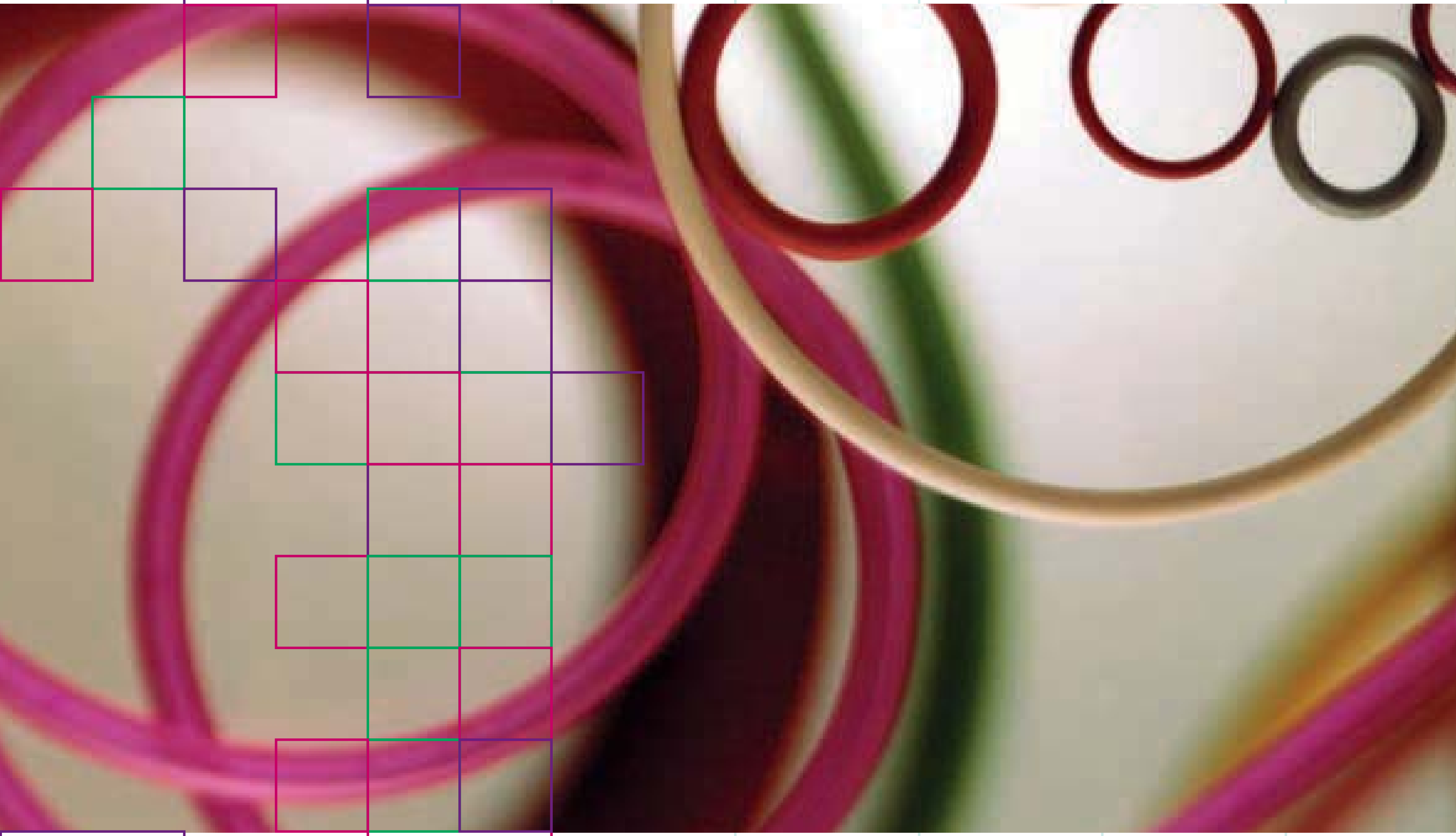
Fabricating with SILASTIC® High Consistency Silicone Rubber



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Rubber Fabrication
Solutions

DOW CORNING

Preface

This manual is designed to help both new and experienced rubber goods manufacturers work with *Silastic* high consistency rubber (HCR) and Fluorosilicone Elastomers. Detailed descriptions are provided on the principal techniques used in the production of rubber parts. Moulding, extruding, calendering, and other fabrication techniques are presented. Information on vulcanization, oven curing, and manufacturing equipment is also provided.

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THE *SILASTIC*® SILICONE RUBBER COMPOUND RANGE

Materials

Dow Corning offers a large variety of ready-to-use compounds designed for all fabrication methods as described below. Hereunder fall: moulding, extrusion, calandering, dispersion coatings and special fabrications.
The product range includes high consistency rubber peroxide or polyaddition curing mechanisms, fluorosilicone compounds and combinations of these product groups.

Based on our long years experience in numerous markets we offer tailor made products for most applications. Should it be necessary those products can be additionally modified. Please contact us for optimum recommendation to fit your own production requirements.

Colours

***Silastic* silicone rubber compounds are available as standard in the following colour shades:**

- Translucent or natural coloured
- White RAL 9010
- Yellow RAL 1021
- Orange RAL 2004
- Red RAL 3000
- Red RAL 3016
- Blue RAL 5002
- Blue RAL 5010
- Green RAL 6017
- Brown RAL 8015
- Grey RAL 7035
- Black RAL 9011
- Violet RAL 4004

Other colour shades may be obtained on special request

Preforms

For optimal adaptation to specific production conditions or technology requirements we offer a variety of rubber preforms

- Round logs diam. 150 mm
- Round logs diam. 120 mm
- Round logs diam. 75 mm
- Round logs diam. 60 mm
- Square logs 70 x 70 mm
- Square logs 100 x 60 mm
- Slab on plastic film rolled up
- 20 kg rolls of slab in plastic bags
- Strips 80 x 20 mm talced, coiled like a snail
- Strips 60 x 20 mm, talced, coiled like a snail
- Strips 40 x 15 mm, talced, coiled like a snail

- Strips 80 x 20 mm wrapped in polyethylene foil
- Strips 60 x 20 mm wrapped in polyethylene foil
- 500 kg continuous strip 80 x 20 mm talced
- 500 kg continuous strip 60 x 20 mm talced
- 500 kg continuous strip 40 x 15 mm talced
- 500 kg continuous strip 40 mm round, talced
- 500 kg continuous strip 30 mm round, talced
- 500 kg continuous strip 80 x 20 mm wrapped in polyethylene
- 500 kg continuous strip 60 x 20 mm wrapped in polyethylene

Packaging

As for technical reasons not every material can be supplied in every preform, please contact us for the specific case.

The package sizes available are

- 20 kg cardboard boxes on euro pallet
- 500 kg cardboard box on euro pallet

VULCANIZING *SILASTIC* SILICONE RUBBER

The Vulcanizing Process

Most silicone and fluorosilicone rubber products that require heat for vulcanizing use some form of peroxide as a vulcanizing agent. When the rubber is heated to the required temperature, the peroxide decomposes to form free radicals that react with organic groups in the silicone polymer. This results in crosslinking between organic groups the number and position of these linkages determine the degree of vulcanization. The process can be completed in 30 seconds to 10 minutes after the critical temperature is reached -the length of time depending on the peroxide used and the thickness of the part produced. As a rule of thumb for HCR with a peroxide curing system one can calculate with **15 sec/mm** for the vulcanisation. Peroxide cured *Silastic* compounds have a minimum usage time (shelf stability) of 6 months.

Newer developed materials use an addition cure system, are sold as ready-to-use compound under the brand name *Silastic* Rapid Cure. Vinyl and hydrogen functional polysiloxanes are cured using a platinum catalyst. This reaction is inhibited at room temperature but is accelerated rapidly by increasing the temperature. The cure speed compared to peroxide cured materials is significantly higher. For an article made out of addition cured RapidCure a value of **5 -7 sec/mm** can be considered as typical cure time. When stored at room temperature, 1 component *Silastic* Rapid Cure moulding grades have a minimum usage time (shelf stability) of 3 months.

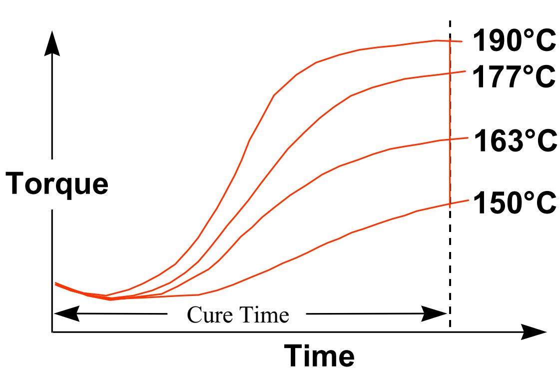

Fig. 1: Peroxide cure at different temperatures

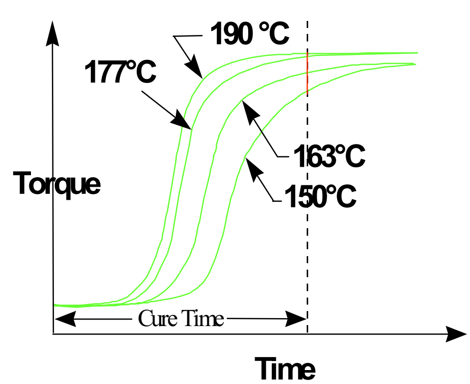

Fig. 2: Addition cure at different temperatures

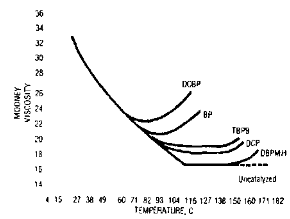

Fig. 3: Mooney viscosity vs. temperature for a silicone rubber with various vulcanizing agents.

