A Practical Approach to Matching Fluorosilicone Compound Technology to Fabrication Processes and Equipment

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Abstract

Fluorosilicone compounds have never been more popular as they are today. These unique materials are most noted for applications that require low and high temperature performance in contact with jet and automotive fuels, many solvents and engine oils. This family of elastomeric materials has found increased use in many high volume automotive applications. As more and more fabricators attempt to manufacture products using fluorosilicone compounds, processing issues and acceptable part yields become an important but difficult hurdle. In addition, compound technology has become more flexible to meet demands of finished part economy using standard rubber finishing equipment. This paper will explore the practical approaches to understanding the material process requirements as they relate to various elastomer fabricating equipment and techniques. The part manufacturing (process) requirements will be used to determine a good approach in developing a fluorosilicone compound that not only meets end user performance requirements, but also can be easily processed on typical elastomer fabricating equipment.
A Practical Approach to Matching Fluorosilicone Compound Technology to Fabrication Processes and Equipment

*Michael A. DiPino, Application Engineering & Technical Service, Dow Corning Corporation*

The unique physical properties of fluorosilicone compounds (FVMQ) have never been in greater demand than they are today. Most noted for service in applications requiring contact with jet or automotive fuels, solvents and/or engine oils, fluorosilicones are finding increased use in a number of high-volume automotive applications. As more fabricators begin to manufacture products using fluorosilicone compounds, optimized processing and acceptable part yields become important but difficult issues. At the same time, compound technology has evolved to offer greater flexibility in meeting the requirements of standard rubber finishing equipment. For these reasons, it’s important to understand the practical approaches to material process requirements as they relate to elastomer finishing equipment and techniques. By considering the features of the specific process and equipment before compounding the material, the fluorosilicone supplier is better equipped to develop a compound that not only meets the end user’s performance requirements, but also processes efficiently on typical elastomer finishing equipment.

**Fabrication**

In the mixing and fabrication of fluorosilicone rubber, it’s essential to understand the relationship between current compounding technology and the various fabrication processes employed. Whether starting from standard fluorosilicone bases and developing compounds for internal use or buying a ‘ready-to-use’ material that is fully-compounded, pigmented and catalyzed, the more information at hand, the better. Even in the absence of mixing equipment suitable for fluorosilicone compounding, a fundamental understanding of the ingredients used and their potential process effects will always be helpful, especially when troubleshooting manufacturing floor problems. Even if troubleshooting efforts fail to resolve a specific or unique problem, a basic knowledge of fabrication and compound dependencies will help in defining the problem to your fluorosilicone supplier technical service resource, and aid in resolving the problem quickly and minimizing downtime.

Like many other endeavors, there is no single correct way to optimize manufacturing or solve a problem using fluorosilicone compounds in any one of the many processes currently employed. The recommendations made here may be used to help provide direction toward resolving a specific issue, but are not meant to cover all situations. Fluorosilicone rubber compounds can present unique challenges in matching particular compounding technologies to various fabricating processes. What works for an extrusion house may not apply to injection molding, for example. And calendering fluorosilicone rubber on fabrics brings a different set of issues than solution (dispersion) coating FSR on the same fabric. To complicate things even more, products supplied from different manufacturers or compounders to meet the same specification may act completely different on a specific piece of equipment.
Basic Compounding Ingredients
For the most part, fluorosilicone compounding ingredients are very similar to those used in
dimethyl-vinyl silicone (VMQ) compounds. Fillers, pigments, cure systems and some of the
modifiers are exactly the same, with minor exceptions.

Base materials
Fluorosilicone (FVMQ), like VMQ, is usually supplied ‘pre-compounded’ as a base, and is not
generally sold for use in its pure polymer form. Bases are designed to cover a multitude of
physical property requirements, and families of fluorosilicone bases (designed to meet similar
property and processing guidelines) are typically sold with two or more durometer choices.
Blending bases within a product family allows fabricators to obtain intermediate properties that
meet particular specifications and optimize cost efficiency. If the specification (fuel, solvent or oil
resistance) allows, economic blends of fluorosilicone and VMQ types can also be used.

