#### **Surface Treatment** by: Eric Finson and Stephen L. Kaplan

### **Packaging Technology**

## **Introduction**

In almost every industry, the nature of a material's surface can drastically affect a product's success. The reasons can be quite different, varying from purely aesthetic to functional. This is particularly true for packaging applications. To the consumer at the point of purchase a package must appear attractive and clean as well as preserve its contents. Obtaining the appropriate balance of structural, aesthetic, and functional (barrier) properties often requires compounding specific additives into the bulk material or combining several separate materials into a composite structure. The type of specialty additives often used as bulk treatments may include but are not limited to antistats, antiblocks, slip modifiers, plasticizers, fillers, and stabilizers for UV, oxygen, and heat. The operations or procedures used to take various raw materials and fashion them into packaging structures is often referred to as "converting." A surface treatment is frequently employed as part of the conversion process to alter the surface characteristics of the specific material being used. Typical surface treatment processes include altering the wettability of a substrate, improving the bondability of an applied material or the elimination of accumulated static charge. Surface treatment technologies can play a key role in the preparation of surfaces of the most commonly used packaging substrates such as paper, plastic, foil, or metal/inorganic depositions for subsequent processing steps. In many cases, packaging producers are required to select specially formulated and expensive materials (eg, printing inks, adhesives, polymer films, or structures) to ensure satisfactory performance. The alternative to this scenario is to choose a material or combination of materials for their bulk properties and then modify their surfaces to achieve appropriate performance attributes. Surface treatments can allow the necessary modifications to packaging material surfaces without altering their bulk properties so that individual or multilaminate composite packaging structures can meet or exceed end use requirements.

### **Surface Preparations**

Achieving adequate adhesion to polymers is a recurring and difficult problem throughout the packaging industry. Historically, various surface treatments have been used to improve the adhesion of coatings to plastics, including flame and corona, mechanical abrasion, solvent cleaning or swelling followed by wet chemical etching, or the application of specialized coatings in the form of chemical primers. Also, high energy density treatments (1) such as ultraviolet (uv) radiation, electron-beam and cold-gas-plasma methods have gained greater acceptance on a larger scale for substrate surface modification. They provide a medium rich in reactive species, such as energetic photons, electrons, free radicals, and ions, which, in turn, interact with the polymer surface, changing its chemistry and/or morphology. These processes can be readily adopted to modify surface properties of webs, films, and rigid containers, which are commonly incorporated into packaging structures. The available surface treatment technologies are summarized in Table 1.



# **Table 1. Surface-Treatment Technologies**

**Abrasive techniques.** Among conventional surface treatment techniques, mechanical abrasion serves only to increase the surface area of the material by "roughening" the exposed areas prior to coating or adhesive bonding. Mechanical abrasion can be achieved through dry blasting, wet blasting or hand/machine sanding. These processes can be very operator. sensitive, labor-intensive, dirty, and difficult to perform on the high-production volumes normally associated with packaging applications. To remove particulates or residues, a solvent wash usually follows mechanical abrasion. In many cases, the spent abrasive materials fall under the classification of hazardous substances and must be disposed of accordingly.

