Solutions from DuPont

Rubber-to-Substrate Bonding Agents Technical Guide

General Use of Specialty Adhesives

Introduction

MEGUM™ and THIXON™ bonding agents together have been used for more than 120 combined years to bond rubber-to-metal substrates. Today, MEGUM™ and THIXON™ bonding agents are used to bond a variety of elastomer compounds to metal and plastic substrates, including steel, stainless steel, aluminum, zinc, copper, polyamides, polyacetals, and polyesters.

The MEGUM™ and THIXON™ adhesive process bonds the substrate to the elastomer during the vulcanization process. This guide explains the application of the bonding agent and the rubber bonding/vulcanization process. The pre-treatment and preparation of the metal and plastic substrates prior to bonding is described in the Rubber-to-Substrate Bonding Technical Guide, “Substrate Preparation” section.

Typical bonded components

MEGUM™ and THIXON™ bonding agents are used in many bonding applications combining the unique properties of the rubber with the properties of the metal or non-metal substrate. Components bonded with MEGUM™ and THIXON™ are used in a wide variety of industrial applications including:

- Vibration control elements: springs, bumpers, bushes, suspension systems, clutch mounts, flexible couplings, and engine mounts
- Corded-rubber belts and hoses
- Seals and gaskets
- Bearing elements for buildings and bridges
- Rollers
- Solid rubber tires
- Track pads for caterpillar vehicles, linings, and protective coverings
- Metal-supported profiles

General description and properties

The majority of MEGUM™ and THIXON™ bonding agents are low-viscosity, organic, solvent-based solutions and/or dispersions of polymers and other reactive chemicals. They are used as one-coat bonding agents or two-coat primer and cover-cement systems. Some products within the range are clear non-pigmented solutions.

Water-borne MEGUM™ bonding agent products are also available. The “Rubber-to-Substrate Bonding” brochure describes the most commonly used products.

MEGUM™ and THIXON™ bonding agents are applied as coatings to substrates, including mild and stainless steels, non-ferrous metals, plastics, natural and synthetic fabrics and fibers, glass fiber, and ceramics. Generally, a pre-treatment is applied to the substrate to ensure that it is clean and that it provides a suitable bonding surface for the application of the bonding agents.

- The MEGUM™ and THIXON™ bonding agent range is extensive and versatile, including products that will bond all types of natural and synthetic rubber compounds to a variety of ferrous and non-ferrous metals, plastics, synthetic and natural fabrics and fibers, ceramic, and glass substrates.
- In-mold techniques such as compression, transfer, and injection molding can be used with MEGUM™ and THIXON™ bonding agents.
- Lay-up techniques, including those used for pipe coating and lining, tank and valve lining, roller bonding, and techniques involving the fabrication of fabric-reinforced hoses and pipes, can all be used with MEGUM™ and THIXON™ bonding agents. MEGUM™, and THIXON™ bonding agent systems are resistant to mold sweep and have a good resistance to in-mold prebake.
- MEGUM™ and THIXON™ bonded components are resistant to the stresses caused by adverse in-service environmental conditions such as heat, oils, chemicals, humidity, and salt-spray climate.
- MEGUM™ and THIXON™ bonded components retain their bonding capability at low service temperatures.

Storage and Handling Prior to Use

Storage

MEGUM™ and THIXON™ bonding agent containers should be stored in dry conditions, indoors and away from direct sunlight. Since most MEGUM™ and THIXON™ bonding agent products contain flammable, volatile organic solvents, they should be stored in an area conforming to the storage of highly flammable liquid regulations.

Avoid solvent evaporation

After use, containers should be carefully closed to minimize volatile organic solvent evaporation, to prevent the ingress of contaminants, and to reduce the build-up of dangerous volatile organic solvent vapor in the working atmosphere.

Evaporation of volatile organic solvents will occur from open-topped MEGUM™ and THIXON™ bonding agent containers. Although this will not cause an immediate reduction in product effectiveness, gradually the product viscosity and solids content will increase. If not controlled, the viscosity increase will cause application – and eventually bonding-performance – problems.

The product should be adjusted back to its specified viscosity- and solids-content range using the recommended diluent.

Agitate before use

Most MEGUM™ and THIXON™ bonding agent products contain dispersed solids that can settle during storage. It is important to displace and re-disperse any solids settlement by adequate and frequent stirring. Bonding agent containers should be stirred using an electric or air-driven motor fitted with an impeller; electric stirrer motors must be explosion proof.

