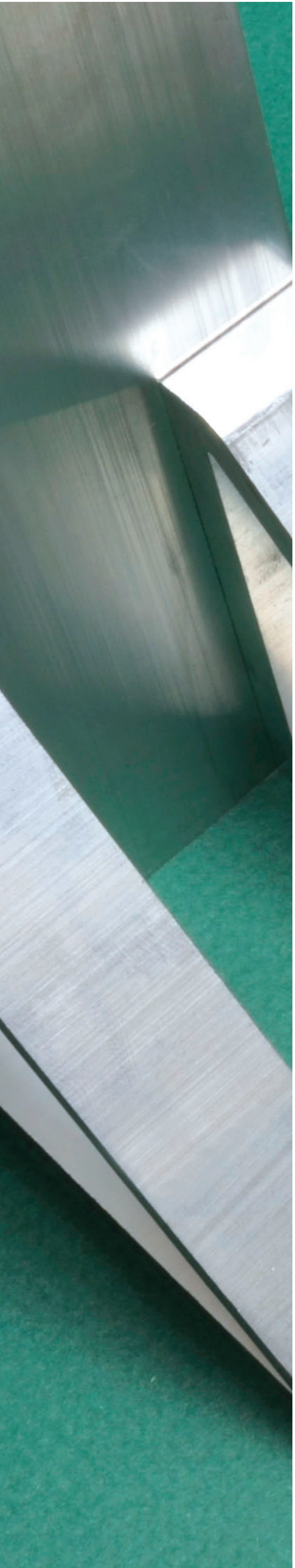

MASTERBOND®
TECH SPOTLIGHT

Achieving Better Adhesion with Proper Surface Preparation

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Achieving Better Adhesion with Proper Surface Preparation

Achieving good adhesion not only depends on selecting the right adhesive, but also on implementing the best surface preparation techniques. In this context, the term "surface" refers to the specific area and depth where the adhesive interacts with the substrate. The affinity of the adhesive with this surface ultimately determines the strength of the bond. Whether you are bonding metals, plastics or rubbers, proper surface preparation always plays an essential role in achieving good adhesion.

Adhesion can occur either mechanically, chemically or in some cases with a combination of each. A mechanical bond, in large part is determined by the area of the substrate that comes in contact with the adhesive. Basically, roughening up the surfaces would not only remove any pre-existing deposits, but more importantly provide a larger surface area, ultimately resulting in better bond strength. Alternatively, the adhesive could attach to the substrate chemically. Typical forces acting between the substrate and the adhesive include ionic, static, polar and van der Waals forces. To obtain optimum bonding in this scenario, the surfaces must be free of all dirt, oils and other contaminants.

A bond can fail either cohesively or adhesively. A cohesive failure (i.e. breakdown of the adhesive itself) typically indicates inappropriate adhesive selection. Alternatively, an adhesive failure (i.e. bond breakage between the adhesive and the substrate) is more difficult to assess. Essentially, it could be due to inadequate surface preparation, improper choice of adhesive, or a substrate that is inherently refractory to adhesive bonding (e.g. gold). In a practical sense, if an adhesive is properly chosen and if the substrate is receptive to bonding, a bond failure would be invariably due to insufficient surface preparation.

Effective wetting of the surface by the adhesive is equally paramount. Therefore, one of the goals of surface preparation is to increase the surface energy of the substrate to be bonded. Higher surface energy substrates allow for easier bonding as compared to lower surface energy substrates because of the wettability factor. Hence, good surface preparation is vital in order to provide easily wettable and cohesively strong surfaces with improved bond strength.

Surface Preparation Techniques

Degreasing

Degreasing is one of the principal steps in surface preparation. Degreasing is carried out in order to remove any loosely held dirt or other contaminants from the surface. Often times, surfaces are degreased using solvents such as acetone and methyl ethyl ketone. Although, depending on the nature of the substrate involved, alternative solvents such as isopropyl alcohol can also be used for degreasing. It is important to make sure that all the environmental, health and safety regulations are met prior to selecting a solvent. The most common methods of degreasing, typically include these two main steps:

1. Depending on the degree of contamination, either vapor degrease, wipe clean or rinse the parts repeatedly, using the appropriate solvent, until the surface is free of all contaminants.

