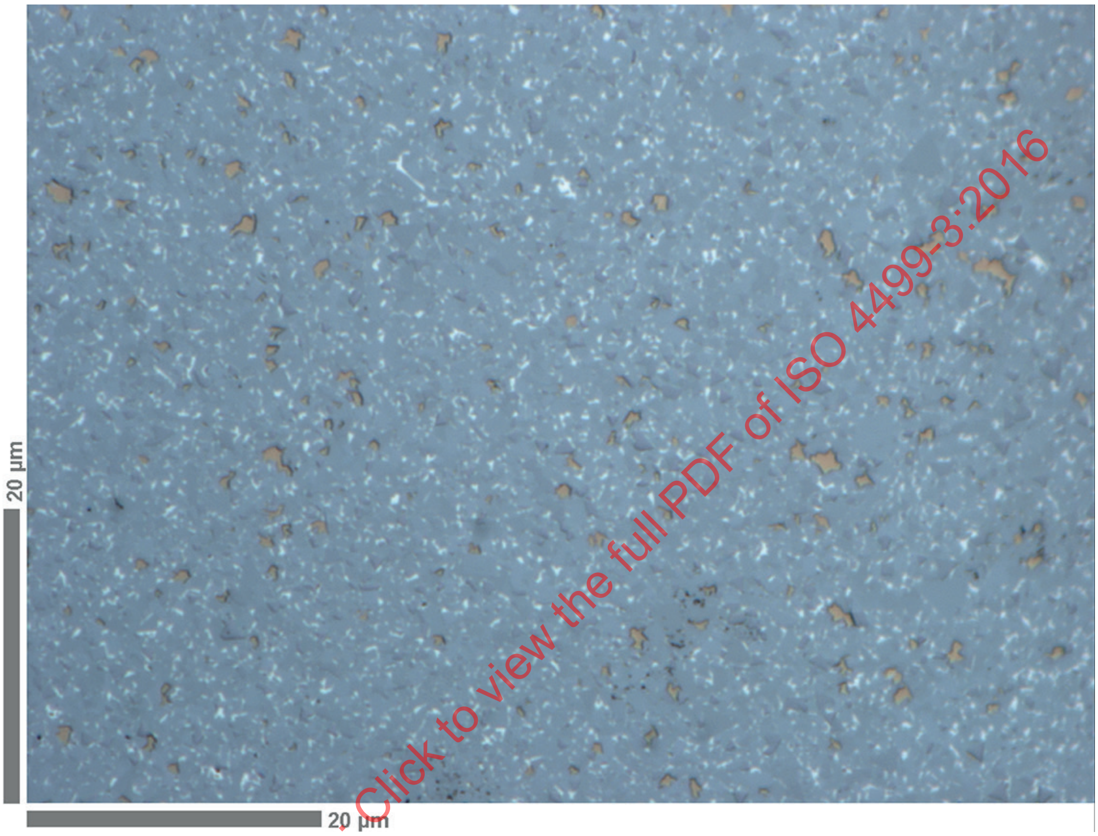


Figure 5 — Material 1, optical micrograph, original magnification ×1 000

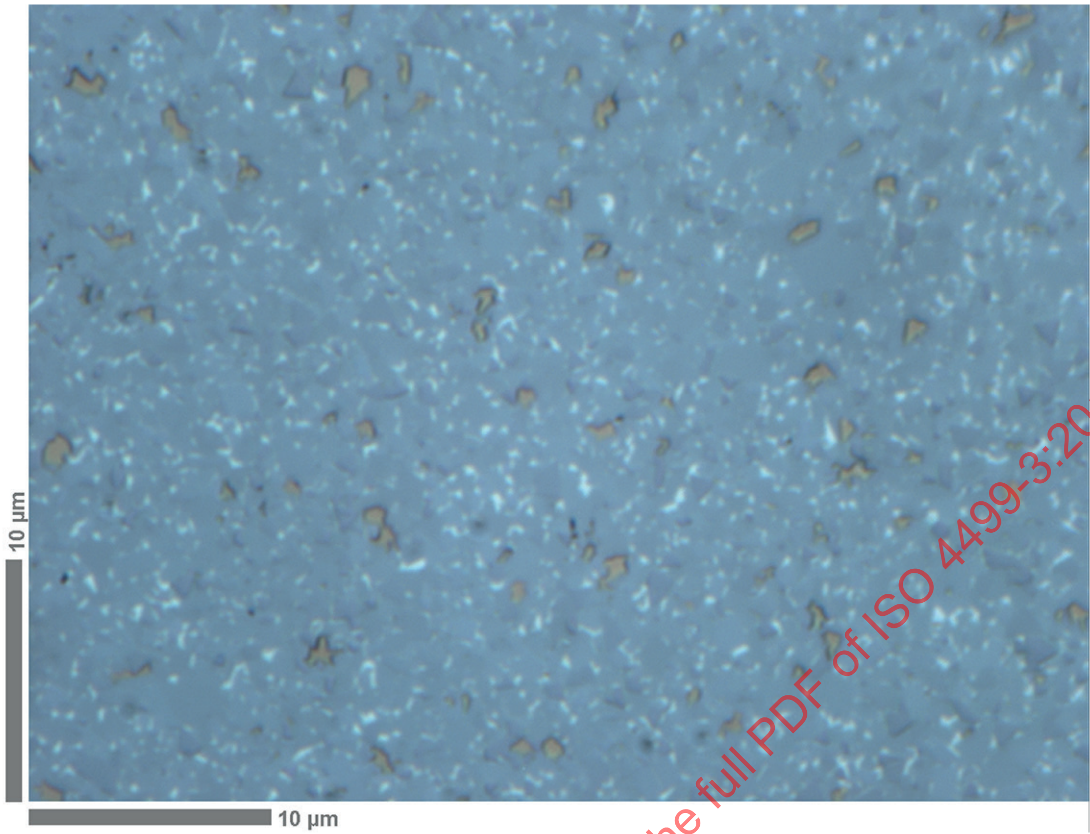


Figure 6 — Material 1, optical micrograph, original magnification ×1 600