**Liquid cleaning techniques.** Liquid cleaning can be very useful for removing gross contamination. Fluid cleaning techniques for polymer surfaces fall into three main categories: hand wiping, solvent cleaning, and water-based washing. Hand wiping can be done with a variety of solvents, combination of solvents, or an aqueous solution of various chemicals. This process is very labor-intensive and is usually only employed in situations with low production volumes. Hand wiping can result in inconsistencies in quality due to either human error or the redeposition of soils onto the surfaces being cleaned from contaminated rags used in the process. Surface treatment by solvent cleaning is most beneficial in those cases where swelling of the polymer surface, due to solvent absorption, results in a rougher morphology that can improve the adhesion of coatings without adversely affecting the substrate's mechanical properties. The process uses inexpensive equipment and works reasonably well in many cases such as in the surface preparation of molded polymer parts for subsequent paint adhesive, or coating application. Solvent treatment processes can be conducted through wiping, immersion, spraying, or vapor degreasing. Typically, high-vapor-pressure organic solvents (alcohols, ketones, toluene, etc), chlorinated hydrocarbon solvents (eg, Freons, or 1,1,1-trichloroethane) or low-vapor-pressure organic solvents (terpenes, isoparaffins, lactates, esters, etc) are used in these processes (2). The major drawbacks of the technology are the environmental and process hazards associated with the use of large quantities of volatile chemicals, to the extent that any savings in equipment cost are usually offset by the increased cost of obligatory environmental controls. Also, solvent-based surface treatment has limited utility when a distinct change in the chemical nature of the substrate surface is desired. Water-based cleaning processes operate with relatively low costs, have low environmental impact, and are well suited for high production volumes. An industrial power washer usually consists of an overhead or floor conveyor with parts mounted on racks that pass through various spray stages. Most systems are composed of seven separate functions: precleaning, cleaning, rinsing, conditioner or rinse aid, deionized water rinsing, air blow off, and oven drying (3). However, for most commercial applications this technology requires capital investment for new equipment.

**Chemical etching.** Generally, chemical or acid etching is more effective in improving adhesion to polymers than liquid cleaning or solvent swelling. These processes cause specific chemical changes to the substrate surface, allowing greater chemical and physical interactions to adhesives or coatings. Some common examples of chemical etching processes for various polymer materials are listed in Table 2 (4). The chemical treatment of polyolefins in many cases incorporates the use of chromic-sulfuric acid mixtures (5). Previous studies have shown that for LDPE and HDPE severe roughening of the surface occurs. The effect of such treatments on polypropylene depends strongly on the prior thermal history of the polymer, and higher etch rates have been observed in areas of low crystallinity. There can also be changes in the polymer surface chemistry after chromic acid oxidation. Reflection ir spectra (6) show that this treatment results in the incorporation of oxygen (hydroxyl, carbonyl, and ester groups) and sulfur (SO3H)-containing functional groups in the LDPE surface. However, the precise chemical state of a polymer surface after chemical exposure is dependent on the nature and thermal history of the polymer, the composition of the etchant solution, and the time and temperature of the exposure. Often a process will work well for one material but will not be effective for another, necessitating specific treatments for each type of substrate. Also, chemical etching processes must be monitored closely as overexposure can result in overtreated, discolored, or damaged materials. In many instances, the etchant materials used can pose serious safety, hazard, and disposal problems. Although many of these processes can be effective in treating specific polymer materials, numerous users are seeking alternatives because of the concerns for operator safety and the complications of use and disposal.



#### **Table 2. Chemical-Etching Processes**

**Chemical priming**. Chemical primers can provide improved printing and adhesion characteristics by applying a chemically distinct layer on the substrate. This is usually accomplished by applying a liquid material in the form of a thin film and then drying off the solvents to leave a desired resin coating. Many polymer surfaces in the form supplied by the manufacturer can generate problems with respect to printability or the adhesion of decorative or functional coatings. Many packaging grade polymers are treated for improved adhesion, but chemical priming can also be used to improve productivity of converting processes. When primers are used on low-surface-energy substrates such as polyolefins, printing defects can be greatly reduced and issues such as screening, mottling, and "fisheyes" can be virtually eliminated. In the printing industry, press-speed limitations are seldom a function of solvent retention, but rather of adequate ink adhesion (7). As press speed increases the effectiveness of highenergy density treatments decreases. The fact that printing primers have the same surface tension characteristics at all press speeds provides a productive advantage as long as there is adequate drying capacity. As a result, maximum press or laminator speeds are attainable as long as the primer and subsequent printing inks can be dried. Unlike corona or flame treatment methods, primed surfaces tend to remain unaltered and the effect of additive migration to their surfaces appears to be limited. Primers can fall under various chemical classes such as polyethyleneimine, polyurethanes, acrylates, and chlorinated polymers. To prime foil substrates for printing or other subsequent converting operations, solvent-based solutions of nitrocellulose and shellac are still used. However, the trend is toward specific high-performance water-based primers such as ethylene acrylic acid. The main drawback to chemical priming is that there is no universal primer and different materials are needed for specific end-use requirements.

