

SINTERED CARBIDE TOOLS

Foreword—This Document has also changed to comply with the new SAE Technical Standards Board format.

1. **Scope**—This recommended practice covers methods for measuring or evaluating five properties or characteristics of sintered carbide which contribute significantly to the performance of sintered carbide tools. These properties are: hardness, specific gravity, apparent porosity, structure, and grain size. They are covered under separate headings below.

2. **References**—There are no referenced publications specified herein.

3. Hardness

3.1 **General**—The Rockwell hardness tester provides a simple, rapid, and reliable means of measuring the hardness of sintered carbide tools. A hardness value is easily obtained, but is subject to error if precautionary measures are not taken in making this test. Hardness determinations, therefore, shall be made according to the requirements outlined below and in ASTM E 18, Methods of Test for Rockwell Hardness and Rockwell Superficial Hardness of Metallic Materials.

3.2 Apparatus

- Rockwell hardness testing machine with 60 kg load and diamond brale penetrator for use with the A scale¹.
- Two tungsten carbide test blocks with a hardness of 90.0 and 92.0 Rockwell A (RA) respectively.

3.3 **Material - Sample**—Preparation of the surface of the specimen prior to making the hardness test is of major importance. It is recommended that a finish equivalent to that produced with a 220 grit diamond grinding wheel be obtained on the surface which is to be checked for hardness. Because of the shallow penetration of the diamond penetrator used in making this test, the surface being tested for hardness must be parallel to the surface opposite of that being tested. Both surfaces must be smooth and devoid of any bulge or other irregularity affecting parallelism. If these two surfaces are only slightly out of parallel, an error will be obtained in the hardness reading.

1. It is recommended that a diamond brale especially selected for use with the Rockwell "A" scale be used for this type of testing. This type of penetrator is of higher quality, free from chips and other imperfections, and should be specified for Rockwell "A" scale use. Slowing down of the rate of speed at which the major load is applied during testing will aid in increasing the life of the diamond brale. This change in load application does not affect the accuracy of the hardness reading. The use of the superficial scale is not recommended for hardness testing of sintered carbides unless extreme care is exercised with regard to parallelism and smoothness during surface preparation of the specimen.

It is important that the Rockwell hardness testing machine is located in such a manner and area that it is free from vibration while hardness tests are being performed. Vibration is detected by the bounding effect transmitted through the needle of the indicator after the major load has been applied.

- 3.4 Procedure**—The hardness test shall be made using the RA scale. This reading is obtained by observing the deflection of the needle pointer on the black scale with a 60 kg load and the diamond brale penetrator.²

Before making the hardness test on the carbide material, the Rockwell testing machine shall be checked for accuracy, using a tungsten carbide test block of known hardness. Two check blocks of different hardness values are recommended to assure accurate hardness readings over the general range of hardness of the common grades of sintered carbides. The check blocks should have a hardness of 90.0 and 92.0 RA respectively. The check block having the hardness closest to the expected hardness of the carbide material to be checked shall be selected for calibrating the Rockwell tester. The average of five readings should check within ± 0.2 of a hardness number. If the Rockwell tester varies appreciably from the hardness number of the test block, the dial of the machine must be adjusted so that the correct reading is obtained. The amount of variation is noted and this correction plus or minus is applied when taking the hardness reading on the specimens of sintered carbide being tested. This dial adjustment will be made just before the major load is applied. With careful manipulations, hardness readings can be accurately duplicated when the hardness tester is calibrated in this manner.

4. Specific Gravity

- 4.1 General**—The specific gravity of sintered carbide tool materials shall be determined by the immersion method, using as a basis the difference in weight of the carbide in air and in water.

4.2 Apparatus

- a. A standard analytical balance of 200 g capacity and 0.1 mg sensitivity at full load.
- b. A 150- or 250-ml beaker, depending upon the size of the carbide specimen.
- c. Small diameter nonferrous wire.
- d. Thermometer 0–100 °C for room temperatures capable of being read to nearest 0.5 °C.

4.3 Materials

- a. The specimen shall be surface ground all over with a 100 grit diamond wheel before testing.
- b. Distilled water.

4.4 Procedure

- a. Weigh the specimen, to the nearest 0.5 mg.
- b. Support a beaker of distilled water³ over the pan of the balance by a suitable bridge. Water level should be high enough to cover the specimen by at least 1/4 in.
- c. Suspend the specimen and the wire from the beam hook, placing the specimen in the water, and weigh to the nearest 0.5 mg.
- d. Remove the specimen from the wire and weigh the wire alone in water. Subtract this weight from the total weight found in step c.
- e. Observe the temperature of the water to the nearest 1.0 °C.