2. Clean the substrates and let them dry completely in order to remove any residual solvent from the surface.

Abrasion

After degreasing, mechanical abrasion is needed in order to remove heavy loose surface deposits such as dirt, oxide layers or any other contaminants that might be deposited on the surface. A vital purpose for mechanical abrasion is to increases the surface area for bonding. Some of the most effective mechanical methods used include sandblasting, wire brushing or abrasion with sandpaper or emery cloth. The surfaces must be cleaned both before and after abrasion in order to remove any pre-existing contaminants on the surface.

An RMS of 150 (micro-inches) to 250 (micro-inches) is generally recommended for metals. It is advisable to get the parts machined to the proper roughness as early as possible in the manufacturing stage. It is very risky to rely solely on observation to determine whether the surfaces are adequately roughened. It should be noted that the abrasion technique eventually employed might vary depending on the specific plastic, rubber or metal being used. Depending on the material that is ultimately used, caution must always be exercised as some techniques may pose certain health hazards, such as roughening beryllium.

How to Optimize Bond Strength

Good adhesion starts with proper surface preparation. Methods include:

Abrasion

Increases surface area for bonding by the use of sand blasting, wire brushing or sand paper

Chemical

Chemical etching changes the surface's physical/chemical properties to improve adhesion

Degreasing

Removes any contaminants or dirt from surface, often uses solvents

Physical

Common methods include flame treatment, corona discharge and plasma treatment

Desired Results

- Effective wettability
- Removal of dirt, deposits, contaminants
- Increased surface energy
- Greater surface area
- Improved bond strength

RUBBERS

Nitrile Elastomers • Santoprene®
Neoprene • Natural rubber

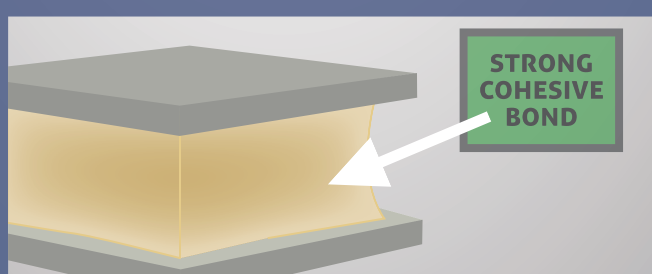
METALS

Aluminum • Cast Iron
Stainless Steel • Titanium

PLASTICS

Acrylics • Fiberglass
Kevlar® • Nylon • PEEK

Final Result



Chemical treatment

An alternative to mechanical treatment is chemical etching. Specific chemical techniques have been developed for treating different substrates. These treatments change the physical and/or chemical properties of the surface in order to improve the adhesion. A wide array of various acids, bases and solvents are used for chemical etching. Table 1, Table 2 and Table 3 list the chemical treatments typically used for some commonly used metal, plastic and rubbers. Each substrate has its own recipe to optimize its bonding capabilities. These recipes and methodologies should be strictly followed in order to optimize adhesion and minimize health related hazards. Obviously, good laboratory skills and extreme care should be taken while handling any chemicals. Wearing appropriate protective equipment and being well-trained in handling chemicals is always recommended. Also, the appropriate environmental, health and safety regulations must be met while handling these chemicals.

Physical methods

Often times--particularly with plastic materials--chemical treatment and mechanical abrasion are simply not sufficiently effective. Other techniques, referred to as "physical methods" can be used to change the surface reactivity of the plastics and modify the surface chemistry to achieve better adhesion. Some of the most common physical methods include:

1. Flame Treatment: Flame treatment involves exposing the surface to be bonded to a gas flame for a few seconds. The flame oxidizes the surface and increases the surface energy by forming higher surface energy functional groups. Warping might be a potential hindrance during processing with this method.