Stirring time

Some MEGUM™ and THIXON™ containers come equipped with internal agitators already. however, all pigmented systems need to be stirred! The stirring time will be dependent on the degree of settlement and on the stirring technique employed. Flat stirrers, about half the diameter of the container, are usually more effective than propellers. Typical stirrer speeds between 60 and 80 rpm are recommended.

The stirring time should be sufficient to obtain a homogeneous material. This is typically in excess of 30 minutes. The material should be at ambient temperature during stirring. Continuous agitation during use is recommended whenever possible.

Preparing MEGUM™ and THIXON™ for Application

Dilution

MEGUM™ and THIXON™ bonding agents are usually diluted to reduce their viscosity prior to spray or dip application. More specific information is available on the individual product technical data sheets.

These recommendations should be used as guidelines. The actual amount of diluent used should be adjusted to suit the application method and the equipment being used. Dilutions can be made by weight or volume, or to a specified viscosity (with less reproducible effects).

Solvent quality

Diluents should be added to the MEGUM™ and THIXON™ bonding agents and stirred to obtain a homogeneous product. The mixing operation should be carried out in a suitable area and with equipment designed for safe use with flammable liquids. Pigmented products should be stirred before diluting. The bonding agents and solvents should be at a similar temperature to the working environment in which the coating operation takes place.

The viscosity of the diluted bonding agents should be regularly checked (at defined temperatures, usually 20 °C or 25 °C) and controlled within the specified viscosity range.

Process Control

Viscosity measurement and control

Control of MEGUM™ and THIXON™ bonding agent viscosity is an important aspect in achieving consistent bonding performance. Viscosity is affected by the thixotropic behavior of the product and by changes in temperature. It is important to carefully control the temperature of the test sample before measuring its viscosity and, if possible, to reduce variation in the working place temperature to a minimum to ensure consistent product viscosity and application characteristics.

The two tools for measuring MEGUM™ and THIXON™ bonding agent viscosity most frequently used are a rotational viscometer or a flow cup.

A rotational viscometer gives accurate and reproducible results, but the flow cup gives a good shop-floor in-process guide to viscosity.

Brookfield Synchro-lectric Rotational Viscometer

The Brookfield Viscometer measures the torque produced on a specified spindle rotating at a set constant speed at a low shear rate. The spindle is immersed to a set depth in a sample of the bonding agents under test.

The temperature of the test sample is carefully controlled. Agitation of the bonding agent sample prior to testing is essential to ensure dispersion of settled solids and to reduce thixotropy.

Flow cups

A flow-cup viscosity determination involves measuring the time taken, in seconds, for a constant volume of the bonding agent to flow through the orifice of the flow cup. The test sample is maintained at a constant, set temperature during the test. The type of flow cup most commonly used is the DIN cup series, with cup numbers 3 and 4 being the most useful.

Measurement of MEGUM™ and THIXON™ bonding agent viscosity using the DIN cup is a simple, comparative method. It is particularly suitable for the measurement of viscosity in shop-floor conditions. However, there are limitations to the accuracy and to the reproducibility of the DIN flow cup method of viscosity determination:

- Measured flow times will be affected by changes in product temperature. The recommended test temperatures are either 20 ± 0.5 °C or 23 ± 0.5 °C. The chosen test temperature should be strictly controlled.
- It is important to thoroughly clean the cup after use. Care must be taken to ensure that the orifice remains clean and undamaged.
- Flow-cup times of less than 12 and greater than 80 seconds may not accurately reflect the true relative bonding agent viscosity. The cup number indicates the size of the orifice. It should be chosen to give a product flow time of between 20 and 80 seconds.

Other flow cups including ISO, AFNOR, IWATA, or Ford cups can also be used to determine viscosity.

Solids content – measurement and control

MEGUM™ and THIXON™ bonding agents are composed of polymers, reactive components, and other ingredients dissolved or dispersed in a volatile organic solvent system. Dry solids content determination is a very accurate method of monitoring dilution ratios and of ensuring that the bonding agent product being used is within the set process specification.

Determination of the “dry solids content” of a product involves evaporating the volatile solvent system from an accurately pre-weighed sample and then re-weighing the resulting dried solids residue. Solids content determination requires the use of a sensitive balance (with a sensitivity of 0.0001 g).