2. See Footnote 1.

3. Care should be used to see that no air bubbles are present on the sample after immersion, and that the wire twist on the sample is completely submerged. Several drops of a suitable wetting agent will aid in eliminating air bubbles.

4.5 Calculations

W_a = Weight of Specimen in Air

W_w = Weight of Specimen in Water

D = Relative Density of Water at Test Temperature (Density relative to that of Water at 4 °C)

$$\text{Specific Gravity} = \frac{W_a \times D}{W_a - W_w} \quad (\text{Eq. 1})$$

5. Apparent Porosity, Structure, And Grain Size

5.1 General—Apparent porosity, structure, and grain size shall be evaluated by metallographic examination, as outlined below.

Apparent porosity is the term applied to the inherent porosity, non-metallic inclusions, and uncombined carbon as observed in the microstructure of the properly prepared surface of sintered carbides.

Structure refers to the type and distribution of the metal carbides and binder material observed in the microstructure of the properly prepared surface of sintered carbides.

Grain size is the term applied to the predominating particle sizes, in microns, of the metal carbides observed in the microstructure.

5.2 Sample Preparation—Select a specimen approximately 1/2 in. square from the area of particular interest of the sample to be tested. Sectioning should be done with a diamond cutoff wheel. Mount unwieldy specimens in hard bakelite or its equivalent, then grind as follows:

- a. Rough grind using a green silicon carbide wheel.
- b. Fine grind using a 320 grit diamond wheel running at a speed of approximately 5500 surface fpm.

Samples should be polished using the ordinary metallographic polishing equipment. Impregnate a paper polishing disc, properly attached to the bronze disc of the polishing lap, with a light (SAE 10) oil. Apply diamond paste to the oiled paper and work it in with the fingertip. At least two polishing laps should be used in the following order:

- a. A diamond lap using a 10 μm maximum diamond powder.
- b. A diamond lap using a 1 μm maximum diamond powder.

Hold the specimen 1-3 in. from the center of the lap running at approximately 1150 rpm. Considerable pressure should be exerted on the specimen while polishing in intervals of approximately 10 sec. Rotating the specimen 90 deg between each interval is recommended. (CAUTION: Light pressure and too much polishing may cause pitting of the specimen.)

Polishing is ineffective when the specimen is above approximately 150 °F; therefore, polishing time should be carefully watched to keep the temperature of the specimen below this point. Extreme cleanliness is necessary to prevent contamination of the diamond laps. The specimen should be washed thoroughly with a suitable solvent after each polishing operation.

- 5.3 Apparent Porosity Evaluation**—After the prescribed sample preparation, the sample shall be examined in the unetched condition at a magnification of 200X.

A porosity rating shall be made by comparing the observed field with the porosity charts of Figures 1–3.

The rating charts depict both the type of porosity, designated alphabetically, and the quantity of porosity, designated numerically. Type A classifies porosity sizes under 10 μm in diameter; Type B classifies porosity sizes between 10 and 40 μm in diameter; Type C classifies cluster porosity or that developed by the presence of uncombined carbon, and is considered the type most detrimental to tool performance.

- 5.4 Structure Evaluation**—After the prescribed sample preparation the sample shall be etched and examined at a magnification of 1500X. The etchant shall consist of a fresh solution having equal parts of 10% potassium hydroxide and 10% potassium ferricyanide. The sample shall be immersed in the etchant for 2 minutes, then rinsed with water and the polished surface swabbed with wet cotton. A second immersion for approximately another 2 minutes shall be made to delineate the structure. The sample shall then be washed with water and dried with alcohol and air to prevent staining. Examination of the prepared surface shall be made with a metallographic microscope utilizing an oil immersion objective. Figures 4A and 4B are typical photomicrographs of tungsten carbide (WC) with 6% cobalt and 13% cobalt, respectively. Figures 4C and 4D are typical photomicrographs of tungsten carbide (WC) plus solid solution carbide (WC-TiC-TaC) with 4.5% cobalt and 11% cobalt, respectively. The tungsten carbide particles are angular and gray in appearance, the solid solution particles, where present, are rounded and usually darker gray, and the cobalt binder appears white. The abnormal "eta phase" carbide is not depicted by the photomicrographs. It is a brittle, carbon deficient carbide detrimental to tool performance but is readily detected as a very rapid etching, black constituent.