**Flame treatment**. In flame treatment, the polymer surface is passed through a flame generated by the combustion of a hydrocarbon (typically natural gas). Flame treatment can be conducted in a variety of configurations (illustrated in Fig. 1). Usually, containers or polymer webs are passed through a bank of flame jets at a given speed to provide the desired properties. In direct flame treatment, the high temperature (adiabatic flame temperature is approximately 33,000'F) is sufficient to dissociate nitrogen and oxygen molecules into free atoms (8). In addition, this high-temperature plasma contains carbon,

free electrons, positively charged oxygen, and other ions and excited species. Because of this reaction, polar functional groups such as ether, ester, carbonyl, carboxyl, and hydroxyl are contained in a flame plasma; these are incorporated into the surface and affect the electron density of the polymer material. The result is that the polymer surface is polarized. By changing the polymer surface from nonpolar to polar, the ink adhesion, laminating, and metallizing characteristics are enhanced. Also, exposure to the open flame oxidizes the surface and burns off surface contamination such as material additives, processing aids, or organic contamination such as oils or grease (9). It is probable that some of the polymer chains actually undergo melting, which "locks" their positions on cooling with respect to the three-dimensional configuration of the substrate, restricting rotation of the polymer molecules. Polar functional groups tend to stay in place on the surface, which can explain why the surface change due to flame treatment does not decay like that due to corona treatment. This process is somewhat energyintensive, and it may be difficult to reach recessed areas and to evenly treat complex shapes. Also, care must be taken to prevent thermal damage to sensitive materials such as thin-walled plastics or film substrates, and higher-energy output is necessary as production speed or throughput are increased.



(b)

**Figure 1.** (**a**) Ring burner for round-bottle treatment; **(b)** burner arrangement for treatment of round plastic bottles.

**Corona treatment.** In the case of corona treatment, the surface is exposed to a discharge between a grounded and powered electrode at high voltage. A low-frequency (typically 10-20 kHz) generator and step up transformer usually provide the high voltage to the electrode. In each half-cycle the applied voltage (20 kV peak) increases until it exceeds the threshold value for electrical breakdown of the air gap, causing the atoms and molecules to become ionized and creating an atmospheric plasma discharge. The voltage eventually peaks and falls below the conducting threshold. Each cycle consists of two such events involving current flow in each direction. In continuous operation the discharge appears to be a random series of faint sparks in a blue-purple glow (uv radiation). The point discharge generated across the pair of electrodes ionizes the gas present in the gap, which subsequently induces changes in the chemistry of the surface. Researchers (10) have demonstrated through derivatization reactions that carbonyl, enol, and carboxylic acid groups are formed on polyolefin materials after corona treatment. The most likely mechanism is free-radical in nature. The corona discharge contains ions, electrons, excited neutrals (atoms and molecules, and photons. All of these have sufficient energy to cause bond cleavage in the polymer surface. The resulting polymer chain radicals react extremely rapidly with 02.. Chain scission is involved in the formation of many of these groups, leading to a progressive reduction in the average molecular weight and finally to the production of CO, CO2, and H2O. In addition to oxidative degradation, there will also be direct degradation by ion-induced sputtering. These changes can have dramatic effects on the surface energy and functionality of polymer materials. Both dielectric polymer and conductive substrates can be treated with this method as illustrated in Figure 2. With nonconducting polymer films, the grounded roller is covered with a dielectric insulating material and a linear electrode is used. However, with conductive metallic substrates, the process is simply reversed by using a rotating electrode covered with a dielectric insulating material to prevent short-circuiting to ground. In either case, the electrode is always connected to a source of high voltage power, and the roller always remains grounded. However, the corona is a shower of arcs or sparks and each discharge point has the capability of causing localized damage and is difficult to apply consistently on threedimensional components or structures. With corona treatment the effect on many materials is reported to be short-lived. This can represent a problem in some packaging applications where treatment stability is important.