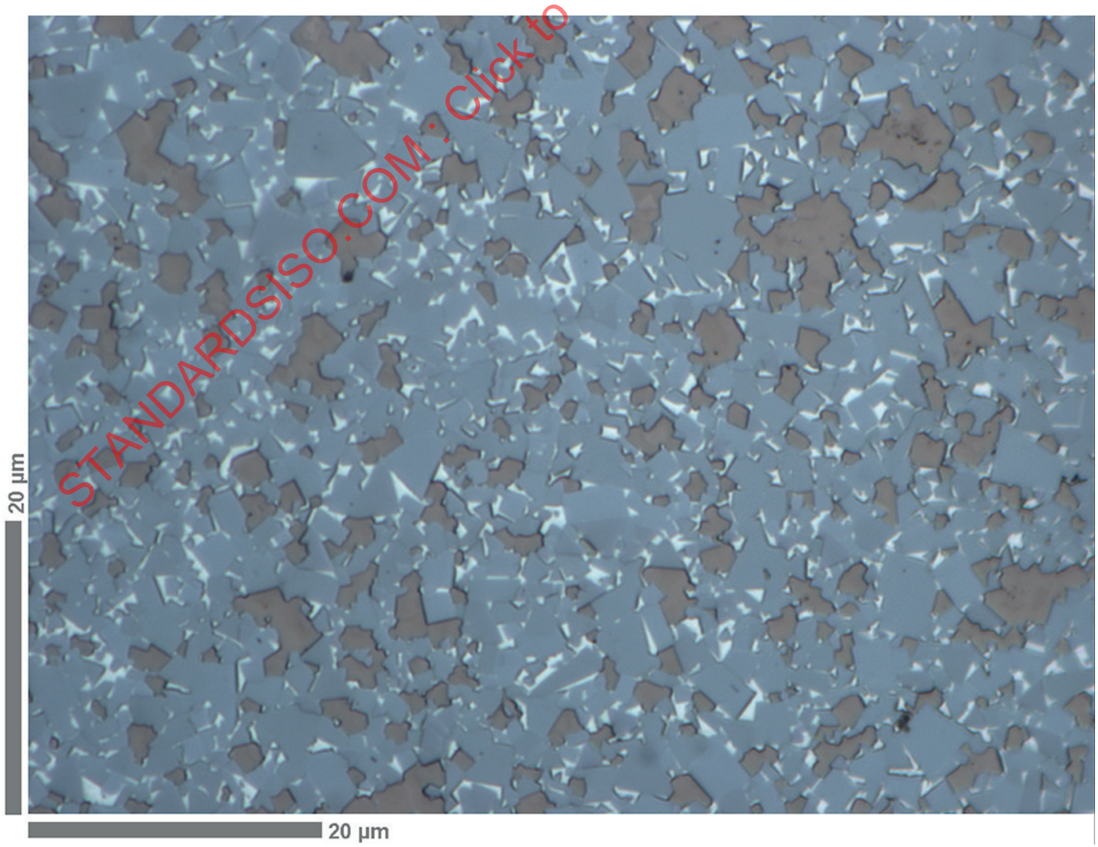


Figure 7 — Material 2, optical micrograph, original magnification ×1 000

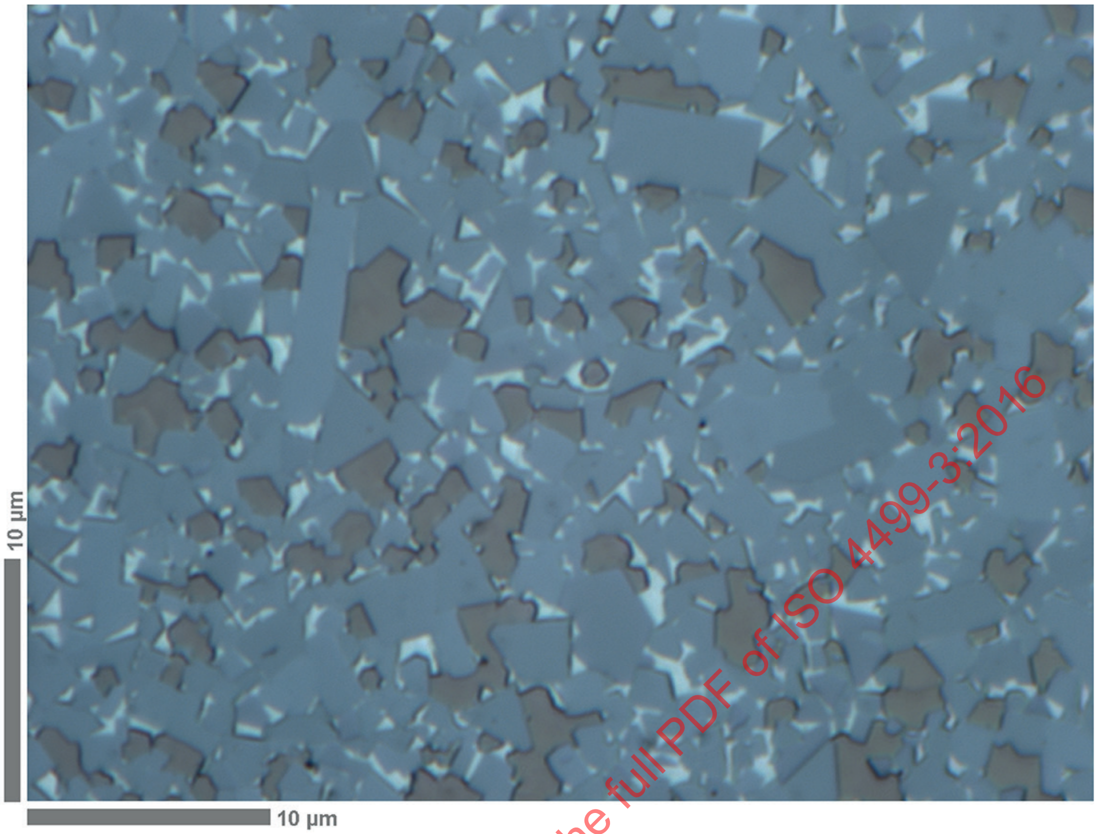


Figure 8 — Material 2, optical micrograph, original magnification ×1 600

