



# Standard Specification for Metallized Surfaces on Ceramic<sup>1</sup>

This standard is issued under the fixed designation F 44; 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 specification covers requirements for powdered refractory metal coatings produced with or without additives. These coatings are applied to ceramic bodies in order to join ceramic bodies to metals or to other metallized ceramics by means of brazing or soldering. Included in this specification are requirements for a secondary metallic layer which is usually applied over the refractory metal layer.

1.2 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

## 2. Referenced Documents

### 2.1 ASTM Standards:

- E 3 Methods of Preparation of Metallographic Specimens<sup>2</sup>
- F 19 Test Method for Tension and Vacuum Testing Metallized Ceramic Seals<sup>3</sup>

## 3. Terminology

### 3.1 Definitions of Terms Specific to This Standard:

3.1.1 *ceramics*—as referred to in this specification are understood to be inorganic, nonmetallic materials, the major phase of which must be crystalline. A glassy intercrystalline matrix may be present as one of the minor phases.

3.1.2 *metallizing—on a ceramic*, is a process whereby a sintered matrix of metal particles firmly adheres to the ceramic.

3.1.3 *refractory metals*—those metals with melting points equal to or higher than that of chromium. Therefore, this group includes chromium, columbium, molybdenum, rhenium, tantalum, and tungsten.

## 4. Ordering Information

4.1 The manufacturer and purchaser shall agree upon specific quality levels as outlined in the following sections:

- 4.1.1 Uniformity of metallizing (Section 5),
- 4.1.2 Thickness of metallizing (Section 6),
- 4.1.3 Secondary metal layer on the metallizing (Section 9),

4.1.4 Values for bond strength (Section 10), and

4.1.5 Vacuum tightness of brazed metallized ceramic assemblies (dependent on allowable use of manufacturer's product) (Section 11).

## 5. Uniformity of Metallizing

5.1 Either of the following two levels of quality may be agreed upon between manufacturer and purchaser, depending upon end use, seal area, and geometry.

### 5.2 Level A—Less Demanding Application:

5.2.1 Thin areas where the ceramic substrate can usually be seen without magnification through the metallizing will be acceptable only if all of the following conditions are met:

5.2.1.1 There are no more than two such areas on any one coated band, spot, or pattern detail.

5.2.1.2 Their extremities are no closer than 10 % of the total band length to each other.

5.2.1.3 They are no wider than 10 % of the width of the band but not exceeding 1 mm.

5.2.1.4 Their cumulative length does not exceed 25 % of the total band length.

5.2.2 Defects such as brush marks, screen marks, marks in the metallizing left by foreign matter such as lint, dust, etc., and pits or blisters, will be acceptable if they meet the four conditions outlined in 5.2.1.1 through 5.2.1.4. Such defects will also be acceptable if their raised edges do not interfere with proper assembly of the joint.

5.2.3 Projections on metallized surfaces, such as oversized particles or agglomerates will be objectionable if they interfere with assembly.

5.2.4 Continuous coatings over sharp edges or corners with a radius less than  $\frac{1}{32}$  in. (0.8 mm) will not be required unless by specific agreement between manufacturer and purchaser.

### 5.3 Level B—Demanding Application:

5.3.1 Thin areas will be unacceptable where the ceramic substrate can be seen through the metallizing when examined at 40× magnification.

5.3.2 Defects through which the underlying ceramic can be observed, such as brush or screen marks, marks left by foreign matter such as lint or dust, and pits or chips, will be acceptable only if all of the following conditions are met:

5.3.2.1 There are no more than two such defects on any coated band, spot, or pattern detail.

5.3.2.2 Their extremities are not closer together than 10 % of the length of the metallized area in which they occur.

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<sup>2</sup> Annual Book of ASTM Standards, Vol 03.01.

<sup>3</sup> Annual Book of ASTM Standards, Vol 10.04.