Fillers
Fillers used in fluorosilicone compounding are essentially identical to those used in VMQ
materials. The most common are inert, and based on SiO₂ (silica). The silica can be fumed,
precipitated or ground. A difference observed in compounding fluorosilicone rubber (vs. VMQ
types) with fumed silica is that untreated, fumed silica is less reactive, and more of the filler can be
used before poor handling or crepe become factors. Because of this, crepe hardening of
fluorosilicone rubber compounds occurs more slowly than similarly filled VMQ compounds. In
addition to the most common silica fillers, mica, talc and other mined silicas can be used. Most of
these fillers are extending in nature, and are used to meet specific fluid resistance, compression set
or economic targets.

Pigments
Pigments can be either organic or inorganic, most commonly supplied as masterbatches in fluid or
polymer (gum) carriers. As a group, organic pigments may be affected by elevated temperatures
during cure or post-cure, and some can even change color as a reaction with peroxide catalysts.
When evaluating an organic pigment for use, evaluate the final color only after the part or slab
receives a typical cure and post cure (if necessary). Inorganics are generally more color stable and
less reactive with peroxide or platinum cure systems. A notable exception is carbon black, which
can greatly shorten the shelf stability and effectiveness of some peroxides (especially diacyl types).
With platinum cure systems, carbon black may also inhibit cure.

A consideration not to be overlooked in the inorganic family of pigments is the presence of
heavy metals in some colors (refer to the MSDSs of pigment masterbatches or fully mixed
compounds). Although stable colors can be obtained with their use, restrictions on these metals
may be an issue if the product is intended for export. European and Asian chemical restrictions are
becoming a part of formulation review for global or export products. Since most pigments are sold
in masterbatch form, the carrier may also potentially contribute to sensitivity in specific fabrication
methods

Modifiers
This group of ingredients is added to fluorosilicone compounds to alter a cured property or correct
a processing deficiency. These modifiers can be used for improved high temperature performance,
mold release, or reduced bloom in unpostcured parts that use an acid (diacyl) type peroxide
catalyst. Like pigments, some modifiers may be unsuitable for export, and care should be taken to
review the source of modifiers used. Something as simple as a stearate-based internal mold release derived from animal by-products may restrict a cured part from being sold or used in Europe.

**Curing Agents**
In addition to the well-known peroxides commonly used for fluorosilicones and VMQ type silicones [see Figure 5], platinum (addition) cure is another option for fluorosilicones. In most applications, more than one curing agent will fit a particular process, and in some cases, there may be benefits to blending two types of these curing agents in a single compound. The choice of which curing agent to use is closely tied to the process being employed to fabricate parts. Specifics will be explored in the fabrication sections.

**Reclaim**
In processes where excessive flash or low yield generates an appreciable amount of scrap (>5-10%), reclamation may be an option. Generally, using reclaimed fluorosilicone will improve compound economies, but extra care should be taken to avoid cross-contamination. The high cost of virgin compound and the critical nature of many fluorosilicone applications should limit the addition of reclaimed rubber to use in the same virgin compound only.

The use of reclaimed material also affects how a compound is manufactured. It is important to consider reclaimed fluorosilicone as a non-vinyl rubber with no vulcanizing agent. If using a vinyl-specific curing agent, the reclaimed material can only be considered an elastomeric filler, contributing no positive effect to cured physical properties. When using a non-vinyl specific curing agent, include the reclaim in calculations to determine the optimum level of agent. In addition, materials compounded with reclaim may be undesirable for extrusion or calendering operations, where surface finishes can be affected by the reclaimed material. The reclaimed portion of a fluorosilicone rubber compound will not go into solution well, and should be avoided in solution-coated fabric processing. In molding operations, shrinkage during molding and post cure will be less than in a virgin compound. Compression set values normally increase. Other basic physical properties are also affected. [See Table 1.]
**Mixing**

Fluorosilicone rubber compounds are typically mixed on a 2-roll rubber mill. For high volume compounds, sigma blade (dough) mixers can also be employed. The techniques used to mix fluorosilicone compounds are similar to those for VMQ materials, but different from organic rubber compounds. With fluorosilicone compounds, the ingredients are dispersed into the rubber base, and high shear is unnecessary. Fluorosilicone does generate more heat than VMQ materials (due to higher base viscosity, relatively), but far less than organics. Due to the temperature sensitivity of some peroxides, it is important to keep all mixing equipment cool. Overmixing of fluorosilicone rubber compounds should also be avoided, as it has a tendency to slightly break down the polymer, causing increased tackiness and some reduction in cured physical properties.