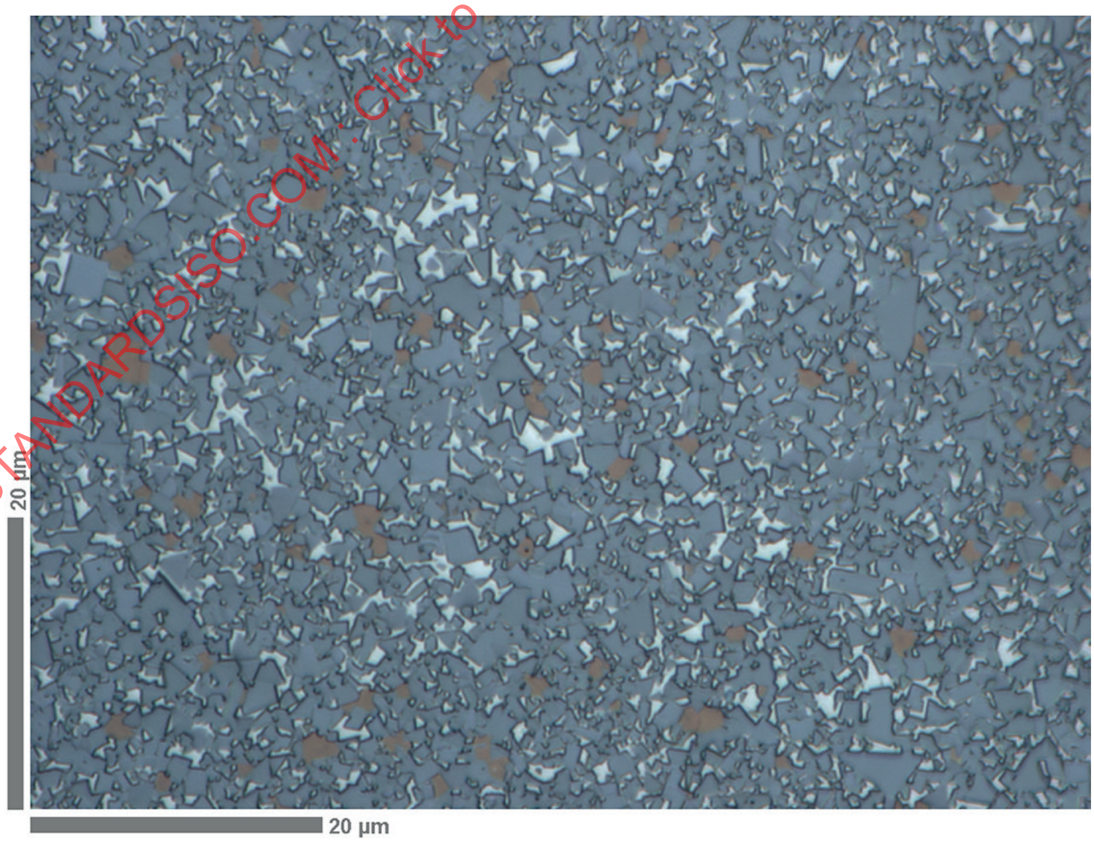


Figure 9 — Material 3, optical micrograph, original magnification ×1 000

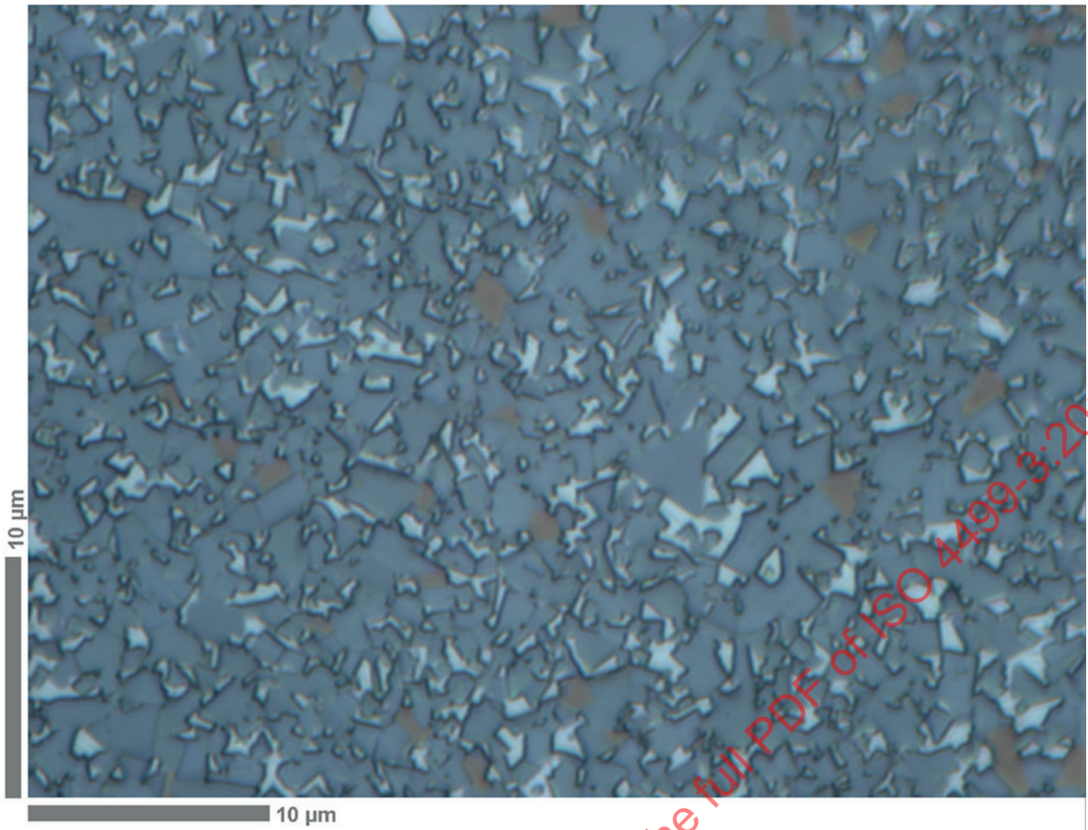


