



Standard Specification for Electrolytic Plasma Treatment Processing of Conductive Materials¹

This standard is issued under the fixed designation A1093/A1093M; 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 the requirements for cleaning, coating, or surface modification, or combinations thereof, of conductive materials, primarily metals.

1.2 This specification covers any conductive material treated or processed by the electrolytic plasma process (EPP) including: products designated as long products, including wire and fine wire; flat-rolled materials; fasteners; connectors; bolts; assemblies; structural materials; hardware items; and medical items.

1.3 Products created under this process shall specifically specify requirements for the specific product being processed using the EPP process.

1.4 This specification is applicable for orders in either inch-pound or SI units.

1.5 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

1.6 *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:²

A90/A90M Test Method for Weight [Mass] of Coating on Iron and Steel Articles with Zinc or Zinc-Alloy Coatings

A924/A924M Specification for General Requirements for Steel Sheet, Metallic-Coated by the Hot-Dip Process

B487 Test Method for Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of Cross Section

D2200 Practice for Use of Pictorial Surface Preparation Standards and Guides for Painting Steel Surfaces

F519 Test Method for Mechanical Hydrogen Embrittlement Evaluation of Plating/Coating Processes and Service Environments

2.2 SSPC Standard:³

SSPC-VIS 1 Guide and Reference Photographs for Steel Surfaces Prepared by Dry Abrasive

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 *anode, n*—positively charged device within the plasma reactor.

3.1.2 *applied potential, n*—direct current (dc) voltage applied between two electrodes, anode and cathode.

3.1.3 *aqueous plasma, n*—use of water as the source of the $H_2 - H^+ + H^+ + 2e^-$.

3.1.4 *cathode, n*—negatively charged work piece within the plasma reactor.

3.1.5 *electrolyte, n*—aqueous solution containing ions capable of conducting electric current.

3.1.6 *micro-roughness, n*—features formed as a result of the kinetic energy produced from the implosion and rapid quenching phenomenon that occur during electro-plasma technology (EPT) processing.

3.1.7 *plasma, n*—collection of free moving electrons and ions capable of conducting electric current.

3.1.7.1 *Discussion*—In this case, the gas is hydrogen and the ion is H^+ . Energy is required to make plasma. Without sustaining energy, plasma recombines into molecular hydrogen, H^+ .

¹ This test method is under the jurisdiction of ASTM Committee A05 on Metallic-Coated Iron and Steel Products and is the direct responsibility of Subcommittee A05.12 on Wire Specifications.

Current edition approved Oct. 1, 2015. Published November 2015. DOI: 10.1520/A1093A1093M-15.

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.

³ Available from the Society for Protective Coatings, 40 24th St., 6th Floor, Pittsburgh, PA 15222.

3.1.8 *plasma reactor, n*—device housing that confines the plasma within the work zone containing the electrolyte, which allows the formation of plasma.

3.1.9 *salts, n*—refers to the soluble metal salts used within the aqueous plasma process.

3.1.10 *surface morphology, n*—unique surface characterized by the presence of micro-craters and spheroids created by the electro-plasma process.

3.2 Acronyms:

3.2.1 *EPP*—Electro-plasma processing

3.2.2 *EPT*—Electro-plasma technology

4. Electro-Plasma Process (EPP)⁴

4.1 Electro-plasma processes (EPP) are a hybrid of conventional electrolysis and atmospheric plasma processes. All the independent studies lead to a common observation that, at a certain value of voltage between two electrodes in an aqueous electrolyte, will lead to deviation from Faraday's normal electrolytic regime. Applied voltage is significantly greater when compared to the conventional electrolysis leading to the analogous phenomenon that is accompanied by the formation of a continuous plasma envelope around either the cathode or the anode with the presence of luminous discharge or glow plasma. The critical factors that influence the formation of the continuous plasma envelope include applied potential, electrolyte temperature, geometry of the electrodes, nature and properties of the electrolyte, and flow dynamics of the electrolyte and work piece. Most of the studies are concentrated on the anodic regime.