Mills used to mix fluorosilicone compounds should have water-cooled rolls (preferably chilled). High temperatures (>55°C) can cause the formulation to experience some loss of curing agent (through volatilization), or it may cause premature crosslinking. Probably the most significant difference between organic and fluorosilicone compounds is seen when a fluorosilicone material warms up on a mill. Fluorosilicone compounds normally band on the slow roller initially, but when they become warm they will transfer to the fast roller and stay there for the remainder of the mixing cycle. For that reason, many mills used for fluorosilicone and VMQ type silicones have roll gap adjustments on the fast side, since this is where the operator will be at the end of the mix. Due to the fact that fluorosilicone mixing is a dispersive rather than shearing action, mill roll speed ratios only need to be in the range of 1.2:1 to 1.4:1.

The use of a nylon scraper or doctor blade instead of a mill knife is also suggested, as fluorosilicone rubber compounds tend to stick aggressively to mill surfaces compared to most organic elastomer compounds. Mills used for both organics and fluorosilicone should be thoroughly cleaned before material changes. Any organic materials contaminating the fluorosilicone can inhibit vulcanization and may carbonize during post cure, yielding dark spots on a colored or unpigmented fluorosilicone part. In either mill or sigma blade mixing of fluorosilicone compounds, tight fitting edge plows on the mill and sealed packings in the mixer are very important. Fluorosilicone compounds have a tendency to extract any trace of contamination from under a mill plow (which will almost always be a different color), or from a mixer’s packing glands. Again, with a mixer, adequate cooling is recommended, even though heat build-up will be slow.
Molding
Fluorosilicone compounds can be fabricated in methods similar to organic thermoset compounds. In some cases, the same molds can be used. There are three primary types of molding methods to fabricate fluorosilicone rubber parts. All three are similar in that they form the rubber into a finished part through the application of heat and pressure. Differences between the methods relate to the mold loading techniques and reliance on manual labor vs. automation.
Compression Molding

![Diagram of Typical Compression Mold](image)

Figure 1. Diagram of Typical Compression Mold

Probably the most common method of molding fluorosilicone, compression molding techniques have been optimized over many years of production. The following table should be part of any review of a fluorosilicone compound for compression molding:

<table>
<thead>
<tr>
<th>Ingredient Type</th>
<th>Potential fabrication effect</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base</td>
<td>Any fluorosilicone base can be used for compression molding.</td>
</tr>
<tr>
<td>Fillers</td>
<td>In compression molding, the uncured rubber generally does not have a long travel and therefore is not very sensitive to flow characteristics. Therefore, high filler (particularly ground quartz) loadings can be used in soft fluorosilicone bases. In other words, fabricators can use a pigmented and catalyzed 60-durometer fluorosilicone base as-is, or start with a 30- or 40-durometer base and load it with inexpensive extending filler. Extending fillers increase modulus, however, and reduce tensile strength and elongation properties. Compression set is usually improved. Fumed silica can also be used, but at a much lower level and higher cost than ground quartz. Precipitated silica lies somewhere in between (maximum usage level and cost).</td>
</tr>
<tr>
<td>Pigments &amp; Modifiers</td>
<td>These compound ingredients are generally added in smaller levels and typically have only a small effect on compression molding variables.</td>
</tr>
<tr>
<td>Reclaim</td>
<td>Reclaim can be used successfully, if the molded part doesn’t have particularly thin cross-sections where reclaimed particles may restrict flow of the fluorosilicone compound into the mold.</td>
</tr>
<tr>
<td>Cure Mechanisms</td>
<td>All cure mechanisms may be suitable for compression molding, but there are considerations. Many compression molds are multi-cavity designs, often hand-loaded. Even if loading boards are used, the time from preform loading to mold closure may be too long for low temperature peroxides or some platinum-catalyzed fluorosilicones, which could begin to cure prematurely. Scorching can affect surface condition and cause distorted or nonfilled parts, due to viscosity increase cure begins.</td>
</tr>
</tbody>
</table>
Compression Molding Tips & Techniques:
The compression molding process is well understood. As they relate to fluorosilicone compounds, the following are important considerations:

1. If using molds for both fluorosilicone and organic rubber, fluorosilicone may exhibit more shrinkage than an organic compound. Also affecting shrinkage are filler loading (high loading results in less shrinkage) and cure temperature (high temperature gives more shrinkage), which are variables that can be used to offset shrinkage for tight-tolerance parts. Ask the peroxide supplier or custom compounder about cure temperature ranges for your specific compound.

