In-Line Plasma Treatment of Wire Insulation Materials

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Durability & legibility of marks made by ink-jet printers depend on system parameters & wire surface characteristics. Polymer surface modification using plasma treatment is an effective way to improve this consistency & durability.

Polymer materials are successfully used in virtually every industry. Applications range from thin-film technology and protective coating to composite materials and fiber optics.

Plastics offer good physical, chemical and weather resistance, and are low-cost and easy to process. But, low surface energy and low wettability limit their applications when strong bonding is required between polymer surface and adhesive. Permanent bonding, coating, printing, etc., on many plastic surfaces is impossible without some surface pretreatment.

Wire insulation materials are no exception. Beside the inherent bad polymer surface properties, the wire surface incurs other problems during manufacture. As wire extrusion is a nonequilibrium process, small variations in baking temperature, line speed, humidity, etc., cause local changes in surface roughness, porosity, crystallinity, uniformity and other characteristics on micro and macroscopic levels. The process does not take place in a clean room environment, so dust particles in the air can also influence surface properties. Insulation surface layers are the most sensitive to these variations, and surface physical and chemical characteristics define adhesion strength and other properties of the interface layer that are critical for marking, striping or coating on the wire surface.

Plasma parameters & gas mixture vary depending on polymer surface properties & surface modifications.

This is a main reason why permanency of marks printed on wire or cable can vary from manufacturer to manufacturer, gauge to gauge, batch to batch and even along the same wire segment. To get the durability and consistency required by most military and commercial aerospace specs, special wire surface treatment is needed. This treatment increases wire surface printability before the marking process and reduces dependence of marking quality on the wire insulation's surface properties.

Polymer Surface Modification

For the last 50 years, different modification techniques have been used to alter polymer surfaces without affecting the bulk properties of the material. Among these methods are chemical treatment, flame treatment, coronas, low pressure plasmas, infrared (IR), ultraviolet (UV), X-ray and γ-ray irradiation, electron and ion beams bombardment, ozone exposure and others.

Plasma treatment is one of the most universal methods. Here, charged particles (ions or electrons) accelerate to energies comparable to or exceeding polymer surface bond energies. When these particles strike a solid surface, they could be reflected and neutralized or the impact could cause the target to eject electrons or atoms. They might be trapped into the target. Also, the impact could cause some structural surface rearrangement varying from point defects (missing atoms), displacement of atoms from original positions (iterstitials) to gross effects like dislocations, changing crystal lattice, altered stoichiometry and other order-disorder phenomena. Impact could promote surface chemical modification if the plasmas are chemically reactive. Or all of these things could happen.

A minimum of charged particles is always present in the plasma to maintain discharge. Their loss is compensated by production of ion-electron pairs during their collisions with neutral molecules in the gas phase and by photoionization. This is why most plasmas are characterized by intensive UV radiation that could affect the surface modification process.

Plasma parameters and gas mixture vary depending on polymer surface properties and surface modifications. For example, oxygen plasma treatment raises, while fluorine plasma lowers, the polymer's surface energy. Ammonia plasma modifies PTFE. Argon and other inert gases induce crosslinking.

Plasma treatment is usually fast and affects only the surface layer. Studying effect of treatment conditions in a glow discharge on PTFE wettability found that more film wettability is not due to UV influence or oxidation of functional groups. In addition to topographic changes and surface oxidation, plasma treatment causes surface charges. Correlation between polymer surface charging and wettability has been seen. Atmospheric and vacuum plasma treatments cause the same changes in wettability and adhesion when exposed for under a second. This is independent of specific plasma system features. So dielectric barrier discharge at atmospheric pressure seems the most effective in-line treatment process.

Plasma Treatment System for Wire & Cable

The PT-1000 Wire and Cable Treatment System improves wettability characteristics of various insulation materials including polyamides, fluoropolymers and polyesters. It creates a plasma curtain around the wire and bombards the dielectric surface with charged particles and high energy UV photons.

