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**ISO 4499-2**

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# 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*

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# Contents

Page

# 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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 4499-2 was prepared by Technical Committee ISO/TC 119, *Powder metallurgy*, Subcommittee SC 4, *Sampling and testing methods for hardmetals*.

ISO 4499-2, together with ISO 4499-1, cancels and replaces ISO 4499:1978, which has been technically revised. A new section has been added for the quantitative measurement of the WC grain size of 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*

# Hardmetals — Metallographic determination of microstructure —

## Part 2: Measurement of WC grain size

## 1 Scope

This part of ISO 4499 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 as the hard phase. It is also intended for measuring the grain size and distribution by the linear-intercept technique.

This part of ISO 4499 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.

The part of ISO 4499 is supported by a measurement case study to illustrate the recommended techniques (see Annex A).

The part of ISO 4499 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 referenced documents are indispensable for the application 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 3326, *Hardmetals — Determination of (the magnetization) coercivity*

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 4505, *Hardmetals — Metallographic determination of porosity and uncombined carbon*

## 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 grains 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.

**3.2.1**
**nano**
with WC grain size $<$ 0,2 µm

NOTE Measured by the mean-linear-intercept method described in this part of ISO 4499.

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

NOTE Measured by the mean-linear-intercept method described in this part of ISO 4499.

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

NOTE Measured by the mean-linear-intercept method described in this part of ISO 4499.

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

NOTE Measured by the mean-linear-intercept method described in this part of ISO 4499.

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

NOTE Measured by the mean-linear-intercept method described in this part of ISO 4499.

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

NOTE Measured by the mean-linear-intercept method described in this part of ISO 4499.

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

NOTE Measured by the mean-linear-intercept method described in this part of ISO 4499.

## 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 $(mm)^2$

$d_{WC}$ 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)

$\sum l_{\mathrm{i}}$ 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

$m_{\mathrm{max}}$ is the maximum magnification

$m_{\mathrm{min}}$ is the minimum magnification

$s_{\mathrm{m}}$ is the measured size, in millimetres (mm)

$s_{\mathrm{a}}$ 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] in the Bibliography), 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[1)]; this is obtained by measuring grain areas and then taking the diameter of a circle of equivalent area.

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 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 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] in the Bibliography). Careful specimen preparation for good images is vital for these materials, and often a combination of etching methods is helpful (see ISO 4499-1).

1) 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] in the Bibliography.

## 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] in the Bibliography).

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.

## 7 Grain-size measurement by the linear-intercept method

### 7.1 General

It is recommended that the arithmetic mean-linear-intercept be used as the parameter to define WC grain size. This is the simplest procedure to use and has the added advantage of providing data that can be used to quantify distribution width.

This method requires a straight line to be drawn across a calibrated image. In a single-phase material the length of line ($L$), starting at a random position, traversing a number of grain boundaries ($N$), and ending at another random position, is measured. The mean-linear-intercept distance LI is thus:

$$\mathrm{LI} = L / N \tag{2}$$

As can be seen from the above equation, only the the mean-linear-intercept distance is calculated, there is no information obtained on grain-size distribution.

For a nominally two-phase material such as a hardmetal ($\alpha$ and $\beta$ phase), the linear-intercept technique is less straightforward because each phase has to be measured independently, but it can provide information on grain-size distribution. A line is drawn across a calibrated image of the microstructure of a hardmetal. Where this line intercepts a grain of WC, the length of the line ($l_i$) is measured using a calibrated rule (where $i = 1, 2, 3, \ldots, n$, for the 1st, 2nd, 3rd, …, $n$th grain). It is advisable to count at least 100 grains, preferably at least 200 grains in order to reduce the uncertainty to below 10 %.

The mean-linear-intercept grain size is defined as:

$$d_{\mathrm{WC}} = \sum l_i / n \tag{3}$$

Hardmetal grain sizes generally fall in the range 0,1 µm to 10 µm. Because of the uncertainties of measurement, it is good practice to report the the mean-linear-intercept grain size to one decimal place for values $>$ 1,0 µm and to two decimal places for values $<$ 1,0 µm. i.e. the results are reported to two significant figures, such as 3,4 µm or 0,18 µm.

A worked example is given in Annex A.