2. Preforms should be uniform in shape and contain enough material to fill the cavity and produce a small amount of flash. In many molds, a weight of 5 to 10% over actual part weight is adequate.

3. Mold surface finish can also affect material flow in compression molding. Highly polished steel normally facilitates good flow. Chrome or other finishes may also improve part removal time, and are particularly helpful in transfer and injection molding of fluorosilicone compounds.
Transfer Molding

This method is particularly well suited for large numbers of small parts where flow is difficult to design into the mold or where the part configuration tends to trap air. A well-designed mold can generate nearly flashless parts (although not completely waste free). Factors influencing fluorosilicone compound ingredients for transfer molding include:

<table>
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<tr>
<td><strong>Base</strong></td>
<td>All fluorosilicone bases can be used for transfer molding, although high tear strength bases would be preferable for intricate parts or designs with extreme undercuts.</td>
</tr>
<tr>
<td><strong>Fillers</strong></td>
<td>High levels of extending filler may be tried, but part dimensions and sprue size need to be taken into consideration. Very small sprues may create turbulence and increase trapped air. In addition, a small sprue with high filler loading may generate excessive frictional heat, resulting in premature scorching.</td>
</tr>
<tr>
<td><strong>Pigments &amp; Modifiers</strong></td>
<td>These compound ingredients are generally added in smaller levels and typically have only a small effect on transfer molding variables.</td>
</tr>
<tr>
<td><strong>Reclaim</strong></td>
<td>Reclaim can be used in most cases. Larger parts with thicker cross sections will be less affected by any potential surface or flow issues. Very small parts or parts with small sprues should be tested carefully before committing to the use of reclaimed fluorosilicone rubber.</td>
</tr>
<tr>
<td><strong>Cure Mechanisms</strong></td>
<td>Generally, high temperature (dialkyl) molding grade peroxide is the preferred curing agent. Low temperature peroxides can scorch in the transfer pot, even when thermally isolated. Residence time in a warm transfer pot can also be a problem with platinum cure mechanisms.</td>
</tr>
</tbody>
</table>
Transfer Molding Tips & Techniques:

1. If at all possible, it’s preferred to have the transfer pot thermally separate from the mold. A water-cooled transfer pot is even better.

2. If using a warm or hot transfer pot, try to minimize the time that the fluorosilicone compound is in contact with heat before it fills out the mold. In almost all cases, the compound will not require warming to improve flow.
Injection Molding

Figure 3. Diagram of Typical Injection Mold

Short cycle cure times and low viscosity (good flow) make injection molding an excellent fit for fluorosilicone compounds. Mold design and sprue size / location are critical factors in optimizing the injection molding process. Due to the high pressures and quick mold fill, short cure dwell times can be designed into a fluorosilicone compound. Other ingredient factors are included in the following table:
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<tbody>
<tr>
<td>Base</td>
<td>All fluorosilicone bases can be used for injection molding, although high tear strength bases would be preferable for intricate parts or designs with extreme undercuts</td>
</tr>
<tr>
<td>Fillers</td>
<td>Due to the pressures realized during the injection part of the mold cycle, care should be given to the type and amount of filler used. With small sprues, highly filled compounds generate significant frictional heat and can begin to form a skin before filling out the mold. Even a slight degree of premature curing will increase the viscosity, causing further buildup of frictional heat.</td>
</tr>
<tr>
<td>Pigments &amp; Modifiers</td>
<td>Because of chemical interactions with some pigments, careful choices should be made when using colored fluorosilicones. The degree of shear realized during the injection phase of the molding cycle can cause color striations, blotches or streaking. Sometimes higher levels of pigments will solve this problem. One modifier to consider is an internal mold release for injection molding. The injection molding process is the least labor-intensive of all the molding processes, and time saved during the molding process is generally worth the effort. Evaluate more than one internal mold release ingredient and usage level before deciding on a modifier, as both of these factors can affect molding efficiency and cured physical properties (especially compression set resistance).</td>
</tr>
<tr>
<td>Reclaim</td>
<td>Reclaim can be used, but due to the particle size, thin cross section materials may end up having a textured surface. Also, with the most common cure agent choices (vinyl-specific peroxides or platinum), the reclaim portion of the compound will not be crosslinked to the virgin polymer.</td>
</tr>
<tr>
<td>Cure Mechanisms</td>
<td>The most common (and recommended) cure agents are of the high temperature variety; both peroxide (dialkyl) and platinum. Dialkyl peroxides have a slower cure rate and usually provide ample time to fill the mold cavity before crosslinking. Platinum cured compounds have the flexibility of the scorch time being adjusted to match the fill rate by increasing the inhibitor level. Platinum cured compounds have an extremely fast cure rate, which is not inhibited by oxygen. Unlike the high-temperature (dialkyl) peroxides, thin flash is fully cured when using platinum catalyzed compounds.</td>
</tr>
</tbody>
</table>
**Injection Molding Tips & Techniques:**