Figure 10 — Material 3, optical micrograph, original magnification ×1 600

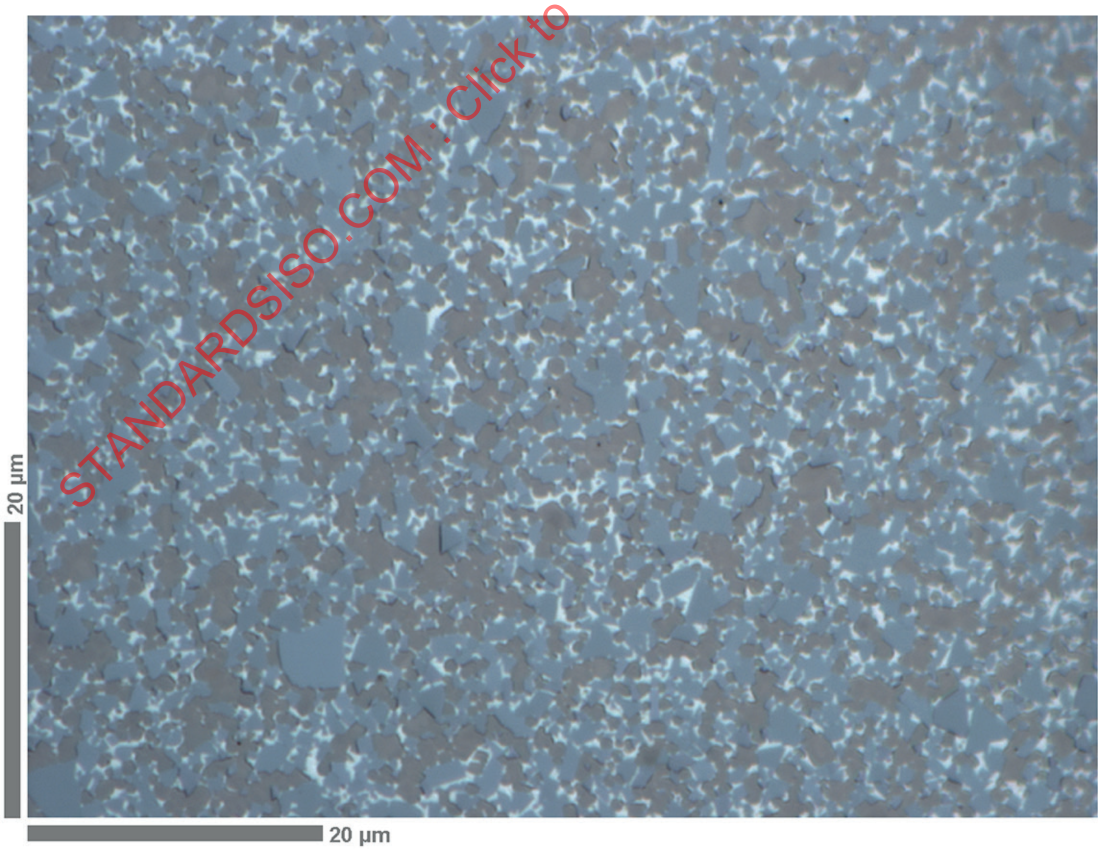


Figure 11 — Material 4, optical micrograph, original magnification ×1 000

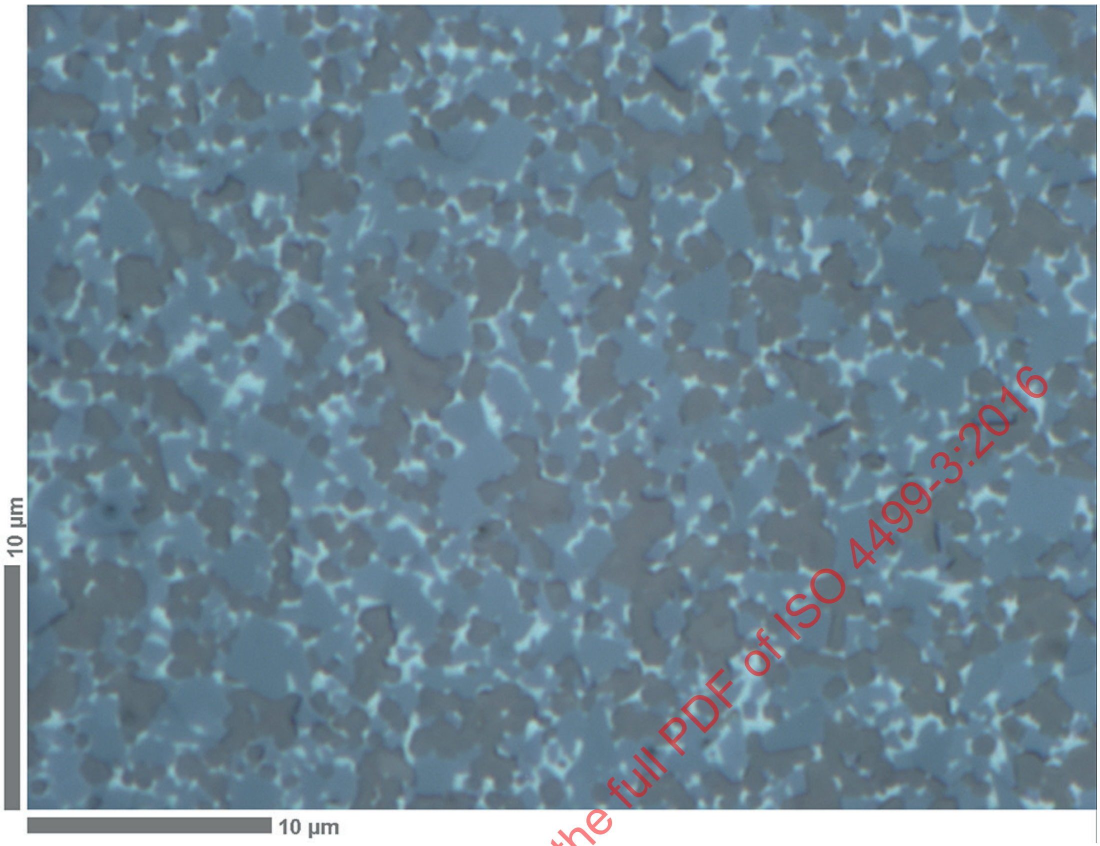