NOTE 1—In the EPT process described here, the work piece is the cathode and it is negatively charged. Plasma forms on the cathode. The anode is the positively charged electrode.

Electro-plasma technology (EPT) is used to engineer metal surfaces in the cathodic regime, but it can be used in the anodic regime depending on desired results. EPT processing is a dynamic process that involves delivery of aqueous electrolyte into a confined chamber (EPT reactors) on the surface of the work piece. Balance between electrolyte flow and plasma generations ensures a uniform treatment of the metal surfaces.

4.2 EPP is an environmentally friendly technology applied in a closed-loop system.

NOTE 2—There are no solid wastes generated from EPT. Vapor generated during the process can be captured and returned.

5.1 In a cathodic regime with an electrolyte of near neutral salts, cleaning of a metal surface can be achieved. EPT can effectively remove lubricants, dirt, metal oxide scale, and so forth from metal surfaces. Organic materials are disassociated

into their gaseous components, thick oxides are broken and blasted off the surface, and thin residual oxide layers are reduced back to their pure metallic element.

5.2 EPT cleaning uses thermal shock, electrical current, chemical reaction, and kinetic energy in the form of cavitation.

5.3 Plasma formed on the surface of the work piece in a liquid electrolyte results in unique surface characteristics and a unique morphology (micro-roughness).

6. Electro-Plasma Cleaning Process

6.1 Electro-plasma cleaning of a continuous length of a conductive metal is accomplished by passing the item through an appropriately designed reaction chamber. The number of reactors required is speed-dependent and they are run in tandem.

6.2 The reactor chamber is supplied with electrolyte in a controlled method.

6.3 A large potential is set between the anode and cathode (work piece), normally in the range of 25 to 250 V.

6.4 Luminous plasma is generated at the work piece surface and the work piece is cleaned of contaminants and oxides.

6.5 The work piece then passes through a hot (70 ~ 80°C [160 ~180°F]) water spray rinse and is dried.

6.6 The product can now be collected on a typical package or it can go directly into EPT deposit reactors for coating with a variety of metals and alloys.

7. Testing Requirements: Cleaning

7.1 The degree of cleaning is typical for a specific industry but is typically done by comparing the appearance of the dirty component to the cleaned component and, therefore, is an attribute rating. In this case, it is recommended that the surface be examined under a magnification of 20× for signs of residual lubricant, soils, and oxides. A minimum of at least three areas should be observed and the estimate of cleanliness recorded. A rating is developed by estimating the percent of the field that contains no signs of residual lubricant, soils, or oxides. The average of these estimate observations are recorded and used to rate the level of cleanliness. The actual cleanliness level of a specimen may vary widely and still be acceptable for the intended end use. The user will be responsible for determining the minimum level of their particular application. It has been the experience when using EPT that the surface is 99 to 100 % free of any visual contamination.

8. Electro-Plasma Deposition

8.1 EPT has the ability to deposit metal and alloy coatings such as zinc, nickel, zinc-nickel, nickel-copper, molybdenum, tin, and so forth. EPT coatings exhibit excellent adhesion with the substrate and are deposited at significantly high deposition rates as compared to conventional electrolytic processes. EPT has also been used to alloy metals such as molybdenum onto

⁴ The electrolytic plasma process, Patent number US 6585875 is covered by a patent. Interested parties are invited to submit information regarding the identification of an alternative(s) to this patented item to the ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.

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another metal's surface. The science of electro-plasma deposition is reviewed in the publication "Electro-Plasma Technology: Science and Engineering – An Overview."⁵ EPT deposition does not follow typical electro-plating principles. The transport of ions from the electrolyte to the cathode is not limited by what is known as the boundary layer. In EPT the metallic ions are transported on plasma bubbles and, in simple terms, hurled at exceptionally high velocities to the cathode. This accounts for the higher deposition rates compared to normal electro-plating rates and eliminates the phase boundary diffusion layer that limits the rates in conventional systems.

