3M Surface Preparation and Pretreatment for Structural Adhesives

chnical Bulletin

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Working directions for advanced surface preparation techniques to obtain optimum adhesion between materials with 3M[™] Scotch-Weld[™] Structural Adhesives.

Structural adhesives depend on the interfacial relationship of the adhesive and the substrate. To optimize the performance the substrate surface preparation is critical. Bonds of high strength on metals, plastics, etc. can be obtained after removal of grease and loose surface deposits, e.g. rust, from the surfaces to be joined, but when maximum strength is required, a more thorough mechanical or a chemical pretreatment is recommended.

Surfaces are prepared by one of the following procedures (listed in order of increasing effectiveness):

- 1. Degrease only.
- 2. Degrease, abrade, and solvent clean.
- 3. Degrease and chemically pre-treat.

After cleaning, care must be taken to avoid contaminating the pretreated surfaces prior to bonding. Contamination may be caused by finger marking, or by cloths which are not perfectly clean, or by using sub-standard degreasing or chemical solutions. Whatever the pretreatment procedure used, it is good practice to bond the surfaces as soon as possible after completion of the pretreatment when surface properties are at their best.

This bulletin will offer suggestions for surface preparation of the most commonly bonded materials. Materials less commonly used and not specifically dealt with in this manual may require only simple degreasing and abrading (as described below) but if other pretreatments appear necessary advice should be obtained from 3M.

While the advice in this manual is intended to cover most situations, you should verify with testing that suggested pretreatments are effective with your specific substrates.

Degreasing The removal of all traces of oil and grease from the surfaces to be bonded is essential for the highest strength, most durable bonds. Degreasing by one of the three methods given below should be carried out even when the surfaces to be bonded appear clean.

(1) Suspend in 3M[™] Novec[™] 72DE Engineered Fluid or 3M[™] Novec[™] 72DA Engineered Fluid (or equivalent vapor degreasing product), (2) Wipe the joint surfaces with a clean cloth soaked in 3M[™] Novec[™] Electronic Degreaser, MEK, acetone or isopropyl alcohol (IPA)* and allow to stand for a minute or two to permit complete evaporation from the joint surfaces. (3) Or scrub the joint surfaces in a solution of detergent or, for metals only, immerse or spray in a suitable alkaline degreasing agent.

After degreasing, wash with clean hot water and allow to dry thoroughly - preferably in a stream of hot air. Ultrasonic degreasing has been found to give excellent results when pretreating very small components with 3M[™] Novec[™] 72DE Engineered Fluid, 3M[™] Novec[™] 72DA Engineered Fluid, and detergent-based degreasing solutions. Alcohol, gasoline, and paint thinners are not effective in removing grease, or may leave a residue.

To find out whether a surface has been properly degreased, drip distilled water onto it. If the water forms a film, the surface is free from grease. If it forms drops, the surface will have to be degreased again. Note: Not reliable on anodized light alloys as water will sometimes form a film on their surfaces even though not properly degreased.

- *Note: When using solvents, extinguish all ignition sources and follow the manufacturer's precautions and directions for use when handling such materials.
- BondingCertain Structural Adhesives, such as 3M™ Scotch-Weld™ Epoxy AdhesivethroughDP920 and 3M™ Scotch-Weld™ Acrylic Adhesives, are designed to bondOilsthrough many oils on the surface of metals, thus allowing protective oils to
remain in place and/or reducing the effort needed for surface preparation prior
to bonding. Results will vary depending on the oil used; testing should be
performed to confirm bond strength. In general, even with these adhesives,
stronger bonds are usually seen if the surfaces are properly prepared as outlined
in this bulletin.

Abrading Lightly abraded surfaces give a better profiled surface for adhesive bonding than do highly polished surfaces. Properly abraded surfaces show no smooth, polished areas.

Abrasion treatment should always be followed by a solvent cleaning to ensure the removal of loose particles.

Metal surfaces are freed from surface deposits, e.g. tarnish, rust or mill scale, by grit-blasting. If grit-blasting equipment is not available or the metal is too thin to

withstand blast treatment, clean the joint surfaces with a wire brush, emery cloth or glass-paper. (Use grade 120-220 abrasives for steel and materials resistant to scoring, but grade 300 abrasive for light alloys and less resistant materials, and very high viscosity adhesives.) ScotchBrite[™] 7447 pads can be effective in many situations.