The “dry solids content” of the bonding agent under test is expressed as a percentage of the original weight of the product sample.

Test procedure for determining dry solids content

Accurately weigh a suitable, clean container. Disposable aluminum cups with a diameter of approximately 50 mm and a height of 12 mm are recommended. They should be clean and stored under dry conditions at ambient temperature.

Accurately weigh approximately 3 g of the bonding agent under test into the solids-determination cup. Ensure that the sample has been properly stirred and is representative of the batch of product being assessed. Make sure that the weighing of the bonding agent proceeds quickly and accurately. This will reduce the possibility that pre-evaporation of the volatile solvent content will occur and lead to an inaccurate solids content determination.

Place the cup with the accurately weighed wet-bonding agent sample into an air-drying oven set at 130°C. If the bonding agent has a ketone or an alcohol solvent base, it should be dried for two hours. If the bonding agent solvent base is xylene or one of the other relatively slow-evaporating solvents, then the oven-drying cycle should be extended to three hours.

Remove the dried bonding agent sample from the oven and allow the sample to cool to ambient temperature in a dry area. Accurately reweigh the dried bonding agent solids content sample cup. Calculate the percentage of dry solids content as follows.

$$\text{Total solid content} = \frac{(m_2 - m_0)}{(m_1 - m_0)} \times 100$$

m_0 weight of clean, dry, solids cup
m_1 weight of wet MEGUM™/THIXON™ sample and cup
m_2 weight of dried MEGUM™/THIXON™ sample and cup

Upon request, each batch of bonding agent is supplied with a certificate of analysis that gives details of its dry solids content, viscosity, and density at the time of manufacture.

Film thickness – measurement and control

The thickness of the applied MEGUM™ and THIXON™ bonding agent film can affect bond performance. Carefully monitoring and controlling applied film thickness within the set specification is recommended. The dry film thickness can be determined using non-destructive methods similar to those used in the paint industry. Equipment is available for ferrous and non-ferrous metal substrates. Film thickness can be measured, for example, by heat-capacity determination via laser techniques, or with the well-known X-ray method allowing the parallel investigation of primer and cover layers. Proper calibration of the equipment, using components with similar surface profile to those being coated, is important to ensure consistent film thickness determination results. The average film thickness can be calculated using a minimum of five measurements.

An alternative method involves accurately weighing a component before and after coating and then using the surface area of the coated area and the dry film thickness of the bonding agent to calculate the applied dry film thickness.

Coverage

Theoretically, MEGUM™ and THIXON™ bonding agent coverage can be calculated using solids content, dry film density, and dry film thickness applied.

(DSC x 10)
Total solid content = (d x t) m2 kg

DSC = dry solids content weight %
d = dry film density in gcm-3
t = dry film thickness in micrometer (μ)

Practical coverage is dependent on wastage and evenness of coating; both are affected by the method of application. Coverage values can be used to estimate product usage and the economics and efficiency of the application method.

Application Methods

Spraying

Spray coating is used to apply MEGUM™ and THIXON™ bonding agents to large numbers of components or to large surface areas. With suitable jigs and masks, bonding agents can be applied to selected areas of a component. Spray application requires considerable technical control and will ensure a consistent, uniform applied film. To ensure even coverage, parts should be rotated. Automatic conveyor-spray application machinery is used for maximum application efficiency. Hand-spray guns are useful for smaller runs. MEGUM™ and THIXON™ bonding agents can be applied using the following types of spray equipment: air, airless, airmix or high-volume, low-pressure (HVLP). Electrostatic-spray and hot-spray technologies can also be used.

Brushing

Brushing is recommended when applying MEGUM™ and THIXON™ bonding agents to small numbers of components. Applied film thicknesses can be variable, but brush coating does allow the application of thicker film coatings for layup bonding methods. Care should be taken to avoid contaminating coated components with gloves or fingerprints. The viscosity of the bonding agent should be monitored. Solvent lost by evaporation should be replaced using the recommended diluent.

Dip coating

Dip coating is suitable for coating large numbers of components that require total coverage. The shape of the component is a major factor in determining whether it is suitable for dip coating. MEGUM™ and THIXON™ bonding agent viscosity and the rate of withdrawal of the coated component from the dip tank are critical to avoiding excessive build-up on the edges of the component. Once set up, dip coating requires minimal supervision and maintenance. For small runs, hand dipping is usually most convenient and requires no special equipment. When handling large numbers of components, a conveyor dip line may be used with in-line metal cleaning and drying sections.