9. Electro-Plasma Deposition Process

9.1 Electro-plasma deposition of a continuous length of a conductive metal is accomplished by passing the item through an appropriately designed reactor chamber. The number of reactors required is dependent upon speed and the thickness of deposit desired, and they are run in series. Deposits can be made as a single layer of a mono-metal or an alloy. Deposits can also be in separate and distinct layers of different metals. Very thin layers of some metals can be alloyed with the surface of the substrate such as molybdenum.

9.2 The reactor chamber is supplied with electrolytes containing the metal ions to be deposited and supplied in a controlled method.

9.3 A large potential is set between the anode and cathode (work piece), normally in the range of 25 to 250 V. (In EPT, Faraday's law does not apply as with normal electro-plating processes.)

9.4 Luminous plasma is generated at the work piece and the wire or other work piece is coated with the metal ions contained in the electrolyte.

9.5 The work piece then passes through a hot (70 ~ 80°C [160 ~180°F]) water spray rinse and is dried.

9.6 The product can now be collected on a typical package or it can go directly into a second set of deposition reactors for coating another metal.

9.7 The last step is to rinse the coated product with hot water and dry before packaging.

9.8 *Intermetallic*—With EPT deposition, there is no inter-metallic zone created.

9.9 Mechanical properties such as tensile strength and torsion will not change because of the process. EPT also will not cause any hydrogen embrittlement. The process parameters should be controlled to make sure that the work piece is not overheated during the process. The tensile test data for as-drawn compared to as-EPT-zinc-coated and as-EPT-cleaned wire shows that the tensile strength is not changed. Tests according to Test Method **F519** have shown that the EPT process does not cause hydrogen embrittlement.

10. Testing Requirements: Coating

10.1 Testing is highly dependent on the user requirements.

10.2 *Adhesion*—The coating shall show no signs of flaking when the samples are bent or twisted as described in the following:

10.2.1 *Round*—Wraps should be made around mandrel of the same diameter as the test sample (minimum of five turns) and examined under 10× magnification.

10.2.2 *Wire*—To determine ductility and coating adhesion, wire can be wrapped around its own diameter, five wraps on without breaking of the wire or delamination of the coating.

10.2.3 *Sheet*—A 180° bend examined under 10× magnification.

10.3 *Ductility*—The coated samples shall be broken in tension and the fracture observed at 10× magnification, and the coating will show ductile flow to point of fracture without separation between coating and substrate.

10.4 *Thickness*—The specific thickness shall be agreed between the purchaser and manufacturer. The following methods may be used to verify the average thickness.

10.4.1 *Electrical Permeability*—Using a standard of known thickness of the same coating material can provide a relative measurement.

10.4.2 *X-Ray Diffraction*—Using a standard of known thickness of the same coating material can provide a relative measurement.

10.4.3 *Metallographic Measurement*—Test Method **B487**. A sample can be prepared in cross section, polished, and viewed at an appropriate magnification to allow microscopic measurement of the perpendicular thickness of the coating. A minimum of three points spaced around the cross section evenly but including thicker and thinner spots to arrive at an average thickness.

10.4.4 *Weight Measurement by Stripping the Coating*—Test Method **A90/A90M**. Selecting a sample of appropriate size, normally 5 to 20 g, and the initial weight is recorded. The coating is dissolved with the appropriate chemical agent. The sample is rinsed in deionized (DI) water and dipped in acetone and allowed to dry. Reweigh the sample and record the new weight. Using the density of the coating material, calculate the average thickness on the cross section.

11. Coating Properties

11.1 *Coating Weight (Mass)*—Use the following relationships to estimate the coating thickness from the coating weight for zinc (mass):

$$1 \text{ oz/ft}^2 \text{ weight} = 1.7 \text{ mil coating thickness} \quad (1)$$

and

$$17.14 \text{ g/m}^2 \text{ weight} = 1 \text{ } \mu \text{ coating thickness} \quad (2)$$

where:

$$1 \text{ oz/ft}^2 = 305.15 \text{ g/m}^2$$

11.2 Coating Weight (Mass) Tests

11.2.1 Coating weight (mass) tests shall be performed in accordance with the requirements of Specification **A924/A924M**.

