INTERNATIONAL STANDARD 4499

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Hardmetals — Metallographic determination of microstructure

Métaux-durs — Détermination métallographique de la microstructure

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FOREWORD

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

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It has been approved by the member bodies of the following countries :

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Austria	Italy	Sweden
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No member body expressed disapproval of the document.

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Hardmetals — Metallographic determination of microstructure

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies methods of metallographic determination of the microstructure of hardmetals.

2 SYMBOLS AND DESIGNATIONS

TABLE 1

Symbol	Designation
α-phase	Tungsten carbide
β-phase	Binder phase (for example based on Co, Ni, Fe)
γ-phase	Carbide having a cubic lattice (for example TiC, TaC) which may contain other carbides (for example WC) in solid solution
η-type phases	Multiple carbides of tungsten and at least one metal of the binder phase

3 APPARATUS

3.1 Metallographic microscope, permitting observations at magnifications up to 1 500 X.

3.2 Equipment for preparation of test-piece sections

4 PREPARATION OF TEST-PIECE SECTION

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 may lead to a wrong evaluation of microstructure.

NOTE — There are several methods for preparation of hardmetal surfaces for metallographic examination. 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.

5 PROCEDURE

The microstructure is examined by the gradual development of phases by etching. There are several methods for etching hardmetal surfaces for metallographic examination. Examples of suitable etching techniques are given in table 2. Care shall be taken to ensure that the true microstructure is revealed.

5.1 The existence of η-type phases is determined after lightly etching the section, for example by technique 1 (see table 2). Phases of η-type are coloured orange to brown, while other phases are unetched. Etching by technique 1 does not prevent subsequent etching by techniques 2 and 3.

TABLE 2

Etching technique	Composition of etchants	Conditions of etching	Application of etching
1	**A** Freshly prepared mixture of equal quantities of 10 to 20 % (*m/m*) aqueous solutions of potassium hexacyanoferrate(III) (potassium ferricyanide) and potassium or sodium hydroxide	Etch in mixture A at approximately 20 °C for 1 to 20 s. Flush the test-piece section with water immediately, without removing the oxide layer. Dry the surface carefully with acetone or alcohol without wiping	Identification of η-phases
2	**A** See technique 1 **B** A mixture of equal volumes of concentrated hydrochloric acid and water	Etch at approximately 20 °C in mixture A for 3 to 4 min. Then wash in water and etch in mixture B for approximately 10 s. Next wash in water, then in alcohol, and dry the test-piece section. Finally, etch in mixture A for approximately 20 s	Identification of γ-phase
3	**A** See technique 1	Etch in mixture A at approximately 20 °C for 3 to 6 min	Identification of α-phase

NOTE — The separate solutions of potassium hexacyanoferrate(III) and potassium or sodium hydroxide may be stored for a long time, but must be freshly mixed each day when used.

The whole surface is examined at a low magnification and, if necessary, at magnifications up to 1 500 X. The existence and distribution of η-type phases is noted and recorded.

5.2 The existence of γ-type phases is determined after etching the surface, for example by technique 2 (see table 2). This phase is coloured light yellowish-brown and has a typically rounded shape (see figure 1). The etched section is examined and the existence of γ-phase is noted and recorded. Its size is estimated and recorded according to figure 1 as γ-fine, γ-medium or γ-coarse.

5.3 The existence of α-phase is determined after etching the surface, for example by technique 3 or, in the case of the presence of γ-phase, by technique 2 (see table 2). The α-phase appears grey and often has an angular shape. The etched section is examined and the existence of α-phase is noted and recorded. Its size is estimated and recorded according to figure 2 as α-fine, α-medium or α-coarse.

5.4 The β-phase is identified after etching the surface, for example by technique 2 or 3 (see table 2). This phase remains white.

