Designation: D 6105 – 04

Standard Practice for Application of Electrical Discharge Surface Treatment (Activation) of Plastics for Adhesive Bonding[1]

This standard is issued under the fixed designation D 6105; 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 practice covers various electrical discharge treatments to be used to enhance the ability of polymeric substrates to be adhesively bonded. This practice does not include additional information on the preparation of test specimens or testing conditions as they are covered in the various ASTM test methods or specifications for specific materials.

1.2 The types of discharge phenomena that are used for surface modification of polymers fit into the general category of nonequilibrium or non-thermal discharges in which electron temperature (mean energy) greatly exceeds the gas temperature.

1.3 The technologies included in this practice are:

Technology	Section
Gas plasma at reduced pressure	8
Electrical discharges at atmospheric pressure	9
AC dielectric barrier discharge	9.1
High Frequency Apparatus	9.1.1
Suppressed Spark Apparatus	9.1.2
Arc Plasma Apparatus	9.2
Glow Discharge Apparatus	9.3

NOTE 1—The term “corona treatment” has been applied sometimes in the literature to the different electrical discharge treatment technologies described in Section 9. This practice defines each electrical discharge treatment technology at atmospheric pressure presented in Section 9 and draws the necessary distinctions between them and corona discharge. See Test Method D 1868 for “corona discharge.”

1.4 The values stated in SI units are to be regarded as the standard.

1.5 *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.* Specific hazard statements appear in Section 6.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 724 Test Method for Surface Wettability of Paper (Angle-of-Contact Method)

D 907 Terminology of Adhesives

D 1868 Test Method for Detection and Measurement of Partial Discharge (Corona) Pulses in Evaluation of Insulation Systems

D 2578 Test Method for Wetting Tension of Polyethylene and Polypropylene Films

D 2651 Guide for Preparation of Metal Surfaces for Adhesive Bonding

D 5946 Test Method for Corona-Treated Polymer Films Using Water Contact Angle Measurements

3. Terminology

3.1 *Definitions*—Many terms are defined in Terminology D 907.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *AC dielectric barrier discharge*, *n*—a self-sustaining AC discharge in relatively short gaps with a solid dielectric layer, where the discharge bridges the entire air gap.

3.2.2 *contact angle*, *n*—the angle in degrees between the substrate surface and the tangent line drawn to the droplet surface from the three-phase point.

3.2.3 *corona*, *n*—visible partial discharges in gases adjacent to a conductor.

3.2.4 *corona treatment*, *n*—see Note 1.

3.2.5 *electrical discharge*, *n*—any of several types of electrical breakdown of gases, primarily air.

3.2.5.1 *Discussion*—The type of discharge depends upon several controllable factors, such as electrode geometry, gas pressure, power supply impedance, etc. When, at atmospheric

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[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.

pressure, the voltage reaches a certain critical value, the current increases very rapidly and a spark results in the establishment of one of the self-sustaining discharges, such as corona, arc, glow and dielectric barrier discharge. In many electrical discharges, ionized regions called plasma exist.

3.2.6 *electrical discharge treatment*, *n*—activation of a polymer surface using electrical discharges to increase surface energy and create polar functional groups on the polymer surface; nonequilibrium discharges are used primarily for surface treatment.

3.2.7 *electric arc*, *n*—a self-sustaining discharge in the gap between two electrodes having a low voltage drop and capable of supporting large currents.

3.2.8 *gas plasma*, *n*—extremely reactive, partially ionized gas consisting of free electrons, positive ions, free radicals, metastables and other species; plasmas exist over a wide range of temperature and pressure and are capable of inducing chemical modifications on polymer surfaces.

3.2.8.1 *Discussion*—The positive ions, the electrons, and the neutral gas atoms of a plasma may or may not be in thermal equilibrium. Since plasma is usually established by an electric field, the temperature of the positive ions is usually greater than the gas temperature, and the electron temperature may be very high.

3.2.9 *glow*, *n—in electrical discharges*, a self-sustaining discharge in the air gap, where the gas near the sharply curved electrode surfaces breaks down at a voltage less than the spark breakdown voltage for that gap length.

