Viton™ Fluoroelastomers

Processing Guide

Technical Information

Introduction

This report provides guidelines for the efficient mixing and processing of Viton™ fluoroelastomers. Suggested machine settings are given for various processing techniques. Since mixing and processing equipment can vary with age, maintenance, manufacturer and design, this report only purports to offer typical conditions, and not absolutes.

Polymer Form

Viton™ typically is supplied in sheet form, either as gum polymer, or as a precompound (gum polymer, plus curatives). Sheets of Viton™ are nominally 1.25 cm (0.5 in) thick and 36 cm (14 in) wide. The length of the sheet may vary but there are typically 10 sheets per box. Some Viton™ gums are supplied as free-flowing pellets.

Packaging

Viton™ comes packaged in rectangular 61 x 41 x 22 cm (24 x 16 x 8.75 in) Kraft boxes. The boxes are designed to fit a standard 1.0 x 1.2 m (40 x 48 in) pallet, using a 5 box per layer pattern. The standard package weight is 25 kg (55.1 lb) per box.

Handling and Storage

Before handling or processing Viton™ polymers or precompounds the user should read and understand the Chemours technical bulletin "Handling Precautions for Viton™ and Related Chemicals." It is generally advisable to avoid having physical contact with the polymer, all compounding ingredients and/or any of the fumes or dust associated with the processing of the polymer/compound.

Like most rubbers, Viton™ and the ingredients used in compounding should be stored in cool dry locations and kept free of contamination. Although the Viton™ polymers tend to have excellent storage stability, storage in hot and/or damp areas should be avoided. To avoid contamination, such items as atmospheric moisture, oils and greases from machinery or other compounds, other polymers and/or polymeric compounds should be prevented from coming in contact with Viton™ polymers.

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Processing

Viton™ polymers can be mixed using conventional rubber processing equipment. Demands in the market place for high quality goods that meet increasingly stringent performance criteria place special emphasis on product quality. Economic constraints, coupled with increased competition have made it essential that products be made properly from the beginning of the process, rather than relying on end-of the line inspections, where rejects invoke higher added costs. For rubber, getting it right from the start means getting it right during the mixing cycle.

Mill Mixing

Mill mixing is one of the oldest and most basic methods of mixing rubber compounds. It consists of two, counter-rotating steel rolls turning at different speeds. The different speeds of the rolls create a shearing action at the point where the two rolls are closest. By passing the polymer through the nip between the two rolls, the polymer is masticated and squeezed to form a band on one of the rolls. After forming a band, the dry and liquid ingredients are incorporated, by the grinding and shearing action of the two rolls. To further enhance the mixing and dispersion of the ingredients, the mill operator must cut, fold and refine the compound.

It is generally recommended that Viton™ be mixed on as cool a mill as possible (23 °C or 75 °F). The use of chilled water minimizes scorch and promotes higher shear, thus improving dispersion. Once the polymer has been banded, the addition of the other ingredients can start immediately. If two polymers of dissimilar viscosities are to be blended, the higher viscosity polymer should be banded first, followed by the addition of the lower viscosity polymer. Once a band of the polymers has been formed, the band should be cut several times to enhance the blending and then the other ingredients may be added. It is important that the batch being mixed on the mill is properly sized for the mill being used. Too large or too small a batch tends to reduce dispersion and/or significantly increase the milling time required to obtain good dispersion. The following are suggested batch sizes for various mill sizes:

Mill Roll Length	Batch Weights
0.91 m (36 in)	13–15 kg (28–33 lb)
1.02 m (40 in)	16–18 kg (35–40 lb)
1.22 m (48 in)	21–25 kg (47–55 lb)
1.52 m (60 in)	34– 41 kg (75–90 lb)

Recommended Mill Mixing Practices

- Always use a clean mill, free of contamination from other elastomers, oils, greases and sulfur-bearing chemicals.
- Viton™ is typically a "back roll" polymer. This means that the polymer will go to the back roll (fastest roll) when banded. Forcing the polymer or compound to the slower roll can sometimes be an option, but the compound may exhibit extensive bagging and splitting to both rolls if this is attempted.
- Use chilled water (10 °C [50 °F]) whenever available. The best dispersion occurs at maximum shear. As the polymer heats up during the mix cycle, the viscosity will decrease along with shear stress. Keeping the stock temperature cool is especially important for polymers that have Mooney viscosities that are 30 and lower, in order to prevent mill roll sticking and to maximize dispersion.
- Typically, ingredients that melt are easier to disperse if added after the filler has been incorporated. These materials can be added late in the mix, when the batch temperature has increased sufficiently to melt them.
- Generally, it is best to allow the mixed stock to condition for at least 12 hr prior to molding parts. This conditioning allows the interaction between the filler system and polymer to take place, resulting in noticeable improvements in mold flow characteristics and physical properties.

Curative Addition

- Diak™ No. 1 should be added to the batch last, just prior to the cross cutting and refining steps. Diak™ No. 3 is a scorch safer cure and can be incorporated at the same time the fillers are added.
- VC-20 and VC-30 should be added to the banded polymer as early in the mixing operation as possible. Neither of the chemicals will melt under normal mixing conditions, and depend on good shear for dispersion. Adding these chemical master batches late in the mix will result in dispersion problems.
- VC-50 is supplied in the form of free flowing pastilles that will readily break up into smaller particles during incorporation. However, the VC-50 must melt in order to be properly dispersed. VC-50 melts at 80 °C (176 °F). Therefore the batch should reach a temperature of 100 °C (212 °F) to ensure melting.

Acid Acceptors

- Always pre-blend the acid acceptors (MgO, $Ca(OH)_2$, ZnO, etc.) with the filler(s) for the best dispersion. The addition of metal oxides alone (especially MgO) can cause caking and sticking to the rolls.
- Moist magnesium oxide will badly cake on the mill rolls and will be more difficult to disperse. Further, moist acid acceptors can cause scorch and cure problems.
- These materials are hygroscopic and should be kept dry.

Processing Aids

- Do not use stearate types of process aids, such as zinc stearate; these materials reduce the processing safety of bisphenol cure systems.
- Add process aids late in the mix, or last. Processing aids, in general, are easily dispersed, but, if added too early in the mix, they can reduce the mixing shear that is needed for good dispersion of ingredients that do not melt.
- Recommended process aids for bisphenol-cured types of Viton™ include Struktol® WS280, Carnauba Wax, VPA 1, VPA 2, and VPA 3. The VPA 3 should not be used at levels higher than 1.25 phr. Levels of VPA 3 at or above 1.50 phr will result in poor scorch safety.
- For peroxide-cured types of Viton™, Struktol® WS280 and HT290 (<1.25 phr) are recommended for improved mill handling and mold release.

Mill Mixing Troubleshooting Guide

Excessive Sticking of Stock to Mill Rolls

- Do not add MgO separately to the batch, at any stage of the mix.
- Increase flow, and/or reduce the temperature of water passing through rolls.
- Do the rolls have excessive scale deposits, thus reducing the heat transfer efficiency of the rolls?
- Add 0.5–1.0 phr of carnauba wax to bisphenol-cured Viton™ formulations. If peroxide cured formulations are being used, add 0.5–1.0 phr of Struktol® WS280 or HT290.
- If possible, use a higher viscosity grade of Viton™, or blend current polymer with a higher viscosity version.

Excessive Bagging of the Stock

- Try warming the mill rolls, above the current temperature, and/or using a tighter mill nip opening.
- The formulation should not contain excessive amounts of process aids: e.g., no more than a total of 1.5 phr. Struktol® HT290 should not be used at levels higher than 0.5 phr.
- If bagging occurs on the front roll, move the batch to the back roll.

Suggested Mill Mixing Procedure

Time, min	Operation
-10	Sweep the mill pan, clean the rolls and guides to eliminate potential contamination. Adjust mill roll temperature to 32 ± 5 °C (90 ± 10 °F). Water on full.
-5	Premix filler(s), magnesium oxide and calcium hydroxide, or other metal oxides in a clean rigid container to obtain a homogeneous blend.
0	Band the polymer and adjust the width of the nip to obtain a rolling bank.
2-3	Add all the premixed ingredients into the nip at a rapid, but uniform rate across the length of the rolls. Allow loose ingredients to fall on the pan. Sweep the pan of any ingredients and reintroduce them into the nip. Sweep and re-add the loose ingredients as often as necessary until none remain.
9-12	When all the ingredients have been fully incorporated, close the mill nip. Cut and blend the stock three times from each side of the mill roll. "Cigar" the stock and pass it through a tight nip setting at least 4 times.
13-15	After refining the stock, open the rolls to the desired width for final sheet thickness and cut the stock across the width of the roll to remove the stock in a sheet. Allow the stock to air cool. Note: Air-cooling is preferred. However, the stock may be water cooled if precautions are taken to limit the amount of time the stock is dipped and if forced air is used to completely rid the stock of any residual water before storing.

Poor Dispersion of Curatives

- VC-20, VC-30: Add these curatives to the batch immediately after banding the polymer. Make sure that the batch does not get too hot, too quickly.
- VC-50: Make sure that the batch reaches a minimum temperature of 100 °C (212 °F), in order to ensure that the curative will melt.