⁵ Gupta, P., G. Tenhundfeld, E.O. Daigle, and D. Ryabkov. "Electro-Plasma Technology: Science and Engineering – An Overview," *Surface and Coatings Technology*, Vol. 201, No. 21. 2007; p. 8746–8760.

11.2.2 The referee method to be used shall be Test Method [A90/A90M](#).

11.3 *Coating Bend Test*—Any bend with tension on the outside surface that does not cause fracture of the base metal can be performed with the EPT coatings to reveal the potential delamination, cracking, or peeling of the coating.

11.3.1 The bend test specimens of coated sheet or bar designated by prefix “G” (“Z”) shall be capable of being bent through 180° in any direction without flaking of the coating on the tension side of the bend only. Flaking of the coating within 6 mm [0.25 in.] of the edge of the bend specimen shall not be cause for rejection.

11.4 *Corrosion*—The customer and supplier should agree on the corrosion properties.

11.4.1 *Wire Products*—The coated rod or wire can be drawn to the maximum limit possible within the properties of the original material.

11.4.2 Since no coating is lost during drawing, the final coating thickness will depend on the starting coating thickness and the total diameter reduction during drawing. The amount of coating on the finished product should be agreed between the supplier and customer.

11.5 *Appearance*—The coating should be continuous and reasonably uniform. It should be free of any imperfection.

12. Surface Modification/Cleaning Characteristics

12.1 The surface of the work piece will have unique characteristics because of the plasma process. This is identified by “spheroids” and “craters” that are created on the surface. The size and distribution of these characteristics can be changed by altering the process parameters that includes voltage and electrolyte properties.

13. Coating Weight and Thickness

13.1 The coating weight and thickness for the final product should be agreed upon between the customer and manufacturer.

14. Coating Characteristics

14.1 The coating deposited by EPT is free of any intermetallic layers. For example, in the case of zinc coatings, only pure zinc is present in the coating (see [Fig. 1](#) and [Fig. 2](#)). No zinc-iron intermetallic layers are present in the coating.

14.2 Steel chemistry does not have any influence on the coating properties.

14.3 The coating is adhered to the substrate by a metallurgical bond that is formed because of the action of plasma.

15. Retests and Disposition of Nonconforming Material

15.1 Retests, conducted in accordance with the requirements of the section on retests and disposition of nonconforming material of Specification [A924/A924M](#), are permitted when an unsatisfactory test result is suspected to be the consequence of the test method procedure.