The PT-1000 incorporates a programmable generator that produces a high-voltage, high-frequency signal applied to a cylindrical electrode embedded in a protective dielectric chamber. When a wire passes through this electrode, the voltage is applied at above its threshold value to cause an electrical breakdown inside the chamber and this forms a uniform plasma curtain completely surrounding the wire. In most cases, this plasma produces a blue color glow that can easily be seen.

Plasma treatment intensity is defined as the amount of energy transmitted to the unit area of the wire surface per unit of time and depends on the driving signal's voltage and frequency. Plasma treatment level at a given intensity is proportional to exposure time and inversely related to wire diameter. The time dependence is usually exponential, with saturation occurring after a long period of exposure (10 sec or more) and linear for short periods of time (0.1 sec or less). For the same treatment quality on thick wire insulation or higher wire throughput speeds, plasma intensity must be increased. Adjusting the PT-1000's electrode voltage from 1 to 15 kV can do this. Despite high potential applied to the electrode, the active currents inside the chamber are very low. At normal operating conditions, average system power consumption is 100 W.

Threshold conditions, plasma density and composition depend on the pressure and nature of the gas in the dielectric chamber, wire diameter, dielectric constant, wire insulation thickness, insulation material properties, etc. A long wire passing through the chamber may be considered a grounded elec-

trode covered with a thin layer of dielectric material. So the electrical discharge in the gap between wire and outer cylinder is similar to the dielectric barrier-controlled discharge usually produced in plane geometry conditions. Electric field intensity on the surface of the wire is $E = 2V/Dw\ln(Dc/Dw)$ where V is applied voltage, Dw is diameter of the wire and Dc is internal diameter of the cylindrical high-voltage electrode.

Breakdown voltage in air at atmospheric pressure for different high-voltage electrode diameters is seen in **Figure 1**. Different wire diameters could require different electrodes for effective treatment. Characteristic discharge oscillograms are

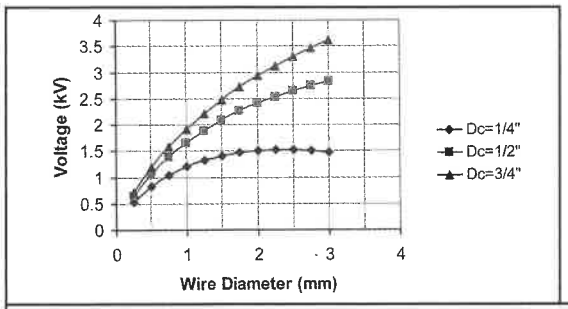

Fig. 1 — Dependence of breakdown voltage in air on wire diameter for different cylindrical electrodes.

shown in **Figure 2**. Discharge behavior differs for positive and negative voltage waves applied to the outer cylinder. For negative, current amplitude is higher and overall discharge duration is shorter. In the negative high-field region near the wire, positive ions achieve high energy before striking the wire surface. Secondary electrons liberated from the surface by ion bombardment start outward-moving electron avalanches, that produce many positive ions, initiating new electron avalanches, and so on. This continues until localized positive charge reduces electrical field below breakdown value. When the wire is positive, the cathode area field is too low to support ionization by gas electrons and discharge is maintained by other mechanisms. This corresponds to the excessive positive charge of the wire exiting the plasma chamber.

Tables 1, **2** and **3** show marking durability test results made for various wire types at different facilities. Marking was done in-line with Tri-Star's Plasma Treatment System. The data clearly show significant improved durability when plasma treatment is used. For Tefzel® insulated wire, the relative improvement could be up to 1000%. UV exposure and alignment of the wire passing through the UV oven could also affect marking quality since most inks used in aerospace are UV curable. Although the marking durability test is an ultimate acceptance test, it only shows qualitative advantage of plasma treatment. Quantitative analysis of the effects of plasma treatment on different types of wire insulation requires more objective test forms, independent of the features of the marking system.

