Designation: B 577 – 93 (Reapproved 2004)

Standard Test Methods for Detection of Cuprous Oxide (Hydrogen Embrittlement Susceptibility) in Copper[1]

This standard is issued under the fixed designation B 577; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope*

1.1 These test methods describe procedures for determining the presence of cuprous oxide (Cu_2O) in products made from deoxidized and oxygen-free copper.

1.2 Inch-pound units are the standard. SI values given in parentheses are for information only.

1.3 *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.*

1.4 The test methods appear in the following order:

	Sections
Microscopical Examination without Thermal Treatment	9-11
Microscopical Examination after Thermal Treatment	12-14
Closed Bend Test after Thermal Treatment	15-17
Reverse Bend Test after Thermal Treatment	18-20

2. Referenced Documents

2.1 *ASTM Standards:* [2]

E 3 Practice for Preparation of Metallographic Specimens

E 883 Guide for Reflected-Light Photomicrography

3. Terminology

3.1 *Definitions:*

3.1.1 *deoxidized copper*—material produced substantially free of cuprous oxide, by the use of metallic or metalloidal deoxidizers, as determined by metallographic examination at 75× under polarized light.

3.1.1.1 Oxygen may be present as residual deoxidation products.

3.1.2 *embrittlement*—the reduction of the normal ductility in a metal as a result of a physical or chemical change.

3.1.2.1 *Embrittlement,* as it relates to these test methods, is the loss of ductility caused by the reaction of cuprous oxide in the copper material when exposed at elevated temperatures to a reducing atmosphere.

3.1.3 *oxygen-free copper*—electrolytic copper produced substantially free of cuprous oxide without the use of metallic or metalloidal deoxidizers as determined by metallographic examination at 75× under polarized light,

3.1.3.1 Oxygen may be present up to a maximum of 5 ppm in Copper UNS No. C10100 and 10 ppm in Copper UNS No. C10200.

4. Summary of Test Methods

4.1 The presence of cuprous oxide is determined either by microscopical examination under polarized light or by methods that involve heating the test specimens in a hydrogen-rich atmosphere and rapidly cooling the specimens without undue exposure to air followed by a microscopical examination or a suitable bend test.

5. Significance and Use

5.1 These test methods determine whether copper products will be resistant to embrittlement when exposed to elevated temperatures in a reducing atmosphere.

5.1.1 It is assumed that all who use these test methods will be trained personnel capable of performing these procedures skillfully and safely. It is expected that work will be performed in a properly equipped facility.

6. Apparatus

6.1 *Test Method A*—Metallographic equipment of the type described in Practice E 3 and Guide E 883 suitably equipped with a polarized light illuminating device.

6.2 *Test Methods B, C, and D*:

6.2.1 Metallographic equipment of the type described in Practice E 3 and Guide E 883 provided with normal illumination.

[1] This specification is under the jurisdiction of ASTM Committee B05 on Copper and Copper Alloys and is the direct responsibility of Subcommittee B05.06 on Methods of Test.

Current edition approved May 1, 2004. Published May 2004. Originally approved in 1973. Last previous edition approved in 1998 as B 577 – 93 (1998).

[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.

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

6.2.2 A furnace of sufficient capacity, capable of maintaining the required reducing atmosphere while the specimens are being heated. A rapid cooling device using either water or a reducing atmosphere is required.

6.2.3 A machinist vise with replaceable matching pairs of jaw mandrels of various radii contours.

7. Sampling

7.1 Sampling shall be in accordance with the requirements of the specification under which the material was ordered.

8. Test Specimens

8.1 Longitudinal specimens, that is, specimens whose axes are parallel to the direction of working are preferable. However, equally reliable results can be obtained with specimens in which the axis is perpendicular to the directions of working.

8.2 Specimens shall be of dimensions suitable for the performance of the required tests. Where necessary to cut a specimen from an oversize piece of material, at least one of the original surfaces of the material shall be retained in the test specimen. Suggested dimensions for test specimens are given in the following table (for Procedures C or D):

Wrought Products	Suggested Dimensions for Test Specimens
Flats (wire, strip, sheets, bar, and plate)	thickness—that of the product but should not exceed ½ in. (13 mm) width—approx. ½ in. (13 mm) length—approx. 6 in. (152 mm)
Shapes and forgings	To the extent that the dimensions of the material permit, the dimensions of the test specimens are those suggested for the flat products specimens. (Where the product dimensions, particularly length, as in the case of forgings, do not permit taking a specimen, the total product may then become the test specimen for examination by Procedures A or B.)
Wire or rod	diameter or distance between parallel surfaces—that of the product but not to exceed ½ in. (13 mm) length—approx. 6 in. (152 mm)
Tubular products: Diameter or distance between parallel surfaces:	
Up to 5⁄16 in. (8 mm), incl.	full section of tube, approx 6 in. (152 mm) long
Over 5⁄16 in. (8 mm) to 1 in. (25.4 mm), incl.	a slit half section of the tube, approx 6 in. (152 mm) long
Over 1 in. (25.4 mm)	a slit section approx ½ in. (13 mm) wide and 6 in. (152 mm) long taken either transverse or parallel to the tube axis
Refinery shapes	a 0.080-in. (2.03-mm) diameter wire specimen made by forging, swaging, hot rolling, and cold drawing as may be necessary

8.3 All specimens made by cutting from larger stock shall have their corners or edges deburred to a slight radius before testing.

TEST METHOD A—MICROSCOPICAL EXAMINATION WITHOUT THERMAL TREATMENT

9. Scope

9.1 This test method describes a procedure by which the presence of cuprous oxide is determined by polarized light microscopy examination at a minimum magnification of 75×.