15.2 Disposition of nonconforming material shall be subject to the requirements of 9.2 of Specification [A924/A924M](#).

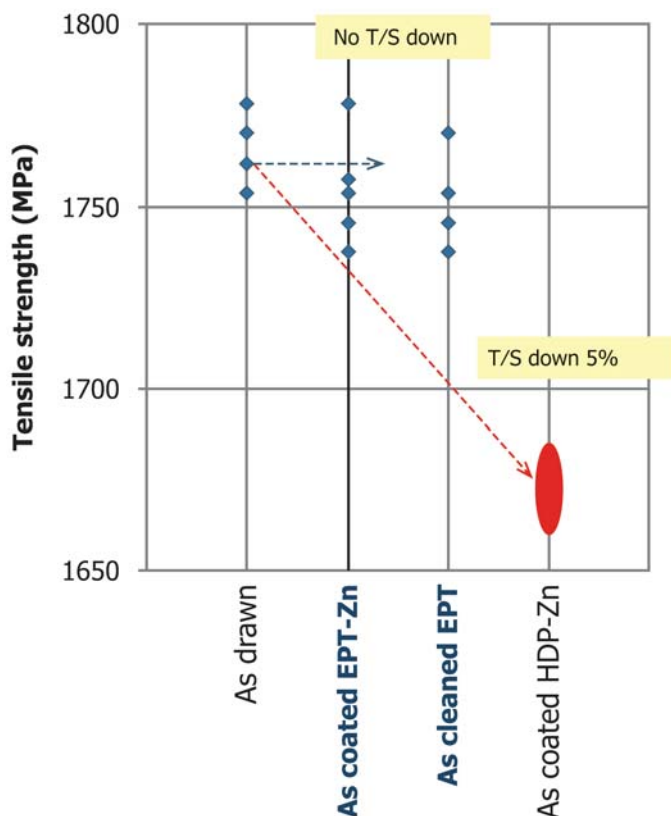


FIG. 1 Tensile Test Data Comparing As-Drawn, EPT Zinc-Coated, EPT Cleaned, and Hot Dip Galvanized Zinc-Coated Wire

16. Dimensions and Permissible Variations

16.1 All dimensions and permissible variations shall comply with the requirements of Specification [A924/A924M](#).

17. Sampling

17.1 Test specimens shall be selected randomly from each inspection lot.

17.2 The method of selection and sample size shall be agreed upon between the processor and the purchaser. Testing requirements shall determine the test specimen size.

18. Specimen Preparation

18.1 Test specimens shall be prepared in such a manner as to meet the necessary testing standards required to meet the purchaser’s requirements for cleaning and coating.

18.2 Cleaning shall be determined by the percentage of the contaminant found to remain on the processed sample, including oxide scale. Oxygen content should not be used since time and atmosphere can cause abnormal readings.

19. Test Methods

19.1 Cleaned samples can be observed under an optical microscope or scanning electronic microscope (SEM) to determine the level of cleanliness.

19.2 SSPC-VIS-1 can be used as a visual standard to determine the level of cleaning; however, the starting condition

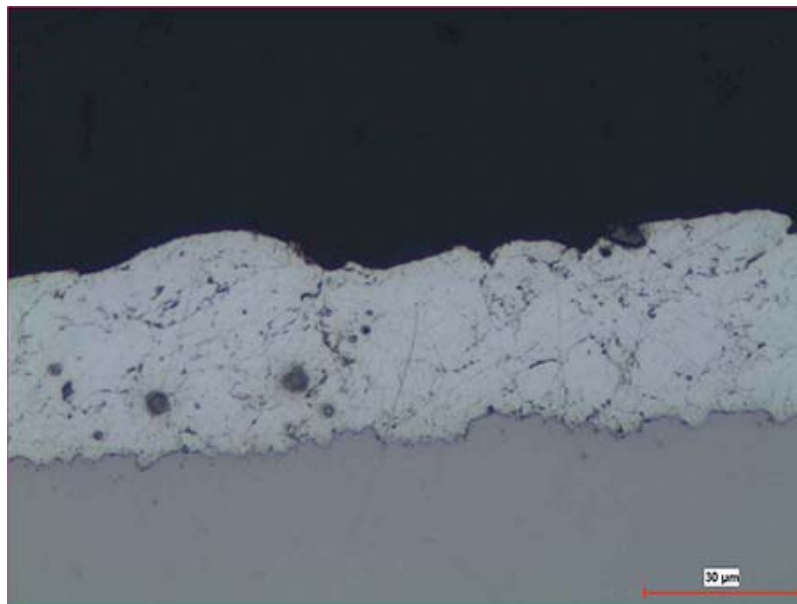


FIG. 2 Photomicrograph of EPT Zinc Coating (x750) Showing that Only Zinc is Present in the Coating and Intermetallic Layers are Absent

of the material to be cleaned has to be noted. This standard conforms to Practice **D2200**.

20. Certification

20.1 When specified by the purchaser or within the contract, the processor shall submit test specimens and reports for each specimen to the purchaser. The purchaser shall have to the option to accept the processor's reports or request additional test specimens for certification, in which case, the purchaser is obligated to pay for the cost for the additional testing and reporting.

21. Packaging

21.1 The purchaser shall specify packaging requirements at the time of placing the order, otherwise, the processor shall use a best practices for the product to be packaged.

22. Keywords

22.1 cleaning; coatings zinc; electro-plasma; steel products-metallic coated; steel wire; zinc-nickel

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