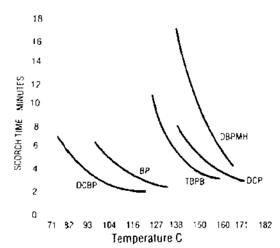

Fig. 4: Mooney scorch time vs. temperature for a silcone rubber with various vulcanizing agents.

After vulcanizing, some silicon rubber products are oven cured to stabilize the rubber and obtain the desired physical properties. However some products do not require the oven-curing step. Refer to specific product data sheets for recommended oven-curing conditions.

Extrusions are usually vulcanized in continuous-process equipment -most commonly, in a hot air vulcanizing (HAV) unit, where the extruded material is carried through a heated chamber. Continuous hot liquid vulcanizing (CV) units can also be used; and small lengths of extrusion of appropriate shape and thickness may be vulcanized in an autoclave.

Vulcanizing Methods

Calendered sheet is vulcanized, when unsupported, by wrapping it on a hollow drum and exposing to steam. When calendering onto a supporting (reinforcing) fabric, pass it over a hot drum or through a hot air vulcanizing unit. Details are given in the section on calendering. Duct and hose made by mandrel wrapping may be vulcanized in an autoclave or steam vulcanizer, as described in the Fabricating Hose section.

Causes and Cures of Vulcanizing Inefficiencies

If at times your products are incompletely vulcanized, check your processing for volatilization and for preferential reactivity of the peroxide (the two most common causes).

Volatilization of the vulcanizing agent can result from the following:

- Too much delay in starting the moulding cycle after loading a heated mould. This may cause trouble in two ways: (a) The peroxide may start to decompose, resulting in scorch (premature vulcanization); or (b) some of the peroxide may volatilize, leaving too little in the rubber for adequate vulcanization.
- Moulding at a temperature too low for the peroxide being used. This is the most common reason for poor vulcanization, and causes part of the peroxide to evaporate before it is activates
- Allowing preforms to stand in warm areas for long periods. Peroxides gradually diffuse from the rubber upon exposure to air. The warmer the air, the faster the diffusion.

Corrective Measures

Once the reason for the loss of peroxide has been determined, there are several corrective measures that might be taken. The most obvious, of course, is to change the production conditions; but this is not always practical. For example, taking the time to completely cool a mould before each loading will be too costly.

Note, that a low vulcanizing temperature can give poor properties regardless of the quantity of peroxide added. Sometimes a different peroxide can be used. Contact Dow Corning in case of this problem.

When vulcanizing problems occur and the cause is not readily determined, the first and most important step is to perform a thorough study and evaluation of the entire manufacturing process. If you desire technical service, it is available on request by calling your local Dow Corning sales office.

How Viscosity of Rubber is affected by Vulcanizing Agent and Temperature

Silastic silicone rubber products flow more readily than other rubbers at room temperature. The viscosity, as measured in a Mooney viscometer with a large rotor, ranges from 15 to 75 units, depending on composition. Heat produces further softening -as shown by the lower curve in the chart for a typical uncatalyzed silicone rubber.

This thermal softening is altered by the presence of vulcanizing agent in the rubber. At temperatures where vulcanization is very rapid, thermal softening is offset by an increase in viscosity due to vulcanization. In such cases the rubber does not reach its normal minimum viscosity, and does not flow as readily. At a given temperature, the vulcanization rate for a silicone rubber can vary widely, depending on the vulcanizing agent used; and this can affect the viscosity and rate of flow. This is shown in Fig. 1. For example, a rubber containing a vulcanizing agent that is activated at high temperature such as 2, 5-dimethyl-2, 5-di(t-butyl peroxy) hexane will reach a lower viscosity (and thus flow better) than the same rubber using a vulcanizing agent that is activated at a lower temperature.

Scorch Time

Mooney scorch time is the length of time required, at a given temperature, to raise the viscosity of a rubber 5 points above its minimum value. In Fig. 4 the curves show scorch time vs. temperature to compare the vulcanization rates obtained with the vulcanizing agents commonly used with silicone rubber.

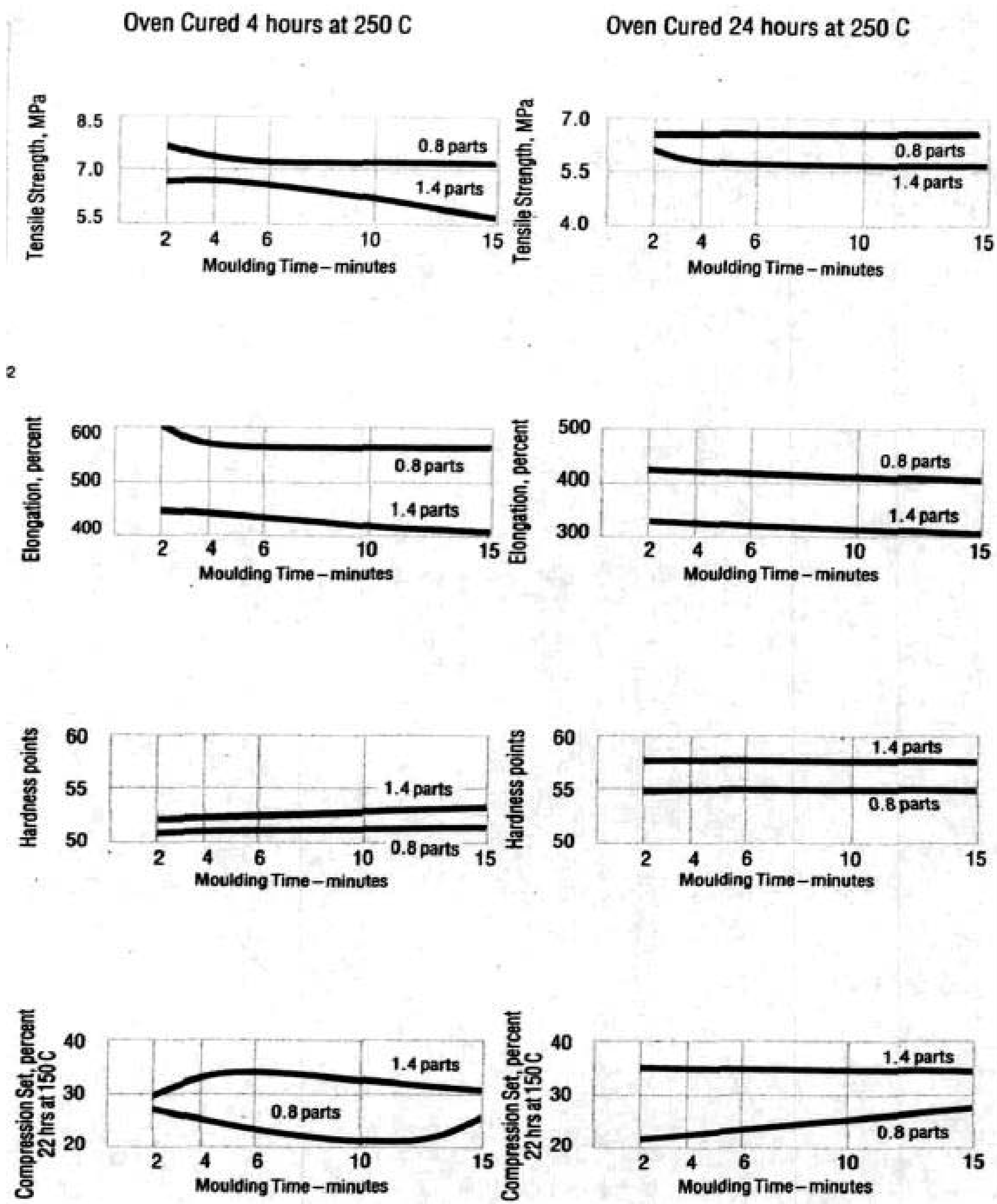

Fig. 5: Effect of vulcanizing time and oven cure on properties of compression moulded parts – vulcanization at 115°C with 2,4 dichlorobenzoyl peroxide (50).
Concentration shown in parts of 50% peroxide per 100 parts of rubber base

Effect of Vulcanizing Time, Oven Cure and Concentration of Vulcanizing Agent on Physical Properties of finished Parts

For a given vulcanizing temperature, the length of vulcanizing time and curing time and temperature affect the properties of silicone rubber. So does the concentration of vulcanizing agent (expressed as parts per 100 parts of rubber, by weight). These facts are illustrated in Fig. 5 and Fig. 6. All tests were made using a formulation of general purpose (VMQ) rubber plus the vulcanizing agent specified.