1. Whether using a stuffer box or a strip feed, the feed section generally doesn’t require added heat; viscosity reduction is unnecessary.
2. In multi-cavity molds, balancing the gates is an important step in optimizing the flow. Gate location and number should be evaluated to ensure good flow and quick fills, designed to match cure scorch.
3. Vents (with or without vacuum) may be necessary to eliminate trapped air, due to the quick fill times and short scorch times.
4. Highly polished steel, chrome or other plating can be a benefit in shortening overall mold cycles by reducing part removal time.
5. Reduced injection speed (ram pressure) should be used in highly filled compounds to control frictional heating through the sprues. Lower mold temperatures may also be needed to balance the effects of frictional heat.
6. With fluorosilicone compounds, cold runner systems are strongly recommended.
7. Generally, the fresher the compound, the easier the flow, the quicker the fill. Refreshening the compound before molding may improve part yield.
General Molding Process Troubleshooting

**Backrind:** This condition is a distortion of the molded part, normally occurring at the mold parting line. Typically, the flash in this area is heavy and leaves a ragged indentation in the part. Backrind is most commonly experienced in compression molding, but can happen in transfer and injection mold processes, as well. Potential causes include burrs or roughness in the mold parting area, or a warped mold that doesn’t seal off completely. Molding at too high a temperature can also be a prime cause of this condition. The mechanical solutions are obvious: if molding at too high a temperature for the selected curing agent is the cause, lower the mold surface temperature. Switching to a higher temperature (dialkyl) peroxide or platinum cure system may also help. In injection molding, slightly lower injection pressures can reduce frictional heating, which may alleviate the backrind.

**Scorch:** This phenomenon is due to premature curing of the fluorosilicone rubber before the mold completely fills. Symptoms include heavy flash and incomplete cavity fills in the same part, distorted parts, cured ‘ripples’ or texture at the surface. Knit lines may also be noticed at the end of compound travel in a mold. Check for hot spots in the mold first, using a pyrometer on the mold surfaces. (Don’t always rely on the machine’s thermocouple readings.) Make certain that little or no heat is applied to the fluorosilicone compound before injection or transfer molding. In compression molding, the time from placing the preforms on the hot mold surface until full mold closure should be as short as possible. In all cases of scorch, one alternative would be to evaluate a higher temperature (dialkyl) peroxide or a platinum-cured fluorosilicone compound. With the addition cure systems, increased amounts of inhibitor or reduced platinum concentration may be an option.

**Entrapped air:** When air is trapped in a mold, it may alter the look or feel of the part in several ways. When using acid (diacyl) type peroxides or platinum, air usually shows up as a light, opaque spot. The part will be fully cured, but cutting through the opaque (or light colored) area will reveal a spongy-looking section. When using higher temperature peroxides (dialkyl) that can be inhibited in the presence of oxygen, the part may appear tacky to the touch and undercured. To minimize entrapped air in compression molds, quickly reduce and reapply mold pressure to “bump” the molds. Also make sure the shape and placement of the preform will fill the cavities from one side, effectively ‘pushing’ the air out the other side as the compound fills the mold. Although entrapped air is less likely in injection molding due to the high injection pressure, it can be a problem in parts with extreme undercuts and/or sharp edges. In these cases, the mold should be vented at the sharp edges and in the undercut area. If the problem persists, vacuum can be used to remove air through the vents. If this still doesn’t solve the problem, changing the sprue pattern and size or including an extra tab on the molded part (designed to collect any entrapped air) may be necessary. The tab is then removed, and discarded with the flash. If the in-house capabilities exist, transfer molding may be an option worth evaluating.
**Spotting:** Small, brown (or yellowish) blotches in a molded fluorosilicone part are almost always due to one of two causes. The first is excessive mold release. It is important to use an external mold release agent designed for hot molding of fluorosilicone or VMQ silicone only. If the correct type is being used, reduce the amount of mold release being applied, or spray the molds less often (not every heating cycle). The second cause of spotting is contamination, either of the preform (especially in compression molding) or from the mixing / re-milling) process. The presence of organic lubricating oil in the compound is the most common contaminant. Preform contamination is an issue of housekeeping techniques. Oil entering the fluorosilicone usually finds its way into the compound from a mill or mixer. The spots may be seen after molding, but are normally most apparent after post cure. The higher temperature of the post cure carbonizes the organic content of the contamination, yielding the discoloration.