Poor Dispersion of Metal Oxides/Acid Acceptors

- Premix acid acceptors with filler(s), prior to adding to the banded polymer.
- Keep the mill rolls as cool as possible.
- Make certain the metal oxides have been stored in a cool, dry place and kept in tightly closed containers.
- Check the metal oxides for lumps, which could be an indication of moisture contamination.
- Use masterbatch metal oxides.
- Allow the stock to condition for 12–24 hr after mixing and then refine the stock.

Cooling and Storage

- Compounds based on Viton™ should be cooled as quickly as possible after mixing. Cooling can be accomplished by immersing the sheeted stock into a dip tank, by exposure to fans, or by water spray.

  The best cooling results from using water spray and fans. If water is used, it is critical that ALL the water be removed from the surface of the stock before placing into storage.
- Under no circumstances should the stock be stacked for storage if the internal temperature is above 32 °C (90 °F). The stock should be cool to the touch. A partitioning agent is required to prevent mixed compound from sticking to itself, especially for compounds made with lower viscosity polymers. Talc is preferred. Do not use a dusting agent that contains stearates.
- Mixed stock should be kept in a cool (18 °C [65 °F]), dry location and protected from airborne contaminants and moisture. When taken out of storage, keep the stock covered while allowing it to come to room temperature. Any moisture that condenses on the stock should be quickly removed, to prevent blistering and scorch problems during the curing process.

Internal Mixing

Although there are several variations of internal mixers, the basic actions they perform are the same. Internal rubber mixers consist of two intermeshing, counter-rotating bladed rotors turning at the same speed. The rotors are set at a specific distance from each other and the walls of the chamber, in which they are housed. A ram is positioned in the throat of the unit, leading to the chamber. During the loading process, the ram is retracted to allow the various ingredients to be charged into the chamber. However, during the mixing cycle, the ram is lowered to help feed the materials to the rotors and to force the materials against the rotors while mixing. The polymer and compounding ingredients are charged into the chamber via chute or throat. The mixing is accomplished by shearing action of the rotors against the walls of the chamber and by the squeezing and shearing action between rotor blades. When the polymer is in contact with the rotors, the speed of the rubber is equal to the speed of the rotor, whereas the speed of a particle located on the fixed walls of the chamber is zero. Due to the peripheral speed of the rotor tip and the static nature of the mixers internal chamber, the shear rate values are very high.

Mixers of this type can range in batch sizes from approximately 2.2–363 kg (5–800 lb). Mix times will generally run from 3–5 min, depending on the condition of the mixer and the formulation to be mixed. It is preferred that mixers of this type have chilled or refrigerated water to provide ample cooling of the batch. As with mill mixing, the best dispersion occurs when the polymer viscosity is the highest. As the mix temperature increases due to shear, the viscosity will decrease along with the dispersion efficiency.

It is generally recommended that for processing uniformity and consistency, an internal mixer should be outfitted with the following:

- A timer for measuring the cycle time
- A method for measuring the internal temperature of the batch
- Variable rotor speed
- Variable ram pressure
- A method for determining power consumption during the mixing cycle
- A method to extract heat from the batch

Definitions and Recommended Practices

Total Mix Cycle Time—This is the time recorded from the start of feeding the materials into the banbury, to the moment the batch is discharged. Because the rate of temperature increase of the work input to the batch can be affected by the conditioning of the raw materials, feedstock and mixer temperature, "total mix cycle time" is not normally a primary control parameter. However, it can be used as an effect alert or indicator that the desired processing or work input is different.

Rotor Speed—The rotor speed will determine the rate of temperature increase. Typically, the ability to monitor, adjust and record rotor speeds during the mixing cycle will allow for significant improvements in mixing uniformity and performance.

Ram Down Cycle Time—The mixing in the banbury is initiated with the pressurization of the batch, caused by the ram being lowered under pressure.

Power/Work Input—The work input to the material is a measure of the energy consumed by the mixer, while combining the ingredients. In theory, the conversion of mechanical energy into heat through the shearing action of the materials passing between the rotor tips and the mixer's chamber wall should be uniform for a given set of operating conditions and uniform feedstock conditions. Variations in the feedstock temperature, either from seasonal changes or process variations, and changes in the mixer temperature will have a significant affect on energy consumption. Other ways to measure energy consumption or work input:

- Peak Power or the maximum instantaneous power utilized by the mixer. Usually the maximum dispersion occurs at the peak power plateau.
- Motor Torque is a calculated value and can be used at various points during the mixing cycle as an indication of the composition's viscosity.
- Batch Gauge Temperature—A continuous indication of the batch temperature is essential for internal mixers. Batch temperature defines and determines the point in the cycle that the various ingredients can be added to the batch, when materials will be better dispersed, when the melting of various ingredients will occur and when the compound can be discharged from the mixer. It is very important that this instrument be properly maintained and checked for accuracy. Generally, the internal temperature of a compound will be 10–25 °C (20–50 °F) higher than that indicated by the mixer's gauge. Batches should be discharged when the mixer's internal temperature gauge is no higher than 110–115 °C (230–240 °F) range.

With few exceptions, most compounds based on Viton™ can be "one pass" mixed in an internal mixer. Mixing times generally range from 3–5 min. If the proper mixing procedures are followed, mixing in an internal mixer can be as safe as mixing on a large mill. As with mill mixing, availability and use of chilled or refrigerated water is very important. The use of chilled water allows better control of the stock temperature during mixing, including keeping the viscosity of the stock higher for increased shear and dispersion.

The recommended load factor for most compounds of Viton™ is approximately 70–72%. Too high a load factor can result in scorching the batch and poor dispersion. Likewise, if the load factor is too low, the mix time will increase and poor dispersion can result. The load factor is the percentage of the mixer's chamber to be filled by the stock. The actual volume of the chamber is the chamber volume minus the volume occupied by the rotors or "net chamber volume." Since rubbers are incompressible, a certain volume of the chamber must be left unfilled, to facilitate movement of the stock and mixing.

Batch Size (Weight) = (Net Chamber Volume) x (Load Factor) x (Compound Specific Gravity)

The load factor defines that portion of the net chamber volume actually occupied by the mixed batch.

It does not take into account such variables as:

- The condition of the mixer. Wear on the mixing chamber and rotors increases the net chamber size and thus may require a slightly higher load factor.
- The rheological properties of the polymer. Polymers that tend to quickly thermally soften may require a higher load factor.
- The stock viscosity or stiffness. High viscosity compounds generate heat quickly and make control of the stock temperature more difficult and may require a slightly reduced load factor in order to have better temperature control of the stock.
- The cooling efficiency and available mixing power. The load factor may have to be reduced to improve control of the temperature and to remain within the power capabilities of the mixer. It should be noted however, that less than optimum load factors tend to increase the mixing cycle and increase the risk of poor dispersion.

Internal Mixing General Recommendations

When mixing any compound based on Viton™, a properly cleaned mixer is required in order to avoid contamination. A thorough cleaning of the mixer is particularly important if sulfur or sulfur-bearing compounds were previously used in that mixer. Sulfur is extremely detrimental to the bisphenol cure system. The presence of as little as 0.15 phr of elemental sulfur can cause the complete loss of cure activity in a bisphenol-cure compound. Likewise, aromatic oils can cause decreased cure activity in peroxide cured polymers. Therefore, care should be used in selecting the oils used for lubricating the rotor seals.

Chilled water (10 °C [50 °F]) is typically recommended for obtaining the best dispersion of ingredients that do not melt below 100 °C (212 °F). Maximum shear is obtained by keeping the polymer viscosity as high as possible.

For highly filled compounds of Viton™ it is recommended that an "upside down" mix or a "sandwich mix" be used. The "upside down" mix involves loading the fillers and acid acceptors first, followed by the polymer(s). A "sandwich" mix involves charging half the polymer into the mixer first, followed by the fillers and acid acceptors, and then the remaining polymer.

If variable rotor speed control is available, a slow rotor speed is recommended over that of a fast rotor speed. Typically, rotor speeds of 25–45 rpm are preferred for most mixing. Rotor speeds that are too high will generate heat too quickly, reducing the viscosity of the polymer and the ability of the mixer to disperse the ingredients. Further, the excessive heat that is generated may cause scorching of the compound. Too slow a rotor speed may cause excessively long mix cycles and failure to reach a temperature high enough to melt specific ingredients.

It is a good practice to determine the actual batch temperature with a probe, after the batch has been discharged from the mixer. All internal mixers tend to exhibit some discrepancy between the actual batch temperature and the batch gauge temperature in the mixing chamber. The difference between the two temperatures may be caused by the location of the mixer's thermocouple, the batch size and/or the batch viscosity. Knowing the differences between the two types of temperature measurements will influence when the batch should be dropped, to avoid scorch problems.

Use the drop-mill only for sheeting and taking heat out of the batch. Mixed compound dropped from an internal mixer will typically be relatively low in viscosity, and will not provide sufficient shear for effective, additional dispersion of the ingredients. If additional mixing is required, use the drop mill to lower the batch temperature, allow the batch to come to room temperature and then refine it on the mill later, in a second pass.