Tumble coating

Tumbling components in a rotating barrel containing diluted MEGUM™ and THIXON™ bonding agents provides low-cost, uniform coverage. Best results are obtained with non-pigmented bonding agent products applied to relatively small-sized components. The coated components are dried by warm-air circulation in the tumbling barrel or after being discharged into drying trays.

The shape of the component, and the effect of any abrasion between the components taking place during tumbling, can affect the uniformity of the applied bonding agent.

Roller coating

Roller coating is used to apply MEGUM™ and THIXON™ bonding agents to flat areas or onto cylindrical components. It is an efficient method to combine maximum coverage with minimum waste and uniformity of film thickness. Bonding agents are usually applied undiluted. Roller coating can also be used to coat cylindrical shafts or pipes using felt or foam rollers.

Preheating and drying of coated components

Metal parts can be heated before and after the MEGUM™ and THIXON™ bonding agent has been applied to speed up drying. To ensure proper wetting of the component and to avoid bonding agent precure, preheat and drying temperatures should not exceed 80 °C.

Storage and handling of coated components

Coated components can be stored for several days before bonding without affecting bond performance, but they must be protected from contamination. Unprotected components are particularly vulnerable to contamination by mold-release spray, hydraulic oil, and dust.

A temperature change from a cold storage area to a relatively warm bonding area can lead to atmospheric moisture condensing on the component bonding area and may result in poor bonding performance.

Methods of Bonding

In-mold vulcanization/bonding

The majority of bonded components are manufactured by in-mold vulcanization/ bonding methods using compression, transfer or injection molding (CM, TM or IM).

MEGUM™ and THIXON™ bonding agent systems provide the following advantages for in-mold bonding:

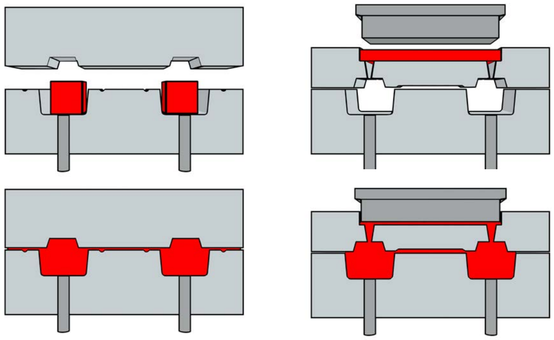
- Excellent lay-over of coated components that can be bonded several days or even weeks after coating as long as they are protected from contamination during storage.
- Resistant to prebake, allowing a time delay between loading of the mold and closing of the press. Stability to prebake is an advantage in loading multi-cavity molds.
- Effective with both low-temperature/ prolonged-cure time and high-temperature/ short-cure time vulcanization cycles. The vulcanization temperature-time cycle may be set according to the requirements of the rubber compound.
- Uncured films are resistant to the various physical stresses encountered in the in-mold vulcanization/bonding process.
- High-pressure IM process reduces mold fouling significantly.
- Hot-bond strength of the bonding agent to rubber bond is excellent, resisting the bond stresses experienced during the removal of the freshly bonded component that is still at molding temperature.
- MEGUM™ and THIXON™ bonding agents create a high crosslinking density between the bonding agent layer and the substrate, as well as the bonding agent layer and the rubber, which results in high bond strength of the composite.
- MEGUM™ and THIXON™ bonding agent films have good thermal stability at molding temperatures of up to 200°C.

Compression molding

CM is the oldest method of in-mold vulcanization/bonding and is still widely used when a relatively small number of bonded components is required. It is also the preferred method for large bonded items. Compression molds are usually less expensive than other methods.

Transfer molding

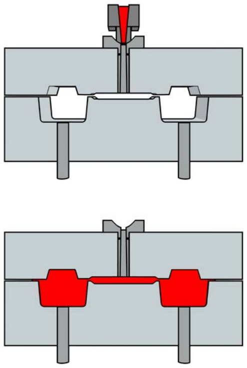
TM is more efficient than CM but can still use the same press equipment. It is more suited to producing large numbers of relatively small components. Mold costs are higher than for CM.