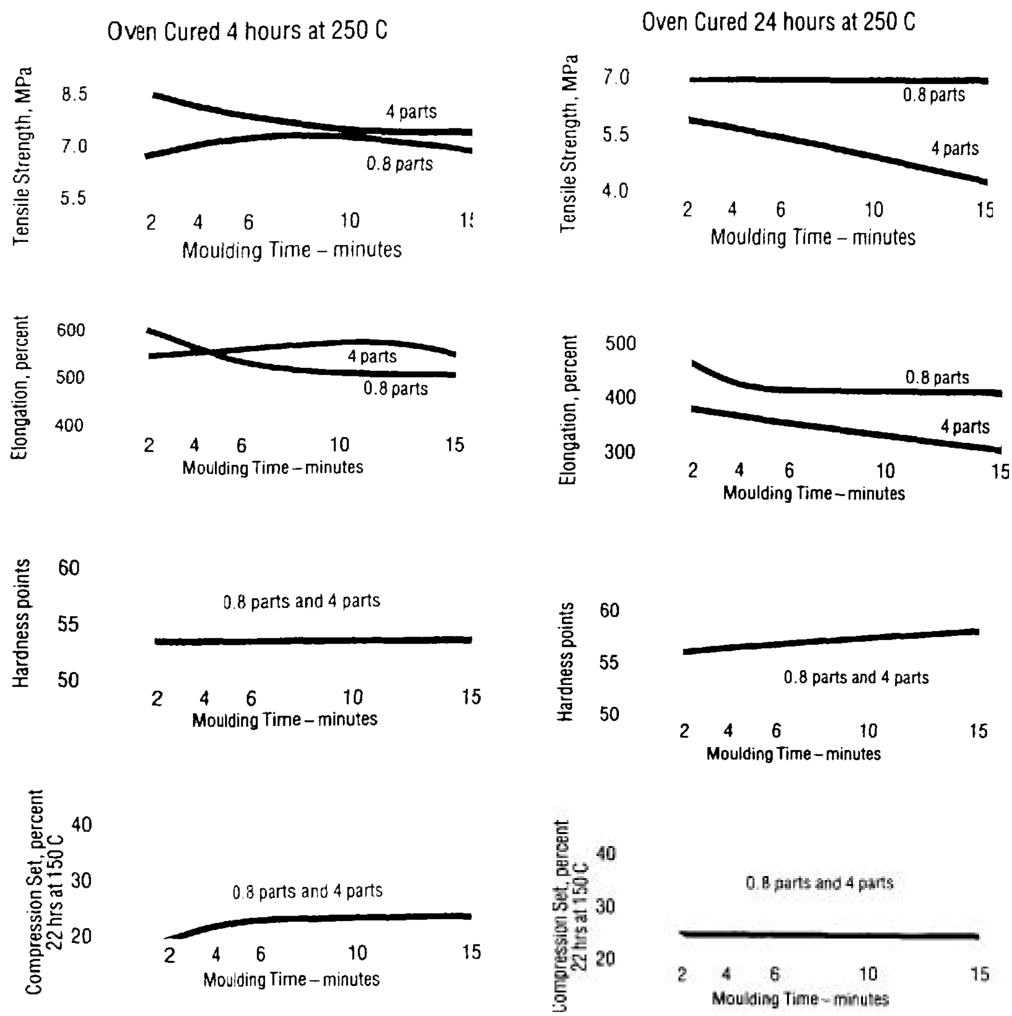

Fig. 6. Effect of vulcanizing time and oven cure on properties of compression-moulded parts-vulcanizing at 171°C with 2,5-dimethyl-2,5-di(t-butylperoxy) hexane.
Concentration of vulcanizing agent is marked for each curve, in parts per hundred.

Formulation: 100 parts of a 40 durometer Silastic® VMQ brand silicone rubber 10 parts Cab-0-Sil MS-7

MOULDING OF *SILASTIC* SILICONE RUBBER

Silastic silicone rubber is moulded to produce a broad range of parts in many shapes and sizes. There are three principal moulding methods: (1) compression moulding, (2) injection moulding, and (3) transfer moulding. All three methods are similar in that each forms the rubber in a mould by application of pressure and heat, which shape and vulcanize the parts. They differ as to mould loading method, time and temperature of moulding cycle, and other details, which are discussed in following paragraphs.

The design of moulds for silicone rubber is generally speaking similar to the design of moulds for thermoplastic. Nevertheless, a few important differences of the behaviour of silicone rubber should be noted. Silicone rubber does not shrink in the mould like a thermoplastic material. The silicone rubber expands in the hot mould. Therefore, the articles do not necessarily remain on a core or more generally on the positive side of the mould as desired. Usually, the articles remain in the cavity half with the larger surface area.

Mould Design

Accuracy

Dimensional accuracy of moulded silicone rubber parts is usually very important; and in such cases, the design of the mould must allow for shrinkage of the parts. Linear shrinkage values can be obtained from test moulded samples of flat rubber sheets and can be used as a rough guideline to mould design values for very simple parts only.

Determining the Shrinkage of complex Moulded Parts

With parts of complex shape, shrinkage is difficult to predict. It depends on several factors:

- tool temperature and demoulding temperature
- pressure in the cavity and consequently the compression of the materials
- location of injection point (shrinkage in the direction of the material flow is usually somewhat higher than perpendicular to the direction of flow)
- the dimension of the part (the shrinkage of thicker articles is lower than of thinner articles)
- post curing the article causes additional shrinkage of about 0.5-0.7 %

Therefore, in designing a mould for producing such parts, it is often helpful to mould a part in an existing mould of similar shape and size -using the same rubber, vulcanizing agent and moulding temperature that will be used with the new mould. The shrinkage values obtained on this part can then be used as a guide in designing the new mould. Conventional rubber moulding presses are used for all methods; and in some cases, moulds used for organic rubber

may be employed.
However, silicone rubber shrinks more than organic rubber during vulcanization; so where dimensional accuracy is critical, moulds designed for silicone rubber are required. This and other factors of mould design are discussed below. In many cases, the parts produced require an oven cure after moulding, to stabilize the physical properties and remove decomposition products formed during press vulcanization.

NOTE: The following sections state basic principles that apply in all cases;
and some give processing data for specific parts. Your moulding operation may differ significantly as to part dimensions, rubber or processing equipment. Accordingly, these figures may not be the best for your particular conditions; but they will be helpful as a starting point in determining what values will give you best results.

Air venting

Moulds should include provision for the release of air trapped in the mould cavity. This can be done by designing the mould so that it is split at undercuts or sharp corners.
The clearance between mould parts should be large enough to allow the air to escape from the mould cavity, but not so large that the *Silastic* silicone rubber also flows out. Generally, smooth-machined surfaces are satisfactory. The air which is enclosed in the cavity is first compressed by the injected silicone rubber and than expelled through the venting channels. If the air can not escape entirely, air entrapments in the article occur which can often be recognised by a white edge along the article. In this case special venting channels with 1 -3 mm width and 0.004 -0.005 mm depth can be inserted into the parting line so the air can escape.
Optimum venting is created by a vacuum. To produce such a vacuum, the mould stops during the closing movement at 0.5 -2 mm before it is completely closed. A gasket is built into the parting line, so a vacuum pump can draw the air from the cavities. Only when the vacuum has reached a certain reduced pressure or a time cycle has come to its finish, the machine closes the mould completely and the injection process is started.

Multi-Cavity Moulds

In designing multi-cavity moulds, it is important to quickly load the mould to avoid scorching. If the total loading time is too long, the moulding temperature may have to be reduced, and this will lengthen the moulding cycle, reducing the advantage of using multiple cavities.

Mould Material and Finish

- Retainer plates are fabricated from unalloyed tool steel. **Steel-No.: 1.1730** DIN-Code: C 45 W
- Moulding platens, which are exposed to temperatures between 170 and 210°C should be made out of pre-tempered steel.
- **Steel-No.: 1.2312** DIN-Code: 40 CrMnMoS 8 6
- The mould platens which contain the cavities are preferably produced from hot work steel, tempered later and possibly nitrided.
- **Steel-No.: 1.2343** DIN-Code: X 38 CrMoV 5 1
- For highly filled silicone rubbers, for example for HV insulators, the use of even harder materials such as flash chrome plated steel or powder metallurgy steel, which has been developed especially for this application, is recommended.
- **Steel-No.: 1.2379** DIN-Code: X 155 CrVMo 12 1

The surface of the cavities influences the process in different ways:

- The article duplicates exactly the surface of the steel and thereby fulfils various optical requirements. Polished steel must be used for the production of transparent articles.
- An eroded surface normally provides less adhesion between the LSR and the mould than a polished surface, so demoulding can be specifically designed.
- Titanium/Nickel surface treated steel has a very high wear resistance.
- PTFE/Nickel renders easier demoulding of the articles possible.