## 7.2 Sampling

### 7.2.1 Sampling of products

Sampling is the procedure whereby an item of hardmetal or a region within an item is chosen for testing. Random sampling is defined such that, in selecting an individual from a population, each individual in the population has the same chance of being chosen (see [9] in the Bibliography).

ISO 4489:1978, Clause 4 states that "For confirmation of the grade of hardmetal, it is usually sufficient to take a test sample of one unit" for the following tests:

— Determination of coercivity ISO 3326;

— Determination of density ISO 3369;

— Determination of Rockwell hardness HRA ISO 3738-1 and ISO 3738-2;

— Determination of Vickers hardness HV ISO 3878.

and tests which may be carried out in special cases:

— Determination of microstructure ISO 4499;

— Determination of porosity and uncombined carbon ISO 4505.

### 7.2.2 Sampling of microstructure

Sampling for microstructural purposes has to be carefully considered depending on the reason for undertaking the measurements:

a) General check measurement of a sectioned isolated object

— The images chosen for analysis should be representative of the whole section and should be obtained by random positioning. The number of images to be prepared is recommended to be at least four, which can be intensively analysed so that in total, at least 200 grains are measured.

b) Determination of homogeneity of grain size

— In this case, a systematic set of images from defined locations within the section shall be obtained and intensively analysed so that at least 200 grains are measured from each location. This will allow for example, trends in grain size greater than the likely error of measurement at each position (fractional error is proportional to $1/\sqrt{N}$, where $N$ is the number of grains at each location) to be determined.

c) Inhomogeneous materials

— In cases where the microstructure is inhomogeneous from one field of view to the next, it is good practice to increase the number of images evaluated, but to evaluate them less intensively, while still achieving a total feature count of $>$ 200.

The magnification of the image obtained should be such that there are between 10 WC and 20 WC grains across the field of view, permitting individual intercepts to be measured to better than 10 % accuracy. This will usually allow 3 or 4 linear-intercept lines to be drawn across the image without intercepting any individual WC grain more than once. Most hardmetals have little or no anisotropy of structure, so it is unimportant if more or less parallel lines are used. If anisotropy is suspected, then it is better to orientate the lines randomly and permit their intersection (see [11] in the Bibliography). Thus, from each image, about 50 linear grain-size intercepts may be obtained.

## 7.3 Measurement errors

### 7.3.1 Systematic and random errors

Measurement errors can have several sources:

— systematic, such as during calibration of the microscope;

— experimental or accidental, such as during data transfer or calculation of actual intercept lengths;

— statistical, such as due to the random nature of the microstructure.

A possible cause of a systematic error is that of calibrating the image from which measurements are to be made. In general, a single number is obtained for the magnification of an optical microscope. But, if the calibration is over different lengths or by different operators, the results will vary, producing a mean magnification and associated standard deviation. Errors are likely to be larger when using an SEM owing to magnifications not being fixed steps.

Accidental or personal errors will occur when measuring individual linear intercepts of WC grains. Different operators measuring along the same intercept line will not choose exactly the same intercept positions or may not detect all boundaries, and this will lead to an uncertainty in the measurement. Accidental or personal errors are more difficult to quantify than systematic errors.

Statistical errors can arise if microstructures are inadequately sampled, for example, too few micrographs are used, or too few grains are measured. A useful test of the adequacy of the statistics is to perform a running-average test. As measurements are made, the mean-linear-intercept size or other parameters are continually re-computed to give running averages which are plotted against the total number of measurements made. The mean result will be seen to fluctuate but to converge towards the true mean for the microstructure with increasing numbers of measurements. Measurements can be halted when the residual fluctuations in the mean are adequately small.

### 7.3.2 Large WC grain sizes

Before deciding which magnification to use for grain-size measurement, a preliminary scan of the etched surface is useful to determine if there are large grains of WC present. If too high a magnification is used, these grains may not fit into the field of view and this will thus affect the measurement statistics. Ideally, the magnification used should be such that the largest WC grain imaged is at most (giving 10 to 20 grains across the view) a third of the field of view as a general guide. In practice, there will always be large grains that intercept the edges of the field of view and thus are not measured. However, if sufficient fields of view are measured and the running-average technique (see Figure A.4) is used, the affect of large grains is minimised.