Painted surfaces May need to be stripped with a paint stripper prior to preparation; otherwise the strength of the joint may be limited by the comparatively low adhesion of paint to metal. Adhesives will generally adhere well to most paints; however, adhesion of paint to underlying metal may lead to lower joint strengths than desired. If so, removal of the paint is necessary.

ChemicalThe surface preparation described above, i.e. degreasing alone or degreasingPre-followed by abrasion and further degreasing, is sufficient for most adhesivetreatmentwork.

But to obtain maximum strength, reproducibility and resistance to deterioration, a chemical or electrolytic pretreatment is required. Care must be taken in the preparation of the chemical solution, not only because of the materials involved, but also because

incorrect proportioning may lead to bond strengths inferior to those that would have been obtained if there had been no chemical pretreatment whatsoever.

Time of application is also critical: too short an application does not sufficiently activate the surfaces, whereas overlong application builds up a layer of chemical reaction products which may interfere with adhesion. Both surface structure and chemistry play a significant role in determining the strength and permanence of bonded structures. It is therefore advisable to bond or prime freshly primed clean surfaces as soon as possible after surface preparation in order to avoid contamination and/or mechanical damage. Please contact your 3M sales representative for primer recommendations.

MetalsMetals not specifically listed below should be prepared by degreasing, abrading
(preferably grit blasting with a fine grit abrasive) and solvent clean.

<u>Aluminum</u>

Bare aluminum requires removal of all oxide for environmentally durable bonds. Etching is the best way to do this; a suggested etchant procedure is as follows.

Optimized FPL Etch Procedure:

Degreasing using an Oakite 164 solution (9-11 oz./gallon water) at 190°F \pm 10°F (88°C \pm 5°C) for 10-20 minutes. Rinse immediately in large quantities of cold running water.

| Material | Amount |
|----------------------------|-----------------------------------|
| Distilled Water | 700 mL plus balance of liter (see |
| | below) |
| Sodium Dichromate | 28 to 67.3 grams |
| Concentrated Sulfuric Acid | 287.9 to 310.0 grams |
| Aluminum Chips | 1.5 g/L of solution |

To prepare 1 liter of this solution, dissolve sodium dichromate in 700 ml of distilled water. Add sulfuric acid and mix well. Add additional distilled water to fill to 1 liter. Heat mixed solution to 66 to 71°C (150 to 160°F). Dissolve 1.5 grams of 2024 bare aluminum chips per liter of mixed solution. Gentle agitation will help aluminum dissolve in about 24 hours. To FPL etch panels; place them in the above solution at 150 to 160°F (66 to 71°C) for 12 to 15 minutes.

Rinse immediately in large quantities of clear running tap water.

Dry – air dry approximately 15 minutes followed by force dry at 140°F (60° C) maximum for 10 minutes (minimum).

Anodized Aluminum: Degrease thoroughly. Adhesion will depend on the thickness and structure of the oxide layer as well as on the type of pore sealer used. For bonds of maximum strength, it may often be necessary to abrade or etch the surfaces to be bonded.

Copper and Copper Alloys (excluding Brass)

If degreasing and abrasion is not sufficient, etching following degreasing can be performed with the following procedure:

| Material | Amount |
|-------------------------------|--------|
| Ferric Chloride, 42% solution | 3.75 L |
| Concentrated Nitric Acid | 7.5 L |
| Water | 50 L |

Immerse for 1-2 minutes at room temperature, wash with plenty of clean cold water and dry promptly with a room temperature forced air system.

Galvanized Steel

In many cases abrasion of galvanized steel is not advisable. However, degreasing or removing any organic coatings may be required.

Galvanized steel may require etching or caustic cleaning. Etch procedure:

Degrease. Etch in a solution of 15 parts concentrated Hydrochloric acid and 85 parts water (by volume). Immerse 2-4 minutes at room temperature, rinse with clean hot water, followed by clean cold water, and dry thoroughly in an oven (60°C-70°C) or with hot forced air.

Some galvanized metals respond to a wipe with household bleach after degreasing.

Magnesium and its Alloys

Optional Etching procedure:

Immerse for 5 minutes at 70°C-75°C in a solution of 6.2 kg of Caustic soda in 50 Liters of water. Wash with clean running water, then etch in the following solution:

| Material | Amount |
|----------------------------------|--------|
| Chromic Acid (CrO ₃) | 5.0 kg |
| Sodium Sulfate (anhydrous) | 31 g |
| Water | 50 L |

Wash with clean cold water, followed by clean hot water, and dry with hot air.