2. Corona Discharge: The corona discharge generated by ionizing the air between two closely spaced electrodes, reacts with the surface of the substrate to form free radicals. These free radicals quickly react with oxygen

Table 1: Surface treatment for metal substrates

Substrate	Etching Solution	Composition (Wt%)	Pretreatment Conditions
Aluminum	DI Water Sulfuric Acid (96%) Sodium Dichromate	68.83 24.51 6.66	Immersion: 20 min. @ 25°C Rinse: Tap water followed by DI water Oven Dry: ~30 min. @ 70°C
Beryllium	Sodium hydroxide Distilled Water	20 80	Immersion: 3 - 4 min. @ 79°C - 85°C Rinse: Tap water followed by DI water Oven Dry: ~30 min. @ 70°C
Copper	Nitric Acid (69%) Ferric Chloride Distilled Water	12.4 6.2 81.4	Immersion: 1 - 2 min. @ 21°C - 32°C Rinse: Tap water followed by DI water Oven Dry: ~30 min. @ 65°C Max
Magnesium	Chromic Acid Distilled Water	20 80	Immersion: 10 min. @ 71°C - 88°C Rinse: Tap water followed by DI water Oven Dry: ~30 min. @ <60°C
Nickel	Nitric Acid (69%)	100	Immersion: 5 sec. @ 20°C Rinse: Tap water followed by DI water Oven Dry: ~60 min. @ 40°C
Stainless Steel	Nitric Acid (69%) Distilled Water	20 80	Immersion: 25 - 35 min. @ 21°C - 32°C Rinse: Tap water followed by DI water Oven Dry: ~30 min. @ 65°C Max
Steel (mild)	Ethyl alcohol (denatured) Orthophosphoric Acid (85%)	66.7 33.3	Immersion: 10 min. @ 60°C Rinse: Tap water followed by DI water Oven Dry: ~60 min. @ 120°C
Titanium	Nitric Acid (69%) Hydrofluoric Acid (60%) Distilled Water	28.8 3.4 67.8	Immersion: 10 - 15 min. @ 38°C - 52°C Rinse: Tap water followed by DI water Use a nylon brush to brush off residual carbon while rinsing Oven Dry: ~15 min. @ 70°C - 80°C
Zinc	Hydrochloric Acid (37%) Distilled Water	20 80	Immersion: 2 - 4 min. @ 20°C Rinse: Tap water followed by DI water Oven Dry: ~30 min. @ 66°C - 70°C

in the atmosphere and increase the surface energy of the substrate to be bonded. This treated substrate with higher surface energy allows the surface to be easily wet by the adhesive. Corona treatment is the most popular method of choice for polyolefins and polyolefinic type materials.

3. Plasma Treatment: Plasma treatment differs from corona discharge and flame treatments because plasma treatment is typically carried out under partial vacuum. In plasma treatment, gas plasma is activated by the appropriate techniques to produce a highly excited, ionized gas that reacts with the plastic substrate.

Often plasma treatment tends to provide substrates with better stability as compared to corona discharge, chemical treatment or flame treatment.

With the exact substrate and treatment employed, the effective time period for adequate bonding is limited and very much dependent on the technique. It is critical to determine the shelf life for good bonding i.e. the window of opportunity for bonding, before the treatment loses its effectiveness. For example, plasma treatment of certain plastics might be effective only for a few hours. Hence, it might be a good processing procedure to treat the parts in line.

Table 2: Surface treatment for plastic substrates

Substrate	Etching Solution	Composition (Wt%)	Pretreatment Conditions
Polyphenylene Sulfide (PPS)	Conc. Chromic Acid	100	Immersion: 2 min. @ 71°C Rinse: Tap water followed by DI water Dry by evaporation
Acetal (Delrin)	Conc. Sulfuric Acid (69%) Sodium dichromate Distilled Water	84.2 0.6 15.2	Immersion: 5 min. @ Room Temperature Rinse: Clean cold water followed by clean hot water Dry in warm air
Nylon	Aq. Phenol (80%)	100	Immersion: 15 – 20 min. @ 70°C - 90°C Rinse: Tap water followed by DI water Air dry @ 60°C - 70°C
Acrylonitrile Butadiene Styrene (ABS)	Conc. Sulfuric Acid (69%) Sodium dichromate Distilled Water	84.2 0.6 15.2	Immersion: 20 min. @ 60°C Rinse: Tap water followed by DI water Dry in warm air
Polytetra-fluoroethylene (PTFE)	Tetrahydrofuran Sodium Naphthalate	85.5 12.3 2.2	Immersion: 1 - 2 min. @ 70°C - 90°C Rinse: Ketone followed by DI water Dry in warm air @ 65°C
Polyethylene (PE) or Polypropylene (PP)	Potassium dichromate* Conc. Sulfuric Acid (69%) DI Water - OR - Plasma or Corona treatment Alternatively, use X17 primer	4.42 88.50 7.08	Immersion: 60 - 90 min. @ Room Tempera-ture/ 30 – 60 sec. @ 71°C Rinse: Tap water for at least 3 min. Air circulating Oven Dry: ~1 hour @ 38°C
Polystyrene	Plasma or Corona treatment	—	—