**Other:** In general, fluorosilicone rubber compounds can benefit from mill freshening before molding. The shearing action from this relatively quick procedure should reduce the viscosity of the fluorosilicone compound, improving mold flow characteristics. Using old compounds without refreshening may cause flow marks and one or more of the other problems mentioned. If refreshening is not an option, standing inventory of mixed fluorosilicone compounds should be kept to a minimum.
Extrusion
Fluorosilicone rubber compounds can be extruded to fabricate a number of products, including gaskets, tubing, wire insulation and various profiled seals. Although the process is similar to organic elastomer extrusion, there are significant differences. In general, fluorosilicone compounds should be extruded at room temperature, due to their low viscosity as compared to organic materials. Spot temperatures above 50°C during extrusion may produce scorching in the extruder, causing different profile measurements (due to higher viscosity in the extruder) and may also result in a less complete cure from partial loss of the curing agent to volatilization. In general, fluorosilicone rubber compounds flow easily in the extruder and expand as they leave the die. Fluorosilicone parts typically have lower green strength than organic elastomers and should be cured (or ‘B’ staged) immediately. Most fluorosilicone compounds are cured via hot air vulcanization (HAV) immediately following extrusion. Continuous vulcanization (CV) is used for some wire insulating applications, but is not the norm for fluorosilicone compounds.
From a compounding ingredient perspective, the following table offers typical formula considerations for fluorosilicone extrusion:

<table>
<thead>
<tr>
<th>Ingredient Type</th>
<th>Potential fabrication effect</th>
</tr>
</thead>
<tbody>
<tr>
<td>Base</td>
<td>Many fluorosilicone bases can be compounded for extrusion, however unfilled compounds (or those without modifiers) may not produce a smooth surface or an even cross-section.</td>
</tr>
<tr>
<td>Fillers</td>
<td>Fillers can improve the extrudability of most fluorosilicone compounds. Using any type of filler will increase the viscosity of the compound and produce a more consistent backpressure in the extruder barrel. The use of extending fillers will tend to break down some of the ‘nerve’ seen in certain unfilled fluorosilicone compounds.</td>
</tr>
<tr>
<td>Pigments &amp; Modifiers</td>
<td>Due to the limited number of curing agents available for HAV curing of fluorosilicone extrusions, the potential for pigment interactions should be evaluated before use. Various cure meters (rheometer, MDR, RPA) can give a good indication of potential interactions. Modifiers can play an important role in optimizing the extrudability of fluorosilicone compounds. A softening additive can make a highly-filled compound a little smoother coming out of the die, while an unfilled compound may benefit from an additive designed to increase green strength. If using an acid (diacyl) type peroxide for an extrusion that does not receive oven post curing, a modifier that contains an acid acceptor is recommended to improve high temperature properties and reduce peroxide bloom.</td>
</tr>
<tr>
<td>Reclaim</td>
<td>Reclaim is generally not recommended for thin-walled extrusions, but may be used in thicker profiles. The use of screens in the extruder to control and maintain a consistent back pressure may have a tendency to trap reclaim particles, resulting in higher or uneven barrel pressure, variations in profile cross section, and variations in profile output.</td>
</tr>
<tr>
<td>Cure Mechanisms</td>
<td>For HAV-cured extrusions, either a low temperature, acid (diacyl) based peroxide or platinum catalyst may be used. In some cases, dual peroxide systems using high temperature (dialkyl) peroxides can be employed to obtain some improvements in heat aging and compression set resistance. Due to the nature of an HAV curing oven, high temperature peroxides, which can be inhibited by oxygen, are usually not recommended. Parts extruded with oxygen-inhibited high temperature (dialkyl) peroxides usually have a sticky surface and high porosity, and are often under cured.</td>
</tr>
</tbody>
</table>
Extrusion Tips & Techniques:

1. The screw used for extruding a fluorosilicone compound should be one designed specifically for fluorosilicone or VMQ silicone. Extruder manufacturers can recommend the best design for the type of tubing or profiles planned.