One such method is contact angle measurement. Results of measurements on the surface of 22759/11-20 wire taken using a Video Contact Angle System made by **ASC Products**, Billerica, MA, USA, are shown in **Tables 4** and **5**. This system performs contact angle analysis and surface energy calculation. From 24 to 33 contact angles were recorded for each liquid on each wire sample for a statistically valid contact angle value. Angle measurements and surface energy calculations consistently indicated an increased wettability for plasma treated samples. The best results were achieved for the plasma treatment in argon atmosphere with small amount of ammonia.

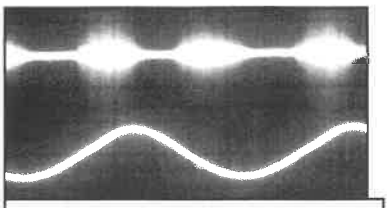

Fig. 2 — Discharge currents in air at atmospheric pressure at sinusoidal high-voltage signal.

Another method developed at Tri-Star specifically measures wettability on wire surfaces. When a droplet of liquid falls from the end of a glass capillary, its mass is proportional to the surface tension of the liquid and radius of the capillary. Knowing the capillary's radius and droplet's mass just before the droplet falls, it is possible to determine the liquid's surface tension. When the droplet falls from the surface of horizontally held wire, the mass of the droplet depends on wire diameter, insulation surface energy and on roughness, porosity and local surface charge. All these factors could affect marking durability. Measuring the critical volume of a water droplet provides quantitative data that describes the overall ability of the surface to accept ink before and after plasma treatment.

Figure 3 shows dependence of critical water droplet volume (CWDV) on wire diameter and surface energy. More surface energy and larger diameter result in higher CWDV. As diameter increases, CWDV reaches a max value determined mainly by liquid surface tension. Gold coating on copper-nickel contacts has a high surface energy and so retains bigger droplets compared to low surface energy fluoropolymers. But the difference is not as significant as the differences in surface energies. The coating has a much smoother and less porous surface than extruded fluoropolymer wires that seem to compensate high surface energy of the material.

Figure 4 provides plasma treated wire aging results relative to plasma exposure time. The 16878/6-26 extruded PTFE

Table 1. Marking Durability for Flame & Plasma Treated Wires (*Woven Electronics*, Simpsonville, SC, USA).

Insulation	ETFE			ETFE			IR ETFE				IR ETFE			FEP		
Wire Spec	M275000-24TE2U00			M27500-20TG2T14			M27500-24502T23			MIS-20186/1			WS 19199/2-20-1			MIL-C 17G		
Wire Vendor				Thermax			Therma-tics			Judd			Delta			Thermax		
Treatment / Testing	U	Fl	Pl	U	Fl	Pl	U	Fl	Pl	U	Fl	Pl	U	Fl	Pl	U	Fl	Pl
Rub (finger)	F	P	P	F	F	P	P	P	P	P	P	P		P	P	F	F	F
Rub (eraser)	F	P	P	F	F	P	F	P	P	F	P	P		F	P	F	F	P
Alcohol	F	P	P	F	P	P	F	P	P	P	P	P		F	P	F	F	P
1-1-1 Trichl.	F	P	P	F	P	P	F	P	P	P	P	P		P	P	F	F	P
TMC (freon)	F	P	P	F	P	P	F	P	P	F	P	P		P	P	F	F	P
Water+	F	P	P	F	P	P	P	P	P	P	P	P		P	P	F	F	P
Acetone	F	F	P	F	P	F	F	P	P	F	F	P		F	P	F	F	P
Eposolve 301	F	P	P	F	P	P	F	P	P	F	P	P		F	P	F	F	P
Masking Tape	F	P	P	F	F	P	F	P	P	F	P	P		P	P	F	F	P
Therm. Shock	F	P	P	F	F	P	F	P	P	F	P	P		F	P	F	F	P
ISO Oil	P	P	P	P	P	P	P	P	P	P	P	P		P	P	F	F	P
Pen(40dyne/cm)	F	P	P	F	P	P	P	P	P	P	P	P		P	P	F	F	F
Cold Shock	F	P	P	F	P	P	F	P	P	F	P	P		F	P	F	F	P