6 TEST REPORT

The test report shall include the following information :

a) reference to this International Standard;

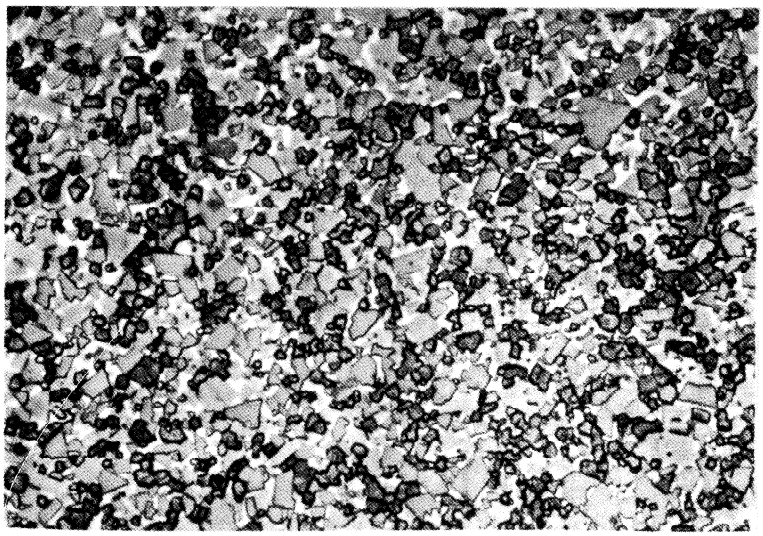
b) all details necessary for identification of the test sample;

c) the result obtained;

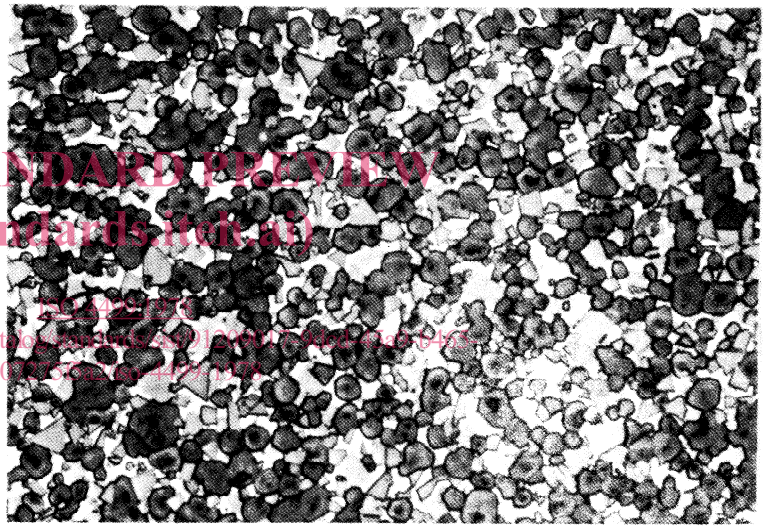
d) all operations not specified by this International Standard, or regarded as optional;

e) details of any occurrence which may have affected the result.