Figure 12 — Material 4, optical micrograph, original magnification ×1 600

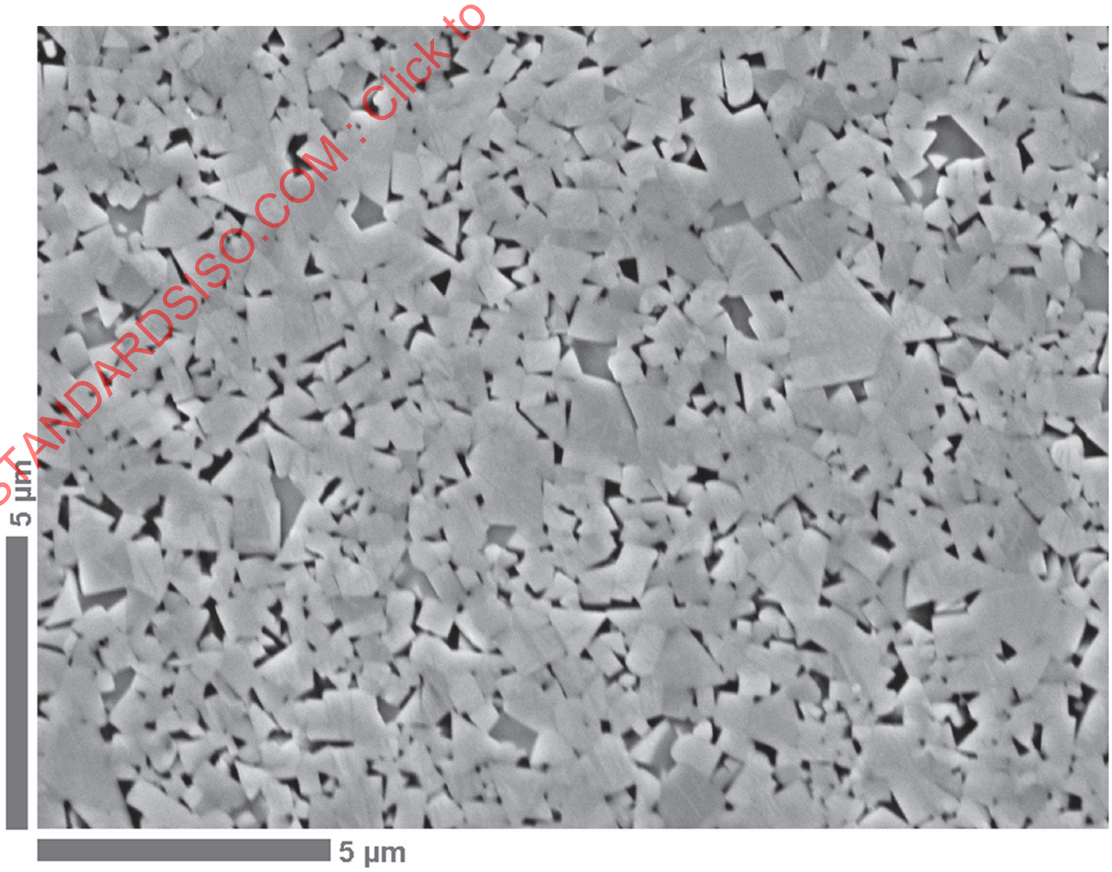


Figure 13 — Material 1, SEM micrograph, original magnification ×20 000

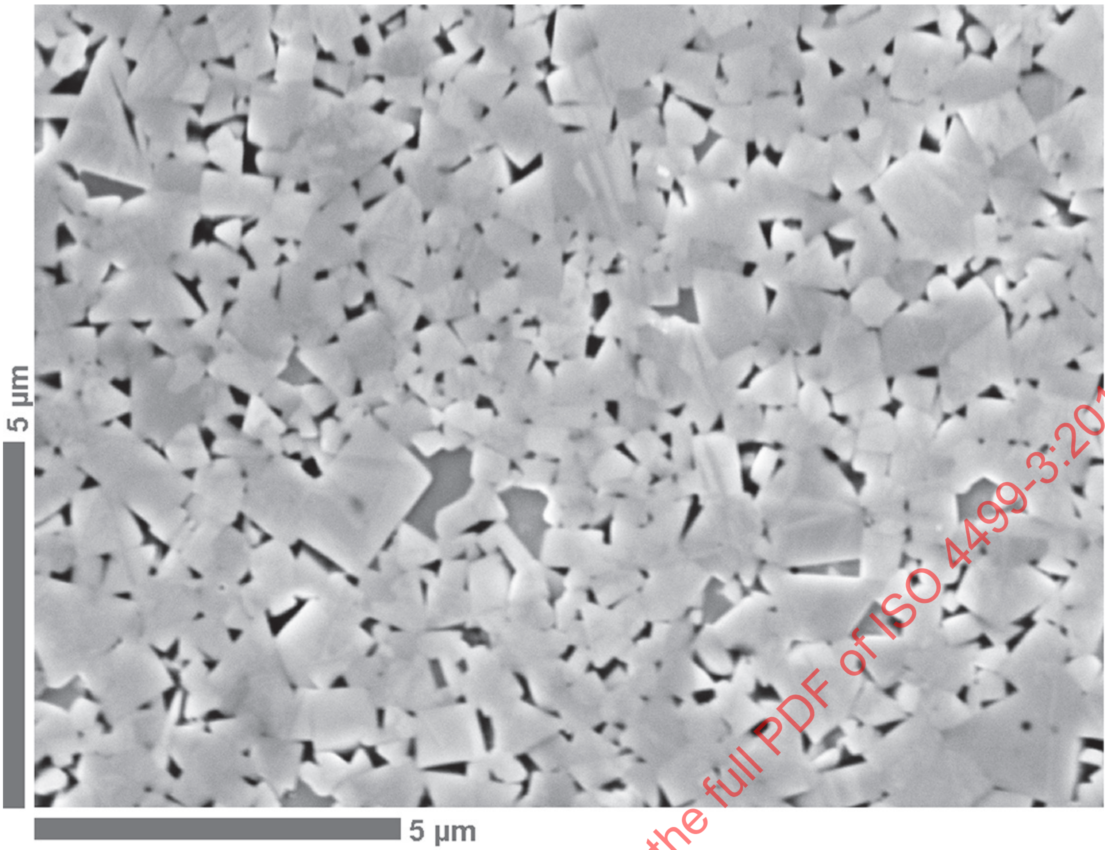