### 7.3.3 Smallest measurable intercept

At present, there are no standards available relating to the smallest intercept size that can be measured by either optical or scanning electron microscopy. As a guide, the resolution of the instrument used can be used to determine the smallest intercept which can be measured. For a particular resolution, the lower limits to which linear intercepts can be measured are given in Table 1. These figures represent the highest resolution which can be obtained under optimum conditions. In practice, poorer resolution may be obtained, particularly when it is difficult to obtain high resolution images because of problems with surface preparation. Thus, the length of the smallest intercept which may be sensibly measured will increase. The smallest intercept which may be sensibly measured will increase. In practice, the smallest intercept measurable is twice the resolution of the instrument and the uncertainty of the measurement is twice the resolution.

**Table 1 — Guide to feasible measurements**

| Instrument | Maximum resolution | Minimum visible intercept length [a] |
|---|---|---|
| Optical microscope | 230 nm [b]<br>350 nm [c] | 500 nm [b]<br>700 nm [c] |
| Scanning electron microscope | 20 nm [b]<br>200 nm [c] | 40 nm [b]<br>200 nm [c] |
| Field emission scanning electron microscope | 1,5 nm [b]<br>10 nm [c] | 3 nm [b]<br>20 nm [c] |

[a] At maximum resolution of the microscope. This will increase at lower magnifications.

[b] Theoretical resolution on a calibration sample.

[c] Practical resolution on typical hardmetal image.

Smaller intercept lengths than those recommended may be measured, but the error in these measurements will increase rapidly. As a guide, the error of measuring the start and finish of an intercept line is twice the resolution, thus to obtain an error less than 10 %, the intercept line should be at least twenty times the theoretical resolution. Thus, for an optical microscope working at the highest numerical aperture, only intercept distances greater than about 5 µm can be measured with an error of less than 10 %. If the microstructure has a large proportion of WC grains whose linear intercepts are less than 5 µm, the error in measuring these could effect the measured mean linear intercept and distort the grain-size distribution. In this case, scanning electron microscopes should be used.

The choice of magnification to accommodate the largest WC grains present also has an effect on the smallest intercept length, which can be measured. Lower magnification objectives for low magnification (LOM) have, in general, lower numerical aperture values and thus lower resolution. For SEM, lower magnification means that the electron beam is sampling with a larger step size. The values in Table 1 represent the maximum resolution that may be obtained. For a LOM, this would be for a ×100 oil immersion objective with a numerical aperture of 1.3.

## 8 Reporting

When reporting the results of grain-size measurement, all relevant information should be given to ensure traceability in those measurements. A typical checklist should, for example, contain the following information:

— sample identification;

— etchant and etching time;

— traceability, calibration graticule number and calibration certificate;

— imaging technique: optical SEM or FESEM;

— magnifications used: one or more;

— number of fields of view measured;

— total number of intercepts;

— arithmetic the mean-linear-intercept size;

— size distribution; as recommended in this guide; if other, then state the method;

— additional comments.

Additional information may also be necessary to fit in with a quality system. These might involve the identification of the image or photo micrograph(s) if archived, as well as information about the source of the material and the customer requesting the measurement to be made.

It is also useful to consider the following additional points:

— largest intercept measured;

— smallest intercept measured;

— largest grains;

— numerical aperture of objective for optical microscopy;

— accelerating voltage, working distance, illuminating aperture for SEMs.

Finally, it is recommended to add a comment on measurement uncertainties. These are usually about ± 10 % when a population of 200 WC grains is measured to obtain a mean value of intercept length.

# Annex A
(informative)

# Measurement case study

This annex describes the measurement of WC grain size using the arithmetic mean-linear-intercept technique.

The image in Figure A.1 was chosen for clarity in describing the method for measuring grain size and size distribution using the linear-intercept technique. Smaller grains have been removed from the image so as not to confuse the technique. In practice, all grain intercepts should be measured.