Many mixers are designed to pump a lubricant into the rotor dust seals, to prevent various ingredients in the compound from entering the bearings. Make certain that the internal mixer is set up to pump a minimum amount of the lubricant, to avoid contamination of the batch. Dioctylphthalate is recommended for use as a bearing lubricant, but excessive amounts of this material in a compound of Viton™ can cause molding defects, shrinkage variations, and can potentially degrade the physical properties or lower the heat resistance of the final vulcanizates.

If a blend of polymers is to be used, the higher viscosity polymer should be added first, allowed to warm-up, and then the lower viscosity polymer added. This technique is only required if the difference in polymer viscosity is greater than 10 points. Polymers that have 10 points or less differences in their viscosities may charged into the mixer at the same time.

Viton™ curatives, VC-20 and VC-30 should be added as early as possible (after the polymer) to the batch, in order to get good dispersion. Viton™ curative VC-50 can be premixed with the acid acceptor/filler blend.

Time, min	Operation
-5	Clean the mixing chamber. Run Full cooling circulating water through chamber shell and rotors. Rotor Speed: 25–35 rpm Ram Pressure: 420–500 kpa (60–80 psi)
0	Add all fillers and acid acceptors followed by the polymer(s)
0.5	Allow all fillers, acid acceptors to dry blend. Sweep the throat of the mixer and lower the ram.
1.0	Mixing begins
1.5	Raise the ram, add internal processing aids if required
2.0	Lower the ram
2.5	Continue the mixing process
3.0–4.0	Dump or drop the batch (110 ± 5 °C [230 ± 10 °F])

In the case of peroxides, and Diak™ No. 7, the two materials can be preblended with the fillers, or the Diak™ No. 7 may be preblended with the fillers and the peroxide added after the fillers.

The best dispersion of acid acceptors is obtained by blending them with the fillers before adding them to the mixer.

Do not use stearate (i.e., zinc stearate) type processing aids. These types of materials will cause premature curing with bisphenol cure systems. Process aid(s) should be added late in the mixing cycle, or during the last sweep. If added too early in the mix, they tend to reduce the shear that is required for good mixing of the other ingredients.

Internal Mixing Troubleshooting Guide

Poor Dispersion

- If the batch fails to go together, the load factor may be too small for the mixer and/or the wrong chamber size is being used in the calculation of the batch size. Check to make certain that the batch is properly sized for that particular mixer. Also make certain that the batch is not too large for the mixer. A load factor between 70 and 72% is generally recommended.
- Make certain the processing aids are added late in the mixing cycle.
- Check the dust seal lubrication system to make certain it is not over lubricating the bearings, and thus leaking into the mixing chamber and contaminating the stock.
- Check the ram pressure. It should be pressurized high enough to force the material into the mixing chamber of the mixer, but low enough to allow movement during mixing. Some slight movement of the ram allows the batch to turnover during the mixing process, thus preventing dead spots in the chamber.

Poor and/or Changed Scorch Characteristics

- Check the rate at which the batch is heating up. If heat is generated too fast, the polymer's viscosity may become too low to provide effective shear for adequate dispersion. Also, the stock may precure before adequate dispersion is obtained. To prevent this, one or all of the following may be required:
- Increase the amount of cooling water flow.
- Clean the water channels of buildup to improve the cooling efficiency.
- Reduce the rotor speed.
- Check the differences between the mixer's gauge temperature and the actual (probe) temperature.
- Make certain the mixing chamber is clean. Contamination from other compounds may affect the cure rate and state.
- Acid acceptors and many mineral fillers are hygroscopic. Introducing moisture into a bisphenol cure FKM compound will decrease the scorch safety. All moisture sensitive materials should be stored in tightly closed containers, in a cool dry location, especially during the months of high humidity. Further, it is a good practice to minimize the number of times a container is opened and closed, and use pre-packaged/weighed units of the chemicals.
- Care must be taken to prevent moisture from condensing on the stock during storage or after being removed from storage.

Extrusion

The extrusion characteristics of compounds made with Viton™ are a function of the compound viscosity, type and amount of processing aid, extruder conditions, as well as the size and shape of the extrudate. In order to obtain smooth extrudates, the incorporation of 1.25–1.50 phr of an extrusion aid, such as VPA No. 2 or carnauba wax, is recommended. It is very important to recognize that compounds of Viton™ extrude best over a comparatively narrow temperature range, and good temperature control is essential for consistent results. Simple screw designs may be effective under some conditions but the best performance will come from mixing/plasticizing configurations.

Compounds based on low viscosity Viton™ types may give easy extrusion, but may also exhibit collapse after extrusion or during the early stages of curing. The importance of this feature will depend on the shape of the extruded profile. The best results are obtained from enhanced rheology copolymer precompounds of medium viscosity, e.g., blend of A-201C/A-401C or Viton™ A-331C.

When extruding preforms of compounds made with Viton™, a relatively cool barrel and screw will increase the stiffness of the stock, thus minimizing the tendency to entrap air. The stock should be free of moisture and at ambient temperature when being fed to the extruder. If the compound is taken from refrigerated storage, water or moisture will sometimes condense on the stock. This moisture must be removed before the stock is fed to the extruder. Moisture will manifest itself as blisters on the surface of the extrudate.

One of the most critical factors affecting the smoothness of the Viton™ extrudate is the extrusion speed. Extruding at too high a speed can cause melt fracture. The critical conditions for the onset of melt fracture depend on the

die entry geometry, die dimensions, temperature and the polymer. Typically, the onset of melt fracture depends on the nature of the polymer and its macromolecular characteristics.

Another, equally important factor in obtaining smooth extrudates with Viton™ is the die temperature: bisphenol cure compounds, in particular, require a hot die—between 115–125 °C (239–257 °F).

Screw Extruder Setup	
Stock Temperature	15–27 °C (60–80 °F)
Screw Temperature	27–90 °C (80–195 °F) Typically the screw is cool at the feed section and gradually increases in temperature as it nears the head of the extruder.
Screw Speed	10–25 rpm
Barrel Temperature	27–71 °C (80–160 °F)
Head Temperature	65–85 °C (150–185 °F)
Die Temperature	115–125 °C (239–257 °F)

Calendering

IMPORTANT: Do not use silicone-treated release paper as liner material when calendering peroxide-cure types of Viton™. Peroxide-cure types of FKM have been known to exhibit a significant degree of adhesion to these types of paper.

The degree of quality obtained in calendering sheet stock compounds of Viton™ depends upon many factors, but largely on the viscosity of the compound at the calender. For best results, every effort should be made to ensure that the compound to be calendered is uniform in dispersion, viscosity, temperature and volume of flow.

Typically, compounds of Viton™ tend to calender smoothly and without roll sticking, using the following suggested conditions:

The temperatures outlined below are only suggested conditions. The actual temperatures employed will depend on the compound formulation (i.e., with or without process aids), the desired sheet thickness, compound viscosity, and the speed of operation.

Stock Warm-Up and Calender Feed

The warm-up mill temperature should be within –5 or +10 °C (–10 or +20 °F) of the top roll temperature of the calender. A sufficient amount of stock should be loaded on the mill to complete one full pass. Particular care should be taken not to over load the mill with too much stock. Once the stock is banded, it should be cut from alternate sides of the mill to ensure a uniform temperature (viscosity). If more stock is required to feed the calender, the warmed stock should be moved to one side of the mill and the fresh stock added to the other side. Once the fresh stock has been warmed to the proper temperature, it can then be moved to the feed side of the mill and blended with the other material. Only uniformly warmed stock should be fed to the calender.

Uniform feed into the calender nip is important for producing air-free calendered sheets, with good surface integrity. The amount of feedstock in the nip should be kept as small as possible. Large banks of stock will have large temperature variations, due to surface cooling. This results in changes in the stock viscosity and variations in the smoothness and thickness of the sheet.

The calender should be continuously and evenly fed across the width of the rolls, to maintain a uniform bank. Feeding the compound into the calender nip with large amounts of compound should be avoided.

After calendering, it is good practice to allow the wrapped stock to stress relax in the liner for approximately 24 hr before further handling.

Curing Calendered Sheet

Calendered sheet, under proper tension (pressure) may be cured in steam or hot air. The curing time required generally depends upon the steam or hot air temperature, number of wraps on the curing drum and the insulating effect of the liner material. It is important that the core of the bundle reach cure temperature for an adequate amount time. When steam curing, it is best to slowly increase and lower the pressure, in order to prevent blistering. Also, it is best to protect the stock from direct contact with the steam by covering the outer layer of compound with an impermeable membrane (PTFE, or FEP sheet, for example).

To facilitate release of the cured sheet stock from the liner with a minimum of distortion, the liner should be stripped from the stock as quickly as possible after curing. To post-cure the sheet, it is best done by festooning in a forced hot air oven. A distance of 10 cm (4 in) between the festoons is suggested, to allow proper air circulation and even heat transfer. Sheets greater than 6.4 mm (0.25 in) should be step post-cured to prevent the formation of blisters in the stock.