**Figure 2.** Corona discharge treaters with **(a)** segmented electrodes; **(b)** driven electrode rolls.

**Cold-gas-plasma treatment.** This process consists of exposing a polymer to a low-temperature, lowpressure glow discharge (ie, a plasma). The resulting plasma is a partially ionized gas consisting of large concentrations of excited atomic, molecular, ionic, and free-radical species. Excitation of the gas molecules is accomplished by subjecting the gas, which is enclosed in a vacuum chamber, to an electric field, typically at radio frequency (rf). Free electrons gain energy from the imposed rf electric field, colliding with neutral gas molecules and transferring energy, dissociating the molecules to form numerous reactive species. It is the interaction of these excited species with solid surfaces placed in the plasma that results in the chemical and physical modification of the material surface (see Fig. 3).

The effect of a plasma on a given material is determined by the chemistry of the reactions between the surface and the reactive species present in the plasma. At the low exposure energies typically used for surface treatment, the plasma surface interactions only change the surface of the material; the effects are confined to a region only several molecular layers deep and do not change the bulk properties of the substrate. The resulting surface changes depend on the composition of the surface and the gas used. Gases, or mixtures of gases, used for plasma treatment of polymers can include air, nitrogen, argon, oxygen, nitrous oxide, helium, tetrafluoromethane, water vapor, carbon dioxide, methane, or ammonia. Each gas produces a unique plasma composition and results in different surface properties. For example, the surface energy can be increased very quickly and effectively by plasma-induced oxidation, nitration, hydrolyzation, or amination. Depending on the chemistry of the polymer and the source gases, substitution of molecular moieties into the surface can make polymers either wettable or totally nonwettable. The specific type of substituted atoms or groups determines the specific surface potential. For any gas composition, three competing surface processes simultaneously alter the plastic, with the extent of each depending on the chemistry and process variables: ablation, crosslinking, and activation (11). Ablation is similar to an evaporation process. In this process, the bombardment of the polymer surface by energetic particles (ie, free radicals, electrons, and ions) and radiation breaks the covalent bonds of the polymer backbone, resulting in lower-molecular-weight polymer chains. As long molecular components become shorter, the volatile oligomer and monomer byproducts boil off (ablate) and are swept away with the vacuum-pump exhaust. Crosslinking is done with an inert process gas (argon or helium). The bond breaking occurs on the polymer surface, but since there are no free-radical scavengers, it can form a bond with a nearby free radical on a different chain (crosslink). Activation is a process where surface polymer functional groups are replaced with different atoms or chemical groups from the plasma. As with ablation, surface exposure to energetic species abstracts hydrogen or breaks the backbone of the polymer, creating free radicals. In addition, plasma contains very high-energy uv radiation. This uv energy creates additional similar free radicals on the polymer surface. Free radicals, which are thermodynamically unstable, quickly react with the polymer backbone itself or with other free-radical species present at the surface to form stable covalently bonded atoms or more complex groups.



**Figure 3.** Plasma surface-modification mechanism.

Figure 4 illustrates the components of a typical plasma surface-treatment system. In a conventional plasma process, the chamber is evacuated to a specified pressure using a mechanical vacuum pump and gas is introduced into it through flow controllers. Once the gas flow has stabilized and the desired operating pressure has been reached, the rf power is applied to the electrodes and the gas is ionized. A capacitance-matching network tunes the chamber impedance to a constant load. During normal operation, gas is being continually introduced into the chamber and the unreacted species and byproducts are continuously evacuated. The chamber thus operates in a steady state. Cold-gas plasma offers the engineer a means of reengineering the polymer surface and introducing the desired functional groups in a controlled and reproducible manner. The nature of plasma surface modification lends itself to precise control and process repeatability. In a majority of applications, plasma surface treatment employs innocuous gases that allow the engineer or scientist to radically modify the surface while maintaining workplace and environmental cleanliness and safety (12). The treatment effect can be very long-lived and can be applied to a variety of configurations such as webs of plastic films or three dimensional containers.



