

Designation: F 68 - 99

# Standard Specification for Oxygen-Free Copper in Wrought Forms for Electron Devices<sup>1</sup>

This standard is issued under the fixed designation F 68; 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  $(\epsilon)$  indicates an editorial change since the last revision or reapproval.

#### 1. Scope \*

- 1.1 This specification establishes the requirements for wrought and fabricated shapes made from Copper UNS<sup>2</sup> No. C10100, conforming to the chemical requirements of Specification B 170. Grade 1, and suitable for use in electron devices.
- 1.2 The requirements of this specification are in addition to those prescribed in the following product specifications appropriate to the material being ordered: B 1, B 2, B 75, B 75M, B 152, B 152M, B 170, B 187, B 187M, B 272, and B 372. In case of conflict, however, this specification shall take precedence.
- 1.3 The inch-pound units are the standard for this specification except for grain sizes which are in metric units. Metric values given in parentheses are for information only.
- 1.4 The following safety hazard caveat applies to sections 17.4, 17.5 and 18.7 of this specification: 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 and health practices and determine the applicability of regulatory limitations prior to use.

#### 2. Referenced Documents

- 2.1 The following documents in the current issue of the Annual Book of ASTM Standards form a part of this specification to the extent referenced herein:
  - 2.2 ASTM Standards:
  - B 1 Specification for Hard-Drawn Copper Wire<sup>3</sup>
  - B 2 Specification for Medium-Hard-Drawn Copper Wire<sup>3</sup>
  - B 3 Specification for Soft or Annealed Copper Wire<sup>3</sup>
  - B 75 Specification for Seamless Copper Tube<sup>4</sup>
  - B 75M Specification for Seamless Copper Tube (Metric)<sup>4</sup>
- <sup>1</sup> This specification is under the jurisdiction of ASTM Committee B-5 on Copper and Copper Alloys and is the direct responsibility of Subcommittee B05.02 on Rod, Bar, Wire, Shapes, and Forgings.
- Current edition approved April 10, 1999. Published July 1999. Originally published as F 68 66 T. Last previous edition F 68 93.
- <sup>2</sup> The UNS System for copper and copper alloys (see Practice E 527) is a simple expansion of the former standard designation system accomplished by the addition of a prefix "C" and a suffix "00". The suffix can be used to accommodate composition variations of the base alloy.
  - <sup>3</sup> Annual Book of ASTM Standards, Vol 02.03.
  - <sup>4</sup> Annual Book of ASTM Standards, Vol 02.01.

- B 152 Specification for Copper Sheet, Strip, Plate and Rolled Bar<sup>4</sup>
- B 152M Specification for Copper Sheet, Strip, Plate and Rolled Bar (Metric)<sup>4</sup>
- B 170 Specification for Oxygen-Free Electrolytic Copper— Refinery Shapes<sup>4</sup>
- B 187 Specification for Copper Bar, Bus Bar, Rod and Shapes<sup>4</sup>
- B 187M Specification for Copper Bar, Bus Bar, Rod and Shapes (Metric)<sup>4</sup>
- B 188 Specification for Seamless Copper Bus Pipe and Tube<sup>4</sup>
- B 193 Test Method for Resistivity of Electrical Conductor Materials<sup>3</sup>
- B 248 Specification for General Requirements for Wrought Copper and Copper-Alloy Plate, Sheet, Strip and Rolled Bar<sup>4</sup>
- B 248M Specification for General Requirements for Wrought Copper and Copper-Alloy Plate, Sheet, Strip and Rolled Bar (Metric)<sup>4</sup>
- B 249 Specification for General Requirements for Wrought Copper and Copper-Alloy Rod, Bar, Shapes, and Forgings<sup>4</sup>
- B 249M Specification for General Requirements for Wrought Copper and Copper-Alloy Rod, Bar, Shapes, and Forgings (Metric)<sup>4</sup>
- B 250 Specification for General Requirements for Wrought Copper-Alloy Wire<sup>4</sup>
- B 250M Specification for General Requirements for Wrought Copper Alloy Wire (Metric)<sup>4</sup>
- B 272 Specification for Copper Flat Products with Finished (Rolled or Drawn) Edges (Flat Wire and Strip)<sup>4</sup>
- B 372 Specification for Seamless Copper and Copper-Alloy Rectangular Waveguide Tube<sup>4</sup>
- B 577 Test Methods for Detection of Cuprous Oxide Hydrogen Embrittlement Susceptibility in Copper<sup>4</sup>
- E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications<sup>5</sup>
- E 112 Test Methods for Determining Average Grain Sizes<sup>6</sup> E 527 Practice for Numbering Metals and Alloys (UNS)<sup>7</sup>