5.3.2.3 They are not wider than 5 % of the width of area nor longer than 2 % of the length of area.

5.3.3 Continuous coatings over sharp corners with a radius less than  $\frac{1}{32}$  in. (0.8 mm) will not be required so long as subsequent plating covers the radius. The specific gap permissible at such a corner shall be agreed upon by manufacturer and purchaser.

5.3.4 Projections on metallized surfaces, such as oversize particles or agglomerates will be objectionable if they interfere with assembly or alignment.

## 6. Thickness of Metalizing

6.1 Measure thickness by observation through a calibrated micrometer eyepiece in a metallurgical microscope. Measure a polished cross section that is perpendicular to the metallized surface. The measurement shall consist of the average of at least three representative readings taken where the upper and lower edges of the layer are parallel. Magnification must be at least 400 $\times$  to obtain satisfactory accuracy. A suggested method for specimen preparation is shown in Appendix X1.

6.2 Thickness of the sinter-fired metallize layer shall range from 0.0004 in. (0.01 mm) to 0.002 in. (0.05 mm) unless otherwise mutually agreed upon between the manufacturer and the purchaser.

## 7. Continuity of Metalizing

7.1 The particles in the metallized layer must be sintered in order to form a dense matrix when viewed in cross section at 400 $\times$ . This cross section will indicate the extent of three-dimensional sintering.

## 8. Discoloration of the Ceramic

8.1 Almost invariably some discoloration of the ceramic adjacent to the metallized layer will exist (Note 1). The manufacture will not be required to eliminate discoloration completely.

NOTE 1—The actual color and depth of penetration depend on the metallizing formula and processing cycle. Where a given degree of discoloration is objectionable to the user, the manufacturer may be able to substitute a practice which will yield acceptable results. It is recognized that metallized coating properties vary with the formula and processing. If the user requires such a change to reduce coloration compromise may be necessary.

## 9. Secondary Metallic Layer on Metallizing

9.1 A secondary metallic layer on the metallizing shall be applied by the manufacturer unless otherwise specified.

9.2 All metallized areas must be completely covered unless otherwise specified.

9.3 The purchaser shall specify the metal to be applied.

9.4 Manufacturer and purchaser shall agree upon the layer thickness, including maximum and minimum thickness limits. Measurement shall be by microscopical examination as described in 6.1.

9.5 Brightness of clean matte deposits may not be specified.

9.6 Oxide films on the secondary metallic layer will not be cause for rejection.

9.7 Defects such as blisters developed in subsequent heating operations will be acceptable only in 5.2 Level A application and only if all the following conditions are met:

9.7.1 There are no more than two blisters on any one coated band, spot, or pattern detail.

9.7.2 No blister is closer than 10 % of the total band length to another blister.

9.7.3 Maximum dimension of any blister does not exceed 10 % of the width of the band; in any case not to exceed  $\frac{1}{32}$  in. (0.8 mm).

9.8 The secondary metallic layer must adhere to the metallizing underneath to the extent that no separation exists at this interference in tension or torque peel tests after sintering of the layer in a suitable protective atmosphere.

9.9 Cleanliness such as absence of fingerprints, dirt, etc., which may interfere with brazing or soldering, is not a requirement for acceptance unless agreed upon between manufacturer and purchaser.

9.10 When the secondary metallic layer is omitted from the purchaser requirements, the following items shall become a part of this specification:

9.10.1 Metallized surfaces shall be reasonably free from dirt, grease, fingerprints, etc., in keeping with normal good handling practice, unless this condition is specifically agreed upon between manufacturer and purchaser.

9.10.2 The degree of oxidation acceptable on the metallizing shall be agreed upon between the manufacturer and purchaser.

## 10. Bond Strength

10.1 The manufacturer and purchaser shall agree on specific values of bond strength of brazed metallized ceramics.

10.2 Quantitative measurements of the bond strength may be made by the following, or other methods agreed upon between manufacturer and purchaser.