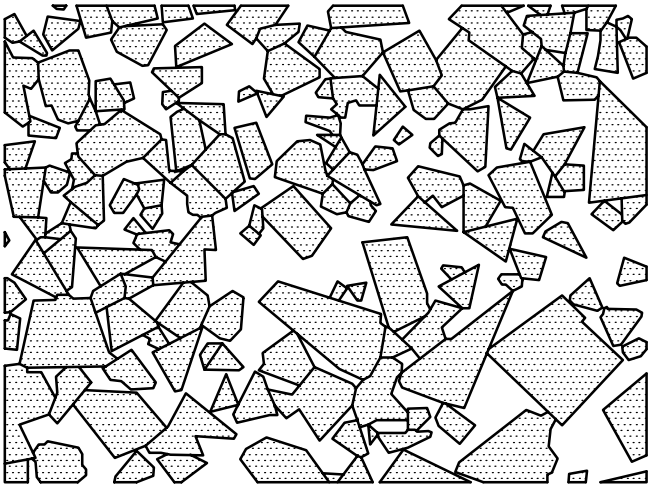


**Figure A.1 — Idealised WC/Co hardmetal structure**

**Step 1**

Obtain an image of the microstructure; this should be representative of the material and free from polishing artefacts. The image may be obtained from an SEM or optical microscope.

The choice of magnification will depend upon the grain size, but typically should contain about 10 to 20 grains across the field of view, as shown in Figure A.1.

**Step 2**

Obtain an image of a scale or stage micrometer traceable to National Standards. This should be at the same magnification and conditions as used to obtain the image of the hardmetal microstructure as in Step 1.

**Step 3**

From the calibration image obtained in Step 2, determine the magnification of the image using a Steel Rule (traceable to National Standards). Measure the distance between set features on the image of the calibration stage micrometer, to the nearest 0,5 mm. Calculate the distance between the set features on the stage micrometer. In the case of the graticule shown in Figure A.2, the distance between lines is 10 µm, so if the distance between the centres of lines 1 and 81 is measured, the actual size measured is 800 µm. The magnification obtained will depend upon the subsequent technique used to print the image. Equation (A.1) shall be used to determine the magnification:

$$m = \frac{s_{\mathrm{m}}}{s_{\mathrm{a}}} \qquad \text{(A.1)}$$

An estimate of the error of magnification may be made by measuring over different lengths of the calibration graticule and obtaining a mean and standard deviation of the magnification. Alternatively, a maximum and minimum error of the magnification may be obtained by assuming that the visual error of measurement is ± 0,5 mm.

Equation (A.2) shall be used to determine the maximum magnification:

$$m_{\max} = \frac{s_{\mathrm{m}} + 0{,}5}{s_{\mathrm{a}}} \tag{A.2}$$

Equation (A.3) shall be used to determine the minimum magnification:

$$m_{\min} = \frac{s_{\mathrm{m}} - 0{,}5}{s_{\mathrm{a}}} \tag{A.3}$$

For this particular example, Figure A.2 was reproduced from Figure A.1 and enlarged. The image of the calibration graticule was also enlarged by the same amount and the final mean magnification was calculated as × 5750.

**Step 4**

A series of parallel lines are drawn across the image of the hardmetal microstructure as shown in Figure A.2. These lines should be placed a sufficient distance apart, such that only one line can intercept any particular grain. The number of lines placed on an image therefore depends upon the grain size.

**Step 5**

Measure the length of line overlaying each carbide grain. These are the linear-intercept lengths. The linear-intercept lengths are shown in the image measurement mask shown in Figure A.3. Grains which touch the border of the image should not be measured as the intercepts lengths are incomplete. The measured intercept lengths are entered into a table as shown in Table A.1. The use of a spreadsheet is recommended.

**Step 6**

For each of the measured intercept lengths, the true intercept length is obtained by dividing by the mean magnification. From these, the mean intercept length and standard deviation can be calculated as shown in Table A.1. At least 200 intercepts should be measured, see 7.1.

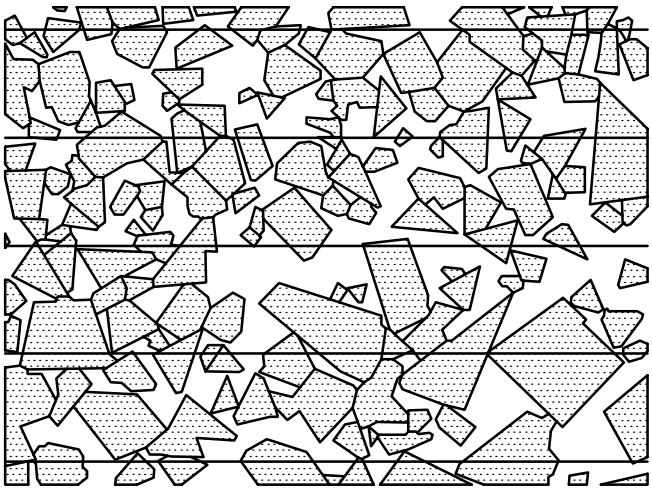


**Figure A.2 — Linear-intercept lines drawn across Figure A.1**

The lines in Figure A.2 have been placed far enough apart such that no carbide grain is intercepted by more than one line. More or fewer lines may be drawn on the image, depending upon the grain size.