CM = Compression Molding TM = Transfer Molding

Injection molding

IM is the most sophisticated technique for producing high volumes of bonded components. It is the most expensive method in terms of equipment and technical control requirements. Generally, to obtain maximum production efficiency, injection-molded components are produced using relatively short, high-temperature vulcanization cycles – typically 5 to 15 minutes at temperatures between 170°C and 200°C.



IM = Injection Molding

Influencing Factors for In-mold MEGUM™ and THIXON™ Bonding

Parameter	CM	TM	IM
Rubber release agent	++	+	0
Degassing	+	+	+
Pressure build-up	+	0	0
Sweep resistance	0	+	++
Short cure times	0	+	++
Scorch time	0	+	++
Mold design	+	+	+
Metal heat transfer	+	+	+
Mold load time	0	++	+
Cure temperature	0	0	+
State of cure	++	++	++

++ Major influencing factor
+ Medium influencing factor
0 Minor influencing factor

Extrusion bonding

Special-purpose MEGUM™ and THIXON™ bonding agents are roller coated (coil coated) onto metal strips, usually steel or aluminum, and then bonded to rubber with a continuous extrusion/bonding technique using a cross-head extruder. The bond is formed during the vulcanization of the extruded profile. Various techniques are used for the continuous vulcanization of the rubber-/metal-bonded profile. These include: hot-air, liquid-sale, and fluidized beds. Typical applications of this technique are automotive weatherstrips or other metal-reinforced profiles.

Pipe and tank lining

The lining of pipes and tanks uses MEGUM™ and THIXON™ bonding agents in combination with tacky adhesive "tie cements." These provide the initial tack necessary while the rubber lining is rolled on in the lay-up process. The bond is formed while the rubber lining is being vulcanized using hot air, hot water, and/or steam. Generally, bonding pressure at the vulcanization stage is low; therefore, it is very important that good initial contact is made between the uncured rubber sheet and the bonding agent tie cement during the lay-up procedure. Ambient temperature-cured rubber compounds with chemically initiated cure systems can also be used with MEGUM™ and THIXON™ bonding agents.

Rubber-lined tanks and pipes are used in the chemical and mineral industries, where the resistance of rubber to abrasion and chemical attack is particularly important.

Roller-pipe covering

MEGUM™ and THIXON™ bonding agent systems are used to bond rubber coverings to rollers, pipes and other process equipment. Usually during the vulcanization process, bonding pressure is provided by a shrinkable fabric wrapping. Several methods can be used to vulcanize the rubber, including steam autoclave, hot water or dry heat. For liquid rubbers such as polyurethanes, a casting process is used. Rubber-covered rollers are used in the paper and food industries (rice rollers), as well as for printing and roller coating of paints. They are also used in the mining industry. Rubber-covered pipes are used in the oil and gas industries for offshore applications.

Post-vulcanization bonding

The post-vulcanization (PV) bonding process can be used to bond cured rubbers to coated metal surfaces or to other cured rubber substrates. The bond is formed by applying temperature and pressure to the assembled parts. The advantage of PV bonding is in reduced mold costs. Typical parts produced with this technology are bushes and mounts with concentric metal tubes, where the inner tube is in-mold bonded and the outer is bonded using a PV-bonding technique.

Rubber Vulcanization – Process Control

The following factors should be considered when selecting the type of MEGUM™ and THIXON™ bonding agent and choosing the process required for a specific application:

- Rubber type and compound formulation
- Substrate composition
- Substrate preparation method
- Number of bonded components required
- Equipment available for applying MEGUM™ and THIXON™ bonding agents
- Methods of molding or fabrication available
- Vulcanization cycle
- Post-bond treatments
- Component in-service function
- In-service environmental resistance requirements

Influence of compounding ingredients

As part of the rubber compound design and production development stages of the bonded component, it is advisable to carry out a MEGUM™ and THIXON™ bonding agent bond feasibility study. As part of this study, the various combinations of substrate type and preparation, application and molding method, mold design, vulcanization cycle, and post-bond treatments can be evaluated as to their effect on the final bond performance.

General rules for the formulation of rubber compounds for bonded components are outlined as follows:

- Keep sulfur concentration high.
- Replace 2 to 5 phr of carbon black with silica.
- Limit concentration of waxes and paraffinic and aromatic oils.
- Low-durometer (less than 45 IRHD) rubber compounds are more difficult to bond than medium- or high-hardness compounds.