<sup>&</sup>lt;sup>5</sup> Annual Book of ASTM Standards, Vol 14.02.

<sup>&</sup>lt;sup>6</sup> Annual Book of ASTM Standards, Vol 03.01.

<sup>&</sup>lt;sup>7</sup> Annual Book of ASTM Standards, Vol 01.01.



#### 3. Terminology

- 3.1 For definitions of terms related to this product, refer to the terminology sections of Specifications B 248, B 249, B 250, or B 251.
  - 3.2 Definition of Terms Specific to This Standard:
- 3.2.1 *extrusion pipe*, *n*—a lamination resulting from the flow of the oxide surface of a billet into the article being extruded and usually confined to the trailing-end portion of the extruded product.
- 3.2.2 oxygen-free, adj—electrolytic copper produced substantially free of cuprous oxide and containing no more than 10 ppm oxygen, as determined by metallographic examination at 75× under polarized light, and manufactured without the use of metallic or metalloidal deoxidizers.
- 3.2.3 oxygen-free, grade 1, adj—as defined in 3.2.2 except that the oxygen content must be 5 ppm maximum. This copper is also commonly termed "oxygen-free electronic."
- 3.2.4 rms, n—root-mean-square, a statistical measure of surface roughness usually determined by means of a profilometer.

# 4. Ordering Information

- 4.1 Orders for the product shall include the following information:
- 4.1.1 The designation and year of issue of both Specification F 68 and the basic product specification involved,
  - 4.1.2 Shape of product,
  - 4.1.3 Size,
- 4.1.4 Total estimated weight or number of pieces, or both, for each size and shape.
  - 4.1.5 The Specification F 68 Class of material,
  - 4.1.6 Intended application (for example, wave guide),
  - 4.1.7 Temper,
  - 4.1.8 Heat identification or traceability details,
  - 4.1.9 Certification,
  - 4.2 The following options are available:
  - 4.2.1 Mill test report,
  - 4.2.2 Special packaging,
  - 4.2.3 Special marking.

# 5. Materials and Manufacture

- 5.1 Material:
- 5.1.1 The material shall be oxygen-free electronic copper which conforms to the requirements of Specification B 170, Grade 1.
  - 5.2 Manufacture:
- 5.2.1 The manufacturing process shall conform to the requirements of this specification and to the basic product specification to which the product was ordered.

# 6. Chemical Composition

- 6.1 The cast refinery shape shall conform to the requirements specified in Specification B 170, Grade 1, Table 1.
- 6.1.1 Copper shall be taken as the difference between the sum of results for Grade 1 specified elements and 100 %.
- 6.2 These composition limits do not preclude the possible presence of other unnamed elements. Limits may be established and analysis required for unnamed elements by agreement between the manufacturer and the purchaser.

# 7. Temper

7.1 The temper of the wrought or fabricated product supplied shall conform to the requirements of the basic product specification to which it was ordered.

# 8. Physical Properties

- 8.1 Electrical Resistivity:
- 8.1.1 The maximum mass resistivity shall be 0.15176 ohms g/m²(conductivity 101.0 % minimum, International Annealed Copper Standard {IACS} at 20°C when tested in the annealed condition.
  - 8.2 Scaling:
- 8.2.1 The test specimens of oxygen-free copper shall show substantial surface oxide adherence when subjected to test as described in 15.3.

Note 1—The purpose of this test is to distinguish between oxygen free and deoxidized copper. Deoxidized copper will not retain the surface oxide in this test.

# 9. Mechanical Properties

- 9.1 All products shall conform to the mechanical property requirements of the basic product specification to which the item was ordered, with the following exception:
- 9.1.1 Sheet and strip with a thickness of 0.125 in. or less and intended for gaskets or for deep drawing shall conform to the requirements prescribed in Table 1.