10. Procedure

10.1 The test specimens taken transverse to and bounded by an original surface of the material are mounted and polished in accordance with Practice E 3.

10.1.1 Photomicrographs, when taken, are prepared in accordance with Guide E 883.

10.2 The polished, but unetched, surface of the specimens are examined under reflected polarized light at a minimum magnification of 75×.

10.2.1 Cuprous oxide will appear as ruby-red particles.

10.2.2 Cuprous oxide will appear as blue particles under white light.

11. Application

11.1 This test method is applicable to Copper UNS Nos. C10100, C10200, C10300, C10400, C10500, C10700, C11700, and C12000.

TEST METHOD B—MICROSCOPICAL EXAMINATION AFTER THERMAL TREATMENT

12. Scope

12.1 This test method describes a procedure by which the presence of cuprous oxide is determined by microscopical examination under normal illumination at a minimum magnification of 75× after thermal treatment of the specimens.

13. Procedure

13.1 Heat the cleaned and degreased specimens which retain at least one original surface for 20 to 40 min in an atmosphere of at least 10 % hydrogen within a furnace held at a temperature of 1562 ± 45°F (850 ± 25°C).

13.2 After the heat treatment, immediately remove and quench the specimens in water without undue exposure to air or quickly cool the specimens in the same atmosphere.

13.3 The test specimens taken transverse to and bounded by an original surface of the treated material are mounted and polished, and etched when desired, in accordance with Practice E 3.

13.3.1 Photomicrographs, when taken, shall be prepared in accordance with Guide E 883.

13.4 Cuprous oxide (hydrogen embrittlement) when present in the material tested will manifest itself by the open grain structure (gassing) characteristic of embrittlement. For example, the grain structure is outlined by a series of voids at the grain boundary.

13.5 In case of controversy concerning the presence or absence of cuprous oxide (hydrogen embrittlement), Method C or D, as specified in the product specification, shall be followed.

14. Application

14.1 The method is applicable to Copper UNS Nos. C10100, C10200, C10400, C10500, C10700, C10800, C11700, C12000, C12200, and C14200.

TEST METHOD C—CLOSED BEND TEST AFTER THERMAL TREATMENT

15. Scope

15.1 This test method describes a procedure by which the presence of cuprous oxide (hydrogen embrittlement) is determined by bending thermally treated specimens into a flattened "U" shape.

16. Procedure

16.1 Heat the cleaned and degreased specimen that retains at least one of the original surfaces for a period of 20 to 40 min in an atmosphere of at least 10 % hydrogen within a furnace held at a temperature of 1562 ± 45°F (850 ± 25°C).

16.2 After the heat treatment, immediately quench the specimens in water without undue exposure to air or quickly cool the specimens in the same atmosphere.

16.3 Flatten all tubular specimens, after the thermal treatment, in a vise or press with smooth surfaces, to twice the wall thickness, before bending.

16.4 Make the bend test at ambient temperature as indicated in Fig. 1 with an original surface of the material on the outside of the bend.

16.5 Bend the test specimen in such a manner as to form a "U" with the final closure being made by squeezing the legs of the "U" together.

16.5.1 The formation of cracks on the outside surface of the bend are evidence of the presence of cuprous oxide in the copper.

17. Application

17.1 This test is applicable to Copper UNS Nos. C10100, C10200, C10300, C10400, C10500, C10700, C11700, and C12000.

TEST METHOD D—REVERSE BEND TEST AFTER THERMAL TREATMENT

18. Scope

18.1 This test method describes a procedure in which the presence of cuprous oxide (hydrogen embrittlement) is determined by subjecting thermally treated specimens to a predetermined number of bends.

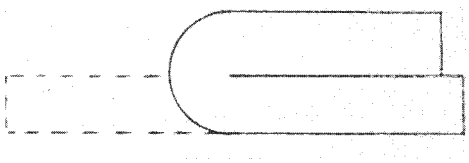

FIG. 1 Bend Test

19. Procedure

19.1 Heat the cleaned and degreased specimens that retain at least one of the original surfaces for a period of 20 to 40 min in an atmosphere of at least 10 % hydrogen within a furnace held at a temperature of 1562 ± 45°F (850 ± 25°C).

19.2 After the heat treatment, immediately remove and quench the specimens in water without undue exposure to air or quickly cool the specimens in the same atmosphere.

19.3 At ambient temperature, clamp the specimen lightly between jaws with edges having a radius of two and a half times the thickness (or diameter) of the material being tested.

19.3.1 An original surface of the material shall be so positioned as to be on the outer bend radius.

19.4 Bend the specimen over one edge of the clamp jaws through an angle of 90° and return the specimen to the original position; this constitutes one bend.

19.5 Bend the specimen in the opposite direction through an angle of 90° and return the specimen to the original position; this constitutes a second bend.

19.6 Continue making bends in alternating directions until the required number of bends have been made or the specimen fractures.

19.7 Failure to withstand the required minimum number of bends is evidence of the presence of cuprous oxide.

20. Application

20.1 This method is applicable to Copper UNS Nos. C10100, C10200, C10300, C10400, C10500, C10700, and C12000.

21. Precision and Bias

21.1 A precision and bias statement is not applicable since the results of these test methods merely indicate whether there is conformation to a criteria for success specified in the particular procedure.

22. Keywords

22.1 cuprous oxide in copper; test methods; hydrogen embrittlement of copper; test methods

SUMMARY OF CHANGES

This section identifies the principle changes to this standard that have been incorporated since the last issue.

(*1*) Title change.
(*2*) Sections describing each test method identified in scope.
(*3*) Each test method self contained.