Figure 14 — Material 1, SEM micrograph, original magnification ×25 000

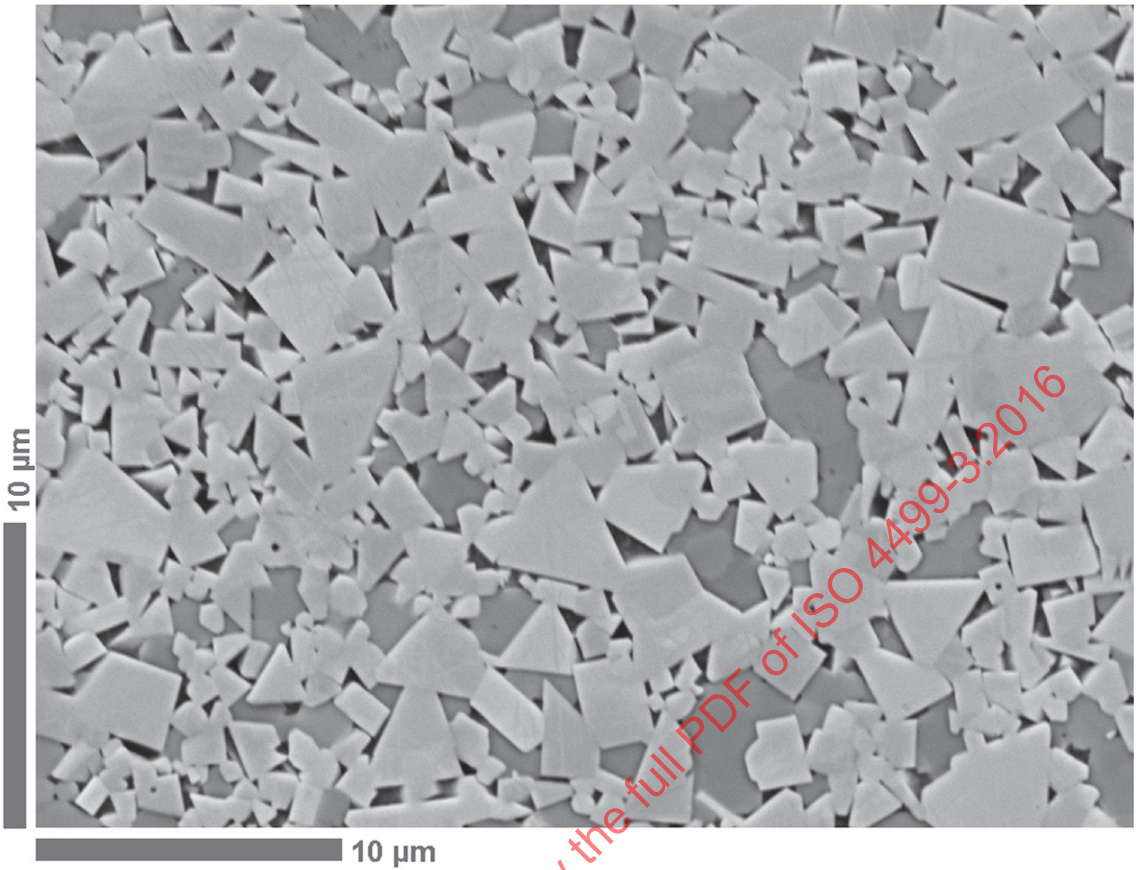


Figure 15 — Material 2, SEM micrograph, original magnification ×10 000

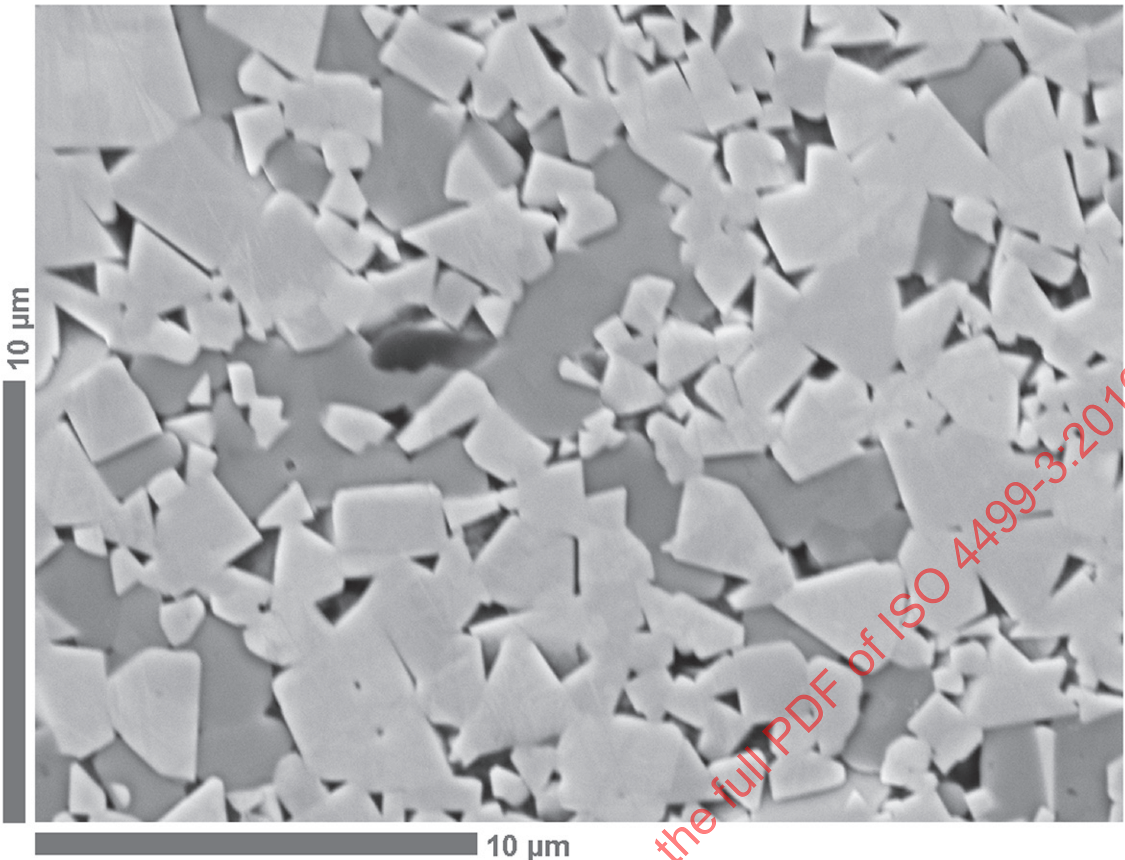