# 10. Hydrogen Embrittlement (Reverse Bend Test Method)

- 10.1 Specimens shall withstand a minimum of ten reverse bends without breaking. Evidence of blisters, upon visual examination, shall be cause for rejection.
- Note 2—Fractured areas of the bent samples should show some reduction in area and have a distorted, smeared surface. The ideal break will have the appearance of a cup and cone tensile failure.

#### 11. General Examination

- 11.1 When examined the samples shall have:
- 11.1.1 A relatively uniform surface free of irregular rough spots sometimes termed open grains,
  - 11.1.2 Edges free of seams, laps and cracks,
- 11.1.3 Cross sections free of breaks, inclusions or extrusion pipe.

# 12. Special Examinations

- 12.1 Special Macro Examination:
- 12.1.1 The samples shall show no subsurface defects in excess of the following limits:
  - 12.1.1.1 Tubular Products:
  - (a) Tubes for Exhaust (Pinch-off) Purposes:

**TABLE 1 Grain Size** 

Thickness	Average Grain Size, max, mm	Hardness HRF <sup>A</sup> max
Less than 0.040 in. (1.0 mm)	0.035	75
0.040 to 0.125 in. (1.0 to 3.2 mm)	0.050	75

A For information only.



- (a.1) For wall thickness over 0.1 in. (2.5 mm), no defects on the external surface deeper than 0.005 in. (0.13 mm).
- (a.2) For wall thicknesses up to 0.1 (2.5 mm) inclusive, no defects on the external surface deeper than 5 % of the wall thickness.
- (a.3) No significant defects on the inside surface when examined with fiber optics or a boroscope.
- (b) Waveguides—No defects on the external surface deeper than 0.005 in. (0.13 mm) or 5 % of the wall thickness, whichever is smaller. No significant defects on the inside surface when examined with fiber optics or a boroscope.
- (c) *Tube for Other Applications*—No defects on either the internal or external surfaces deeper than 0.020 in. (0.51 mm) or 5 % of the wall thickness, whichever is the smaller.
- 12.1.1.2 *Bar and Plate Products*—No defects deeper than 0.06 in. (1.6 mm) or 5 % of the thickness, whichever is the smaller.
- 12.1.1.3 *Sheet, Strip and Flat Wire Products*—No defects deeper than 0.005 in. (0.13 mm) or 5 % of the thickness, whichever is smaller.
  - 12.1.1.4 Rod and Wire Products:
  - (a) Rod and Wire less than 0.19 in. (4.8 mm) in Diameter:
- (a.1) No defects deeper than 0.005 in. (0.13 mm) or 5 % of the diameter, whichever is smaller.
  - (b) Rod and Wire 0.19 in. (4.8 mm) and Over in Diameter:
- (b.1) No defects deeper than 0.06 in. (1.6 mm) or 5 % of the diameter, whichever is smaller.
  - 12.2 Special Microscopical Examination:
- 12.2.1 Samples shall be classified by comparison to Plate 1 which is available from ASTM Headquarters. Request Adjunct No. 12-600680-00.
- 12.2.1.1 Product whose samples have an appearance in conforming to either Class 1 or Class 2 shall be considered acceptable.
- 12.2.1.2 Subsurface microporosity similar to those typical of Class 3, 4 or 5 as illustrated in Plate 1, are permissible in Class 1 and 2 material provided the microporosity does not appear below the metal surface at depths exceeding the following:
  - (a) Tubular Products:
- (a.1) Tubes for Exhaust Purposes and Waveguides—No indications at any depth below the surface.
- (a.2) *Tubes for Other Applications*—Microporosity permissible to depths of 0.020 in. (0.51 mm) or 5 % of the tube wall, whichever is smaller.
- (b) *Bar and Plate*—Microporosity permissible to depths of 0.06 in. (1.5 mm) or 5 % of the thickness, whichever is smaller.
- (c) *Sheet, Strip and Flat Wire*—Microporosity permissible to depths of 0.010 in. (0.25 mm) or 5 % of the thickness, whichever is smaller.
  - (d) Rod and Wire Products:
- (d.1) Rod and Wire less than 0.19 in. (4.8 mm) in Diameter—Microporosity permissible to depths of 0.010 in. (0.25 mm) or 5 % of the diameter or thickness, whichever is smaller.