γ-fine



γ-medium



γ-coarse

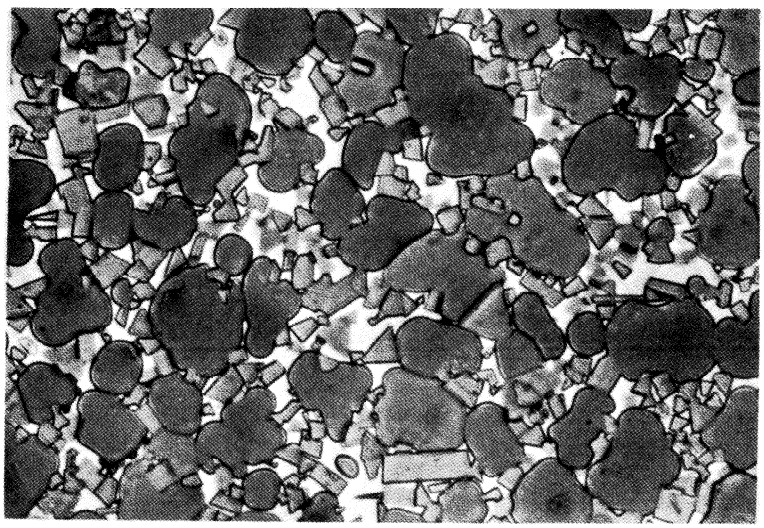
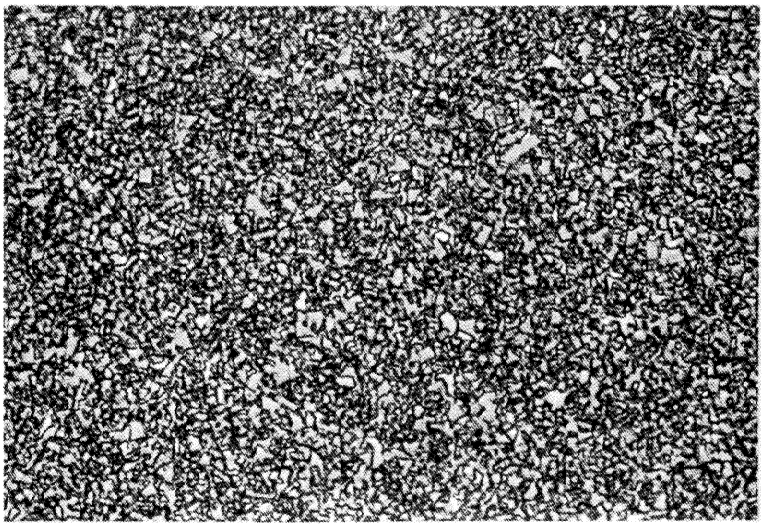
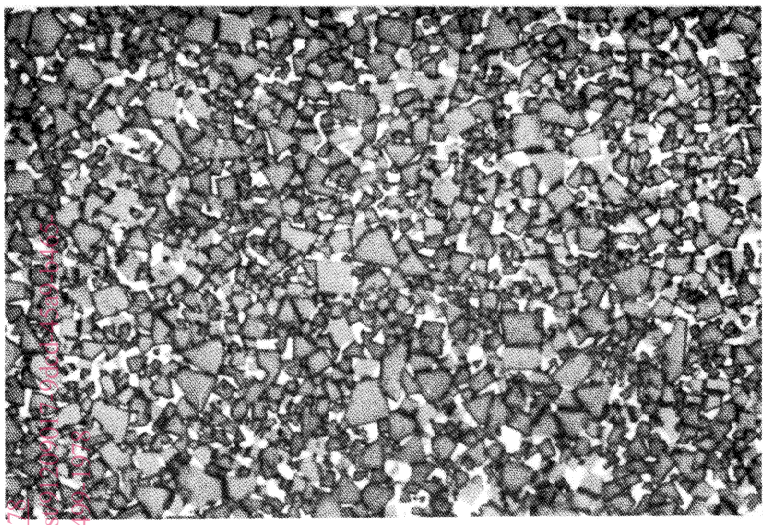


FIGURE 1 — γ-phase X 1 500

α-fine



α-medium



α-coarse

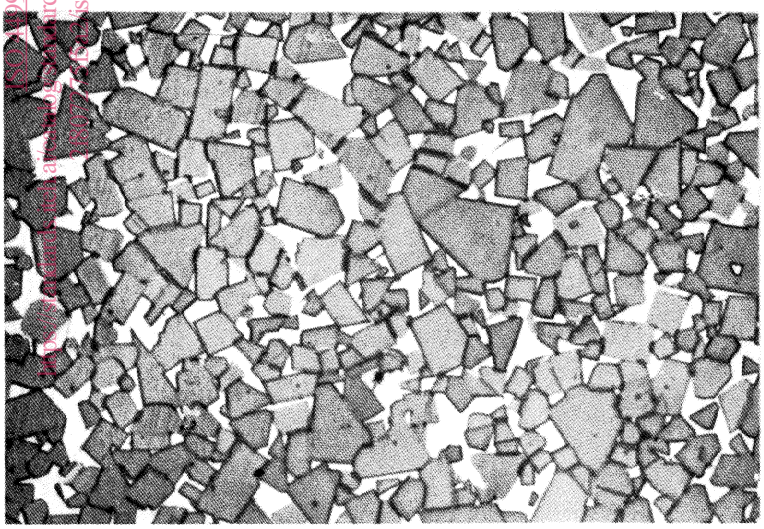


FIGURE 2 – α-phase × 1 500

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