Suggested 3-Roll Calendar Setup

	Temperature, °C (°F)		
Cure System	Top Roll	Middle Roll	Bottom Roll
Diak™ No. 3 (Diamine)	45–50 (113–122)	45–50 (113–122)	Cool or Ambient
VC-20/VC-30 or VC-50 (Bisphenol)	60–75 (140–167)	50–65 (122–149)	Cool or Ambient
Diak™ No. 7, No. 8 (Peroxide)	60–75 (140–167)	55–70 (131–158)	Cool or Ambient

*Different temperature ranges are recommended for compounds based on Viton™ that employ different crosslinking systems: diamine, bisphenol, and peroxide.

Calendering Troubleshooting Guide

Recommended Practices

It is extremely important to supply only the minimum amount of warm compound to the calendar nip that is necessary to maintain a continuous sheet.

Mixed compound must be pre-warmed, prior to being placed in the calender nip. The stock to be calendered should be warmed on a calender mill (minimum shear) to at least 45 °C [115 °F].

Internal release agents should be kept to a minimum, to prevent slipping on the calendar rolls. Carnauba wax and Struktol® WS-280 are effective internal release aids for use in calendering bisphenol-cure types of Viton™. The level of these process aids should not exceed 1.25 phr in the compound.

Rough Surface

If the surface of the calendered sheet is rough, check to make sure that:

- The compound is warm enough before placing in the calender nip
- A minimum amount of material is added to the nip at any given time, and that excess material is not allowed to "hang" at the nip, thereby losing temperature
- The calendar rolls are hot enough

Lacey Sheet

A "lacey" appearance (e.g., has numerous holes and tears, randomly occurring throughout the sheet) can be the result of excessively high roll temperatures and/or using a polymer that is too low in molecular weight to provide the necessary degree of green strength. If this problem occurs, it is recommended that lower calender roll temperatures be evaluated, and/or that a higher viscosity polymer be used in the compound.

Uneven Thickness Across Sheet Width

The higher the viscosity of the polymer, the greater will be the difficulty in maintaining consistent thickness across the width of the calendar rolls. The use of lower viscosity polymer, or increased roll temperature (if practical) should be evaluated, to alleviate this type of problem.

Slipping/Bagging on the Calendar Rolls

If the stock fails to grab, and feed consistently through the nip, it is probably the result of too high a level of internal process aid, and the levels of such materials may have to be reduced.

Molding

General Practices

Typically, compounds of Viton™ exhibit mold shrinkage of 2.5–3.5% after post-cure. All molds should close tightly and cleanly at the flash line. Also, the mold should be designed to give good venting of trapped air. Press platens should be free from distortion.

Typical molding conditions employ temperatures of 160–190 °C (320–374 °F), pressures in the 15–20 MPa (2175–2900 psi) range and the use of delayed press bumping, to allow air to escape. This procedure ensures good compound flow, forces out air and helps to minimize backrinding. The curing cycle time will be dependent on formulation, temperature, part size and configuration.

Mold temperature, as opposed to platen temperature or steam line pressure, should be checked with a calibrated pyrometer or by wax sticks prior to molding. This elementary precaution minimizes the risk of producing defective parts due to undercure. Further, to help minimize temperature lost and/or temperature extremes, the platen heaters should be periodically checked for proper operation and the press/molds should not be subjected to cold air-streams during the molding operation.

Mold staining and dirtying can be a problem with certain types of compound formulations, particularly those that are amine cured (i.e., Diak™ 1 and 3 cures). Careful attention should be given to the condition of molds and a routine inspection/cleaning should be made before the molds are used. Hard chrome plating of mold surfaces is recommended, to minimize mold fouling.

Mold release agents, both internal and external, facilitate the removal of parts and reduce mold fouling. Some suggested systems are detailed in the following Table. In the case of the external mold release agents, pre-conditioning the mold prior to actually making parts is often beneficial.

Compression Molding

Compression molding is the most common process for molding compounds based on Viton™ polymers.

It is recommended that Viton™ polymers having a viscosity of at least 30 (ML 1+10 at 121 °C) be used for compounds that are to be compression molded.

The primary factors effecting molding quality in this process are:

- Preform weight—preforms must be of a weight adequate to provide for complete cavity filling, flow (typically 6–10% higher weight than finished part)
- Preform density (must be dense, and free of trapped air)
- Consistency of pressure, across all cavities in the mold (to assure consistent shrinkage across all cavities)

Transfer Molding

Transfer molding requires the use of relatively low viscosity polymers and compounds. It is recommended that Viton™ polymers having a viscosity lower than 50 (ML 1+10 at 121 °C) be used for compounds that are to be transfer molded.

Transfer molding involves significant shear-heat generation, in transferring the compound form the pot, through the mold sprues, and into the mold cavities. Thus, in addition to providing for low compound viscosity, it is critical that the compound be formulated for adequate scorch safety, such that the compound will exhibit adequate flow (mold filling) prior to the onset of cure.

Four major factors affect mold filling in transfer molding processes, and can be altered singly or in combination to optimize mold filling.

Compound Scorch Safety (ts1, ts2 – onset of cure)—The onset of cure must be balanced against the time required to attain a state of cure adequate for demolding of the cured parts. Too "safe" a compound may require impractically long molding cycles.

Sprue Size—It is desirable to use the smallest size sprue hole practical, to minimize damage to the parts upon demolding, and the tearing of the sprue from the molded part. The sprue size must be large enough, however, to provide for sufficient flow of the compound, given the molding pressure that is available, and given the scorch safety of the compound.

Mold Temperature (165–175 °C [329–347 °F] recommended)—For the same basic mold cycle time, temperatures for transfer molding operations can typically be lower than those used for compression molding, because of the significant amount of shear heat that is generated in the compound, in the act of transferring material from the transfer pot into the mold cavities.

Injection Molding

There are two basic types of injection molding machines used in the rubber industry. One is called the "ram" or "piston" type and the other is called the "reciprocating screw" type.

The ram or piston injection-molding machine is an outgrowth of the transfer molding process. In operation, the rubber compound, in strip or extruded form is fed into a heated cylinder, where it is warmed to a predetermined temperature. The softened elastomeric compound is then forced by a hydraulic ram through a nozzle into mold runners and restrictive gates, into a heated mold cavity. In the mold, the material is shaped, cured and then removed.

In the case of the reciprocating screw injection-molding machine, the rubber compound can be in the form of pellets or strips, however, strips are most commonly used. The compound is fed into a heated barrel, where the material is heated and homogenized by a rotating screw. The rotation and reciprocation of the screw meters a predetermined amount of the compound into the forward portion of the barrel. This warmed material is then injected by the screw, acting as a ram, into the nozzle runners and gates, and into the heated mold. During the early stages of the cure, the screw is maintained in the injection position, at a predetermined pressure to consolidate the molding. Then, at a preset time, the screw starts to turn and moves back to a feeding position, where it prepares more material for the next injection, or shot.

Listed in the following table are some of the advantages of the "ram" type injection-molding machine versus the "reciprocating screw" type.

In general, if the compound is high in viscosity, scorchy, or requires good mixing, then a screw type is more desirable. If the mold design is complex (runner system), then a vertical ram or screw type machine may be more desirable. Typically if a floor space is at a premium, then a vertical machine is dictated. If it is expected that future work will involve high viscosity compounds, then a screw type of machine would be advisable. However, if initial cost is critical, then a ram type is dictated. At the same clamping tonnage, ram type injection-molding machines are generally lower in price. If a lower price per part is critical, then the screw type will generally give you shorter molding cycles. Regardless of the type of injection molding machine purchased, it should be designed for rubber processing.

The projected area of the parts to be made generally dictates the tonnage requirement of a machine.

As general rule, the clamping tonnage can be estimated as follows:

Required Clamp kg = ([Pa x NC] + Ra) x (281.1)

Where: Pa = the area of the parts, cm^2
NC = the number of cavities in the mold
Ra = the runner area in cm^2

The shot capacity requirements can be estimated as follows:

Shot Capacity = ([wt per part] x no. of cavities) + runner wt + 15% material wt retention in the barrel)

Definitions

Injection-Molding Process—A procedure whereby an accurately metered amount of plasticized rubber compound is moved under controlled temperature, pressure and time into a closed heated mold, where it is to be vulcanized.

Ram (Plunger) Type Injection-Molding Machine—A machine that utilizes a ram within a heated cylinder to force heat plasticized rubber compound into a closed, heated mold for the purpose of vulcanization.

Reciprocating Screw Injection Molding Machine—An injection molding machine that utilizes a screw within a heated barrel to plasticize and convey stock to a certain position, and then is used as a ram to force the stock through runners to a closed heated mold for the purpose of vulcanization.

Injection Time—The time required by the screw or ram to force the stock into the mold.

Hold Time—The time immediately following the completion of the injection cycle when a reduced pressure is maintained on the stock in the mold to consolidate the molding during the initial curing stage of the compound.

Cure Time—The time the compound is held in the mold, before the mold is opened.