- (d.2) Rod and Wire 0.19 in. (4.8 mm) and Over in Diameter—Microporosity permissible to depths of 0.06 in. (1.5 mm) or 5 % of the diameter or thickness, whichever is the smaller.
- 12.2.1.3 Product whose samples have an appearance corresponding to Class 3, 4 or 5 and not in conformance with 12.2.1.2, shall be acceptable only by agreement between the manufacturer or supplier and the purchaser.

## 13. Dimensions, Mass and Permissible Variations

13.1 The dimensional tolerances for the product described by this specification shall be as prescribed in the basic product specification to which it was ordered.

# 14. Workmanship, Finish and Appearance

14.1 The product shall be prepared and handled in such a manner as to be substantially free from surface oxide and the presence of foreign material such as metal chips, dirt and grease.

# 15. Sampling

- 15.1 The sampling practice shall be as prescribed in the basic product specification to which it was ordered.
- 15.1.1 The number of sample pieces to be selected, however, shall be twice the number prescribed in the basic product specification.

#### 16. Number of Tests and Retests

- 16.1 *Tests*:
- 16.1.1 The number of samples submitted to all tests shall be twice that required by the basic product specification to which the product was ordered.
  - 16.2 Retests:
- 16.2.1 Retests are permitted as stated in the basic product specification to which the product was ordered for tests associated with the basic product specification.
- 16.2.2 Retests shall be permitted for tests and examinations required by this specification.
  - 16.2.3 Products which fail in retest shall be rejected.

## 17. Specimen Preparation

- 17.1 Chemical Composition:
- 17.1.1 Analytical sample preparation shall be as directed in the basic product specification to which the product was ordered.
  - 17.2 Electrical Resistivity:
- 17.2.1 Specimen preparation shall be as directed in the basic product specification to which the product was ordered.
  - 17.3 Grain Size:
- 17.3.1 Specimen preparation shall be as directed in the basic product specification to which the product was ordered.
  - 17.4 General and Special Macro Examination:
- 17.4.1 Transverse sections of the samples shall be machined to a 63 rms or smoother surface finish and degreased.
- 17.4.1.1 In a properly ventilated fume hood, etch the samples for 3 to 5 min by lowering the samples into a freshly prepared etching solution and slowly agitate.
- 17.4.1.2 Prepare the etching solution by mixing equal parts of concentrated reagent grade nitric acid (HNO<sub>3</sub>) and deionized



or distilled water. Add 5 grams of copper for each liter of the 50 % acid solution and cool the solution to ambient temperature prior to use. The solution shall be kept free of dirt and maintained at 75  $\pm$  5°F (24  $\pm$  3°C) during use. **Caution:** Do not pour the water into the acid.