<u>Nickel</u>

Optional Etching procedure: Etch for 5 seconds in concentrated Nitric Acid. Wash with clean cold water, followed by clean hot water, and dry with hot air.

Alternatively, 3M[™] Surface Pre-Treatment AC-130-2 can be used to improve adhesion.

Stainless Steel

Optional Etch solution:

| Material | Amount |
|----------------------------|---------|
| Oxalic Acid | 14.0 kg |
| Concentrated Sulfuric Acid | 12.2 kg |
| Water | 70 L |

Immerse in etchant solution for 10 minutes at 85°C-90°C, remove from the solution and, under clean running water, and brush off the black deposit with a clean stiff brush. Dry with hot air. Bond as soon as possible.

Alternatively, 3M[™] Surface Pre-Treatment AC-130-2 can be used to improve adhesion

Tungsten and Tungsten Carbide

Optional Etch Solution:

| Material | Amount |
|------------------|--------|
| Sodium Hydroxide | 8.5 kg |
| Water | 20 L |

Immerse for 10 minutes at 80°C-90°C. Wash with clean cold water, followed by clean hot water, and dry with hot air.

Wrought Iron and Mild Steel

Optional etch solution:

| Material | Amount |
|-------------------------|--------|
| o-Phosphoric Acid (88%) | 10 L |
| Denatured Alcohol | 5 L |

Immerse for 10 minutes at 60°C. Remove from the solution and, under clean running water, brush off the black deposit with a clean stiff brush. Dry with hot air. Bond as

soon as possible, before rust can form.

Wash with clean cold water, followed by clean hot water, and dry with hot air.

Zinc and Zinc Alloys

May be treated like Galvanized Steel.

Ceramics, May benefit from priming with 3M[™] Scotch-Weld[™] Metal Primer 3901, unless using
Glass and an adhesive that contains a silane coupling agent.
Stone

Ceramics

Smooth surfaces: Degrease. Abrade with carborundum + water slurry, dry and degrease again.

Glazed surfaces: Remove glaze by grit-blasting or with emery cloth, and degrease. Earthenware: Dry thoroughly, clean with wire brush, and remove all loose particles.

<u>Concrete</u>

Remove heavy grime and laitance by wire-brushing. Degrease with a proprietary detergent solution. Where concrete is deteriorated and weak, the surface must be removed until sound concrete is exposed.

Even where concrete is sound, it should be pretreated wherever practicable by one of the following methods.

Method 1 is more effective than 2, and 2 is more effective than 3.

1. Remove by mechanical scarification 3 mm (1/8 inch) - or more -of all surfaces to be bonded, then remove dust, preferably by vacuum-cleaner.

Or

2. Sand-blast about 1.5 mm (1/16 inch) off all surfaces to be bonded, then remove dust, preferably by vacuum-cleaner.

Or

3. Etch with 15% hydrochloric acid solution* (1 litre per square metre, spread by stiffbristle brooms) until bubbling subsides (about 15 minutes). Wash with clean water by high-pressure hose until all slush is removed and the surface is neutral to litmus. Final rinsing with 1% ammonia solution followed by clean water is good practice - this

ensures thorough neutralization. Allow the surface to dry thoroughly. Remove dust, preferably by vacuum-cleaner.

Glass and Silica

Degrease thoroughly. Better still, grit-blast until matt or abrade with carborundurn + water slurry, dry and degrease. Then apply a silane primer (e.g. 3M[™] Scotch-Weld[™] Metal Primer 3901).

Precious Stones

Degrease.

| Plastics and | Pretreatment of Plastics and Composites |
|--------------|---|
|--------------|---|

Composites

Thermoset Resins: Epoxy and carbon fiber composites, laminates, etc. can usually be bonded without difficulty. To ensure good bond strength, all soil and residual release agent must be removed from the joint surfaces before the adhesive is applied. The surfaces must either be abraded with emery cloth or grit-blasted, or they must be cleaned with a solvent such as acetone, MEK, etc. Abrading or grit-blasting is recommended for glossy composite surfaces since they may otherwise repel the adhesive.

Thermoplastics: These are often difficult to bond. Certain types permit only moderately successful bonding, 3M[™] Scotch-Weld[™] Acrylic Adhesives are specifically designed to bond thermoplastic materials as well as metals. In many cases very high bond strengths can be obtained with these adhesives without the need for surface preparation beyond cleaning.