Notes: All wires marked with UV-curable ink; U (Untreated); Fl (Flame treated); Pl (Plasma treated); F (Fail); P (Pass); Rub (finger), 20 strokes of moderate pressure; Rub (eraser), per Mil-M-81531,4.6.2.; Alcohol; Trichl.; TMC; Water; Acetone; Exposolve per Mil-Std 202; Masking Tape, press on to marking, wait 30 sec., peel off, read marking; Thermal Shock per WS 19185; ISO Oil, soak for 48 hours, rub once; Pen, check spreading on wire surface; Cold Shock, 5 min. at -40°F (-40°C), bend, rub.

Table 2. Results of Rub Test with Skydrol Fluid Done at *McDonnell Douglas*, Long Beach, CA, USA, per DMS 2325 for Marking Kapton® Insulated Wire via Dot-Matrix Marker.

Conditions	No Treatment	Plasma Treatment	Heat Curing	Plasma Treatment/ Heat Curing
Sample 1	12	>30	14	>30
Sample 2	12	>30	30	>30
Sample 3	10	>30	26	>30
Sample 4	14	>30	18	>30
Sample 5	30	>30	26	>30
Sample 6	16	>30	24	>30
Sample 7	22	>30	14	>30
Sample 8	26	>30	22	>30
Sample 9	30	>30	30	>30
Sample 10	30	>30	22	>30

Note: Tests were stopped after 30 rubs.

Table 3. Results of Scrape Abrasion Test at *Raychem*, Menlo Park, CA, USA, for Marking Tefzel® Wire via Ink-Jet.

Line Speed	30 fpm (9 mpm)	60 fpm (18 mpm)	120 fpm (37 mpm)
UV Exposure	1 sec.	0.5 sec.	0.25 sec.
Avg. Cycles Plasma Off	7	11	12
Avg. Cycles Plasma On	58	>125	48

Test Method: Motor-driven, reciprocating cam mechanism, equipped with a reset counter & capable of a 3/8" (9.5 mm) stroke at 60 cycles/min., with a clamp for holding specimens. Contact surface is a smooth cylindrical steel mandrel, 0.025" (0.64 mm) diameter. Applied weight is 500 g (17.6 oz). One cycle corresponds to two strokes. **Note:** Tests were stopped after 125 cycles.

Table 4. Contact Angle (degrees) Measurement for Plasma Treated FEP Insulated Wire (22759/11-20).

Plasma Treatment Conditions	Water Surface Surface Tension 72 dyne/cm	Methylene Iodide Surface Tension 50 dyne/cm	Xylene Surface Tension 30 dyne/cm
No Treatment	101 ±5	75 ±4	46 ±4
Air Plasma	94 ±8	70 ±3	32 ±3
Argon Plasma	90 ±4	66 ±5	23 ±5
Argon/Ammonia	82 ±4	67 ±6	20 ±7

wire has original CWDV of 13 µL and diameter of 0.65 mm (0.026"). A 17 kHz sinusoidal high voltage signal was applied to the cylindrical electrode with internal diameter of 0.25" (6.35 mm) and 4" (102 mm) length. Total power consumed was 35 W. The 1 and 0.1 sec exposure times corresponded, respectively, to the 20 and 200 fpm (6.1 and 61 mpm) line speeds. The plasma treatment at this setting reaches an equilibrium in about 0.3 sec. Effect of plasma treatment achieved on the surface, stored in open air at normal conditions, slowly decays from maximum CWDV=25 µL to the 22 µL in two weeks. The drop of about 15% in the first two weeks is quite similar to the decay of uncompensated electrical charge on the PTFE surface.

CWDV, relative to plasma exposure time for ETFE wires, is shown in **Figure 5**. The curves shown have the same shape as the previous one reaching an equilibrium in about 0.5 sec. CWDV rises from 26 to 57 µL for crosslinked ETFE treated in air and from 29 to 53 µL for the regular ETFE treated in argon.