Moulding Tips

Mould Release Agents

It is usually necessary to use a mould release agent when moulding silicone rubber, though it may not be needed for chrome plated or highly polished mould surfaces.

A light coat of a 2 percent to 5 percent solution of household detergent in water, will prevent sticking in most cases. In making aqueous solutions of release agents, it is best to use distilled water if the plant water has a high mineral content. With rubbers that are formulated to give good adhesion to metals without priming, these detergents are ineffective, but a light coating of fluorocarbon lubricant can give good results.

Mould release agents should be used sparingly, to prevent buildup on the mould. Usually, one application every 5 to 10 moulding cycles is adequate. *Silastic* MRA-2 modifier can be milled into the rubber prior to vulcanization to increase mould release. Use of this modifier will decrease the need for conventional spraying, but may affect compression set of the cured rubber.

CAUTION: Silicone mould release agents, which are highly effective with organic rubbers, do not provide good release of silicone rubber.

Making Preforms

Preforms are roughly formed pieces of unvulcanized rubber that are placed in the compression mould, the cylinder of the injection moulding machine, or in the pot of the transfer mould (explained below). They are normally made by die-cutting or extruding -and in some cases, for compression moulding, by hand-forming, where parts are of highly complex shape.

For compression moulding, preforms should be of approximately the shape of the mould cavity, and should contain enough rubber to fill the cavity and produce a slight flash.

For injection and transfer moulding, preforms can be of any shape that is convenient for feeding the injection cylinder or transfer pot. Rubber compounds are ready-to-use and can be supplied in strip or coil form and cut to length, or in chunks or sheets. Strip preforms are especially suited to injection moulding.

Bumping

To dislodge air bubbles entrapped in the mould or rubber, most moulding operations require "bumping" - that is, sudden release of the moulding pressure, followed by buildup "to full pressure. This is done several times while the parts are being moulded and before vulcanization has started. Some presses bump automatically as part of the moulding cycle.

Compression Moulding

In compression moulding, a preform is placed on one half of a heated mould. When the mould is closed and put underpressure (in a press), the rubber is forced into all parts of the mould cavity; and excess rubber flows into a flash groove around the mould cavity.

Silicone rubber is compression-moulded to form gaskets, seals, 0-rings, flat sheets, fabric reinforced laminates, and many other types of industrial rubber goods, of almost any size desired.

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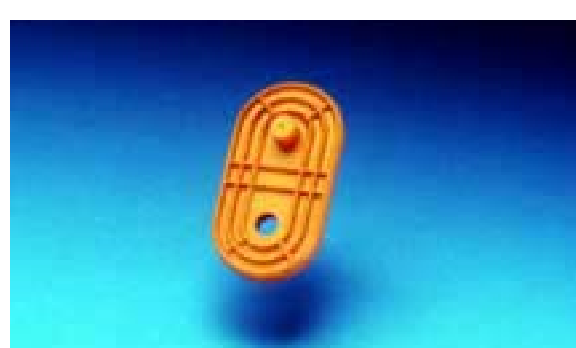

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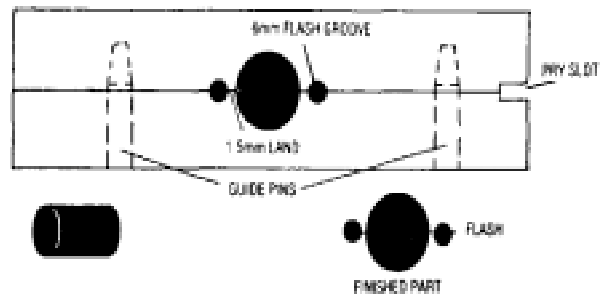


Fig.7. Typical compression mould

Loading the Mould

Single cavity moulds are loaded by hand. With some multiple cavity moulds loading boards may provide faster mould loading, which helps prevent scorching of the preforms. Loading boards are devices on which the preforms are placed in position for simultaneously loading all of the mould cavities.

Moulding Time

Moulding time and temperature vary with the vulcanizing agent used, the thickness of the part being moulded, and other production conditions discussed under "Moulding Problems" The figures given in Table II are typical; but for each specific moulding job, the most favourable values of time and temperature can only be determined by experimentation.

Moulding Pressure

Enough pressure should be applied to obtain sufficiently rapid flow of rubber in the mould. Most *Silastic* silicone rubber products flow well at about 200 psi (1.4 MPa).

Unloading

Silicone rubber parts are usually unloaded hot. However, when moulding thick sections or fabric reinforced parts, it may be necessary to cool the mould before releasing the pressure -to help prevent backrinding, porosity, and delamination.

TABLE I: SUGGESTED CONDITIONS FOR COMPRESSION MOULDING WITH VARIOUS VULCANIZING AGENTS

		Moulding Time, minutes				
Vulcanizing agents	Moulding Temperature	1.5mm	3mm	6mm	10mm	12mm
2,4-dichlorobenzoyl peroxide	116°C	5	5	10	15	20
Benzoyl peroxide	127°C	5	5	10	15	20
Dicumyl peroxide	150°C	10	10	15	18	20
Tertiary-butyl perbenzoate	150°C	10	10	15	18	20
2,5-dimethyl-2,5-di (t-butyl peroxy) hexane	171°C	10	10	15	18	20
Tertiary butyl peroxy isopropyl carbonate	140°C	5	5	10	13	15

Injection Moulding

In injection moulding, a high speed ram or reciprocating screw forces the unvulcanized rubber from a cylinder through a nozzle and into a closed heated mould, by a pressure independent of the pressure that holds the mould closed. The two halves of the mould are attached to heated platens. The ram injects only enough material to completely fill the mould -then retracts so rubber for the next cycle can be loaded into the cylinder.

Advantages of this process include short moulding cycles (much shorter than for compression moulding), little or no preform preparation, little or no flash to remove, and low scrap rate.

For any given *Silastic* silicone rubber, the following production factors must be properly balanced for best results, through experience and experimentation:

Cylinder Temperature

For most silicone rubber products the cylinder or the screw and the barrel should be at room temperature.

Moulding Time

This will vary according to the type of rubber and size of the part. The normal range for peroxide cured materials is 30 to 90 seconds, for *Silastic* Rapid Cure products 15 to 45 seconds .

All vulcanizing agents normally used with silicone rubber can be used for injection moulding. However, the higher temperature peroxides are preferred because they are less likely to scorch.

Injection Pressure

This should be from about 500 to 2000 psi (3.45 to 13.78 MPa), depending on the viscosity of the rubber, size of injection nozzle, mould design and desired injection time. Under most conditions, most rubbers inject satisfactorily at about 800 psi (5.51 MPa)

Injection Time

This depends on the mould cavity size, injection pressure, and viscosity of the rubber. It is usually between 5 and 10 seconds -7 seconds being theaverage for most conditions. A short injection time is desirable, to minimize scorch and total moulding time. Since it is important to keep injection time as short as possible higher pressures should be used with high viscosity rubbers. Fig. 8 shows the relationship between viscosity and injection pressure for a typical moulding setup.

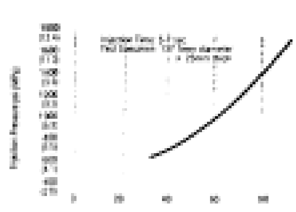


Fig. 8 : Mooney viscosity versus temperature for a silicone rubber with various vulcanizing agents

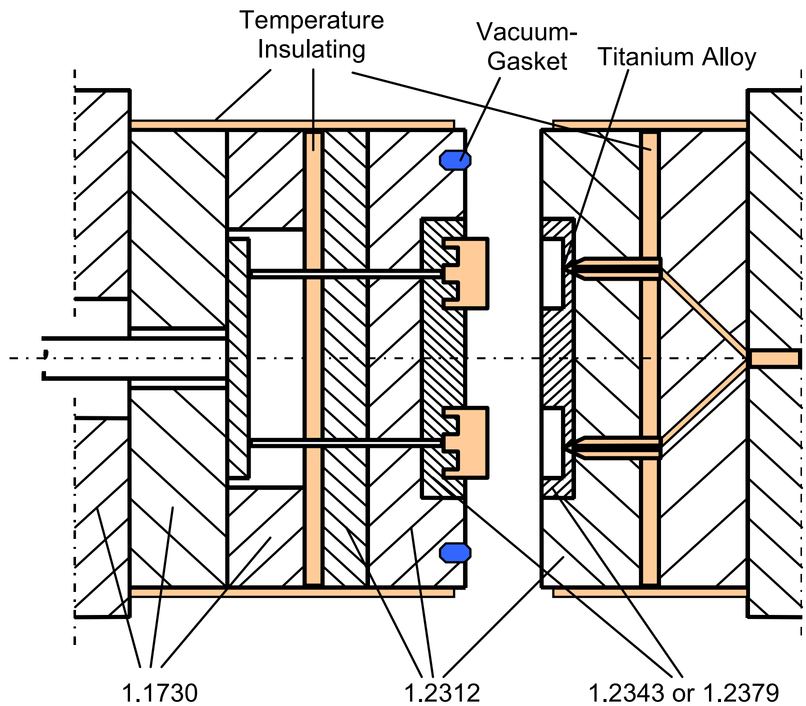


Fig. 9. Sketch of a coldrunner mould for silicone rubber

Transfer Moulding

In transfer moulding, the unvulcanized rubber is placed in a chamber (called a pot), usually at the top of the mould, and the assembly is placed in a press. The press applies pressure to a piston-Ike plug in the open end of the pot, clamping the halves of the mould together and forcing the rubber to flow through one or more sprues into the heated mould.

