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Specialty Adhesives

Rubber-to-Substrate Bonding Technical Guide Substrates Preparation



Preparing Substrates for the Application of MEGUM[™] and THIXON[™] Bonding Agents

Substrate Pre-treatment

MEGUM[™] and THIXON[™] bonding agents are used to bond natural and synthetic rubbers to a variety of rigid, semi-rigid, and flexible substrates.

Cleaning and preparing the substrate surface before the application of MEGUM[™] or THIXON[™] bonding agents is a very important part of the bonding process. It is impossible to achieve consistent bonding performance between the substrate, the MEGUM[™] or THIXON[™] bonding agent, and the rubber unless the substrate is both physically and chemically clean.

Once the substrate has been cleaned, it must be protected from physical and chemical re-contamination before application of the MEGUM[™] or THIXON[™] bonding agent.

The purpose of the substrate preparation process is to:

- Provide and maintain a clean, stable substrate bonding surface before the application of two-coat MEGUM[™] or THIXON[™] bonding agent primer and cover-cement or MEGUM[™] or THIXON[™] single-coat bonding agent systems
- Remove oil and grease that could prevent the MEGUM™ or THIXON™ bonding agent from properly wetting the substrate surface
- Remove contamination, such as oxidative rust, which may fracture and cause bond delamination during the in-service application of the bonded component
- Prevent re-oxidation or re-contamination of the freshly prepared substrate bonding surface

Substrates Used for Bonding

Steel is the most commonly used substrate for rubber-to-metal bonding. Other materials include stainless steel, aluminum, brass, plated metals, and alloys. MEGUM™ and THIXON™ bonding agents are also used to bond rubbers to plastics including glass-reinforced nylons, and phenolic and epoxy resins. Other specialized bonding substrates include natural and synthetic fibers and fabrics, etched PTFE, glass and ceramics, and vulcanized and unvulcanized rubbers.

Choice of Preparation Method

Substrate preparation methods can be either mechanical or chemical, or a combination of methods. The choice between using mechanical or chemical methods is influenced by considering a number of factors. These include substrate composition, size, number, and configuration of components, and the in-service application of the bonded component. The resistance of the bonded component to adverse environments and to under-bond corrosion can be affected by the substrate pre-treatment process used.

Steel substrates that have been phosphated before MEGUM™ or THIXON™ bonding agent application, using recommended pre-treatment methods, result in bonded components that are resistant to severe in-service environmental conditions.

Recommendations for suitable pre-bond and post-bond phosphate treatments can be obtained from DuPont Specialty Adhesives.

This guide gives detailed recommendations for the preparation of substrates for MEGUM[™] and THIXON[™] bonding agent bonding.

Substrate Preparation Methods

Process Outline

The first stage in the pre-treatment process is degreasing, which should ensure that the MEGUM™ or THIXON™ bonding agent primer or single-coat bonding agent will thoroughly wet the substrate surface.

The second stage is removal of contamination, including the oxide layer from metallic substrates, exposing a fresh, relatively oxide-free surface. Failure to remove an accumulated oxide layer, or a time delay between removal of the oxide layer and subsequent MEGUM[™] and THIXON[™] bonding agent primer or MEGUM[™] and THIXON[™] bonding agent single-coat application (allowing re-oxidation of the prepared surface), may lead to in-service delamination of the bond within the oxide layer. Both mechanical and chemical pre-treatment methods are suitable.

Removal of Oil and Grease

Steel parts for rubber-to-metal bonded components are usually supplied to the user with an oil or grease protective coating to prevent corrosion by rusting during transport and storage before use. The first stage in the substrate preparation process is to remove this protective oil and grease layer thereby ensuring that the MEGUM[™] or THIXON[™] bonding agent achieves good contact, or more specifically "wets" the substrate bonding surface.

Most mechanical surface pre-treatment processes include a vapor degreasing unit in which the oil and grease are removed by contact with the vapor of a suitable organic solvent. The solvent vapor condenses on the part, and the grease and oil are dissolved and removed as the condensed solvent drips back into the liquid degreasing bath.

Chemical pre-treatment methods use alkali wash tanks to remove oil and grease, followed by a water rinse to prevent carry-over of contamination into the chemical conversion process tanks.