The data provided by this test are an excellent indicator for identifying a particular producer's product and its uniformity.

- 5.5 Grain Size Evaluation**—Sample preparation, etching technique, and equipment shall be the same as described for structure evaluation.

The grain size shall be determined by comparing representative areas of the observed sample field with the carbide grain size chart (Figure 5). This chart illustrates the relationship of particle sizes from 1 to 10 μm as observed at a magnification of 1500X.

The grain size rating shall consist of a sequence of numbers such as 231. Each number refers to a carbide particle size range; that is a "1" includes all particles which are 1 μm or finer, a "2" includes all particles over 1 through 2 μm , a "3" includes all particles over 2 through 3 μm , etc., as illustrated by the carbide grain size chart. The sequence of the numbers shall be in the order of the sample area they represent, with the first number representing the greatest area. A minimum of 80% of the representative sample area shall be included in the rating.

Grain size and distribution has considerable influence on the mechanical properties of sintered carbide. Thus, materials having similar composition but different grain size and distribution may have very different performance characteristics.

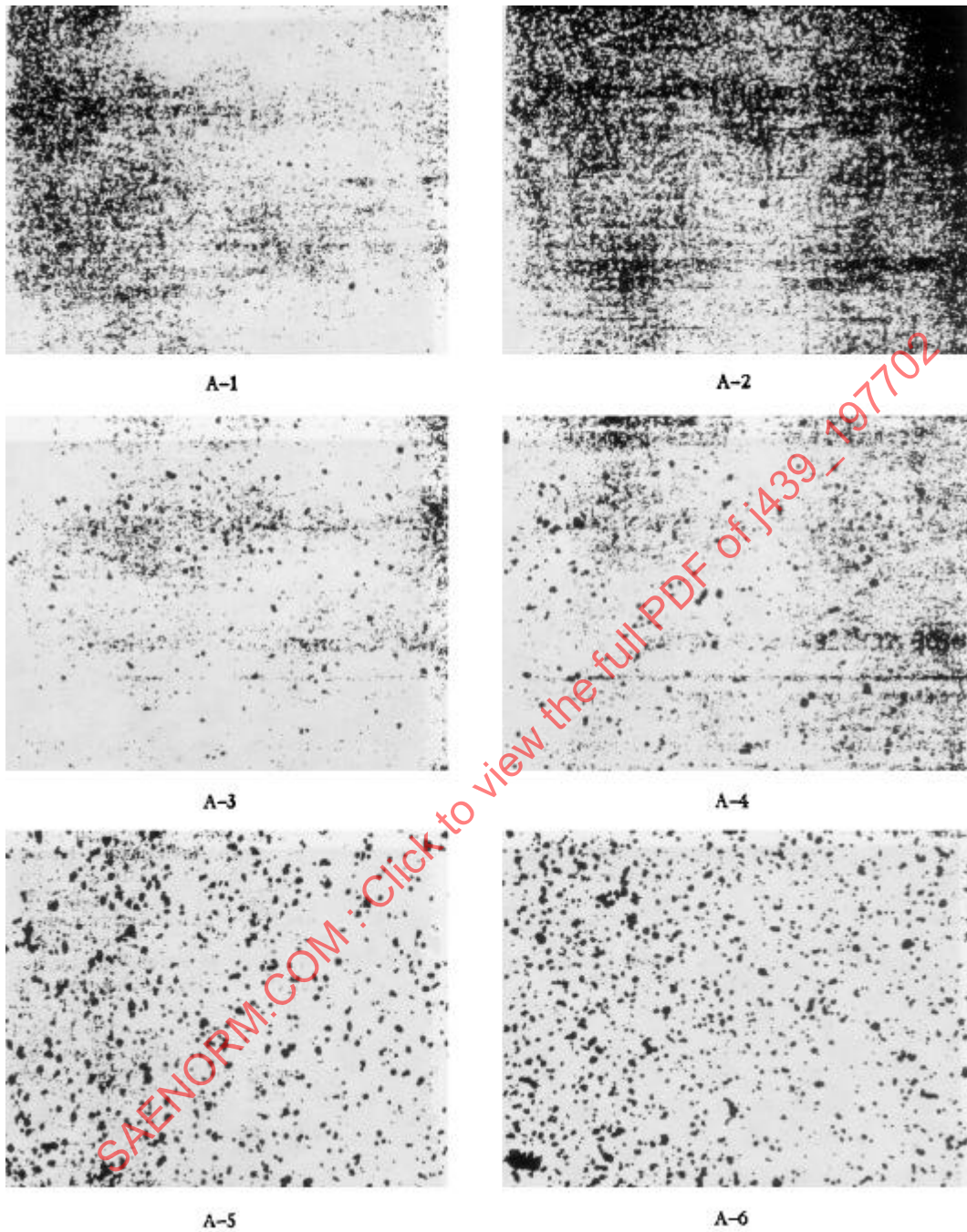


FIGURE 1—TYPE A - APPARENT POROSITY MICROSTRUCTURE OF CEMENTED CARBIDES (X200) (B 276)

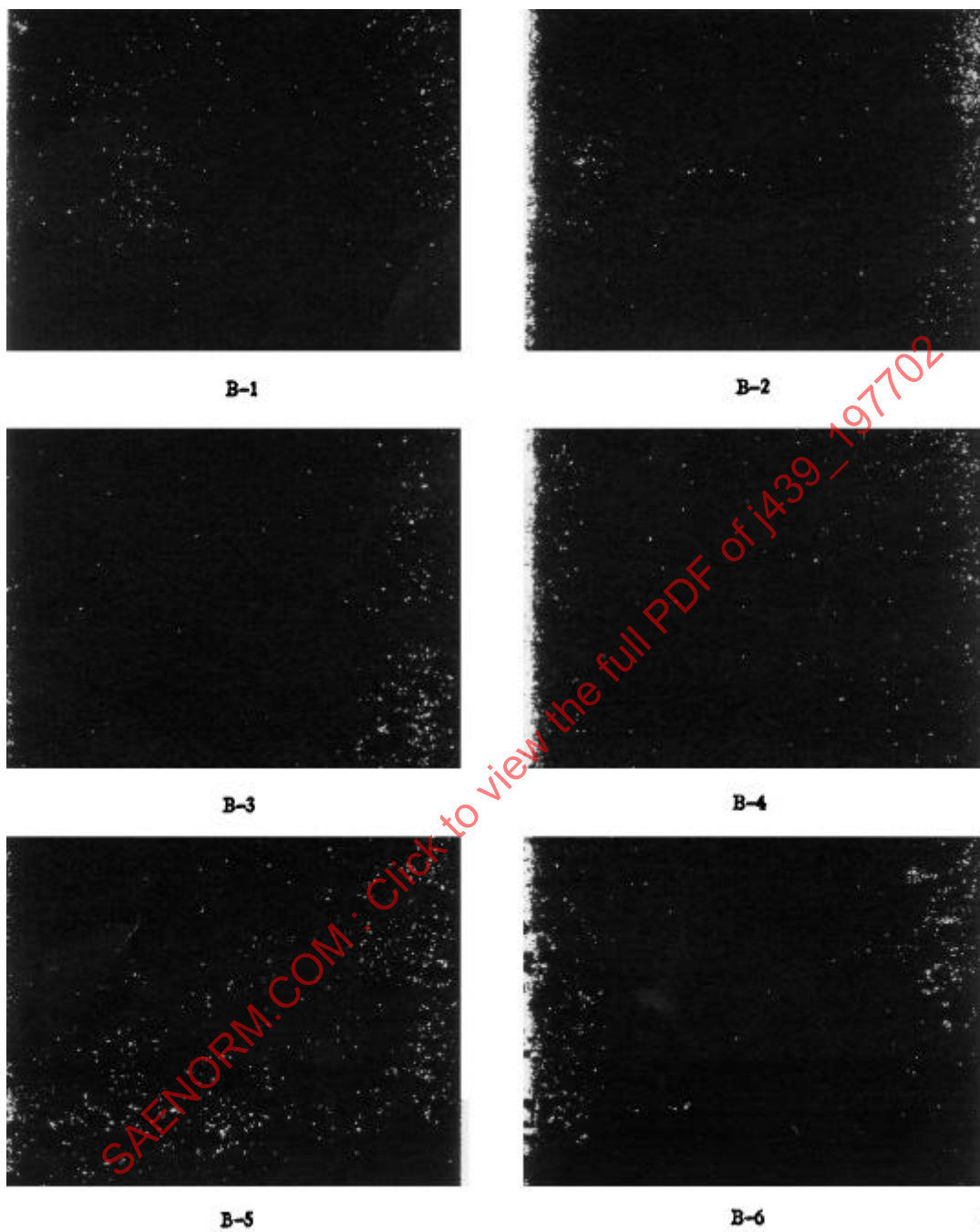


FIGURE 2—TYPE B - APPARENT POROSITY MICROSTRUCTURE OF CEMENTED CARBIDES (X200) (B 276)

