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Standard Specification for Aluminum Diffusion Coating Applied by Pack Cementation Process¹

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1. Scope

1.1 This specification covers the requirements for aluminum diffusion of metals by the pack cementation method. Pack diffusion employs the chemical vapor deposition of a metal which is subsequently diffused into the surface of a substrate at high temperature. The material to be coated (substrate) is immersed or suspended in a powder containing aluminum (source), a halide salt (activator), and an inert diluent such as alumina (filler). When the mixture is heated, the activator reacts to produce an atmosphere of aluminum halides which transfers aluminum to the substrate for subsequent diffusion. The aluminum-rich surface enhances corrosion, thermal stability, and wear-resistant properties.

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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

- B 374 Terminology Relating to Electroplating²
- B 487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of Cross Section²
- B 567 Test Method for Measurement of Coating Thickness by Beta Backscatter Method²
- B 602 Test Method for Attribute Sampling of Metallic and Inorganic Coatings²
- B 697 Guide for Selection of Sampling Plans for Inspection of Metallic and Inorganic Coatings²
- B 762 Method of Variables Sampling for Metallic and Inorganic Coatings²
- C 664 Test Methods for Thickness of Diffusion Coatings²
- D 3951 Practice for Commercial Packaging³
- E 3 Methods of Preparation of Metallographic Specimens⁴

¹ This specification is under the jurisdiction of ASTM Committee B-8 on Metallic and Inorganic Coatings and is the direct responsibility of Subcommittee B08.15 on Diffusion Coatings.

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² *Annual Book of ASTM Standards*, Vol 02.05.

³ *Annual Book of ASTM Standards*, Vol 15.09.

⁴ *Annual Book of ASTM Standards*, Vol 03.01.

F 1330 Guide for Metallic Abrasive Blasting to Descale Surfaces of Pipe⁵

3. Terminology

3.1 Definitions used in this specification are in accordance with Terminology B 374.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *diffusion coating*—a diffusion coating is one produced by causing an element to react with or diffuse into, or both, the surface of a metallic substrate, thus, chemically altering the surface of the substrate.

3.2.2 *retorts*—containers in which powder and parts are packed for processing. They can be constructed of carbon, stainless, or high alloys and fabricated in all shapes and sizes to accommodate parts being processed.

3.2.3 *significant surface*—areas that are essential to the serviceability or function of the article. These surfaces must be identified on a drawing or marked-up sample of product. Areas can fall into one of three categories as follows:

3.2.4 *coating required*—these surfaces must be in accordance with all quality requirements of this specification.

3.2.5 *no coating required*—these surfaces are areas where no coating is allowed due to a number of reasons including dimensional, fabrication, and welding, as well as others. Materials used for masking are commercially available.

3.2.6 *optional*—these surfaces do not require coating, but at the same time do not require masking.

4. Classification

4.1 There are three classes of aluminum diffusion defined by base (basis) metal category.

4.1.1 *Class I*—Carbon steel and low alloy.

4.1.2 *Class II*—Stainless steels.

4.1.3 *Class III*—Nickel-based alloys.

5. Ordering Information

5.1 In order to make the application of this specification complete, the purchaser shall supply the following information to the vendor through a purchase order and drawings:

5.1.1 Title, ASTM designation number, and date of issue of this specification.

⁵ *Annual Book of ASTM Standards*, Vol 01.07.

- 5.1.2 Deposit by classification (see Section 4).
- 5.1.3 Composition and metallurgical condition of substrate to be coated.
- 5.1.4 Location of significant surfaces (see 3.2.3).
- 5.1.5 Samples for destructive metallurgical test (see 8.1).
- 5.1.6 Any post heat treatment required.
- 5.1.7 Acceptance inspection procedure to be used (see Section 9).
- 5.1.8 Any requirement for certification (see Section 11).