Transfer moulding is particularly useful in producing parts whose shape is such that the moulds cannot provide good flow and tend to trap air. It is the best method of moulding parts that contain wires, pins and other inserts that require precise positioning.In some cases, the pot is built into the mould. In others, the pot is separate from the mould and is positioned by pins or location marks. In the latter case, the pot is usually removed immedately after the mould is filled, to prevent vulcanization of the rubber in the pot. With this system, the pot can be filled with enough rubber to load the mould several times.

Moulding Time and Temperature

The time and temperature for transfer moulding are the same as for compression moulding. The figures given in Table II are typical; but for each specific moulding job, the most favourable values of time and temperature can only be determined by experimentation.

Shrinkage

Most products moulded with *Silastic* silicone rubber shrink during vulcanization and oven cure. Thus, the finished parts are smaller than the mould cavities in which they were formed. The amount of linear shrinkage ranges from about 2 percent to 5 percent depending on the composition of the moulding rubber, the moulding temperature, and the size and shape of the part. There are two principal causes of shrinkage:

- The coefficient of thermal expansion of silicone rubber is greater than that of the steel mould. Therefore, when the moulded part cools to room temperature, it is smaller than the mould cavity at room temperature.
- During vulcanization and oven curing, decomposition products of the vulcanizing agent and volatile components of the rubber are driven off, decreasing the size of the moulding.
- Moulded parts can be held within commercial tolerances, by using rubber of consistent shrinkage and controlling the processing variables.

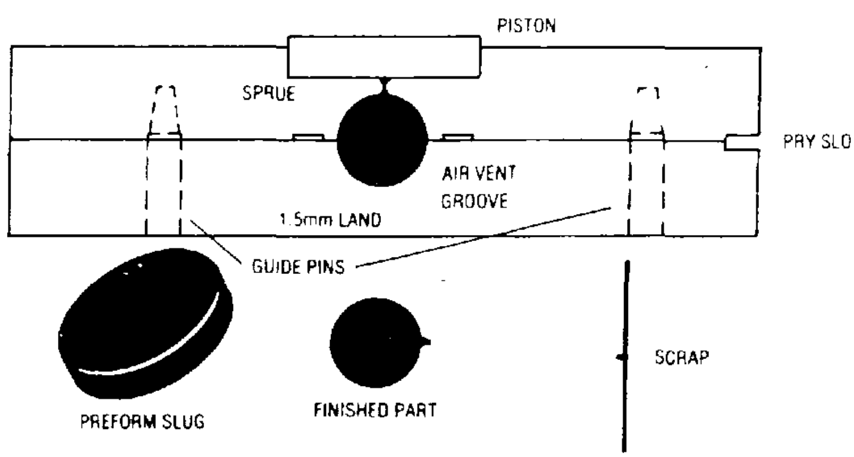


Fig. 10. Typical transfer

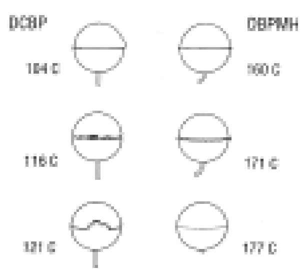
Moulding Problems

Scorch

Scorch is premature vulcanization of the rubber -before the flow of rubber in the mould is completed. The poor mould flow results in distorted or incompletely formed parts.

Scorch may be caused by hot spots in the mould, by moulding at too high a temperature, by too great a length of time between start of mould loading and mould closing, or by restrictions in the mould that substantially reduce the rate of flow. To prevent or eliminate scorch, see that the mould is completely filled before the rubber starts to vulcanize. Specifically:

- Assure the mould cavity is uniformly heated.
- If possible, use a vulcanizing agent with a higher reaction temperature. This will let you bring the rubber to a higher temperature, and therefore to a lower viscosity for better mould flow.
- In general, with simple shapes, high mould temperatures and short cycles can be used; but with intricate shapes or thin mouldings, lower temperature may be necessary. With compression mouding in particular, keeping the moulding temperature near the low end of the vulcanizing temperature range will reduce scorch.
- In compression moulding, increasing the moulding pressure to increase the rate of flow will sometimes help



Typical examples of backrinding and how to prevent this condition. Each part was moulded 10 minutes at the temperature shown. On parts at left, vulcanized with DCBP (2,4-dichlorobenzoyl peroxide), backrinding is lessened by lowering the moulding temperature. By vulcanizing parts at right with DBPMH (2,5-dimethyl-2,5-di[t-butyl-peroxy] hexane) backrinding is greatly reduced

Backrinding

This is distortion of the moulded product at the mould parting line -usually in the form of a torn or ragged indentation -and is mainly a problem of compression moulding.

Backrinding may be caused by burrs or roughness of the mould parting edges, a warped mould that does not close completely, or moulding at too high a temperature for the vulcanizing agent used. In the latter case, the backrinding is caused by sudden release of internal pressure within the part when the mould is opened. To prevent backrinding, make sure there is no roughness or excessive opening at the mould parting line, and keep the moulding temperature as low as possible for the vulcanizing agent used. Cooling the part in the press will sometimes reduce backrinding.

Entrapped air

Air entrapped in the mould or in the rubber may produce soft, discoloured areas on the surface or in the cross section of the moulded part, due to incomplete vulcanization. This is because some of the vulcanizing agent is lost by reacting with air. Remedies include:

- "Bump" the mould.
- Increase the amount of vulcanizing agent.
- Use a high temperature vulcanizing agent that is less

reactive with air. Thermal expansion of the rubber will then expel the air before vulcanization begins.

- Vent the mould at sharp corners and undercuts.
- If air entrapment still occurs with compression moulding, the part may be produced successfully by transfer moulding.

Surface Discolouration

Surface discolouration of the moulded part usually results from excess build up of mould release agent in the mould cavity, or from excessive dust or dirt on the preform.

To prevent it, clean the mould as often as necessary and keep the preforms clean. Use release agents sparingly.

EXTRUSION OF *SILASTIC* SILICONE RUBBER

Silastic silicone rubber is extruded to make tubing, rods, gaskets, seals, wire insulation, and preforms used in compression moulding. With this process, the rubber is continuously forced through a die that forms it to the desired cross-sectional size and shape. Although the general procedure is the same as for organic rubber, there are detailed differences. The following sections cover basic facts on extruding silicone rubber.

Screw Extruders

Screw extruders used with silicone rubber should have the following equipment:

- A screw designed for silicone rubber.
- A feeding roller attached to the screw by a gear or separately driven in the intake zone.
- An extended barrel, suited to the length of the screw. The barrel should be hard metal plated.
- A breaker plate with recess to hold screens that will produce enough pressure in the rubber to ensure removal of trapped air fitted with A 60 to 120 mesh stainless steel screen.
- A spider flange or crosshead to (a) hold a mandrel for forming the inside diameter when making tubing, and (b) hold the die holder and permit radial adjustment for centring.
- A universal die holder.
- A coin or plate die.

Screw Design

For good extrusion work with silicone rubber, it is important to have a screw of proper design. A single flight screw with diminishing pitch is best. Typically (length-to-diameter) ratio of 10:1 to 12:1 is used. The screw should have a compression rate of 1:1.5 to 1:2, which can be obtained with constant core diameter and varying flight distance or with varying core diameter and constant flight distance. For troublefree continuous feeding and high output, the flight should be quite deep and should be hardened or hard metal coated to prevent wear. The screw should be well cooled to prevent scorch due to the shear heat generated during the extrusion process. For improved consistency of volume output some manufacturers recommend to use double flights in the output area of the screw.

Roller Feed

A roller feed to the extruder, with the rubber fed from a "hat" or coil, ensures a more uniform extrusion than hand feeding, and saves considerable labour.

When rubber is roller-fed from a "hat" the coil support should be horizontal and mounted close to the feed roller.

