This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

Designation: B657 – 23

Standard Guide for Metallographic Identification of Microstructure in Cemented Carbides[1]

This standard is issued under the fixed designation B657; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope*

1.1 This guide covers apparatus and procedures for the metallographic identification of microstructures in cemented carbides.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Precautions applying to use of hazardous laboratory chemicals should be observed for chemicals specified in Table 1.

1.3 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

B665 Guide for Metallographic Sample Preparation of Cemented Tungsten Carbides

2.2 *ISO Standard:*[3]

ISO 4499 Hardmetals—Metallographic Determination of Microstructure

3. Terminology

3.1 *Definitions of Symbols:*

Symbol	Definition
α phase	tungsten carbide
β phase	binder (for example, Co, Ni, Fe) that may contain alloying elements in solid solution (for example, W, C, Ru, Re, Mo)
γ phase	carbide of a cubic lattice (for example, TiC, TaC, NbC) that 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

4. Significance and Use

4.1 The microstructure of a cemented carbide affects the material's mechanical and physical properties. This guide is not intended to be used as a specification for carbide grades. Producers and users may use the microstructural information as a guide in developing their own specifications.

5. Apparatus

5.1 *Metallographic Microscope* capable of magnifications up to 1500 times.

5.2 Ordinary metallurgical laboratory equipment.

5.3 Equipment for specimen preparation as outlined in Guide B665.

6. Specimen Preparation

6.1 A suitable procedure is described in Guide B665.

7. Procedure

7.1 Examine the microstructure by gradual development of the phases by etching. Examples of suitable etching techniques are given in Table 1. Examples of the appearance of some expected phases, in fine, medium, and coarse grain sizes, are given in Fig. 1.

7.2 Determine the presence of η-type phases by lightly etching half the section with Technique 1 (see Table 1). Examine the entire section at low magnification and, if necessary, at magnifications up to 1500 times. Phases of η-type

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

*A Summary of Changes section appears at the end of this standard

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TABLE 1 Etching Techniques

NOTE 1—The separate solutions of potassium hexacyanoferrate (III) and potassium or sodium hydroxide may be stored for a long time. Etchant solution of potassium hexacyanoferrate (III) and potassium or sodium hydroxide should be discarded after a week of use or when etching times exceed recommended times in Table 1.

NOTE 2—The times in the conditions for etching are for concentrations listed. Deviation from these concentrations will affect times required to reveal desired structures.

Etching Technique	Composition of Etchants	Conditions of Etching	Objective of Etching
1	Freshly prepared mixture of equal quantities of 10 %(mass/mass) aqueous solutions of $K_3Fe(CN)_6$ (III) (potassium ferricyanide) and potassium or sodium hydroxide	Etch at approximately 20°C for 2 to 10 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 phase
2	A Same as 1A	Etch at approximately 20°C for 2 to 4 min. Swabbing surface may enhance the effects of etchant.	Identification of α, β, and γ phases

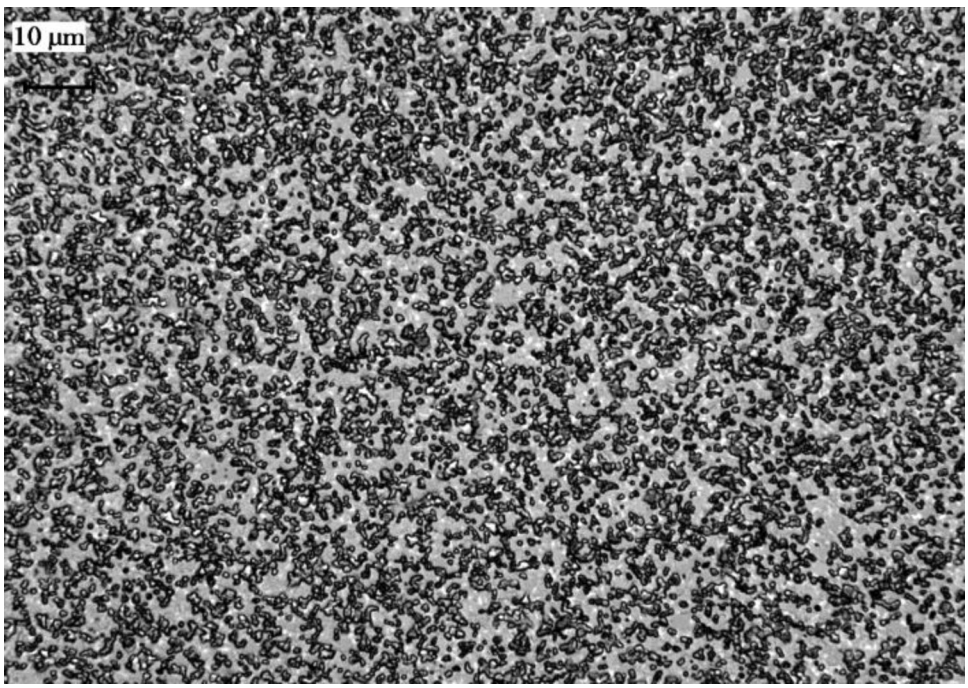

FIG. 1 γ Phase Fine, All 8.5% Co, 5.9% TiC, 5.5% TaC, 2.4% NbC, balance WC

are colored orange to brown in the etched portion, and white to light gray in the unetched portion. γ-Phase may also etch lightly and appear brownish in the etched portion, but will have a brown/gold color even in the unetched portion, thereby differentiating it from η-phase. The other phases remain unetched. Etching by Technique 1 does not preclude subsequent etching by Technique 2. Note and record the existence of η-type phases and their distribution.

7.3 Determine the presence of γ phases by etching with Technique 2 (Table 1). This phase appears light yellowish brown and has a typically rounded shape (see Fig. 1). Examine the etched section and note and record the existence of a γ phase.

7.4 Determine the presence of α phase in the same etched specimen as in 7.3. The α phase appears gray, angular (idiomorphic), and straight-slided (see Fig. 2). Examine the etched section and note and record the presence of α phase.

7.5 Identify the β phase in the same etched specimen as 7.3 and 7.4. This phase remains white and is usually the continuous, interstitial phase between the α and γ grains.

NOTE 1—This procedure follows essentially ISO 4499.

8. Report

8.1 The report shall include complete identification of the specimen and the phases present.

9. Keywords

9.1 cemented carbides; hardmetals; microstructure; powder metallurgy

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