6. Processing Requirements

6.1 *Substrate Preparation*—The metal to be aluminized shall be free of flaws and defects that will be detrimental to the coating. Thorough cleaning is essential to ensure satisfactory diffusion. Materials used for cleaning should not damage the base metal. Oils, dirt, grease, and stains must be removed. When blasting is also required, use aluminum oxide (90 to 220 mesh) at 60 to 80 psi from 4 to 6 in. standoff.

6.2 *Pack Mix*—Can be categorized by the following:

6.2.1 *Types of Activity*:

6.2.1.1 *High Activity*—Deposit aluminum rapidly and perform at temperatures (1000 to 1500°F). This is usually followed by a diffusion heat treatment at (1900 to 2000°F).

6.2.1.2 *Low Activity*—Deposit aluminum slowly and perform at temperatures in excess of 1650°F.

6.2.2 *Materials*:

6.2.2.1 *Masteralloy with 50 to 100 % Pure Aluminum*—Sold under a number of trade names in various mesh sizes. Percentage in mix depends on material being processed.

6.2.2.2 *Activator*—Most commonly used is ammonium chloride, but many others are available. Percentage in mix depends on material and type of activity.

6.2.2.3 *Inert Filler*—Aluminum oxide or calcined alumina is used and commercially available in numerous mesh sizes. Percentage in mix depends on material being processed and mesh size being used.

NOTE 1—It is important that both proper equipment is used and time established to guarantee uniform blend of pack mix.

NOTE 2—It is common practice to rejuvenate used mixes by addition of coating elements, alloys, and activators. Chemical analysis of used mixes facilitates control of critical constituents. Disposal of used mixes must be performed in compliance with environmental regulations.

6.3 *Loading*—Place pack mix and parts in retorts. Retort size is limited by dimensions of furnace used for processing.

6.4 *Furnace Cycle*:

6.4.1 High-activity packs are heated to a temperature between 1100 and 1500°F and held for the amount of time needed to produce a sufficient deposit of aluminum which after a high-temperature diffusion cycle meets thickness requirements. Time is also dependent on activator(s) being used.

6.4.2 Low-activity packs are heated to a temperature between 1650 and 2100°F and held for the amount of time needed to produce the desired depth of diffusion.

NOTE 3—Due to the high temperature of the process, a positive pressure of an inert gas, usually argon, must be maintained in retorts throughout the entire furnace cycle.

6.5 *Post-Cleaning*—Retorts should be allowed to cool sufficiently before opening. When parts are removed from retorts, residual pack mix is removed from surfaces by a supplier-approved method.

6.6 *Diffusion Heat Treatment*—Parts processed in high-activity packs are placed in the retort and under inert atmosphere of argon or hydrogen are heated to 1900 to 2000°F and held for the amount of time needed to produce the desired level of diffusion.

6.7 *Post-Straightening*—Long tubes may have distortion due to the high temperature of process. Use rotary straightener or hydraulic press to restore straightness and ovality.

6.8 *Visual Inspection*—Inspect in accordance with 7.3.

6.9 *Marking and Packaging*—To be defined by the purchaser in the purchase order. Parts processed for the U.S. government and military, including subcontracts, shall be packaged in accordance with Practice D 3951.

7. Coating Requirements

7.1 *Diffusion Thickness*—The following are minimums for the different classes of materials based on standard process parameters. Thicker coatings require special processing and must be called out for in the purchase order if required.

Class	Minimum, in.
Class I (carbon and low alloy)	0.005
Class II (stainless steels)	0.003
Class III (nickel-base alloys)	0.001

7.2 *Aluminum Content*—The outer 15 % of coating shall contain a minimum of 28 % (by weight) aluminum.

7.3 *Appearance*—The diffusion zone shall be nonporous and adherent to the base metal and shall have a uniform surface free from objectionable imperfections. Minor variations in color and surface appearance shall be considered acceptable, providing the requirements of 7.1 and 7.2 are met (see 8.6).

8. Test Methods

8.1 *Special Test Specimens*:

8.1.1 The permission or the requirements to use special test specimens, the number to be used, the material from which they are to be made, and their shape and size shall be stated by the purchaser.