Figure 16 — Material 2, SEM micrograph, original magnification ×15 000

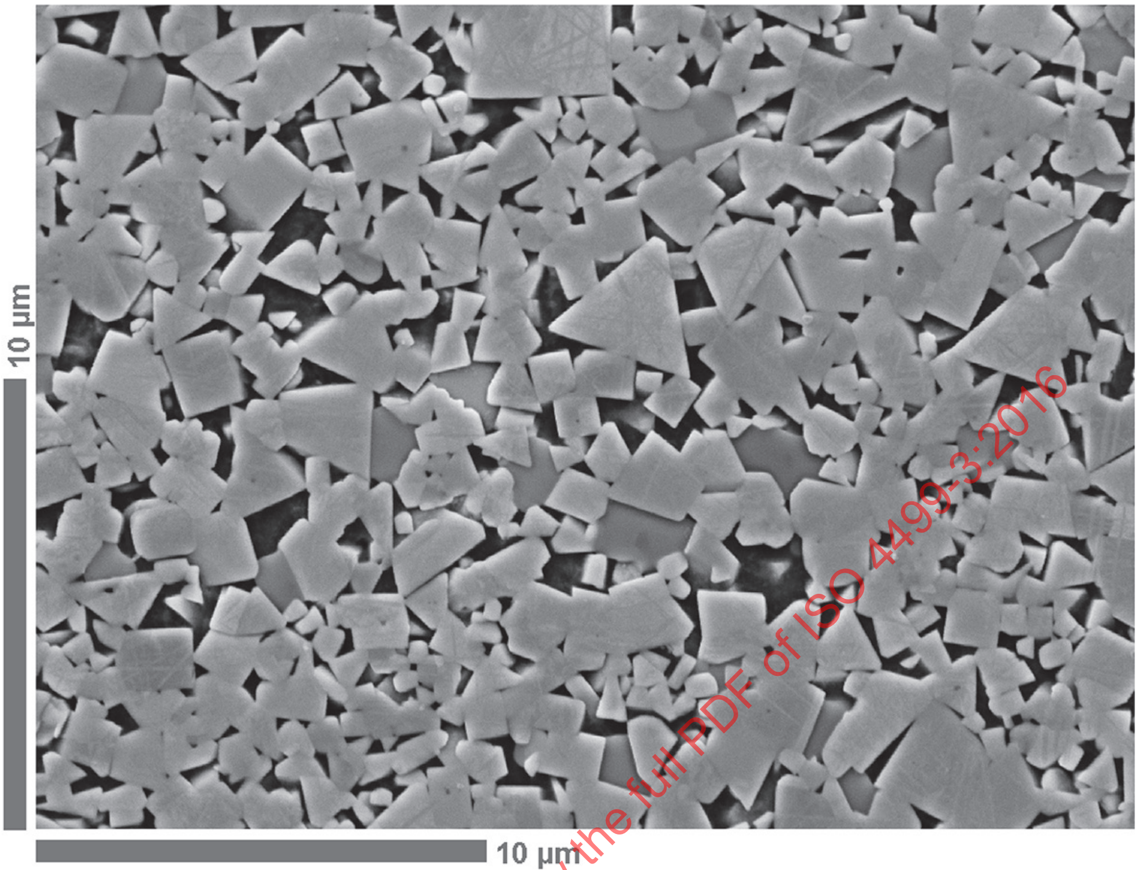


Figure 17 — Material 3, SEM micrograph, original magnification ×15 000