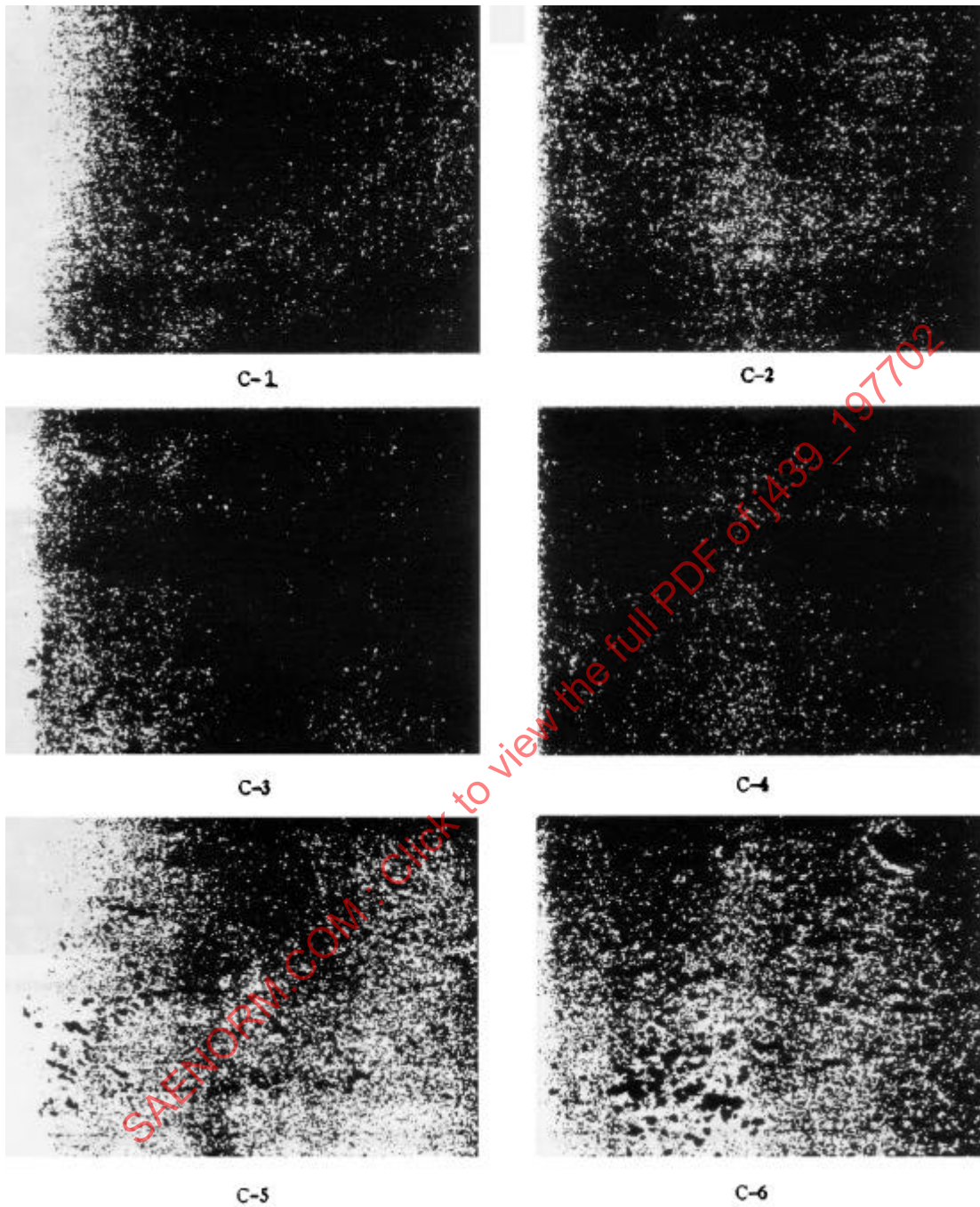


FIGURE 3—TYPE C - APPARENT POROSITY MICROSTRUCTURE OF CEMENTED CARBIDS (X200) (B 276)