Fig. 11: Screw design of a flight progressive screw for silicone rubber extrusion

Gear Extruders

Gear pumps have been used since 15 years to improve output consistency of screw extruders and to increase the die pressure, mainly for thermoplastics. Recently the principle of the gear pump has been used by several machine manufacturers as independent, highly effectice rubber extrusion unit. The gear pump is fitted with two feeding rolls at the entrance side. Advantages of this layout compared to screw extruders is a significantly lower energy consumption, more compact design to save production space, a very low shear inside the machine with a low buildup of heat, minimizing the risk of scorch, and a very constant output. Disadvantage is that air entrapments in the raw material are not removed and the material homogenization is low so that the requirements to the uniformity of the raw materials is very high. For more information on equipment suppliers please contact
Dow Corning AETS.

Extrusion Dies

Mandrel

In extruding tubing, the ID (inside diameter) is formed by a mandrel, which is held by the spider flange. To keep thin wall tubing round during vulcanization, the flange and mandrel are drilled to admit air under light pressure -from less than 7kPa to about 35kPa, depending on tube diameter and wall thickness.

Die Design Principles

Coin or plate dies work well for silicone rubber extrusions. The following pointers will be of value in designing good dies:

- There should be no "dead spots" to catch and hold the rubber as it approaches the die. If a quantity of silicone rubber is trapped in the extruder for an extended period, frictional heat may cause it to vulcanize. This will result in restricted flow, rough extrusions, or other troubles. Tapered inserts are often used to eliminate "dead spots" Silicone rubber normally swells as it leaves the die; so the die opening ordinarily must be somewhat smaller than the size desired in the extrusion. It is well to remember that shrinkage during vulcanization and curing will partially compensate for this die swell. In addition, the cross section of the extrusion may be reduced by stretching the unsupported portion between the die and the vulcanizing chamber.

- Flow of silicone rubber should be uniform through all parts of the die. Sharp corners in the die tend to produce excessive drag, resulting in rough edges on the extrusion. This effect can be overcome by putting a slight radius on all corners. Non-uniform flow also results when some parts of the die opening are smaller than others in a cross-sectional area. This can be corrected by providing a shorter land at the constriction. The land is shortened by drilling or machining away part of the thickness of the die (see Figs. 10 and 11). Conversely, uniform flow can be obtained by slowing the flow through large openings in the die. This is done by installing a dam on the screw side of the die to retard the flow.
- Dies should be designed so that corrections or adjustments can be easily made. Figs. 12 and 13, for example, show dies with adjustable dams to vary the rate of flow.

Extrusion Characteristics of Silicone Rubber

Silastic silicone rubber should be extruded at room temperature. In fact, it should not be allowed to reach a temperature above 54°C during extrusion, since higher temperatures may produce scorching and loss of vulcanizing agent.

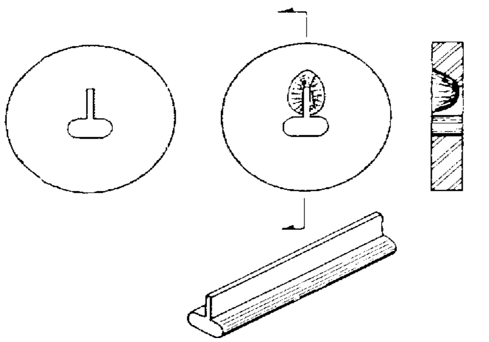


Fig. 12. Die for bulb-type extrusion is machined to have a short land where the thin leg is extruded. This provides the same rate of flow for the thin section as for the heavy section. Another method of balancing flow is shown in Fig. 14 and 15.

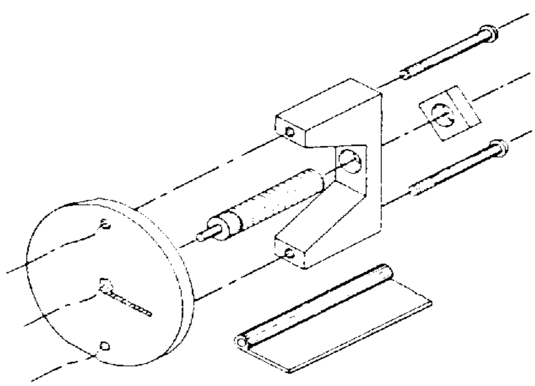


Fig. 14. In this die for a "p" gasket, hole in "p" is formed by pin mounted on bridge. Rate of flow in thick and thin sections of extrusions is balanced by shoulder dam behind small-diameter section of pin. Pin can be positioned along its axis to adjust the rate of flow to suit the rubber used.

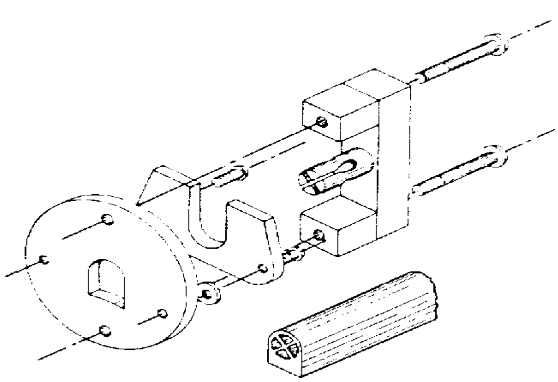


Fig. 15. In this die for a seal, a dam or baffle plate restricts the flow at heavy section of extrusion, to obtain uniform flow for all sections. Here, the rubber must flow between die plate and dam to fill the heavy section. Clearance between dam and die plate can be adjusted as required for different rubber.

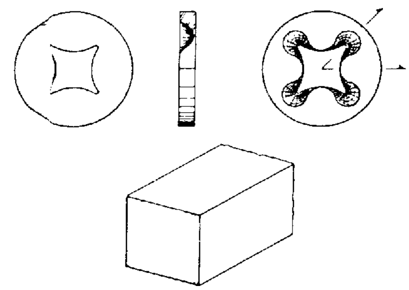


Fig. 13. Die for making square extrusion. Sides of die opening are made convex so that straight sides are formed as the silicone rubber swells upon leaving the die; and comers have a slight radius to help obtain smooth corners on the extrusion. Rear and sectional views show how part of die has been cut away to provide short land at corners to balance the flow of rubber.

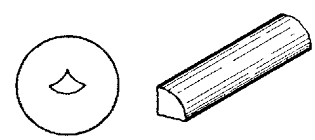


Fig. 16. In die for extruding quarter-round seal, die opening has convex sides to give straight sides on the right-angle portion, and corners have a slight radius to help obtain smooth comers on extrusion.

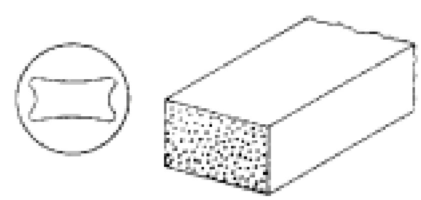


Fig. 17. Die for extruding sponge of square cross-section. Dies for sponge must allow for expansion during blowing (sponge formation) in addition to the expansion that is normal in solid extrusions.

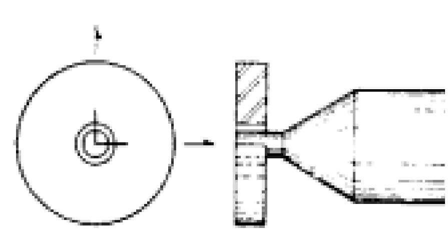


Fig. 19. Die and mandrel for extruding silicone rubber tubing. Mandrel tip is replaceable for easy change of ID of tubing.

Vulcanizing Methods and Equipment

The following paragraphs cover methods and equipment used in vulcanizing extrusions. The vulcanizing agents, their effects on properties of the finished product, and related matters are discussed in the section on vulcanizing.

Hot Air Vulcanizing

This a continuous process, and is the most frequently used method of vulcanizing extrusions. The extrusion is passed through a horizontal chamber heated to 250-500°C or a vertical chamber heated up to 700°C. Extrusions of small cross section require only a few seconds to vulcanize at these temperatures, those of larger cross section require proportionately longer time.

In horizontal HAV (hot-air-vulcanizing) units, the extrusion is carried on a steel or wire-mesh conveyor belt from the extruder through the vulcanizing chamber. In vertical units, the extrusion is dropped downwards by its own weight or pulled upwards through the chamber by a motor-driven drum at the top. In the latter case extrusion is sufficiently vulcanized in the entry area of the chamber to prevent excessive stretching, and as it moves up through the chamber, it continues to vulcanize, gaining strength enough to hold the weight of the material below with little change in dimensions even for heavy profiles.

With both horizontal and vertical units, the size of the unsupported loop of extrusion between the extruder and the vulcanizing chamber should be held constant, to maintain uniformity of cross section.

Both horizontal and vertical vulcanizing chambers can be heated by strip heaters, infrared units, heating mantles, or any other clean heat source. In addition horizontal chambers can be heated with an airstream providing a very fast and efficient heat transfer. The air is heated, and is then blown into the chamber to create turbulence. This provides much faster heat transfer than a static atmosphere. The heating can be electrically or with natural gas heating. To save energy, part of the air stream can be conducted in a circulating system.