3.2.10 *partial discharge*, *n*—electric discharge that only partially bridges the insulation between conductors.

3.2.11 *polarity*, *n—in surface chemistry*, value that quantifies concentration of polar functional groups on the polymer surface and is measured as a polar component of surface energy divided by a sum of polar and non-polar components.

3.2.12 *spark breakdown*, *n*—a sudden transition from the "dark" discharge to one of the several forms of self-sustaining discharge; this transition consists of a sudden change in the current.

3.2.13 *surface energy*, *n—for a given solid*, defines molecular forces of its interaction with other interfaces, J/m^2.

4. Summary of Practice

4.1 This practice identifies and defines several electrical discharge treatment technologies for surface modification of polymers. The practice outlines essential technical aspects of each technology.

5. Significance and Use

5.1 Bonding of many polymeric substrates presents a problem due to the low wettability of their surfaces and their chemical inertness. Adhesive bond formation begins with the establishment of interfacial molecular contact by wetting. Wettability of a substrate surface depends on its surface energy. The surface activation with electrical discharges improves wettability of polymers and subsequent adhesive bonding. The surface activation with electrical discharges results in addition of polar functional groups on the polymer surface. The higher the concentration of polar functional groups on the surface the more actively the surface reacts with the different polar interfaces.

5.2 To achieve a proper adhesive bond the polyolefin substrate's polar component should be raised from near zero to 15 to 20 mJ/m^2.

5.3 The pre-treated surfaces are ready for application of the adhesive immediately after the treatment.

6. Hazards

6.1 *Ozone*—Ozone is a by-product of the electrical discharge in atmospheric-pressure air. The ozone produced during the treatment can be vented into external atmosphere where dilution and subsequent breakdown will occur. If the ozone cannot be vented out, the station should be equipped with an exhaust hood and activated carbon filter or manganese dioxide catalyst.

6.2 *Electrical Hazard:* **Warning**—The users of these practices must be aware that there are inherent dangers associated with the use of electrical instrumentation and that these practices cannot and will not substitute for a practical knowledge of the instrument used for a particular surface preparation.

6.3 *Radio Frequency:* **Warning**—Persons with pacemakers may be affected by the radio frequency.

6.4 Electrical discharge treatments produce no volatile organic compound (VOC) emissions.

7. Procedure - General

7.1 *Surface Cleanliness*—The surface must be clean prior to submitting the specimen to any of the treatment processes. Potential surface contaminants include the following: additives, handling residue (fingerprints), mold release, machine oil, and grease.

7.1.1 *Techniques for Cleaning Surface*—Use a technique for cleaning the surface appropriate for the substrate. If no other cleaning method is specified, use a solvent wipe with isopropyl alcohol and clean, low lint cloth or wipes.

7.2 *Selection of Appropriate Electrical Discharge Treatment*—When making a choice the following factors must be considered:

7.2.1 Necessary treatment level,

7.2.2 Treatment speed,

7.2.3 Treated parts shape and size,

7.2.4 Process type - continuous, batch, etc, and

7.2.5 Economics.

Consult the attribute chart in Appendix X1 for comparison.

7.3 *Procedure for Polymer Surface Treatment*—Surface treatment with electrical discharges involves, in general, applying the discharge, and the plasma generated in the discharge, to the surface to be treated.

7.4 *Procedure for Determining Efficacy of Treatment*:

7.4.1 *Water Break Test*, Guide D 2651, Section 5.5.4. A water-break test is a common method used to analyze surface cleanliness. This test depends on the observation that a clean surface (one that is chemically active or polar) will hold a continuous film of water, rather than a series of isolated droplets. This is known as a water-break-free condition. A break in the water film indicates a soiled or contaminated area.

Distilled water should be used in the test, and a drainage time of about 30 s should be allowed.

7.4.2 *Water Contact Angle Determination*, Test Method D 724 and Test Method D 5946. This is the most precise method to evaluate surfaces. The contact angle data can be easily used for statistical analysis and statistical process control. Perform the test on enough of the treated area to assess treatment uniformity.