Mechanical Pre-treatments

Mechanical pre-treatment methods involve abrasion of the degreased substrate surface to reveal a fresh clean surface for bonding. The abrasion treatment roughens the bonding surface, increasing its surface area and providing a key to the MEGUM[™] or THIXON[™] bonding agent. The mechanically abraded surface will react very rapidly with atmospheric oxygen (the reaction being accelerated in the presence of high humidity). It is important to apply MEGUM[™] or THIXON[™] bonding agent as quickly as possible after the surface abrasion treatment to reduce the effects of oxidation and possible contamination.

Methods of mechanical pre-treatment include wheel abraders or air blast using chilled iron, alumina, silica or carborundum grits. Chilled iron or steel grits are not recommended for the preparation of stainless steel or other non-ferrous substrates. Other methods of abrading the metal surface include grinding, wire brushing or emery cloth.

Chemical Pre-treatments

Chemical pre-treatment methods generally use alkali wash tanks to remove oil and grease, followed by a water rinse to prevent carry over of contamination into the chemical conversion process tanks. For certain types of grease (like animal greases) resisting alkali treatment, alternatives include the solvent degreasing by carbon hydrates or the perchloroethylene degreasing in fully encapsulated units. Run a lab trial before deciding which degreasing treatment to use. Contact DuPont Specialty Adhesives for specific recommendations.

Chemical pre-treatment methods include phosphating, acid etch, anodization, and plating. Phosphating is used on ferrous surfaces and involves a chemical conversion of the substrate surface. This gives the rubber-to-metal bonded component improved corrosion resistance and increased service life.

Checklist for Substrate Pre-treatment before MEGUM[™] or THIXON[™] Bonding Agent Application

Clean and Prepare Metal

Clean and prepare metal by chemical, mechanical or a combination of chemical and mechanical surface pre-treatment methods. Certain substrate types require special pre-treatment processes. Ensure that the correct and the most appropriate pre-treatment method is being used and monitor its performance.

Non-ferrous substrates like stainless steels and plastics should be grit blasted with non-ferrous grits.

Ensure that the prepared substrate surface is degreased before MEGUM[™] or THIXON[™] bonding agent application. If grit blasting pre-treatments are used, it is recommended that the substrate bonding surface is degreased before and after grit blasting. The first degreasing operation reduces contamination of the abrasive grit with oil and grease. The second degreasing operation removes residual grease and dust contaminants resulting from the grit abrasion process.

Handle Carefully

Pre-treated, degreased components must be handled carefully to avoid re-contamination of the bonding surface before applying MEGUM[™] or THIXON[™] bonding agent.

Degreased components should be handled with clean, lint-free gloves to prevent contaminating the bonding surfaces.

Prevent degreased components being contaminated with oil, grease or dust before MEGUM[™] or THIXON[™] bonding agent application. Avoid moisture condensation and ensure that prepared metal substrate components are kept at ambient temperatures. Avoid transferring cold metal components into relatively warm humid atmospheres where condensation of atmospheric moisture vapor is likely to occur.

Avoid Delays

One of the crucial stages in the preparation of parts for rubber bonding is the time delay between the end of the pre-treatment process and the application of the MEGUM[™] or THIXON[™] bonding agent. It is often during this stage that the final quality of the bonded component is determined. Both mechanically and chemically prepared metals are subject to oxidation and contamination.

Mechanically Prepared Metals

Mechanically pre-treated metal surfaces oxidize rapidly after treatment. Aluminum, brass, and stainless steels oxidize more rapidly than mild steel. Metal substrates will oxidize more quickly in humid and/or acidic atmospheres. Mechanically pre-treated components that are subject to atmospheric moisture condensation (typically when relatively cold metal parts are brought into a relatively warm atmosphere in conditions of high relative humidity) will oxidize more quickly.

For steel substrates, a time delay of two hours is acceptable between the pre-treatment and MEGUM™ or THIXON™ bonding agent application. Some oxidation of the steel substrate will have occurred during this two-hour delay period; however, it should not have progressed to such an extent as to be detrimental to the bonding performance of the component, as long as the recommended precautions on handling, storage, and transport have been observed.

The oxidation of mechanically pre-treated non-ferrous metals and stainless-steel substrates can be extremely rapid. A maximum time delay of 30 minutes should be allowed between pre-treatment and MEGUM[™] or THIXON[™] bonding agent application.