Clamp Time—The sum of the injection time and the cure time.

Cycle Time—The time required to inject the compound, cure the part, remove the cured part(s) from the mold and close the mold again. It is the overall times between successive closing of the mold, and may include cleaning of the mold surface, if necessary.

Injection Pressure—The pressure exerted on the plasticized stock, in the barrel or cylinder, to force it into the mold cavities.

Hold Pressure—This is the reduced pressure on the injected stock, used to consolidate the molding during the initial curing stage.

Clamping Pressure—This pressure is usually expressed in tonnage. It is the amount pressure exerted on the material in the mold, by the mold, during the curing or vulcanization cycle.

Back Pressure—The pressure exerted on the compound in the barrel, by the screw, to compact of the stock before injection shot.

Recommended Practices for Injection Molding

The following are typical operating conditions for injection molding compounds of Viton™:

	Ram-type	Screw-type
Temperature, °C		
Barrel		
Feed zone	80–90	25–40
Middle zone	80–90	70–80
Front zone	80–90	80–100
Nozzle	90–100	100–110
Nozzle extrudate	115–120	115–120
Mold extrudate	165–170	165–170
Mold	205–220	205–220
Pressure, MPa		
Injection	(Sufficient to give an injection time of 3–5 sec. Typically this pressure may range from 14–115 MPa.)	
Hold pressure	—	½ injection pressure
Back pressure	—	0.3–1.0 MPa
Clamping pressure	Max.	Max.
Time, sec for thin pieces (<5 mm thick)		
Injection	3.5	3–5
Hold	—	10–15
Cure	45–60	30–45
Clamp (injection and cure)	48–65	33–50
Cycle	58–76	43–60
Screw	—	40–60 rpm

Compounding for Injection Molding

An injection-moldable compound of Viton™ should have good scorch safety characteristics, to withstand the preinjection plasticization temperature in the barrel without scorching. It should flow easily through the narrow nozzle, runners and gates, to allow fast filling of the mold cavities. Its shear/viscosity relationship should be such that the heat build-up that occurs during the passage from the barrel to the mold is controllable. On reaching the hot mold, the compound should then cure quickly. In general, many of the compound characteristics that are favorable for transfer molding are applicable to injection molding.

Although no elaborate stock preparation procedures are required for injection molding compounds, the following recommendations are advisable:

- Care should be taken to prevent the incursion of moisture into the stock. Moisture in compounds made of Viton™ can decrease the scorch safety and cause blisters and internal porosity. It is advisable to make certain that all the ingredients used in the stock are protected during storage to prevent the absorption of atmospheric moisture.
- The actual viscosity, scorch characteristics and cure rate required will depend on the part size, mold design, machine specifications and machine operating conditions.
- The preform that is fed into the barrel should contain a minimum amount of trapped air. This is especially important when using ram type injection-molding machines. With the screw type of injection molding machines, much of the air is able to escape back down the flights of the screw. (See the "Handling Precautions for Viton™ and Related Chemicals" technical information bulletin on the "diesel" effect and other related precautions.)

- The compounds being prepared for injection molding should be exposed to as little heat history as possible prior to actual usage. This would include heat generated during mixing, milling, extruding, storage, etc.
- Each batch should be handled in a consistent fashion to reduce batch-to-batch variations, thus insuring uniform moldings and less rejects or poor quality parts.

Troubleshooting Injection Molding Problems

There are no set rules for trouble shooting injection molding problems, since they may be due to a combination of things. Each problem should be handled on an individual basis and analyzed with regard to:

- The compound being used, and the preparation of the stock
- The part being made
- The injection molding machine and it's operation
- The mold

General Guidelines

- Use a systematic approach when investigating or analyzing a problem
- Examine the compounding parameters carefully for all preparation variations. Also compare the control test results to previous records
- Check the equipment for mechanical failure and/or wear
- Check the machine operation. Go through the cycles; check the temperatures and pressures at each portion of the molding cycle. Also, compare the results to previous records
- Record the cause and corrective action for future references

Troubleshooting Guide—Problems and Corrective Actions

The following is a list of some of the more common problems encountered when injection molding rubber. It is by no means an all-inclusive list. One must also keep in mind that many factors are interrelated and that changing one parameter in the operating conditions may often have an affect on other molding conditions or molding quality.

Problem—Air Entrapment in the Mold

Any air trapped in the mold cavity will prevent the mold cavity from filling properly. To avoid trapping air:

- Make certain the feed stock, especially for ram type machines, is free of entrapped air
- Provide sufficient backpressure at the nozzle to adequately compress the stock in the barrel or cylinder
- Increase the injection time
- Lower the injection pressure, and/or
- Make certain the mold is properly vented

Problem—Backrinding

This type of problem may involve the compound acceleration system (speed of curing), too long a "hold time," too high a hold pressure, and/or too high a mold temperature. In addition to backrinding, "sink backs" may occur at the gates.

- Decrease the level of the accelerator system or formulate a slower cure rate
- Reduce the mold temperature
- Decrease the hold time
- Decrease the hold pressure

Problem—Blisters

Blisters can originate from a variety of causes, such as entrapped air in the feed stock, entrapped moisture in the compound, incorrect stock viscosity, too large a nozzle, too short an injection time, inadequate mold venting and/or the volatilization of one of the compound's ingredients.

- Make certain the stock has not been contaminated with moisture, either from the compounding ingredients or improper storage
- Check the feedstock for trapped air. Remill or extrude at a lower temperature if necessary
- Adjust the viscosity of the stock to insure good compaction in the barrel or in the cylinder. Usually a higher viscosity will help
- Decrease the barrel or cylinder temperature to increase the backpressure, thereby obtaining better compaction of the stock
- Eliminate all compounding ingredients that may volatilize (decompose) or produce a gaseous product at the processing temperature
- Check the nozzle extrudate for air bubbles. Decrease the nozzle size to increase the backpressure if necessary
- Decrease the injection pressure

- Increase the injection time
- Increase the hold time and/or pressure
- Increase the cure state of the article
- Make certain the mold is properly vented

Problem—Distortion or Rough Surface

Scorched stock, too long an injection time, too hot a mold or an undersized runner and/or gates may cause distortion of the molded article after removal from the mold.

- Use fresh stock, the old stock may have precured
- Recompound for better stock flow or lower viscosity
- Decrease the temperature of the stock entering the mold by:
  - Decreasing the compound viscosity by filler or polymer modification
  - Decrease the cylinder temperature
  - Decrease the injection pressure
  - Decrease the injection time
  - Increase the nozzle size
  - Increase the gate size
- Reduce the mold temperature

Problem—Excessive Mold Flash

This problem is usually associated with too low a stock viscosity, too high an injection pressure, too long an injection time, too large a shot size, or a poor fitting mold. To eliminate this problem:

- Increase the stock viscosity
- Decrease the injection pressure
- Decrease the injection time
- Make certain the shot size is not too large
- Check the alignment of the mold halves
- Increase clamping pressure

Problem—Excessive Nozzle Flash

Nozzle flash is usually due to a worn nozzle or nozzle bushing surface, too large a nozzle, too high an injection pressure, or too low a compound viscosity.

- Check the nozzle and/or nozzle bushing surfaces for wear, and refinish them if necessary
- Decrease the injection pressure
- Increase the stock viscosity
- Decrease the nozzle size
- Decrease the back pressure

Problem—Long Cure Cycles

Too low a barrel or mold temperature will cause long cure cycles. Also, an inadequately formulated compound will result in a long cure cycle.

- Adjust the compound cure system to increase the rate of cure
- Preheat the reserve stock (ram machines only).
- Increase the temperature of the stock entering the mold by:
  - Increasing the compound viscosity
  - Increase the barrel or cylinder temperature
  - Increase the injection pressure
  - Reduce the nozzle and/or gate size

Problem—Poor Knitting

Poor knitting may be due to excessive mold release (lubricant), too high a mold temperature, too fast a cure rate, or inadequate stock flow.

- Reduce the amount of internal process aid(s) and/or externally applied mold lubricant
- Lower the compound viscosity, reduce the rate of cure, or increase the scorch safety
- Lower the mold temperature

Troubleshooting General Molding Problems

Backrinding

Backrinding appears as very rough edges on a molded part, caused by the expansion of the part upon demolding, at the nearest area of relief, usually at the parting line of the mold cavity.

Causes and Corrective Measures—Stock viscosity is too high. Reduce the compound viscosity by using a lower viscosity polymer.

Inadequate mold flow—Use a bump cycle on the mold, or change existing bump cycle by lengthening the time when the mold is bumped.

Compound is too scorchy—Reduce mold temperature and/or increase scorch safety of the stock.

If the scorch safety has changed, determine the reason for the change and/or use fresh stock.

Blisters

There are a number of different types and causes of blisters. The appearance, relative size, and number of blisters in molded parts can be quite different depending on the actual cause of the problem.

Blisters Caused by Undispersed Particles

Appearance—Usually a relatively small number of blisters per part, small in size, and usually visible only near the surface of the part. This type of blister is readily identified by the presence of an undispersed particle inside the blister. Such particles usually are readily seen, by cutting mixed stock, preforms, or molded parts with a sharp knife. Undispersed particles large enough to cause blisters in molded parts will be visible to the naked eye.