- 17.4.1.3 Remove the samples periodically from the etching solution and visually examine to determine whether sufficiently etched. Should the samples tend to stain, add 25 mL of concentrated reagent grade hydrochloric acid (HCl) to each liter of etching solution.
- 17.4.1.4 After etching, rinse the samples thoroughly with deionized or distilled water and blow dry with air for examination
  - 17.5 Special Microscopical Examination:
- 17.5.1 Cut and degrease longitudinal samples taken from the finished product.
- 17.5.2 Heat the samples to  $1870 \pm 40^{\circ}F$  ( $1020 \pm 20^{\circ}C$ ) and hold at temperature for 30 min in an atmosphere comprising not less than 10 % hydrogen. Rapidly cool to ambient temperature by quenching in water without undue exposure to air.
- 17.5.3 Polish and etch for examination using the etching solution described in Table 2. CAUTION: With constant gentle stirring or swirling of the water, slowly add the acid. Do not pour the water into the acid.
- 17.6 *Hydrogen Embrittlement* (Reverse Bend Test Method): 17.6.1 Sample preparation shall be in accordance with Test Specimens Section of Test Methods B 577.
  - 17.6.2 Tubular Products:
- 17.6.2.1 For tube with an outside diameter of 0.12 in. (3 mm) or less, the samples shall be flattened by pressing or rolling.
- 17.6.2.2 For tube with an outside diameter greater than 0.12 in. (3 mm) but less than 1 in. (25.4 mm) the samples shall be cut longitudinally from the tube and flattened, when necessary by either pressing or rolling.
- 17.6.2.3 For tube with an outside diameter of 1 in. (25.4 mm) and greater, samples may be cut either parallel or transverse to the axis of the tube and then flattened.
- 17.6.2.4 Should the thickness of any flattened sample be greater than 0.081 in. (2.06 mm), it shall be machined to a thickness of 0.081  $\pm$  0.001 in. (2.06  $\pm$  0.025 mm) and at least one of the original surfaces of the finished tube shall be retained on one of the samples.
- 17.6.2.5 Samples shall be heated to  $1560 \pm 40^{\circ} F$  (850  $\pm$  20°C) and held at that temperature for a period of 30 min in an atmosphere containing not less than 10 % hydrogen and then rapidly cooled by quenching in ambient temperature water.
  - 17.6.3 Bar and Plate Products:
- 17.6.3.1 Samples shall be machined to a thickness of  $0.081 \pm 0.001$  in.  $(2.06 \pm 0.025 \text{ mm})$  and at least one of the original surfaces of the finished product shall be retained on one of the samples.

**TABLE 2 Etching Solution** 

Sodium Dichromate	16 grams
Sodium Chloride	12 grams
Sulfuric Acid (Conc.)	64 mL
Water (deionized or distilled)	100 mL

- 17.6.3.2 Samples shall be heated to  $1560 \pm 40^{\circ}$ F ( $850 \pm 20^{\circ}$ C) and held at that temperature for a period of 30 min in an atmosphere containing not less than 10 % hydrogen and then rapidly cooled by quenching in ambient temperature water.
  - 17.6.4 Sheet, Strip and Flat Wire Products:
- 17.6.4.1 For products with a thickness of 0.081 in. (2.06 mm) or less, the samples shall be prepared without machining.
- 17.6.4.2 For products with a thickness greater than 0.081 in. (2.06 mm), the samples shall be machined to a thickness of  $0.081 \pm 0.001$  in. (2.06  $\pm 0.025$  mm) and at least one of the original surfaces of the finished product shall be retained on one of the samples.
- 17.6.4.3 Samples shall be heated to  $1560 \pm 40^{\circ}$ F ( $850 \pm 20^{\circ}$ C) and held at that temperature for a period of 30 min in an atmosphere containing not less than 10 % hydrogen and then rapidly cooled by quenching in ambient temperature water
  - 17.6.5 Rod and Wire Products:
- 17.6.5.1 For products with a diameter or thickness of 0.081 in. (2.06 mm) and under the sample shall be prepared without machining.
- $17.6.5.\overline{2}$  For products with a diameter or thickness greater than 0.081 in. (2.06 mm) the samples shall be machined to a thickness of 0.081  $\pm$  0.001 in. (2.06  $\pm$  0.025 mm) and at least one of the original surfaces of the finished product shall be retained on one of the samples.
- 17.6.5.3 Samples shall be heated to  $1560 \pm 40^{\circ}$ F ( $850 \pm 20^{\circ}$ C) and held at that temperature for a period of 30 min in an atmosphere containing not less than 10 % hydrogen and then rapidly cooled by quenching in ambient temperature water.

#### 18. Test Methods

- 18.1 Chemical Composition:
- 18.1.1 The test methods used for the determination of composition shall be as specified in the basic product specification to which the material was ordered.
- 18.1.2 The test method(s) used for the determination of elements required by contractual or purchase order agreement shall be as agreed upon between the manufacturer or supplier and the purchaser.
  - 18.2 Grain Size:
- 18.2.1 Grain size shall be determined in accordance with Test Methods E 112. In case of dispute, the intercept method shall be used.
  - 18.3 Electrical Resistivity:
- 18.3.1 Electrical resistivity shall be determined in accordance with Test Methods B 193. Measurement uncertainty shall not exceed  $\pm 0.15$  %.
- 18.4 General Examination and Special Macro Examination:
- 18.4.1 Examination shall be visually with the unaided eye or at  $10 \times$  magnification.
  - 18.5 Special Microscopical Examination:
- 18.5.1 Examination shall be made at  $100 \times$  magnification and classified by comparison to Plate 1.
- 18.6 *Hydrogen Embrittlement Susceptibility* (Reverse Bend Test Method):