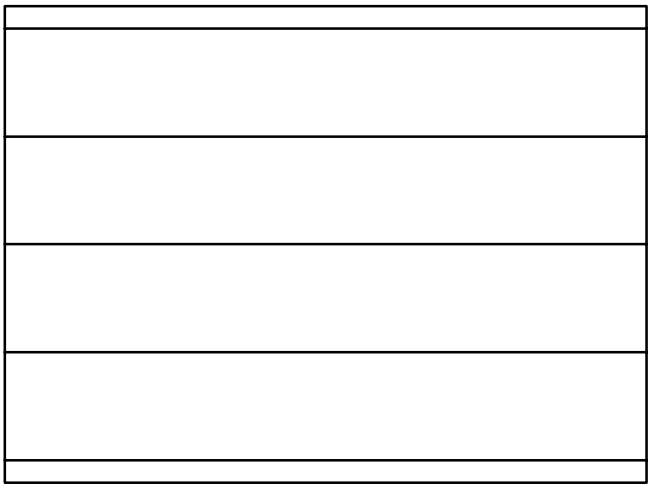
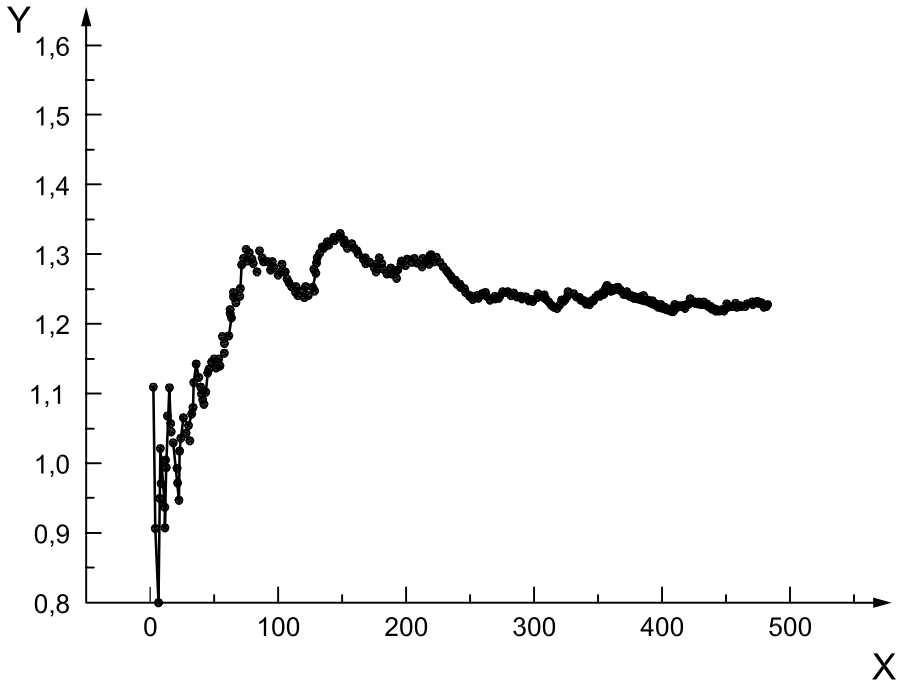


**Figure A.3 — Linear intercepts for measurement from Figure A.2**

Intercepts touching the edge of the image should not be measured.




**Key**

X Number

Y Running-average intercept, µm

**Figure A.4 — Convergence of average mean-linear-intercept**

It can be useful to plot the running-average mean intercept against the number of intercepts measured as a guide to how many intercepts need to be measured. In the example shown in Figure A.4, the mean value converges after about 250 measurements.