Causes and Corrective Measures—Blisters of this type result from poor dispersion. Make certain the stock is refined prior to making the preforms. Review mix procedures to maximize the dispersion of all powdered ingredients. Check compounding ingredients for the presence of hard, undispersable particles/grit, or contamination.

Blisters Caused by Contamination of the Compound with a Different Compound, Based on a Nonfluorinated Rubber

Appearance—These types of blisters are less obvious than those made with undispersed lumps of light colored acid acceptors or mineral fillers. Typically these types of blisters may appear as a crack, or a void inside of a molded part. If the contaminant is near the surface of the molded part, it may pop out upon demolding, thus leaving an irregular-shaped void in the part.

Causes and Corrective Measures—Before mixing or processing (by any method) a compound based on Viton™, make sure to clean the equipment to remove all non-fluoroelastomer based compound. This also includes such contamination as oils, from all equipment involved in the processing of the stock, including mixers, preformers, and molding operations.

Blisters Caused by Trapped Air

Appearance—Trapped air usually appears as a relatively small number of blisters, randomly located throughout the part. Depending on their size, individual blisters may have a spongy appearance, but have no evident particle or contaminant within the blister itself.

Causes and Corrective Measures—In low viscosity stocks, and particularly in the case of using a ram extruder to make preforms, it is possible to trap air in preforms. If the preform is not subjected to adequate flow/squeeze in the molding operation, the air will not be forced out, and will create blisters. Make sure all molding preforms are dense, and do not contain pockets of trapped air. A breaker plate and screen pack at the extruder head may be used to increase pressure to a degree that is sufficient to force air out of the preform at the die entrance. Low preform weight can also result in trapped air. Make certain the preform weight is sufficient to provide adequate stock flow and pressure in the mold cavities.

Blisters Caused by Inadequately Dispersed Process Aid

Appearance—Blisters of this type tend to have a "wet" look on their interior surface.

Causes and Corrective Measures—Pockets of undispersed process aid, such as waxes, may vaporize at molding temperatures, thereby forming blisters. Make certain that adequate mixing time is provided for the complete incorporation and dispersion of all process aids used in the stock.

Blisters Caused by Entrained Water

Appearance—This type of blister usually appears as a number of small-sized blisters per part, with no evident particles or contaminants within the blister itself.

Causes and Corrective Measures—Stocks that are stored in a cold room, and which are then brought onto the shop floor in humid weather can collect significant amounts of moisture on the surface. Subsequent processing may cause this surface moisture to be trapped within the compound.

When a compound is stored under cold conditions it must be covered with plastic, and when retrieved for additional processing, the plastic must be removed only after the compound has come to room temperature. There have also been cases in which internal mixer rotors and extruder screws developed cracks, thus allowing cooling water to be introduced into the compound.

If water is thought to be the cause of blistering, and condensation from cold storage of compound is proven not to be the cause, pressure-test internal mixer rotors and extruder screws for possible cracks/leakage of cooling water or steam.

Blisters Caused by Poor Dispersion of Accelerator or Curative

Appearance—This type of blister typically shows up as small areas of very small blisters. The blistered area will typically have a sponge-like appearance and, when probed, will exhibit obvious signs of undercure.

Causes and Corrective Measures—Poor dispersion of either accelerator or curative will result in an inadequate state of cure in the immediate area of poor dispersion. During molding, these areas will "blow," resulting in blistered or sponged areas. Good dispersion of the accelerator and crosslinking agents is critical in avoiding this type of blister.

Blisters Caused by Undercure

Appearance—Parts that are undercured typically exhibit extensive blistering, throughout the entire part. The interiors of these blisters are spongy in appearance, and if probed with a metal pick, exhibit obvious signs of undercure.

Causes and Corrective Measures—Too low a mold temperature, too short a time in the mold, or grossly under-weight preforms will result in extensive blistering. The corrective action may require one or more of the following:

- Increase the mold temperature
- Increase the molding time
- Increase level of accelerator in the compound formulation
- Make certain the preform weight is adequate

Sponged Areas, Splits, Fissures After Post-Curing

Appearance—This type of molding defect occurs most frequently in parts that have a cross-sectional thickness greater than 5 mm (0.20 in). Such parts may appear to be without flaws when initially demolded, but will exhibit internal voids, or fissures, after post-curing.

Causes and Corrective Measures—Areas having a sponge-like appearance in the interior of parts are usually the result of an inadequate state of cure. Provide sufficient time in the mold to allow complete transfer of heat through the bulk of the part to its interior. For example, parts that are 6–9 mm (0.25–0.35 in) thick may require 15–20 min at 175 °C (347 °F) to fully cure, while a part made using the same compound that is less than 2.5 mm (0.10 in) thick, may need only 2.5 min to be fully cured.

Internal fissures or splits are significantly different in appearance from blisters or sponge. Instead of being rounded, discrete voids, fissures and splits are, as the names suggest, distinctly planar and depending on the cause, their interior surfaces can be very rough in appearance. Fissures typically are caused by either one or two different problems.

Fissures are most commonly seen in post-cured parts that are thicker than 5 mm (0.20 in). Fissuring, or "blowing" is typically the result of the violent escape of volatiles trapped within a part during post-cure. Parts thicker than 5 mm should always be oven "step-post-cured," and the cure cycle should start at 90 °C (194 °F) for 2–4 hr. This will allow any volatiles that are trapped inside the part to escape gradually without "blowing" the part.

If the preforms are built up from multiple plies of stock, and if too much internal process aid is used in the formulation and/or there is insufficient preform weight or mold pressure to provide for adequate compaction (knit) of the layers, the layers may separate, either upon de-molding, or after the post-cure cycle. Multiple plies or layers of stock can be successfully used to make preforms for thick molded parts, but care must be taken to insure that:

- The surfaces of all layers are clean, and free of process aid, which may "bleed" out of the stock while sitting at room temperature
- An adequate amount of preform weight, and mold clamping pressure is used, to insure sufficient compaction and knit of the individual layers.

Knit Lines/Flow Lines

Appearance—Knit, or flow lines, appear as small, groove-like lines in the surface of the molded parts. These flaws are caused by inadequate flow and knitting of the compound.

Causes and Corrective Measures—Knit lines problems are likely to occur in molding applications having the following conditions:

- Where the compound has to flow a relatively long distance to meet with another portion of the part flowing from the opposite direction
- Where the compound only has to flow a short distance, but under very low shear and pressure

One of the two principal causes of knit lines, under long flow conditions, is the onset of cure before completely filling of the mold cavity. Premature cure (scorch) will create a surface that is high in viscosity, thus decreasing the compound's ability to completely fill the mold cavity. Further, this high viscosity interface will also interfere with the compound's ability to knit.

A common cause of knit lines is the use of excessive mold lubricant (release) or internal process aid. In molds where an excessive amount of mold lubricant is used and where

a long flow path is required, the compound can wipe the excess lubricant from the mold. In doing so, the lubricant can buildup in front of the compound and prevent bonding and/or knit. Internal processing aids are effective because they are incompatible with the fluoroelastomer compound. Because of their incompatibility they tend to bleed to the surface of the stock. If an excessive amount of internal mold lubricant is added to the compound, it may bleed to the surface of the part during the molding, and be pushed along in front of flowing stock or coat the compound surface. The surface coat and/or accumulation of the mating surfaces may prevent their knitting.

These knit lines which are the result of excessive external or internal mold lubricant will exhibit decidedly "wet" looking surfaces, and, in severe cases, the presence of excessive mold lubricant can be determined by a wet slippery feel in the area of the knit line. Care should be taken to avoid over-spraying molds. The level of internal mold release agents will be restricted by the type and amount of filler in the compound, the molding process, and the size and design of the mold (especially as regards the stock's flow path).

A compound designed for compression molding a small, simple-shaped part, with little flow involved will accommodate a greater amount of internal lubricant than, for instance, a compound which is to be injection molded into a cavity that involves a long flow path, through runners and gates.

Non-Fills

Appearance—Non-fills usually appear as small, depressed areas on a molded part

Causes and Corrective Measures—Like knit lines, the following may cause this type of flaw:

- Premature curing (scorch) of the compound
- Insufficient preform weight
- Inadequate cavity pressure, and/or
- Uneven mold closure (resulting in varied pressures between mold cavities)

If non-fills occur, check for premature scorch by molding the part at a lower temperature and by running a Mooney scorch test.

Mold temperature or ratio of accelerator to crosslinker (VC-20-VC-30 ratio) can be reduced, to provide for increased scorch safety.

Verify that the preform weight is adequate for the finished part weight and for sufficient flash.

Make sure that all mold cavities are of the same height and receive the same clamping force.

Tearing

Appearance—Tears can occur in molded parts in varying degrees. They can range from small cracks in the surface, or partially separated corners or edges of molded parts, to total separation or breakage of a molded part into two or more separate pieces.