Degreasing
and
AbradingPlastics in general should be abraded and solvent cleaned. However, some plastics can
be damaged by solvents. General recommendations are as follows:

| ABS | Degrease or remove mold |
|--------------------------------|---------------------------------|
| Polycarbonates (e.g. Makrolon® | release with Isopropanol. |
| and Lexan®). | Abrade with ScotchBrite™ |
| PMMA/acrylic (e.g. Plexiglass® | 7447 or grit blast. Remove dust |
| and Perspex®) | with Isopropanol. |

| Polystyrene | |
|--|--|
| Polyvinylchloride (rigid) | |
| Acetal resins (e.g. Delrin®) | Degrease or remove mold |
| Epoxy resins and composites | release with Acetone. Abrade with ScotchBrite™ 7447 or |
| Urea resins | grit-blast. Remove dust with |
| Polyamides (e.g. Nylon, Ultramid® etc.) | Isopropanol |
| Polyester resins | |
| Polyphenylene oxide | |
| Polyurethanes | |
| Chlorinated Polyethers | Remove mold release with |
| Thermoplastic Polyolefins (TPO) | Isopropanol. Do not abrade. See further instructions below. |
| Polyethylene | |
| Polypropylene | |

Additional
SurfacePretreatment of plastics by the following methods has been found to give good
to excellent bonds. However, since the grade of plastic and the manufacturing
process used to make the component may affect the optimum application time
of the chemical pretreatment, it would be advisable to establish by trial whether
the specified time needs to be adjusted.

Acetal (e.g. Delrin®).

Degrease and etch for about 20 seconds in a solution of:

| Material | Amount |
|----------------------------------|----------|
| Concentrated sulfuric acid (s.g. | 3.0 L |
| 1.82) | |
| Potassium Dichromate | 0.150 kg |
| Water | 0.24 L |

Wash with clean water and dry.

Polyamide (e.g. Nylon, Ultramid®)

Flame treatment of a cleaned surface may be beneficial for bonding.

Polyolefins (e.g. HDPE, UHMW, PP)

Polyolefins can be structurally bonded without additional pretreatment when using 3M[™] Scotch-Weld[™] Structural Plastic Adhesive DP8010 or 3M[™] Scotch-Weld[™] Structural Plastic Adhesive DP8005.

Alternatively, the following procedure may be used with other adhesives:

Etch inn a solution of:

| Material Concentrated sulfuric aci 1.82) | d (s.g. | Amount 3.0 L |
|--|-------------|-----------------|
| Potassium Dichromate | | 0.25 kg |
| Water | | 0.15 L |
| Plastic | Immerse for | At Temperature |
| Chlorinated Polyethers | About 5 min | 70°C |
| Polyethylene | 10-15 min | 25°C |
| Polyphenylene oxide | 5-15 sec | 70°C |
| Polypropylene | 1- 2 min | 70°C |

After immersion, wash surfaces with clean cold water and dry carefully. Alternatively, burn off the joint surfaces by moving them through a blue, not yellow acetylene flame until they appear smooth and polished. Do not overheat or melt the plastic.

Alternatively, plasma or corona treatment can be used.

PTFE and other Fluoropolymers

Degrease and etch with a commercial flouropolymer etchant such as Fluoro-Etch® or TetraEtch®.

Or, abrade with a coarse abrasive (e.g. 80 grit) and bond with 3M[™] Scotch-Weld[™] DP8010 Plastic Adhesive.

Rubbers Thorough roughening and degreasing will sometimes suffice, but in most cases the following procedure will be necessary: Use a solvent-containing isocyanate primer OR the following etching procedure:

Rubber, Natural

Treat the surfaces for 2-10 minutes with concentrated sulfuric acid, wash with clean cold water, followed by clean hot water, and dry. Flex the rubber - the appearance of minute hair-line cracks on the surface indicates that it is ready for bonding. (The time of treatment with the acid will depend on the grade of rubber.)

Vertical surfaces may be treated with a paste prepared by adding barytes powder to the concentrated sulfuric acid until it ceases to flow.

Rubber, Synthetic

Treat with concentrated sulfuric acid as for natural rubber. (The time required may be longer.) If the surfaces feel very smooth or greasy, roughen them before treating with acid. If minute surface cracks fail to appear on flexing, continue treatment using concentrated nitric acid until the cracks do appear. Wash with clean cold water, followed by clean hot water, and dry.

Silicone rubber and a few other synthetic rubbers cannot be bonded with Epoxy adhesives.

Wood Smooth with fine-grain sandpaper. Ensure the wood is dry (moisture content not higher than 8-12%). Wood free of grease requires no additional pretreatment.

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