*Dissolve the potassium dichromate in the clean tap water, and then add the sulfuric acid in increments of about 200g, stirring after each addition.

Evaluating the treated substrate

Once the surfaces have been treated, it is important to know how effectively the surface treatment was carried out. The treated substrates can be evaluated for the effectiveness of the treatment in the following ways:

1. **Destructive Test:**

Mechanical strength tests such as tensile test or lap shear test can be performed to evaluate the mode and point of failure. Surface preparation is carried out in order to ensure that the failure occurs in the bulk of the adhesive layer and not at the interface between the adhesive and the adherend. If the failure occurs only cohesively, then we can conclude that good surface preparation techniques were employed. However, if the failure occurs adhesively, the surface preparation technique carried out must be re-evaluated.

2. **Non-destructive Tests:**

Water break test: Spray or coat a thin uniform film of deionized (DI) water on the treated substrate. If there is a break in the film, it may indicate the presence of contaminants on the surface. This technique does not provide any quantitative analysis of treated surface.

Contact angle test: For quantitative analysis, you can measure the contact angle between the surface and a drop of reference liquid.

UV light detection: Another way of detecting contamination on the surface involves coating the surface using fluorescent oil and observing the surface for any other contaminants under a UV light.

What is the right technique for your application?

As mentioned throughout this paper, surface preparation is critical for good adhesion. There are a fairly wide number of techniques that can be used to prepare substrates. Often times, a particular surface preparation technique is dictated by the nature of the substrate. Other times, the choice of surface preparation might depend on safety issues. Ultimately, one must consider all the approaches and choose the appropriate technique based on effectiveness, cost, safety and the shelf life of the substrate being treated.

For further information on this article, for answers to any adhesive application questions, or for information on any Master Bond products, please contact our technical experts at Tel: +1 (201) 343-8983.

Table 3: Surface treatment for Rubber substrates

Substrate	Solvent Cleaning[1]	Bleach based Solution[2]	Sulfuric Acid Solution[3]
Butyl	E	E	—
Ethylene Propylene diene monomer (EPDM)	E	G	G
Natural	G	E	—
Neoprene	—	—	E
Nitrile	E	E	—
Styrene-butadiene	—	—	G
	(E) Excellent	(G) Good	(—) Not Recommended

1. Solvent Cleaning: Degrease the rubber substrate as mentioned above using trichloroethylene solvent.
2. Bleach Based Solution: Prepare the modified bleach solution by pouring cold deionized (DI) water (1 liter) into a clean container made of plastics, glass or similar inert ware. While stirring the water, add concentrated hydrochloric acid (5 ml) in a slow steady stream. Then add household bleach (30 ml), stirring it thoroughly into the diluted acid. (Never mix the household bleach directly with the concentrated acid. Make sure that the acid is diluted. Fresh solution should be made up each day. The solution gives off chlorine: good ventilation is essential.) Immerse the rubber substrate in modified bleach solution for 1-3 minutes at room temperature. Following that, rinse it in cold DI water, followed by a rinse in hot DI water. Finally dry the substrate in hot air.
3. Sulfuric Acid Solution: Immerse the rubber substrate in concentrated sulfuric acid for 2-10 minutes. Following that, rinse it in cold DI water, followed by a rinse in hot DI water. Finally dry the substrate in hot air.