**Figure 4.** Components of a typical plasma surface-treatment system.

Cold-gas-plasma processes can also be used to apply transparent thin-film silicon oxide-based gasbarrier depositions for flexible-packaging applications. Common polymer packaging films such as polyethylene terephthalate (PET) and biaixially oriented polypropylene (OPP) can be used as substrates for plasma-deposition processes. A common process involves the plasma decomposition of 1,1,3,3 tetramethyldisiloxane (TMDSO) or hexamethyldisiloxane (HMDSO) in a helium and oxygen plasma to form a gas and vapor barrier layer of SiO2 (Fig. 5). The process has been successfully commercialized into production equipment that can accommodate 1.5 meter web widths and run up to 100 m/min. (13). The resulting barrier films can then be processed through typical converting steps to form transparent high-barrier flexible packaging. This approach can improve the shelf life of packaged foods or products and offer an alternative to conventional packaging gas-barrier technologies.



**Figure 5.** Low-pressure SiO2 plasma deposition process.

**Ultraviolet/ozone**. For this process, the polymer surface is exposed to both uv light and ozone to increase the number of oxygen functional groups incorporated into the material. This approach can be useful in the surface modification of three dimensional parts. The process has been used on polypropylene and polyester substrates and has shown rapid and reproducible uptake of surface oxygen functional groups (14). The attachment of oxygen groups greatly changes the surface energy and chemistry, which can lead to improved adhesion of functional and decorative coatings. Most of the initial process development has been targeted at the treatment of three-dimensional plastic components, These are suspended or tumbled inside a reaction chamber at room temperature and pressure and exposed simultaneously to uv (mercury-source) light and various concentrations of ozone (O3) gas. After 5 min. of exposure, oxygen functional groups can be attached to as many as 30% carbon atoms on the outermost surface of polypropylene. The stability of this treatment is usually quite good. For example, relatively little change in the receding contact angle occurs on the treated surface of polypropylene after aging in air for a period of 28 days.

**Evaporated acrylate coating**. This is a relatively new high speed process in which thin highly uniform acrylate coatings are applied to the polymer surface in order to smooth the surface of polymer film substrates so that subsequent depositions are more defect-free. This process involves feeding acrylate monomeric fluids into an ultrasonic atomizer connected to an evaporator in a reaction chamber at reduced pressure. The acrylate material is atomized into a mist that contacts the hot walls of the evaporator, where it is transformed into a gas. The molecular vapor that results exits through a slit in the evaporator nozzle and is condensed on a substrate moving in front of the nozzle. This thin liquid is then irradiated with a low-voltage electron gun and the coating is transformed into a hard, tough thin-film coating. The process is claimed to be compatible with various vacuum coating processes such as sputtering, evaporation, or plasma deposition; and can in principle be conducted in series with other deposition processes within the same reaction vessel. The presence of this coating has been shown (15) to greatly improve the coating uniformity of aluminum metallized barrier depositions on both oriented polypropylene and polyester packaging films. Consequently, the oxygen- and water-vapor barrier properties are improved. This can have an impact in numerous applications for extending the shelf life of packaged products, The improvement in barrier can be attributed to three factors:

1. The coating forms a smooth layer on the polymer surface, eliminating any surface irregularities.

2. The coating has very good temperature stability which provides a thermally stable platform on which to apply a barrier material.

3. The acrylate surface is more chemically polar than many polymer films, and the density of the resulting film is higher. Barrier layers composed of metals and inorganic oxides tend to grow more readily on a polar substrate than on a nonpolar substrate.