18.6.1 Hydrogen embrittlement shall be determined in accordance with Test Method D of Test Methods B 577.

18.7 Scaling Test:

18.7.1 The samples shall be heated in air in a clean muffle furnace for 30 min at  $1560 \pm 40^{\circ}$ F ( $850 \pm 20^{\circ}$ C) and immediately quenched in clean, ambient temperature water.

#### 19. Significance of Numerical Limits

19.1 For the purpose of determining compliance with specified limits an observed value or calculated value shall be rounded as indicated in accordance with the rounding method of Practice E 29.

Property

Rounded Unit for Observed or Calculated Value Nearest unit in the last right-hand significant digit used in expressing

Chemical composition

Electrical resist.

Grain size

Up to 0.055 mm, incl.

Over 0.055 to 160 mm

nearest multiple 0.005 mm nearest 0.01 mm

the limiting value

#### 20. Inspection

20.1 The manufacturer shall inspect and make tests necessary to verify that the product furnished conforms to the requirements prescribed in the basic product specification to which the product was ordered and the requirements of this specification.

20.2 Source inspection of the product by the purchaser may be agreed upon between the manufacturer or supplier and the purchaser as part of the contract or purchase order.

20.2.1 The nature of the facilities needed to satisfy the inspector representing the purchaser that the product being furnished is in accordance with the product specification shall be included in the agreement.

20.2.2 All tests and inspection shall be conducted so as not to interfere unnecessarily with the operations of the works.

20.3 The manufacturer or supplier may accomplish the final inspection simultaneously by mutual agreement.

# 21. Rejection and Rehearing

#### 21.1 Rejection:

21.1.1 Product which fails to conform to the requirements of this specification when inspected or tested by the purchaser, or the purchaser's agent, may be rejected.

21.1.2 Rejection shall be reported to the manufacturer or supplier, promptly, and in writing.

21.1.3 In case of dissatisfaction with results of the test upon which rejection is based, the manufacturer, or supplier, may make claim for a rehearing.

#### 21.2 Rehearing:

21.2.1 As a result of product rejection, the manufacturer, or supplier, may make claim for a retest to be conducted by the manufacturer, or supplier, and the purchaser. Samples of the rejected product shall be taken in accordance with this specification and subjected to test by both parties using the test method(s) specified herein or, alternatively, upon agreement by both parties, an independent laboratory may be selected for the test(s) using the test methods specified in this product specification.

## 22. Certification

22.1 When specified in the contract or purchase order, the purchaser shall be furnished certification that samples representing each lot have been tested or inspected as directed in this specification and the requirements have been met.

#### 23. Mill Test Report

23.1 When specified in the contract or purchase order, a report of the test results shall be furnished.

## 24. Packaging and Package Marking

24.1 Packaging:

24.1.1 The product shall be separated by shape, size and temper and prepared for shipment in such a manner as to afford protection from the normal hazards of transportation.

24.1.2 Special packing or packaging required shall be by agreement between the manufacturer or supplier and the purchaser at the time of the contract or purchase order.

24.2 Package Marking:

24.2.1 Each shipping unit shall be legibly marked with the purchase order number, metal designation, temper, size, shape, gross and net weight, and the name of the supplier. The specification numbers shall also be shown when so specified in the contract or purchase order.

# 25. Keywords

25.1 oxygen-free copper; oxygen-free copper for electron devices; wrought copper forms for electron devices; wrought oxygen-free copper for electron devices



#### **SUMMARY OF CHANGES**

Committee B-5 has identified the location of selected changes to this standard since the last issue (F 68 - 93) that may impact the use of this standard.

- (1) A five-year review was conducted with minor technical changes.
- (2) Several editorial revisions were made to clarify wording or comply with the Committee B-5 Outline of Form.

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