7.4.3 *Dyne Solution Method for Wetting Tension*—This test method is based on Test Method D 2578. When applied to other materials, or shaped items, it may produce erroneous results. The results from this method are approximations and should be used with caution.

7.5 *Shelf Life of Treated Surfaces*—Shelf life of treated polymers is determined by the treatment level decay from the surface energy level achieved in the treatment below a predetermined value. Use techniques described in 7.4 to determine the treatment level decay.

7.5.1 In general, surface energy rapidly decreases immediately after the treatment and then stabilizes at a level which is higher than the initial surface energy. The treatment shelf life ranges from hours to years, depending on the plastic, its formation, how it was treated, and the ambient environment of the storage area. It is recommended to do the adhesive bonding operation of the treated material soon after treatment.

8. Plasma Treatment at Reduced Pressure

8.1 Plasmas at reduced pressure 13.3 to 133 Pa (0.1 to 1 torr) are generated in a plasma reactor chamber. This is a pressure vessel designed to support the pressure/flow conditions of the plasma. The material for processing is placed in the chamber and a necessary degree of vacuum is established. A source of high-frequency energy is coupled to the reactor. The most common are radio frequency plasmas (13.56 MHz, an FCC assigned frequency) and microwave plasma (2450 Mhz) and less common are lower frequency range devices (60 Hz and 20 to 100 kHz). The plasma fills the whole chamber, resulting in a three-dimensional treatment of the objects placed inside the chamber.

9. Electrical Discharge Treatment at Atmospheric Pressure

9.1 *AC Dielectric Barrier Discharge*—In the AC dielectric barrier discharge apparatus the discharge is generated between two electrodes located on opposite sides of the treated surface. One or both electrodes is insulated. The treated part itself can serve as an insulator. There are two types of the AC dielectric barrier discharge apparatus, the high frequency apparatus and the suppressed spark apparatus.

9.1.1 *High Frequency Apparatus*—The typical apparatus consists of a high-frequency power generator and high voltage transformer(s). Each generator/transformer(s) set is capable of supporting multiple discharge electrodes.

9.1.1.1 Voltage in the 10 000 to 70 000 V range at 20 to 30 kHz is used.

9.1.1.2 The treatment width depends on the size of the discharge electrode and can range from 1 to 1000 mm and more.

9.1.1.3 Small specimens can be treated as they are positioned and continuously moving through the discharge region between the electrodes.

9.1.1.4 Larger specimens, such as an automotive body panel, can be treated by placing it on an electrode fixture which is formed in the same shape as the inner surface of the part. The treated part is then subjected to the discharge from an array of electrodes.

9.1.1.5 The typical distance between the discharge electrode(s) and the treated surface ranges between 2 to 50 mm, and depends on the applied voltage and electrode geometry.

9.1.1.6 The typical treatment speed ranges from 1.7 to 42 $\times 10^{-2}$ m/s (1 to 25 m/min), and depends on the number of electrodes, their geometry and distance to the treated surface.

9.1.1.7 High frequency apparatus can be effectively used to treat conductive polymers by using insulated electrodes and the treated surface as a second electrode.

9.1.2 *Suppressed Spark Apparatus*—In a suppressed arc apparatus discharge is generated between two insulated electrodes maintained at a very high potential of 50 to 60 Hz. The strong electrical field in the air gap generates a plasma region while a spark breakdown in the air gap is quenched by a dielectric insulation on electrode surfaces.

9.1.2.1 *Apparatus*—A typical suppressed spark apparatus has a tunnel-type treating area with a pair of metal electrodes mounted one against the other on each side of the treating chamber. The conveyor belt made of a dielectric material travels at a slow speed 8.3 to 50 $\times 10^{-3}$ m/s (0.5 to 3 m/min) through the treating area.