All vulcanizing chambers should have an air exhaust system to assure proper removal of volatiles.

Horizontal HAV units are most widely used, and are best suited to extrusions that have one or more flat sides. Vertical units are best for making thin-walled tubing, since there can be no flattening in a vertical unit before vulcanization is complete. In addition, with no conveyor belt, vertical units provide uniform heating around the extrusion, assuring rapid and uniform vulcanization. Thus, in making solid and tubular sponge, they provide a uniform cell size throughout the cross section. They also prevent "snaking" while vulcanizing; and, of course, they produce no belt marks.

Salt Bath

The advantage of a salt bath is an excellent heat transfer and the lack of oxygen inhibition. A mixture of salts (KNO_3, $NaNO_3$ and $NaNO_2$) melting at 140° is used as heat transfer fluid to cure extruded silicone rubber. The extruded profile has to be kept under the surface of the molten salt by guiding rolls. The salt has to be removed of the profile after leaving the salt bath. Typically the profile needs washing, which leads to the requirement of salt recovery for environmental reasons.

Continous Vulcanization in Steam (CV Cure)

For the production of cables a continous vulcanization in steam is common. A pipe is attached to ther extrusion die with a thermic separation, in which pressurized steam at 9 to 15 bar and 180 to 200 °C is blown. Typical length of the steam cure oven is 80 m or more. The wire inside is needed to support the cable in a horizontal CV cure.

Autoclave Vulcanizing

A steam autoclave is often used in vulcanizing small batches of extruded *Silastic* silicone rubber. Loosely coiled in a pan or tray, the extrusion is subjected to steam pressure to provide the required temperature for the required length of time, as determined by the rubber and the thickness of the extrusion.

Oven Curing

To attain the desired properties, most *Silastic* silicone rubber used for extrusions may need to be oven cured after vulcanizing.

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CALENDERING OF *SILASTIC* SILICONE RUBBER

Calendering is used for producing unsupported or supported long, thin sheets of uniform thickness. Many *Silastic* silicone rubber products lend themselves to this process.

Preparing the Rubber

Most *Silastic* silicone rubber requires freshening on a mill before calendering. With soft, sticky rubber that is difficult to calender, let the rubber set 24 hours after milling

Calendering Equipment

Calender

Either a 3-roll or 4-roll calender may be used. The 4-roll unit offers the advantage of working air out of the rubber more thoroughly.

A variable-speed main drive should be provided, to give a centre-roll speed range of .6 to 3 surface metres per minute. In most cases, the calender should be set for skim coating or "even"; i.e. the centre and bottom rolls turn at the same rate and turn faster than the top roll. In rare cases, particularly with stiff or highly filled rubber, an "odd" speed where the centre and bottom rolls turn at different rates gives better results.

Silicone rubber is usually calendered at room temperature. However, a means of heating the rolls should be provided; some rubbers may stick less, and thus process better, with one or more rolls heated. To avoid risk of scorching, the roll temperature must not exceed the decomposition temperature of the vulcanizing agent used.

To keep the rubber from creeping over the ends of the rolls, use nylon ploughs and end plates on all but the bottom rolls. Nylon readily takes the contour of the rolls, and does not give off metal wear particles that might discolour or contaminate the rubber being calendered.

Rotacure

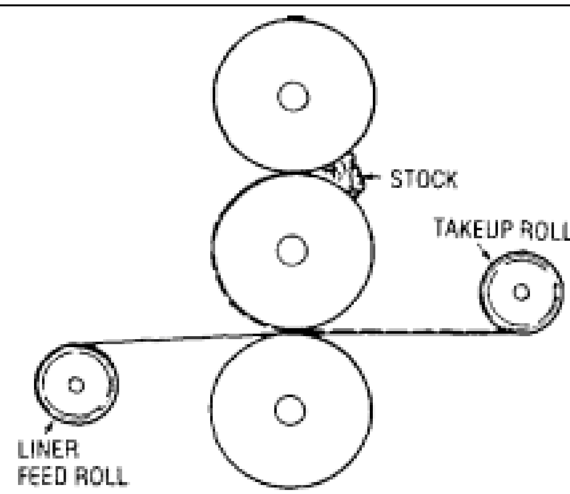
The rotacure unit is a continuous vulcanizing unit consisting of a heated roll with a continuous belt around pressing the sheet to the roll during the vulcanization process. Electrically heated rolls and steam heated rolls are available.

The speed of the rotacure can be adjusted to the speed of the calander so that a continuous vulcanization of calandered sheet is possible. The supporting cloth can be removed in an online process and the finished sheet can be rolled by winding equipment or cut into sheet as needed.

Making Unsupported Sheet

Unsupported sheet is usually made by calendering onto a liner, which is stripped off after vulcanizing. Suitable liner materials include plastic film, holland cloth, and cotton or nylon fabric treated for free release.

Feed the milled rubber to the calender as illustrated, and insert the liner between the calendered rubber and the bottom roll. To start calendering onto the liner, it may be necessary to cut the rubber away from the centre roll whet the liner first comes through the rolls, calander the desired gauge onto the liner, and wind it onto a hollow core.

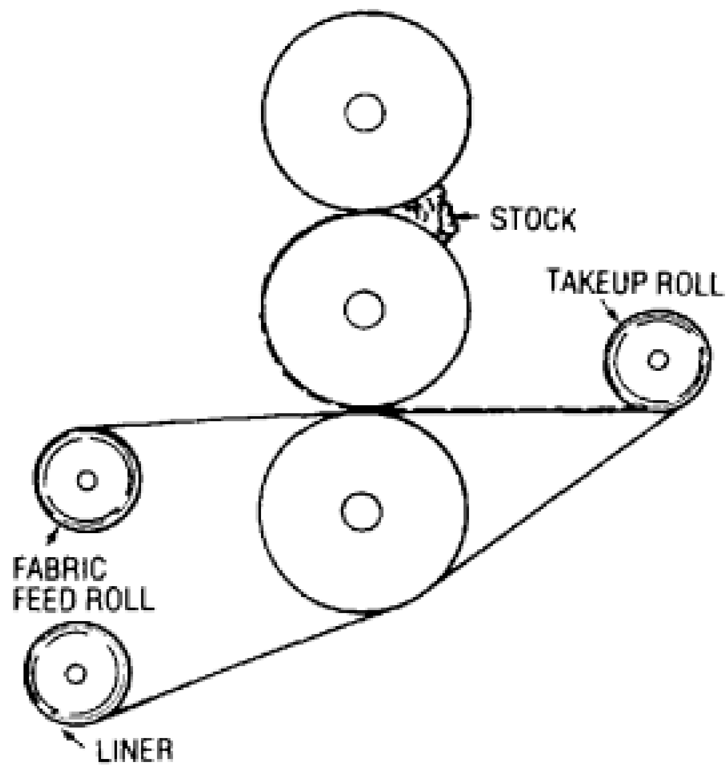


Schematic diagram of calender making unsupported rubber

Making Supported Silicone Rubber Sheet

Silastic silicone rubber may be calendered onto untreated fabrics such as glass, nylon, Nomex high-temperature nylon, Orlon, Dacron, rayon and cotton. To apply *Silastic* silicone rubber to both sides of the fabric, the usual practice is to calender the rubber to the first side and partially vulcanize. Partial vulcanization takes 10 to 30 seconds at 150 to 315°C - the time and temperature depends on the thickness of rubber, vulcanizing agent, and heat-stability of the fabric. Heating for this brief interval can be accomplished by passing the calendered material over a hot drum or by feeding it through a hot-air vulcanizing unit. If the supported sheet is wound onto a core for storage or vulcanizing, use a liner as described under "Making Unsupported *Silastic* Silicone Rubber Sheet" to prevent adhesion between layers.

Insert the liner next to the bottom roll -Polyethylene liner should be used if the calendered sheet is not to be vulcanized. Mylar®, Kodacel, or holland cloth should be used when the calendered sheet is to be vulcanized with a core (aluminium or steel) that will withstand the heat of vulcanization.



Schematic diagram of calender making supported rubber with liner.

DISPERSION COATING WITH *SILASTIC* SILICONE RUBBER

Dispersion-coating of fabric with *Silastic* silicone rubber permits very thin coatings, and gives thorough penetration. As a size coating, the dispersion greatly improves the flex life of glass cloth, and forms an excellent base for calendered coatings of *Silastic* silicone rubber. Fabrics with thicker dispersion coatings may be used for making gaskets, diaphragms, ducts, hose and similar products. The excellent penetration of dispersions is especially valuable with cloth designed for electrical applications.

Silastic silicone rubber dispersed in solvent is applied to fabrics made of glass, cotton, rayon, nylon, Nomex high-temperature Nylon, Dacron, Orlon, Kevlar and other fibres. Such coated fabrics are excellent electrical insulating materials, and improve the flex life of reinforced parts. Also, the coating makes fabrics moisture-resistant, and improves adhesion of calendered silicone rubber. Most silicone rubbers can be dispersed in suitable solvents.