**Table A.1 — Results of measurements made on image shown in Figure A.1 (for example with 50 grains only**

| | Measured intercept length mm | Calculated intercept length µm |
|---|---|---|
| Line 1 | 10 | 1,74 |
| | 12 | 2,09 |
| | 13 | 2,26 |
| | 6,5 | 1,13 |
| | 18 | 3,13 |
| | 30 | 5,22 |
| | 23,5 | 4,09 |
| | 18 | 3,13 |
| | 8,5 | 1,48 |
| | 5 | 0,87 |
| Line 2 | 11,5 | 2 |
| | 5 | 0,87 |
| | 24,5 | 4,26 |
| | 11 | 1,91 |
| | 4 | 0,7 |
| | 9,5 | 1,65 |
| | 5 | 0,87 |
| | 2 | 0,35 |
| | 4 | 0,7 |
| | 18,5 | 3,22 |
| | 7 | 1,22 |
| | 6 | 1,04 |
| Line 3 | 2 | 0,35 |
| | 8 | 1,39 |
| | 14,5 | 2,52 |
| | 13,5 | 2,35 |
| | 1 | 0,17 |
| | 12 | 2,9 |
| | 17,5 | 3,04 |
| | 12,5 | 2,17 |
| | 10 | 1,74 |
| Line 4 | 34 | 5,91 |
| | 4 | 0,7 |
| | 15,5 | 2,7 |
| | 5,5 | 0,96 |
| | 7 | 1,22 |
| | 17,5 | 3,04 |
| | 23 | 4 |
| | 7 | 1,22 |
| | 2,5 | 0,43 |
| | 24 | 4,17 |
| | 49 | 8,52 |
| | 7 | 1,22 |
| Line 5 | 17,5 | 3,04 |
| | 10 | 1,74 |
| | 14,5 | 2,52 |
| | 27,5 | 4,78 |
| | 6,5 | 1,13 |
| | 18 | 3,13 |
| | 37 | 6,43 |
| Mean intercept length: 2,33 µm<br>Number of intercepts: 50 | | |

# Annex B
(informative)

# Report proforma

| GRAIN SIZE MEASUREMENT REPORT | | | |
|---|---|---|---|
| **REPORT NUMBER** | | **DATE** | / / |
| **CUSTOMER** | | | |
| **ADDRESS** | | | |
| **MATERIAL IDENTIFICATION** | | | |
| **SAMPLE PREPARATION** | | | |
| **ETCHANT AND TIME** | | | |
| **IMAGE IDENTIFICATION** | | **NO OF IMAGES USED** | |
| **ARCHIVED** | **YES/NO** | | |
| **MAGNIFICATION** | | **OBJECTIVE N.A.** | |
| **CALIBRATION CERTIFICATE** | | | |
| **RESULTS** | | | |
| **NO OF INTERCEPTS** | | **MEAN LINEAR INTERCEPT** | |
| **OPERATOR** | | **SIGNATURE** | |

The following is a Report proforma containing results from the measurements detailed in Table A.1.

| GRAIN SIZE MEASUREMENT REPORT | | | |
|---|---|---|---|
| **REPORT NUMBER** | CMMT/000/000 | 10 / 04 / 1999 | |
| **CUSTOMER** | National Physical Laboratory | | |
| **ADDRESS** | Queens Road, Teddington<br>Middlesex, TW11 OLW | | |
| **MATERIAL IDENTIFICATION** | CWC25C, Materials Index Code WCX/04/01 | | |
| **SAMPLE PREPARATION** | Prepamatic polishing machine, procedure QPCMMT/B/136.<br>Grinding stages, 120, 65 and 20 μm diamond. Lapping 6 μm<br>Petrodisc M, Polishing 6, 3, and 1 μm diamond on DP Pan cloth. | | |
| **ETCHANT AND TIME** | Murakami's reagent, 10 g KOH in, 10 gms $K_3Fe(CN)_6$ in 200 ml water<br>Etched 6 minutes at room temperature. | | |
| **IMAGE IDENTIFICATION** | CWC25C.jpg | 1 | |
| **ARCHIVED** | YES | | |
| **MAGNIFICATION** | X1600 | 1.4 | |
| **CALIBRATION CERTIFICATE** | | | |
| **RESULTS** | | | |
| **NO OF INTERCEPTS** | 50 | 2.33 μm | |
| **OPERATOR** | E G Bennett | E G Bennett | |

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**ICS 77.040.99; 77.160**

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