For steel parts, as long as the handling, storage, and transport conditions are dry, and the parts are protected from contamination by oil, grease, dust, and other contaminants, then it may be possible to exceed the two-hour delay between pre-treatment and MEGUM[™] or THIXON[™] bonding agent application. For details on metal treatment, see Table 1.

Table 1: Pre-treatment of Ferrous and Non-ferrous Metal Substrates

| Metal | Degreasing and Mechanical Pre-treatment ^{1,2} | Chemical Pre-treatment |
|--------------------|--|--|
| Mild steel | Degrease, abrade, degrease, and apply MEGUM™ or THIXON™ bonding agent within two hours. | Phosphate: Aqueous degrease and phosphate. ³ Apply MEGUM [™] or THIXON [™] bonding agent as quickly as possible after treatment. Acid pickle: With nitric acid – water rinse, dry, and apply MEGUM [™] or THIXON [™] bonding agent as quickly as possible. |
| Stainless steel | Degrease, abrade, degrease, and apply MEGUM™ or THIXON™ bonding agent within 30 minutes. Non-ferrous grits should be used. | Acid pickle: With hydrochloric, nitric acid, oxalic acid or chromic acid pre-treatments, apply MEGUM™ or THIXON™ bonding agent as quickly as possible after pre-treatment. |
| Aluminum | Degrease, abrade, degrease, and apply MEGUM™ or THIXON™ bonding agent within 30 minutes. Non-ferrous grits should be used. | Anodizing or alkali cleaning followed by chromic/sulfuric acid etching. |
| Brass or copper | Degrease, abrade, degrease, and apply MEGUM™ or THIXON™ bonding agent within 30 minutes. Non-ferrous grits should be used. | Acid pickle, or use ammonium persulphate or ferric chloride pre-treatments. |
| Zinc | Degrease, abrade, degrease, and apply MEGUM™ or THIXON™ bonding agent within 30 minutes. Non-ferrous grits should be used. | Phosphate or acid pickle, apply MEGUM™ or THIXON™ bonding agent as quickly as possible after pre-treatment. |
| Plated metal | Degrease, light abrasion, degrease. | Etch with dilute acid. Contact DuPont Specialty Adhesives for recommendations. |
| Sherardized (Zinc) | Degrease, oil and/or grease may be absorbed into the porous sherardized surface. Ensure thorough degreasing. Lightly abrade. | |

¹Mechanical treatment methods: Abrade to remove surface oxidation and other contaminants. Degrease to remove oil, grease, and residual dust from the abrasion process. Recommended abrasives include chilled iron (0.6 to 1.0 mm particle size), alumina (brown, angular 0.7 to 0.9 mm particle size) silica or carborundum grits. Other methods include surface grinding, wire brushing or emery cloth.

²Degreasing agents: Degreasing agents include chlorinated solvents such as perchloroethylene and other high boiling point degreasing solvents. Vapor degreasing processes are preferred.

³Chemical treatment methods for mild steel substrates:

1. Aqueous degrease: using acid or alkali treatments

2. Activation

3. Phosphate treatment: zinc or iron phosphate

Chemically Prepared Metals

Phosphate pre-treatments recommended for MEGUM[™] or THIXON[™] bonding agent bonded metal substrates produce a relatively thin phosphate layer (typically 3 to 5 micrometers). Localized areas of untreated metal can be vulnerable to oxidation. The oxidative process will be accelerated in conditions of high humidity and in the presence of other contaminants either in the atmosphere (acidic components) or as the result of ineffective rinsing of acidic components from the phosphating process itself.

The transport and storage of phosphated parts can be a decisive factor in determining whether oxidation of the bonding surface occurs. Phosphated components should be protected from moisture and other contaminants by being stored in sealed containers before use. If the phosphating process is remote from the MEGUM[™] and THIXON[™] bonding agent application location (for example when contract phosphating is being carried out), then particular care should be taken to protect the parts from moisture and other contamination during transport and storage. Phosphated parts should be kept in suitable sealed containers until required for use.

A rigid inspection procedure should be adopted for incoming phosphated parts to ensure that corrosion of parts is not occurring.