Causes and Corrective Measures—Tears experienced during demolding are usually caused by inadequate amount of hot elongation necessary to extract the part from the mold. It should be noted that elongation values measured at room temperature do not adequately reflect how well a compound will perform at demolding temperatures. To gain a better idea of how the compound will perform under demolding conditions, tensile properties should be measured at or above the demolding temperature, before post-curing. For example, a compound with a 120-130% elongation, measured at 177 °C (350 °F), for instance, is usually adequate for demolding valve stem seals, or shaft seals. Higher elongation values may be required for more complex shapes.

Poor release of the part from the mold surface can create tears that, given good mold release performance, would not otherwise occur. Good mold release is critical in terms of preventing demolding tears from occurring.

Poor mold flow/filling and/or knit lines can also cause tears. See the suggestions for knit lines and flow problems for corrective actions.

The tear resistance of fluoroelastomer vulcanizates is very dependent on the tensile strength and

elongation properties of the compound. Tensile and elongation values decrease, with increasing temperature. Thus lower mold temperatures may significantly reduce part tearing, providing the mold release is adequate. However, increasing the accelerator level of a compound or a faster-curing version of the polymer type may be desirable to avoid any penalty in molding cycle time due to lower molding temperatures.

The elongation at break of a vulcanizate is highly dependent on the amount of cross-linking agent that is added to the compound. Reducing the level of curative (crosslinking agent) will have a significant impact on improving hot elongation at break and, consequently, demolding tear.

Tearing can also be caused by a foreign inclusion, and/or poor dispersion, which can create stress risers. These stress-risers are weak points in the part whereby tear can be easily initiated due to the concentration of force at that location, when extracting the part form the mold. Check for foreign inclusions and poor dispersion. Further, mold cavity design can also have an effect on producing stress-risers. Sharp corners or angles can also create stress-risers and make demolding without tearing very difficult. Where possible, all inserts and mold cavities should have the largest radiuses allowable, to distribute the demolding forces.

Mold Shrinkage

Variable Shrinkage

When variations in shrinkage of finished parts occur, and the cause cannot be traced to any specific, consistent difference, such as batch to batch, or (consistent) variation between mold cavities, then the post-cure oven should be looked at carefully as a possible source of the problem. If the post-cure oven is not set up to provide for proper air flow and internal air circulation, and if it is near its rated limit for high temperature capability, the temperature within the oven can vary as much as 50–75 °C (122–167 °F), from the center of the oven to the various corners. Such variations in oven temperature will cause variations in shrinkage.

Variable Shrinkage Within Molding Heats

Variable shrinkage in multiple-cavity molds can result from number of causes. When investigating variable shrinkage, it is extremely valuable to identify and record the shrinkage for the individual cavities. Often, a pattern or trend can be identified if the following events or factors are at fault in creating the shrinkage variation:

If specific cavities consistently provide different shrinkage compared to other cavities, this is likely the result of either, uneven heights of flash groove cut-off, a cocked platen, or the result of a mold being placed off center of the molding platen or ram. In the case of uneven platens, or off-center mold placement, shrinkage between cavities typically will differ by a consistent, increasing amount, in going from the area of highest clamp pressure to the area of lowest pressure.

Variable Shrinkage Between Molding Heats

When variations in shrinkage occur between consecutive heats, and:

- Occur within the same batch of mixed compound
- Occur using the same batch of mold preps, and
- Are noted between heats which have been post-cured in the same oven, using the same post-cure cycle

The cause of these variations is most likely due to variations in mold temperature between heats. Changes in mold temperature, sufficient to effect differences in shrinkage, can result from having the mold out of the press for different periods of time between heats. Or, significant variations in the mold "loading and demolding cycle" can also affect the temperature. How long the mold is out of the press between heats, or significant "breaks" in the normal cycle of loading, curing, and demolding, as in the case of the first heat being run after a lunch break during which the mold sat unused in the press.

Changes in molding pressure can also cause differences in shrinkage between batches, but it is unusual for presses to be at fault in this manner. This type of shrinkage variation can occur, however, if multiple deck presses are not loaded in a consistent manner. For example, if a press is typically run with two molds, it should always be run this way, and both molds should always be loaded with preforms.

Variable Shrinkage—Batch to Batch

If differences in shrinkage occur between different mixes of compound (and the post-cure ovens have been ruled out as a source of the problem) this will most likely be the result of differences in scorch and/or viscosity between batches. Such differences can themselves result from differences in mixing history and/or the age of the mixed compound.

Process aids can be another possible source of differences in shrinkage between batches, particularly for process aids that are relatively fugitive. In these cases, it is important that heat histories of the batches be as nearly equivalent as possible. That should include the mixing cycles and the sheet-off cycle, and any subsequent preparation of the compound. Variations in mix temperature can result in significant differences in the residual levels of the process aid, which will directly impact the final shrinkage values of the cured parts.

Adhesion

Good adhesion can be obtained between compounds of Viton™ and to other substrates if the surfaces are properly prepared, and if a suitable primer adhesive is used. For detailed information on adhering Viton™ to metal substrates.

Cured compounds of Viton™ can readily be bonded to itself, but, in general, the best bonds are achieved between vulcanizates that have not been oven post-cured.

In order to obtain a bond between two pieces of vulcanized Viton™, the surfaces that are to be mated should be roughened slightly with sandpaper and wiped with a solvent-soaked cloth. Isopropanol, ketones (acetone, methylethyl ketone), or low molecular weight acetates (ethyl acetate) may be used to remove any traces of grease or oil, and to"pre-swell" the surface, allowing better penetration of the adhesive.

"Skive" or angled cuts are strongly recommended, versus "butt" splices, particularly if the bonded splice will be subjected to compressive stresses while in service. Angled cuts on both ends of the pieces, to be bonded together, will provide a considerably larger surface area for adhesion than a "butt" splice. The "skive" cuts should be made such that the plane of the resulting splice is parallel to any compressive force to which the finished part will be subjected.

Adhesives

There are a number of adhesives available that have been expressly designed for bonding vulcanized fluoroelastomer to vulcanized fluoroelastomer. These adhesives are based on fluoroelastomer compounds dissolved in solvent(s) and are commercially available from the following companies:

Eagle Elastomers, Stow, OH
Telephone No.: (330) 923-7070

Pelseal Technologies, Bensalem, PA
Telephone No.: (215) 245-0581

Thermodyn Corporation, Sylvania, OH
Telephone No.: (800) 654-6518

In addition, cyanoacrylate adhesives and two-part epoxy adhesives may be used for this purpose. In the case of two-part epoxies, thinning the adhesive with xylene or toluene provides bonds that are more flexible than those obtained using the undiluted epoxy adhesives.

General Practices

Bonding Viton™ to Metal

High mold cavity pressure is extremely important in bonding, especially for good and consistent bonds to metal inserts. Depending on the pressure capability of the press, the number of cavities in the mold may have to be restricted or reduced, in order to increase the effectiveness of the available platen ram pressure.

Silane-based primers/adhesives are very sensitive to moisture, and metal inserts treated with silane adhesives should be used within several days of being coated. In seasons of high humidity, prepared inserts should be kept covered and/or used within a day or two of being coated.

The thickness of the primer/adhesive layer is critical to obtaining good bonds. Too thick a layer will not provide strong bonds, and therefore most silane-type adhesives must be thinned with a compatible solvent. The percent dilution of silane adhesives will vary, depending on a number of factors. At least two different concentrations should be tried, before settling on, or discounting any particular primer. For example, there have been cases where a 100% Chemlok® 607 treatment provided no bond, but a 25% Chemlok®/75% ethanol mixture resulted in 100% stock tear bonds. In diluting silane-based adhesives, it is critical that dry solvents be used.

Silane types of adhesives/primers are particularly suited for the dipping processes. Dipping allows the treated inserts to dry quickly and tack-free. Spray application is less preferable, as the spraying allows for ambient humidity to have a greater effect on the application. Applying the primers by brush is the least preferred method, as it is difficult to obtain 100% coverage of a consistent thickness.

Wiping of the adhesive by the Viton™ compound as it flows around or on to the insert can occur during the mold filling process. Prebaking the treated metal inserts for approximately 5–15 min at 125–150 °C (257–302 °F) can reduce this problem. Lower viscosity compounds will exhibit less tendency to wipe the adhesive from the insert than higher viscosity stocks.

Compounds with very short scorch times will typically be more difficult to bond to metal than identical compounds having a longer scorch (ts2) time.

Oven post-curing of molded parts must take into account the thermal stability of the adhesive. Typically, post-cure temperatures should not exceed 200 °C (392 °F). Some primers have higher thermal stability than others, check with the manufacturer to be certain of the upper temperature capabilities.

The compound formulation should not contain excessive amounts of waxes or other internal process aids that can migrate out of the polymer under heat and shear. Similarly, care must be taken to assure that external mold spray does not "land" on primer-treated inserts.