**Fluorination treatment.** The fluorination process involves exposing polymeric webs continuously to fluorine gas (F2) diluted with an inert gas (eg, nitrogen) inside a reaction chamber (16). This process can greatly increase the surface energy of polymer materials such that excellent adherence can be attained to other materials such as lacquers and adhesive agents. Diatomic fluorine, an almost colorless gas, is one of the strongest oxidizing agents; it reacts with almost all organic and inorganic substances (except nitrogen and other inert gases). Fluorine's great reactivity is due to the interaction of the low dissociation energy of the molecule and the very strong bonds it forms with other atoms. Electron spectroscopy for chemical analysis (ESCA) data indicate that the activation of polymer surfaces using this process results from the partial fluorination of the hydrocarbon structure of the polymer molecules. An additional application would be the fluorination of high-density polyethylene gasoline tanks to provide hydrocarbon barrier. Fluorine is routinely transported in its liquid state and is commercially available because of use in the nuclear industry for the refinement of fuels. The safeguards used for this technology are similar to safety measures used and approved for ozone generation. Compared with other pretreatment processes, surface fluorination not only has a wide spectrum of applications but also doesn't require the use of electrical equipment such as corona or plasma treatment. Surfaces treated with fluorine exhibit long-lasting, if not irreversible, changes. This can be very important in practical applications in industry, since subsequent converting processes don't have to immediately follow the surface activation.

**Electrostatic discharge treatment**. Plastic, as opposed to metal, substrates make good electrical insulators because they are electrically nonconductive and possess high electrical resistivity. The higher the surface resistivity, the lower the surface conductivity. However, those plastic insulating materials that have high dielectric constants can generate and store static electricity. Static electricity is generated when two materials in intimate contact are separated by a frictional force causing electrons to be preferentially stripped from one surface and transferred to the other surface. This causes the electronrich and electron-deficient surfaces to assume positive and negative charges and this surface polarization results in the generation of static electricity. Unless this charge is dissipated, the static buildup can cause the attraction of dust, lint, sparks, materials-handling problems, shocks, and difficulty in wetting or adhering.

Packaging substrates made from polyethylene, polypropylene, polyester, polystyrene, and other dielectric materials at some time during their manufacture are usually subjected to at least one of the many available static control techniques. These fall into two separate categories: charge dissipation and charge neutralization. With electrically conductive materials, dissipation of static charge can he accomplished by simply grounding the charged material. However, this is difficult with nonconducting materials such as polymer films, so one approach is to humidify the work area so that the exposed surface absorbs a thin layer of water that conducts the charge to ground. An alternative method is to shield the surface with antistatic organic compounds. Most antistatic agents fall under the following types: nonionic ethoxylated alkylamine, anionic aliphatic sulfonate/phosphates, and cationic quaternary ammonium compounds (17). Antistats can be applied topically or blended, and their purpose is to retard static buildup and also to rapidly discharge any accumulated charge.

Another approach to static elimination is to neutralize the accumulated charge using devices capable of ionizing the surrounding air. This works by exposing electrically neutral atoms in air to an applied electric field of voltage high enough to create positively and negatively charged ions. Because of the bipolar nature of the ionized air, the static charge on a material can be neutralized by the oppositely charged ions present in the surrounding air. Basically, there are three types of air-ionizing devices available: nonpowered, powered, and self-powered. The nonpowered induction type of static eliminator consists of brass brushes mounted on ground straps that come in light contact with the charged material, causing the surrounding air to ionize. Electrically powered static eliminators are powered with a lowamperage high voltage power supply for the ionization of the air. Radioactive self-powered units are similar to electrical static eliminators in design and construction except for the source of power. Radioactive devices are self-propagating, usually consisting of a low-energy source of an alpha-emitting radioisotope such as polonium-210 (210Po). The alpha radiation interacts spontaneously with the air molecules, producing ionization of the surrounding environment.

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