10.2.1 *Torque Peel Test*—A torque peel test may be used where a standardized strip of a strong, ductile metal such as nickel is brazed to the metallized and secondary metal surface. See Appendix X2.

10.2.2 A tension test may be made in accordance with Test Method F 19. This tension test really measures the combined effects of all the factors involved in making the joint. It may also measure either the adherence of the metallizing to the ceramic or the cohesive strength of the sintered metal layer itself, depending on where the fracture occurs.

## 11. Vacuum Tightness

11.1 Vacuum tightness, where required by the user, shall be tested in accordance with Test Method F 19, or by other method agreed upon between the manufacturer and the purchaser.

## 12. Keywords

12.1 bond strength; metallizing of ceramics; thickness control

## APPENDIXES

### (Nonmandatory Information)

#### X1. SUGGESTED METHOD FOR PREPARATION OF SPECIMENS FOR MICROSCOPY

X1.1 Cut the specimen with a water-cooled diamond cutoff saw.

X1.2 If the specimen is too small to handle readily during subsequent polishing, it may be mounted by any of the ordinary methods. (Approved methods of mounting are given in Methods E 3.) It is best to mount a complete seal section in a resin which contains fillers so that the relatively soft metal phases adjacent to hard ceramic phases do not become rounded during polishing.

X1.3 Rough grind on 240 and 400-grit diamond laps. This step should remove 0.040 in. and 0.060 in. (1 to 1.5 mm) stock from the surface in order to remove completely all surface damage caused by the cutoff operation.

X1.4 Fine grind with 15- $\mu$ m diamond grinding compound on a hardwood or napless cloth-covered wheel. This step should impart a polished look to the ceramic and reduce the dark “plucked-out” areas considerably when viewed at 100 to 200 $\times$  magnification through a metallurgical microscope.

X1.5 Polish successively with 6, 3, and 1- $\mu$ m diamond compounds on napless cloth or chemotextile-covered wheels. Another polish with submicron diamonds will improve the polish on the metal phases.

X1.6 Perform final polishing with alpha alumina of particle size 0.1 to 0.3  $\mu$ m, on the same type wheels as in preceding steps. This step has two purposes: ( *a* ) to remove scratches from the metal phases, and ( *b* ) to give some relief and therefore contrast to the glassy phases of the ceramic and the metallizing layer.

X1.7 Use heavy pressure with the diamond abrasives. Usually about a minute on each wheel will be sufficient to completely remove the marks made by the previous step. The final alumina stage should be done with light but firm touch; time in this stage should be as short as possible in order to minimize relief of the softer metal phases.

#### X2. TEST FOR METALLIZING QUALITY

X2.1 The following test may be used to determine the quality of metallizing on any ceramic surfaces having metallized areas 0.125 by 0.125 in. (3.18 by 3.18 mm) or larger. The ceramic surface should be flat to 0.001 in. (0.02 mm), although curved metallized surfaces may also be tested. Curved surfaces tend to yield slightly higher values.

X2.2 The ceramic may be metallized by any method. A secondary metallic layer may be deposited as desired or the surface otherwise prepared for brazing. Position a clean, ductile metal tab 0.125  $\pm$  0.001 by 0.025  $\pm$  0.001 by 1 in. (3.18  $\pm$  0.02 by 0.6  $\pm$  0.02 by 25.4 mm) long snugly against

the metallized surface so that 0.125  $\pm$  0.005 in. (3.18  $\pm$  0.01 mm) of its length is held against the metallizing. Braze using silver-copper eutectic solder and good established brazing techniques.

X2.3 Hold the ceramic firmly in a suitable fixture and gently bend the metal tab at right angles to the metallized surface at the point of contact. Grasp the end of the metal tab by a suitable pressure grip and pull at right angles to the ceramic surface until failure occurs. The manufacturer and the purchaser shall agree upon acceptable low limits and maximum range.

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