Bisphenol-cured Viton™ compounds that are to be bonded to metal should be formulated with low (2–3 phr) levels of calcium hydroxide. Typically, Viton™ recipes call for combinations of 3 phr magnesium oxide and 6 phr calcium hydroxide. Using levels of 6–8 phr magnesium oxide and 2–3 phr calcium hydroxide will provide significantly better bonding to metal. In addition, this combination of metal oxides will provide essentially the same cure rates, with slightly better tear resistance, and no sacrifice in tensile properties, or compression set. Also, the use of 15–17 phr of a low activity magnesium oxide, in combination with 2 phr of calcium hydroxide has been found to be particularly effective in promoting metal bonding with bisphenol-cured compounds of Viton™.

Compounds filled with mineral fillers typically provided better adhesion to metal than carbon black-filled stocks. Furnace blacks are noticeably poorer than Thermal grades in this regard.

Various metals will also differ in the ease with which compounds of Viton™ to metal bonds can be obtained.

This is due in large part to how well the surface can be roughened. A relative rating of metals and the relative ease of bonding is as follows:

Steel > Stainless Steel > Brass > Aluminum

Steel can be sand or grit-blasted to create a roughened surface, or it can be treated with a phosphatizing agent. Both techniques will create roughened surfaces, thereby increasing the surface area for better bonding.

Stainless steel must be "pickled"—etched with hydrochloric acid. The end result, like phosphate coating, is to increase the surface area.

It is more difficult to obtain a roughened surface on brass or aluminum, since they are softer metals, however, brass may be etched with ammonium persulfate.

Types of Primers and Adhesives

For multicavity, small part production, silane-type primers are preferred. The silane-type primers are preferred for these types of productions due to the inherent capabilities of the primer to coating large numbers of metal inserts without the tendency for the primed parts sticking together. The following are representative silane-based primers known to be effective in bonding compounds of Viton™ to metal:

Silane Types

Chemlok® 8116—Water based. Peroxide cures.

Chemlok® 5150—Methanol based. Peroxide, bisphenol, and diamine cures.

Chemosil® 511—Ethanol based. Bisphenol cures.

Chemosil® 512—MEK based. Bisphenol cures.

Megum™ 3290-1—Ethanol based. Bisphenol cures.

Megum™ W 2525—Water based. Bisphenol and peroxide cures.

Megum™ 3299—Ethanol based. Peroxide cures.

Monicas® VT-200—MEK/methanol based. Bisphenol and diamine cures.

Monicas® CF-5M—MEK/methanol based. Bisphenol and diamine cures.

Monicas® MP-204—Methanol based. Peroxide, bisphenol, and diamine cures.

Epoxy Types

Epoxy adhesives are effective for very difficult bonding applications. However, two-part epoxy-types of adhesives are not well suited for treating large numbers of metal inserts, since the adhesive coating does not dry tack-free in ambient air. Semi automated adhesive application lines can be set up to take advantage of the excellent bonding capability. These semiautomatic lines must either provide for maintaining separation between the coated inserts or include a pass of the coated inserts through an oven, sufficient to bake dry the epoxy coating. Typically a baking time of 5–10 min at 150 °C is required.

It should be noted that epoxy adhesives are particularly useful in the manufacture of relatively large parts, particularly where limited mold pressure is available. An example of this would be in the case of molding butterfly valve liners.

Cements/Tie-Coats Based on Viton™

For particularly difficult bonding applications, where epoxy type adhesives may not be adequate, cements based on compounds of Viton™ can be prepared. These cements have been proven to provide excellent bonding to metals. Such cements, like epoxies, are best suited for large parts, particularly when limited molding pressure is available. An example of a "tie-coat" formulation is shown below.

Tie-Coat Base Compound	phr
Viton™ A, or Viton™ B	100.0
Low-Activity MgO	15.0
MT Carbon Black	20.0
Diak™ No. 3	3.0

After mixing the above formulation, the compound is dissolved at 20–30 wt%, in methylethylketone (MEK), methylisobutylketone (MIBK), or a mixture of the two solvents. MIBK evaporates more slowly than MEK, and can serve to prevent a "skin" from forming on the coating. By using a mixture of MEK and MIBK the drying time is faster than using MEK by itself.

When the compound is fully dissolved, add 5 parts of Chemlok® 607 or Chemosil® 512 to 100 parts of the solution. The fully mixed cement will typically be stable for several days before showing signs of gelling. The cement should be applied to a grease-free, grit-blasted metal surface, and be allowed to fully air dry, before attempting to mold. The formulation as described is also an effective cement for bonding cured compounds of Viton™ to cured compounds of Viton™.

Oven Post-Curing

General Practices

Parts to be post-cured should be deflashed prior to post-curing, and care should be taken to assure that all loose particles of flash are removed from the parts. Small pieces of flash, falling onto the heating elements in the oven can be a source of fires. Tensile strength and compression set are the two physical properties most affected by oven post-curing. Fluid resistance, in terms of permeation rates or volume increase, is affected only very slightly.

Typically, 80–90% of the improvements that can be obtained in tensile strength and compression set resistance can be achieved within 1–4 hr at 232 °C (450 °F) or 250 °C (482 °F). In many cases, post-cure ovens are run for 24 hr simply for convenience in factory shift scheduling. Refer to the bulletin referenced above for more detailed information on the effects of oven post-cure cycles on vulcanizate properties.

In order to obtain the best possible resistance to compression set, compounds of Viton™ with bisphenol cure systems must be oven post-cured for at least 16 hr at 230–250 °C (450–482 °F).

Substantial improvements in tensile strength and resistance to compression set (compared to values for those properties with no post-cure), can be obtained in oven post-cures at temperatures as low as 150 °C (302 °F), for periods of time as short as 1–2 hr. Depending on the required tensile strength and/or compression set values that are required, post-cure cycles can be considerably shorter than the "standard" 24 hr that is normally used.

Fluoroelastomer parts should not be placed in a post-cure oven with other parts made from different elastomers. In particular, parts made with silicone rubber should not be post-cured with a fluoroelastomer. Post-curing VMQ or FVMQ and FKM parts together can result in the total loss/destruction of the entire batch of parts. This is due to the chemical interactions between silicone rubber and the small amounts of HF generated during the post-curing of the fluoroelastomer compound.

Post-cure ovens must be equipped to provide adequate (fresh) airflow. Generally a minimum of 0.14–0.20 m^3 (5.0–7.0 ft^3) of air per minute should be introduced on a continuous basis into an oven having interior dimensions of approximately 0.23 m^3 (8 ft^3).

Parts to be post-cured must be placed evenly throughout the oven. The parts must not be piled too deeply, thus preventing adequate air circulation around the parts. Adequate airflow around the individual parts is critical, not only to obtain consistent physical properties within a given batch of parts, but also to prevent oven fires.

Post-cure oven temperatures should be monitored on a regular (at least quarterly) basis. It is critical that an even temperature be maintained throughout the entire oven, and that temperature differences between the center of the oven and any corner area not exceed 5 °C (10 °F).

Molded parts having cross-sectional thickness in excess of 5 mm (0.20 in) should be "step" post-cured.

A recommended "step" post-cure cycle would start at 90 °C (195 °F) for 2–4 hr. The oven temperature can then be increased in increments of 25–40 °C each hour, until the desired final temperature is reached. The remaining time can be run at the final temperature. The "step" post-cure cycle is used to allow moisture that is generated in the press cure, to escape gradually.

Bonded parts (Viton™/metal inserts) should be post-cured at temperatures no higher than 200 °C (392 °F), in order to prevent degradation of the primer, and a subsequent loss of the bond.

Post-Cure Oven Fires

Refer to Viton™ bulletin "Handling Precautions for Viton™ and Related Chemicals."

There are several possible causes of post-cure oven fires, which have already been mentioned above, under General Practices. Some of the most obvious techniques to prevent oven fires are by:

- Preventing small pieces of molding flash from falling onto the oven heater elements
- Maintaining adequate rate/amount of airflow through the oven
- Never loading too many parts or by piling them too deeply into an oven, thus preventing the required airflow from reaching each individual part

High levels of hydrocarbon process aids, such as carnauba wax, or low molecular weight polyethylene in fluoroelastomer compounds can, over time, potentially cause oven fires. Such materials may be boiled out of the compounds during the oven post-cure cycle, and can condense or collect on the inside of the oven venting pipe. The resulting accumulated residue can then be ignited by a small flame source, such as a glowing piece of flash being blown up into the oven, and subsequently into the vent pipe. These residues may also cause fires by being heated to their ignition point.

Suggested Mold Release Systems for Viton™

Amine Cures	
Internal	0.5 parts low molecular wt polyethylene[1] or 1.0 part vegetable wax[2]
External	Polyethylene or silicone emulsions
Bisphenol Cures	
Internal	0.5–1.0 parts vegetable wax[2] 0.5–0.8 Nafol® 1822D 0.5–0.8 Struktol® WS280
External	Silicone or PTFE telomer emulsions
Peroxide Cures	
Internal	0.2–1.0 Struktol® HT290 0.2–0.8 Struktol® WS280 0.2–0.5 vegetable wax[2]
External	PTFE telomer emulsions

[1]e.g., AC Polyethylene 1702
[2]VPA No. 2 or carnauba wax flakes

For more information, visit Viton.com

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