Conclusion

The PT-1000 improves durability of marks on Tefzel, Kapton®, Teflon® and other hard-to-print insulations. Treated marks pass most military and commercial specs. The system easily interfaces and operates with any wire marking process.

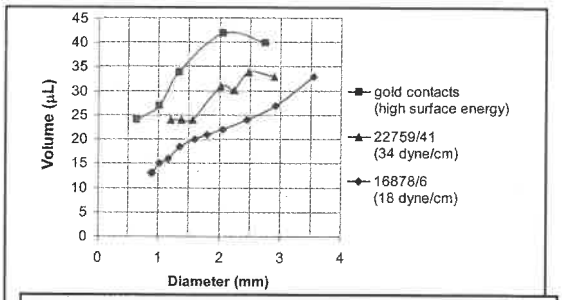

Fig. 3 — Dependence of critical water droplet volume on wire diameter for materials with different surface energies.

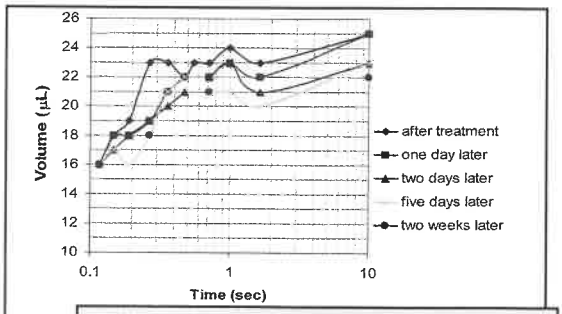

Fig. 4 — Dependence of critical water droplet volume on plasma exposure time for 16878/6-26 wire.

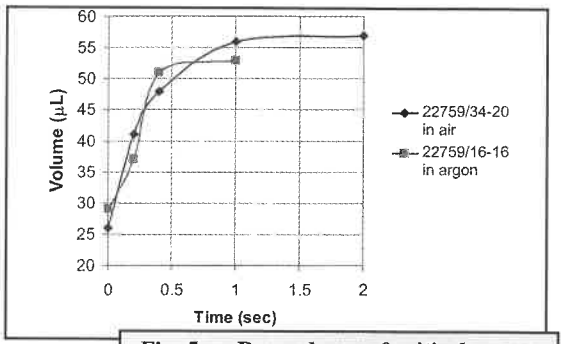

Fig. 5 — Dependence of critical water droplet volume on plasma exposure time for ETFE wires.

Once processed with the PT-1000, wire or cable requires no extra treatment such as alcohol, flame, preheating, etc. Treated surface retains improved wettability for up to several weeks. Long-term life of this treatment depends on storage conditions and insulation type. With fluoropolymers, this effect can last up to a year, but will eventually degrade.

Cables and wires of different outer diameters may need different corresponding electrodes. As the PT-1000 operates with ambient air, it does not need special gas mixtures or pressure settings. It can process many different types and sizes of wires and cables with no damage to insulation, and supports line speeds to 1000 fpm (305 mpm).

Table 5. Surface Energy Based on Contact Angle Measurement of Plasma Treated FEP Wire.

	Harmonic-Mean Approximation			Geometric-Mean Approximation		
Plasma Treatment Conditions	Dispersive Component Dyne/cm	Polar Component Dyne/cm	Total Dyne/cm	Dispersive Component Dyne/cm	Polar Component Dyne/cm	Total Dyne/cm
No Treatment	17.3	5.4	22.7	14.3	2.6	16.9
Air Plasma	20.5	7.2	27.9	17.7	3.9	21.6
Argon Plasma	19.9	9.1	29.0	16.4	6.0	22.4
Argon/Ammonia	23.0	11.5	34.5	19.3	8.4	27.7

WCTI

Company Profile... Tri-Star Technologies D.A.H. Co., makes the PT-1000 plasma treatment system, a fast & safe, stand-alone unit that improves wetting and adhesion characteristics of wire & cable insulation materials.

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