Vulcanizing agents

In general, conventional natural and synthetic rubber vulcanizing agents such as sulfur, zinc oxide, and accelerators do not affect bond performance. However, rubber compounds specifically designed for in-service resistance to heat aging, that are formulated with efficient vulcanization (EV) or semi-EV sulfur-donor accelerator systems, are more difficult to bond.

Alternative cure systems, including peroxide, bisphenol or amine cure systems, may require special-purpose MEGUM™ and THIXON™ bonding agents. Contact your DuPont Specialty Adhesives representative for details.

Fillers

The choice of filler and its concentration within the rubber compound can influence ultimate bond performance. Carbon black is recognized as contributing to improved bonding performance. Low-hardness compounds (i.e., below 45 IRHD) are more difficult to bond than medium- and high-hardness compounds.

Plasticizers

Plasticizers should be used with caution, particularly when used in rubber compounds formulated for oil resistance. Plasticizers and other additives, which tend to diffuse readily to the rubber compound surface, should only be used in very small quantities. Certain ester plasticizers, which soften the bonding agent film and can weaken the rubber-to-metal bond after vulcanization, should be avoided. DuPont Specialty Adhesives supplies a range of plasticizers that are recommended for use in natural and synthetic rubber compounds.

Antioxidants and antiozonants

Antioxidants used in moderate concentrations will not affect bonding performance. Antiozonants are migratory by their nature, and as such may interfere with bonding unless they have been properly incorporated into the rubber compound.

Influence of Vulcanizing Conditions

PV bonding

The quality of the rubber compound should be strictly controlled. The age of the compound is important in that scorched or partly cured compounds may not give optimal processing characteristics. This may affect bond performance.

Rubber compound sheet used for CM should have a clean bonding surface. There should be no surface bloom of accelerators, curatives, plasticizers or other rubber chemicals that may interfere at the MEGUM™ and THIXON™ bonding agent/rubber interface and eventually lead to poor bonding performance. If rubber compound has been stored for long periods, it is advisable to pre-mill before molding to ensure proper incorporation of compounding ingredients and provide a fresh bonding surface.

Vulcanizing temperature

Rubber-to-cement failure ('R/C') can occur if MEGUM™ and THIXON™ bonding agents are allowed to prebake or precure before molds fill and pressurization operations are complete. Prebake of coated components usually occurs when either the mold-loading operation is prolonged, or there is a delay between loading the mold and the subsequent introduction of the rubber, mold closing and, application of molding pressure. High-temperature IM operations are particularly susceptible if mold-loading delays occur. The problem can be overcome by reducing insert loading time, reducing mold temperatures or using a mold-loading jig to speed up loading time.

MEGUM™ and THIXON™ bonding agents designed to give prolonged resistance to prebake are also available.

Bonding pressure

Good contact between the rubber compound and MEGUM™ and THIXON™ bonding agents is essential to obtain maximum bonding potential. Bonding pressure is required to achieve this contact.

- In-mold bonding: For in-mold bonding, rubber fill and pressurization should be completed as quickly as possible after the coated components have been loaded.
- Roller and pipe covering: Bonding pressure is applied by tape wrapping unvulcanized rubber covering, followed by steam autoclave curing.
- Lay-up: Used for the lining of tanks, pipes, and other vessels, bonding pressure is minimal – and surface contact pressure is applied only during the application of the unvulcanized rubber sheet. Tacky tie cements are used, usually manufactured by dissolving some of the unvulcanized rubber sheet in an organic solvent. The tie cement is applied over standard MEGUM™ and THIXON™ bonding agent primer/cover-cement bonding systems.

Mold design

Transfer and injection molds for rubber bonded components should be designed to reduce the possibility of MEGUM™ and THIXON™ bonding agents being removed, or “swept,” from the coated substrate as the unvulcanized rubber enters the mold. The unvulcanized rubber is of relatively high viscosity and moving at considerable speed as it enters the mold. Mold entry ports or pressure relief holes should not be situated adjacent to the rubber/substrate bonding interface.

Molds involving the assembly of a number of parts should be designed to minimize assembly time and reduce the possibility of prebake of the coated substrate between mold loading and mold closure.

Multi-cavity molds may require a loading jig to reduce the mold-loading time of the coated components and avoid prebake.

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