2. A breaker plate to hold screens (60 - 200 mesh) may be necessary to maintain a high, even back pressure, and provide a consistent profile cross-section and aid in trapped air removal from the compound.

3. Extruder barrel cooling is recommended to maintain cool compound temperatures.

4. Some highly filled, light-colored extrusions can produce an extrudate with dark streaks, typically caused by metal wear particles due to the hard, abrasive nature of some extending fillers. In some cases, modifications can be made to the barrel to minimize this occurrence.

5. When manufacturing thin-walled tubing, low-pressure air is normally introduced into the inside of the tube while in the extruder to prevent collapse of the profile.

6. As with fluorosilicone molding compounds, most extrusion grade fluorosilicone rubber formulations will benefit from mill freshening prior to fabrication. This process can further remove any trapped air and provide a consistent viscosity to reduce start-up waste. Without a refreshening step, older inventory may require different extruder set-up conditions than fresh supplies of the same compound.

7. HAV oven temperatures can vary from 250°C - 650°C, and normally operate in the range of 300°C - 425°C. Sometimes a short ‘hot box’ (0.3 - 1 meters in length) is used to ‘set’ the cure before the extrusion enters a horizontal oven on a conveyor. Softer fluorosilicone compounds not using a hot box may experience a slight out-of-round condition, or may take on the texture of the HAV oven belt before scorch. Vertical ovens are not susceptible to this, but a sufficient hot green strength is required.

8. Hot air curing ovens should have an air exhaust system to remove volatiles generated from the fluorosilicone and peroxide by-products.

9. The use of roller feeds (particularly when using strip coils) ensures a more consistent fill of the extruder, resulting in constant backpressure and a more uniform extrusion profile.
Calendering
For calendering fluorosilicone rubber compounds, either a 3- or 4-roll calender may be used. In most cases, the 3-roll calender is a vertical stack design. A 4-roll calender may also be a vertical stack, but inverted ‘L’ or inclined ‘Z’ configurations can be used, as well. The end result of the calendering process is a solid sheet of fluorosilicone, either unsupported, impregnated into a carrier fabric (Figure 4) or laminated onto a film. The main advantage of the calendering process is to produce long, continuous sheets of uniform thickness. A variable speed main drive is recommended, and the ratio of the take-off rolls should normally be 1:1. In some extremely stiff fluorosilicone compounds, a slight ratio change between the rolls at the take-off nip may bring an improvement by making use of friction at the nip. Fluorosilicone rubber compounds are generally calendered at room temperature, however some heating capacity may be helpful in specific compounds and may improve tack to fabric, if supported.

Figure 4: Diagram of calendering process (supported rubber with liner)
Common calender types

3-Roll ‘stack’ calender

Inverted ‘L’ calender

Inclined ‘Z’ calender
The following table lists some effects of compound ingredients on the calendering process:

<table>
<thead>
<tr>
<th>Ingredient Type</th>
<th>Potential fabrication effect</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Base</strong></td>
<td>Most fluorosilicone rubber bases are suitable for calendering. With fabric-supported processing techniques, very thin coatings require some inherent tack to transfer to the fabric. It should be noted that certain bases don’t calender to a smooth finish, and some experimentation and scale-up will need to be done before final decisions are made.</td>
</tr>
<tr>
<td><strong>Fillers</strong></td>
<td>As in extrusion processing, fillers usually help in breaking down the ‘nerve’ sometimes noticed in calendering fluorosilicone compounds. Extending fillers seem to be the most effective.</td>
</tr>
<tr>
<td><strong>Pigments &amp; Modifiers</strong></td>
<td>Generally, this group of ingredients is not a significant factor in the ability to calender fluorosilicone compounds. The exception is green strength additives. A degree of green strength (related to the calendered thickness) may be required to provide an adequate, even film, especially when unsupported.</td>
</tr>
<tr>
<td><strong>Reclaim</strong></td>
<td>Reclaimed fluorosilicone rubber can be used in thicker sheets, but should be avoided in very thin film thickness. Using reclaim in thin films may result in a cured, rough surface.</td>
</tr>
<tr>
<td><strong>Cure Mechanisms</strong></td>
<td>In calendering, there are a number of cure options: HAV, pressure cure on a steel roll (oven or autoclave) or continuous, single layer, pressure cure (e.g. Rotocure®). The available cure process is a consideration in deciding which cure mechanism to use. Depending on the process (and temperature restrictions), the choices of peroxide or platinum cure system will be narrowed down.</td>
</tr>
</tbody>
</table>
Calendering Tips & Techniques:

1. Most fluorosilicone rubber compounds will benefit from mill freshening before processing on a calender.

2. It is recommended that an unsupported fluorosilicone sheet be calendered onto a release liner that will be stripped off after curing. In general, a non-silicone release liner works best.

3. When using a release liner, strip the sheet while warm (whenever possible). If allowed to cool and the liner tears or sticks, rewarmed the roll before attempting to strip the liner from the sheet.

4. If transfer to fabric is poor or intermittent, dip coating in a solution (10-20%) of the compound (then drying and curing) will add sufficient tack to the fabric.

5. Dual peroxides can be used in a calendering process. The low temperature peroxide can be cured (‘B’ staged) to improve subsequent handling operations or impart a more consistent surface finish. This is also one of the more common techniques if two passes are required for 2-sided coating of fabrics on a 3-roll calender.
Conclusions
When deciding whether to purchase a finished fluorosilicone compound or custom mix in-house, it is important to understand the sensitivities of the various fabrication processes for converting a bulk compound into a functional, profitable part. As illustrated, there are numerous compound ingredient considerations and alternatives to balance end part function with processing efficiency. As with many applications involving engineered materials, the least expensive compound to purchase does not always offer the lowest overall cost. The process cost differences between labor-intensive techniques and more automated choices must be part of the overall fabrication decision-making process.

Every fluorosilicone rubber fabricator learns over time how to improve part quality and yield using his/her particular equipment and processing design. There are a large number of variables that come into play between fluorosilicone base manufacturers, equipment suppliers and even vendors of the various compound ingredients. Process experience and material knowledge is critical in matching and troubleshooting fluorosilicone rubber compounds and their associated processes. Using fluorosilicone rubber compounding skills to match economic targets and processing techniques will ensure that the formulations and equipment will be optimized, contributing to higher yields, lower scrap levels and reduced downtime for greater manufacturing profitability and personal sanity.

References
Figure 5: Curing agents for fluorosilicone rubber compounds

Common Peroxides for Fluorosilicone:

**Diacyl types**
- Dibenzoyl peroxide
- Bis (2,4 dichloro benzoyl) peroxide

**Dialkyl types**
- 2,5-dimethyl-2,5-di-(t-butylperoxy) hexane
- Dicumyl peroxide
- \_\_ \_ \_di(t-butylperoxy) diisopropylbenzene

Other Peroxides for Fluorosilicone:

**Peroxyketals**
- 1,1-di-(t-butylperoxy)-3,3,5-trimethyl-cyclohexane
- n-butyl 4,4-di-(t-butylperoxy) valerate

Table 1: Typical Effects of Reclaim on Fluorosilicone rubber

<table>
<thead>
<tr>
<th>% new Fluorosilicone rubber</th>
<th>% reclaimed Fluorosilicone rubber</th>
<th>Durometer Hardness, Shore A</th>
<th>Tensile Strength, MPa</th>
<th>Elongation at Break, %</th>
<th>Compression set, % 22 hrs @ 150°C</th>
</tr>
</thead>
<tbody>
<tr>
<td>100</td>
<td>0</td>
<td>58</td>
<td>6.62</td>
<td>188</td>
<td>24</td>
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<td>10</td>
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<td>180</td>
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<tr>
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<td>27</td>
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<tr>
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<td>30</td>
<td>58</td>
<td>5.52</td>
<td>140</td>
<td>29</td>
</tr>
<tr>
<td>50</td>
<td>50</td>
<td>55</td>
<td>6.00</td>
<td>160</td>
<td>32</td>
</tr>
</tbody>
</table>

Oven cure: 24 hours @ 150°C