If the quality of the phosphating process, transport, and storage is maintained and a rigid inspection-before-use system is imposed, then generally a delay of 24 hours between phosphating and MEGUM[™] and THIXON[™] bonding agent application is acceptable.

As long as the parts have been correctly treated with phosphate, and they are stored in sealed containers protected from contamination by moisture, dust, oil, grease, and other contaminants, then it may be possible to exceed the 24-hour delay period without affecting bonding performance.

Surface Contamination

Contamination of the substrate surfaces is a problem common to mechanically and chemically pre-treated parts. Protection of parts during storage and transport is important in maintaining the overall quality of the bonded component. The greater the time delay between substrate pre-treatment and application of MEGUM[™] or THIXON[™] bonding agent, the greater the possibility of oxidation and/or contamination of the substrate surface by dust, dirt, oils, greases, moisture, and mold release agents.

The location of the pre-treatment area can be significant. If possible, isolate the mechanical pre-treatment area from the MEGUM[™] or THIXON[™] bonding agent application area and the MEGUM[™] or THIXON[™] bonding agent application area from the bonding area. By adopting this compartmentalization technique, cross contamination of untreated, pretreated, and MEGUM[™] or THIXON[™] bonding agent coated parts can be reduced.

Effects of Contamination

Bond failures can be caused by ineffective or inconsistent substrate pre-treatment or due to faults in handling, transport, and/or storage procedures.

By analyzing the performance of bonded components, it is usually possible to determine whether failure has occurred as the result of oxidation or contamination of the substrate. Delamination of the bonded component at the MEGUM™ or THIXON™ bonding agent primer/metal or MEGUM™ or THIXON™ bonding agent primer/plastic substrate interface could indicate a surface contamination problem. However, occasionally, substrate contamination can also affect the bond between the MEGUM™ or THIXON™ bonding agent primer and the MEGUM™ or THIXON™ bonding agent cover cement. Delamination at the bonding agent/substrate interface is usually associated with a reduction in the expected bond strength.

If bond failures persistently occur, then a full re-assessment of the pre-treatment process and environment, storage, transport and potential sources of contamination is essential.

A reduction in the time delay between pre-treatment and application of MEGUM[™] or THIXON[™] bonding agent may be required.

Plastics Pre-treatment

Plastic substrates often become contaminated with mold release agent as part of their manufacture. This contamination is usually removed by degreasing, but, occasionally, this is not sufficient. A light grit blast with non-ferrous grit (such as alumina) followed by degreasing is recommended.

Silicone mold release agents are particularly persistent and difficult to remove. It should be specified to the manufacturer of the plastic parts that silicone release agents should never be used. For details on plastic pre-treatment, see Table 2.

| Table 2: Pre-treatment of Plastic and Cured Rubber Substrates | | | | | | |
|---|--|--------------------|----------------------|-------------------------|-------------------------------------|--|
| Plastic | Degreasing and Mechanical Pre-treatment ^{1,2,3,4} | | | | Chemical | |
| | Vapor Degrease | Dip/Wipe Degrease | Grit Blast Medium | Grade of Emery Cloth | Pre-treatment | |
| Polyamide (including glass reinforced nylon) | Yes | Acetone or Ethanol | Yes | Yes | Formic acid | |
| Polyester | Yes | Acetone | Yes | Yes | - | |
| Acetal | No | Acetone | Yes | Yes | Acid pickle | |
| Phenolic resins | Yes | Acetone or MEK | Yes | Yes | - | |
| PTFE | Yes | Yes | No | No | Proprietary etching process | |
| Polycarbonate | No | Methanol or IPA | Yes | Yes | - | |
| Epoxy resin | Yes | Acetone or MEK | Yes | Yes | - | |
| ABS | Acetone | Yes | Yes | - | Acid pickle | |
| Polyimide | Acetone or Ethanol | Yes | Yes | - | - | |
| Polyethylene, polypropylene | Yes | No | No | - | Flame treatment or corona discharge | |

¹Degreasing agents: The choice of degreasing agent can be critical. Some organic solvents can cause crazing or dissolve certain plastics. ²Vapor degreasing: Perchloroethylene is recommended.

³Dip or wipe degreasing: MEK is methyl ethyl ketone, IPA is isopropyl alcohol.

⁴Grit blasting medium: Recommended particle size is between 0.6 and 1.0 mm.

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