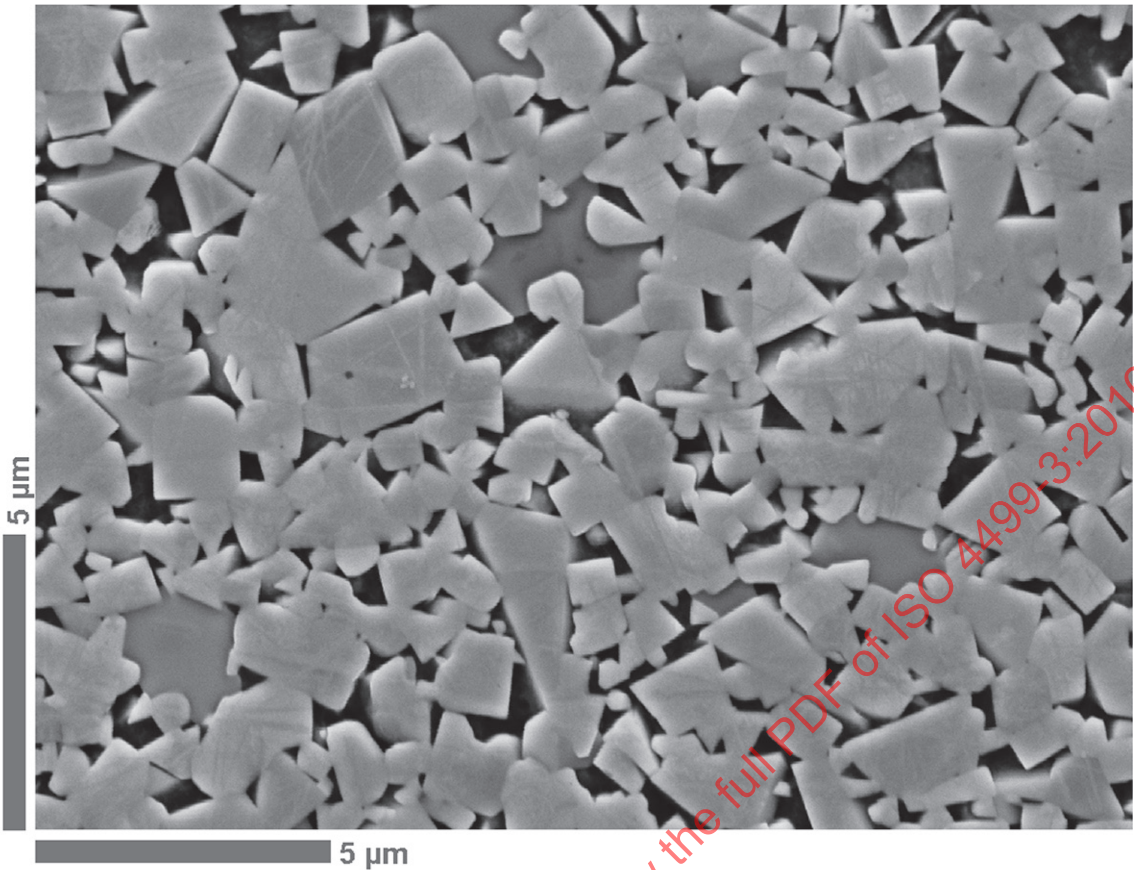


Figure 18 — Material 3, SEM micrograph, original magnification ×20 000

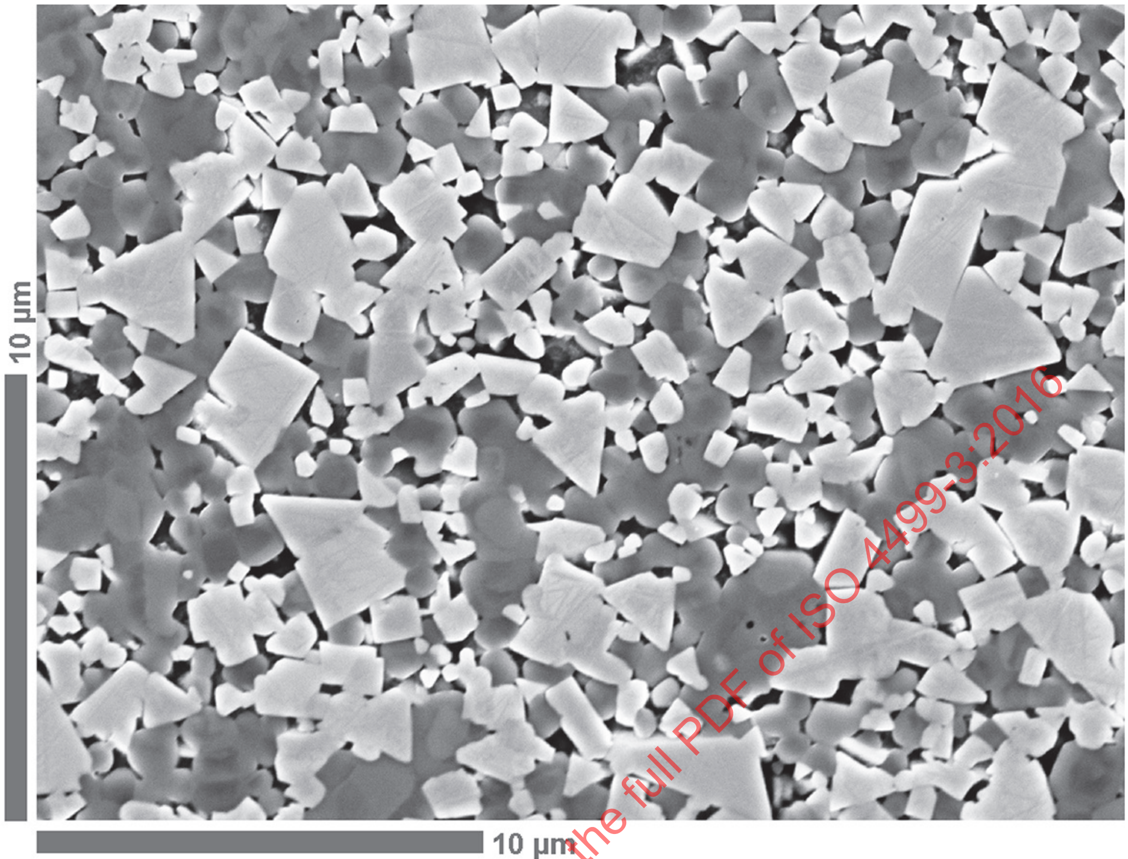


Figure 19 — Material 4, SEM micrograph, original magnification ×15 000