9.1.2.2 Objects to be treated are placed on the conveyor belt.

9.1.2.3 Maximum distance between the electrodes is 400 mm.

9.1.2.4 The treatment width is determined by the distance between the electrodes.

9.1.2.5 The potential difference between the two electrodes reaches 200 000 V at 60 Hz. This voltage produces an electric field capable of breaking a large air gap between the electrodes. The electrodes are covered with insulating layers which help suppress the spark formation.

9.2 *Arc Plasma Apparatus*—A plume of plasma is generated by blowing air through an electrical arc between two metal electrodes. This plume is directed onto the surface to be treated.

9.2.1 *Apparatus*—A typical apparatus consists of a pair of high voltage transformers, a blower and one or more pairs of metal wire electrodes. Each of the two electrodes is maintained at about 5000 VAC RMS at 50/60 Hz, with 180 degrees phase shift between the electrodes. Therefore, there is always a potential difference between the electrodes. The power supply with low impedance supports high current to sustain the arc. A blower fan can be placed immediately behind the electrodes or in the power supply housing and be connected to the electrodes with a flexible hose.

9.2.2 The treatment width ranges from 20 to 50 mm for each electrode pair.

9.2.3 The typical distance between the discharge electrodes and the treated surface ranges between 5 to 20 mm. The treatment level drops as the distance increases.

9.2.4 A typical treatment speed ranges from 1.7 to 8.3 × 10^{-2} m/s (1 to 5 m/min). The treatment level drops as the treatment speed increases.

9.2.5 Arc plasma apparatus should be used with caution when treating conductive polymers as the discharge tends to destroy the surface being treated.

9.3 *Glow Discharge Apparatus*—The glow discharge at atmospheric pressure is generated from sharp electrodes with high voltage of 0.5 to 2 MHz frequency. There is no need for the second electrode to maintain the glow discharge at that frequency

9.3.1 *Apparatus*—Discharge at that frequency is generated by electromechanical means, such as a spark gap generator and a Tesla coil.

9.3.2 The typical distance between the discharge electrodes and the treated surface ranges between 5 to 20 mm. The treatment level drops as the distance increases.

9.3.3 The typical treatment speed ranges from 1.7 to 8.3 × 10^{-2} m/s (1 to 5 m/min). The treatment level drops as the treatment speed increases.

9.3.4 The treatment width ranges from 10 to 70 mm for each electrode.

9.3.5 Glow discharge apparatus should be used with caution when treating conductive polymers as the discharge tends to destroy the surface being treated.

10. Report

10.1 Report the following for all treatments:

10.1.1 Type of treatment,

10.1.2 Type of treatment equipment used,

10.1.3 Equipment settings (voltage, frequency, power, etc.), exposure time, treatment speed, etc.,

10.1.4 Test conditions – temperature, humidity, etc.,

10.1.5 Specimen cleaning procedure,

10.1.6 Confirmation test procedure for cleaning and surface treatment with results, and

10.1.7 Complete identification of the specimen.

11. Keywords

11.1 discharge; corona treatment; modification; polymer; surface; surface treatment

APPENDIX

(Nonmandatory Information)

X1. Attribute Chart

X1.1 Table X1.1 compares attributes of treatment technologies.

TABLE X1.1 Attributes of Treatment Technologies

Technology	Gas Plasma	AC Dielectric Barrier Discharge		Arc Plasma Apparatus	Glow Discharge Apparatus
		High Frequency Apparatus	Suppressed Spark Apparatus		
Treatment Environment	Reduced Pressure	Atmospheric Pressure	Atmospheric Pressure	Atmospheric Pressure	Atmospheric Pressure
Practical Treatment Level	>50 mJ/m^2	>50 mJ/m^2	>50 mJ/m^2	<50 mJ/m^2	<50 mJ/m^2
Uniformity	high	high	average	average	average
Treatment Speed	depends on chamber size and design	high	high throughout	low	low
Treated Part Shape and Size	no limitations	no limitations	rounded, convex surfaces; limited by the size of the tunnel	simple shapes; small parts	simple shapes; small parts
Safety	general safety, RF safety	high voltage safety, OSHA ozone limits	high voltage safety, OSHA ozone limits	high voltage safety	high voltage safety, OSHA ozone limits