NOTE 4—Test specimens are often used to represent coated articles in a test if these articles are of a size, shape, or material that is not suitable for the test, or if it is preferred not to submit articles to a destructive test, because, for example, the articles are expensive or few in number. The specimen should duplicate the characteristics of the article that influence the property being tested.

8.1.2 Special test specimens used to represent articles in an adhesion, weldability, corrosion resistance, or appearance test shall be made of the same material, shall be in the same metallurgical condition, and shall have the same surface condition as the articles they represent, and they shall be placed and processed in the production lot along with the articles they represent.

8.2 *Thickness*—Perform the destructive test on samples in accordance with Test Methods C 664. Read and record the results in accordance with Test Method B 487.

8.3 *Aluminum Content*—Shall be performed by beta backscatter or X-ray fluorescence.

8.4 *Mechanical Properties* (if required)—Tensile testing on samples from each process batch.

8.5 *Hardness Testing* (if required)—Macrohardness and microhardness testing are both acceptable means.

8.6 *Appearance*—The coating can be examined at up to 5× magnification for conformance to appearance requirements.

9. Sampling

9.1 The sampling plan used for inspection of a quantity of coated articles shall be agreed upon by the purchaser and the supplier.

NOTE 5—Usually, when a collection of coated articles, the inspection lot (9.2), is examined for compliance with the requirements placed on the coating, a relatively small number of the articles, the sample, is selected at random and is inspected. The inspection lot is then classified as complying or not complying with the requirements based on the results of the inspection of the sample. The size of the sample and the criteria of compliance are determined by the application of statistics. This procedure is known as sampling inspection. Three standards, Test Method B 602, Guide B 697, and Method B 762, contain sampling plans that are designed for the sampling inspection of coatings.

Test Method B 602 contains four sampling plans, three for use with tests that are nondestructive and one for tests that are destructive. The buyer and seller may agree on the plan or plans to be used. If they do not, Test Method B 602 designates the plan to be used.

Guide B 697 provides a large number of plans and also gives guidance in the selection of a plan. When Guide B 697 is specified, the buyer and the seller need to agree on the plan to be used.

Method B 762 can be used only for coating requirements that have a numerical limit, such as a coating thickness. The test must yield a numerical value, and certain statistical requirements must be met. Method B 762 contains several plans and also gives instructions for designating plans to meet special needs. The buyer and the seller may agree on the plan or plans to be used. If they do not, Method B 762 designates the plan to be used.

NOTE 6—When both destructive and nondestructive tests exist for the measurement of a characteristic, the purchaser needs to state which one is to be used so that the proper sampling plan is selected. A test may destroy

the coating, but in a noncritical area; or, although it may destroy the coating, a tested part may be reclaimed by stripping and recoating. The purchaser needs to state whether the test is to be considered destructive or nondestructive.

9.2 An inspection lot shall be defined as a collection of coated articles of the same kind that have been produced to the same specifications, coated by a single supplier at one time or approximately the same time under essentially identical conditions, and that are submitted for acceptance or rejection as a group.

9.3 If special test specimens are used to represent the coated articles in a test, the number used shall be that required in 8.1.1.

10. Rejection and Rehearing

10.1 Parts that fail to conform to the requirements of this specification may be rejected. Rejection shall be reported to the producer promptly in writing. In the case of dissatisfaction with the results of a test, the producer may make a claim for a rehearing. Coatings that show imperfections may be rejected.

11. Certification

11.1 When specified in the purchase order or contract, the purchaser shall be furnished with certifications that the samples representing each lot have been either tested or inspected as directed by this specification and the requirements have been met. When specified in the purchase order or contract, a report of the test results must be furnished.

12. Record Retention

12.1 The seller is to maintain all job records and samples for a period of two years unless the purchaser requires a longer period.

13. Keywords

13.1 aluminizing; corrosion-resistant coatings; diffusion coating; pack cementation

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