Preparation of the Dispersion

Solvents

Silastic silicone rubber may be dispersed in aromatic solvent such as toluene, xylene or VM&P naphtha although many other solvents may be used.

For dispersing fluorosilicone rubbers, the best solvents are methyl isobutyl ketone and methyl ethyl ketone. See "Precautions in Processing" Page 22.

Mixers

Propeller-type units are most commonly used for making silicone rubber dispersions. Good shearing action is essential to produce a good dispersion. Drive motors should be explosion-proof.

Dispersing

- *Silastic* silicone rubber is best dispersed as follows:
- Soften the compound on a rubber mill.
- Sheet the rubber off the mill and cut into small pieces.
- Place the rubber in a container and add just enough solvent to cover it completely. Then cover the container and let it stand overnight.
- Stir the resulting mass with a propeller-type mixer until a uniform paste is obtained, observing the "Precautions in Processing"
- Add solvent in small amounts, stirring thoroughly after each addition, until the desired concentration is obtained.
- Filter the dispersion, as described below.

Filtering

All *Silastic* silicone rubber dispersions should be filtered before use to remove any foreign matter or undispersed particles. In general, dispersions containing more than 50 percent solids are filtered through an 80-mesh screen. Those with a lower solids content require a finer screen for the same filtering effectiveness. For example, a 35 percent solids dispersion is usually filtered through a 140-mesh screen.

All freshly-filtered dispersions should be allowed to stand before use, long enough to permit the escape of entrapped air. In most cases, standing overnight is sufficient.

Type and Amount of Peroxide

Benzoyl peroxide is the best vulcanizing agent for use in dispersions, because: (1) it is not volatile at room temperature, and (2) its decomposition temperature is high enough to allow the use of heat in removing the solvent after coating.

Any vulcanizing agent used in dispersions will undergo some loss during solvent removal. Furthermore, the half-life of the vulcanizing agent in solvent is relatively short. Thus, a higher-than-normal concentration must be used to maintain the desired concentration. Even with this precaution, the shelf life of the dispersion with vulcanizing agent is reasonably short. About 4 percent benzoyl peroxide (100%) is used for most dispersions, but as much as 10 percent may be needed when making thin coatings or when coating organic fabrics that cannot be heated to temperatures much above 150°C.

Adding Benzoyl Peroxide to Dispersions

With many dispersion applications, the amount of vulcanizing agent needed cannot be predicted, but must be determined through experiment. This is best done by adding vulcanizing agent to the dispersion in increments until the desired vulcanization is reached. Also, the vulcanizing agent may need to be added to make up for losses due to processing or storage. Benzoyl peroxide is readily added to dispersions as described. This material is the best vulcanizing agent for dispersions. It is available commercially in two forms:

- a paste masterbatch, and
- (2) a crystalline solid.

Adding Pigment Masterbatch

Benzoyl peroxide paste is usually a 50 percent active material in a silicone fluid. It is added to dispersions as follows:

- Weigh out the required amounts of paste. CAUTION: Benzoyl peroxide in paste form is an oxidizing material. Observe the manufacturer's recommendation for safety in handling.
- Blend it with an approximately equal volume of the dispersion.
- There is no further filtering; make sure all lumps in the paste are broken down. The resultant mixture should be a

paste are broken down. The resultant mixture should be a smooth homogeneous blend. Lumps will result in voids on the surface of fabric coated with the dispersion.

- Add this masterbatch to the dispersion, and stir until uniformly blended.

Adding crystalline Solid

Benzoyl peroxide in crystalline form is added to dispersions as follows, observing the "Precautions in Processing":

- Weigh out the required amount of crystals.
  CAUTION: Benzoyl peroxide in crystal form is an oxidising material. Observe the manufacturer's recommendation for safety in handling.
- Weigh out the required amount of solvent such as toluene or xylene -preferably, the same solvent as is used in the *Silastic* silicone rubber dispersion -in the ratio of 95 percent solvent to 5 percent benzoyl peroxide, by weight.
- Add peroxide to the solvent and stir until the peroxide is completely dissolved.
- Add this solution to the dispersion, and stir until uniformly blended.

NOTE: Benzoyl peroxide is relatively unstable in the above-named and other solvents, and will decompose and lose its activity within a few days. Therefore, it is best to make a fresh batch of peroxide solution each time the solution is needed. The solution should be used the day it is made -the sooner the better.

Precautions in Processing

Most solvents used in *Silastic* silicone rubber dispersions are flammable, and their vapours are explosive. Mixing with such solvents should be done in closed containers, or under well-ventilated hoods, away from heat, sparks, and open flames; mixer-drive motors should be explosion-proof.

Caution should be taken to avoid heat buildup in blending masterbatches of benzoyl peroxide paste, and in dissolving the peroxide crystals in solvent. A temperature rise of even a few degrees accelerates decomposition to the peroxide in solvent; and this can cause incomplete vulcanization of the dispersion in subsequent processing.

Also, avoid overheating the dispersion while adding benzoyl peroxide and in storage and handling. Temperatures above 55°C can cause partial vulcanization and loss of vulcanizing agent, which will adversely affect the dispersion's performance during coating.

Resoftening stiffened Dispersions

Some high-solids-content dispersions stiffen after standing for an extended period. This is easily remedied by stirring briefly before use, observing the "Precautions in Processing”.

The Coating Process

Selecting the Fabric

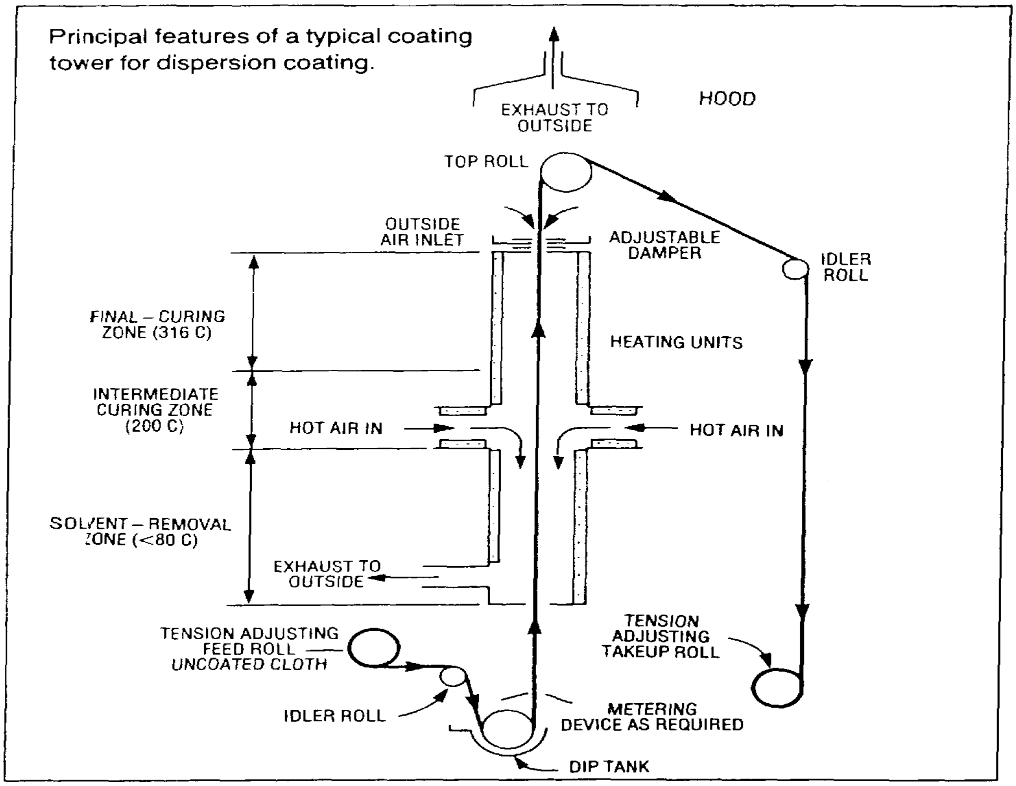
Silastic silicone rubber coatings are often used where heat stability and good electrical properties are required. Since glass cloth is also excellent in these respects, it is the fabric most often dispersion-coated for such application. Usually heat-treated glass fabric with a surface treatment is used. Nylon, Dacron, Orlon and other fabrics can be used where resistance to high temperatures is not required.

Priming

To obtain maximum adhesion of the dispersion, treat the fabric with a primer before coating. Several suitable primers are available from Dow Corning. In some cases, a 5 to 15 percent dispersion of the *Silastic* silicone rubber can be used in place of a primer. The proper concentration of primer depends on the fabric. Hard-finished fabrics require a higher concentration than soft, porous fabrics, since they pick up less of the solution. Primer is conveniently applied by unrolling the fabric and running it through a dip tank.

As the solvent evaporates from the fabric, the primer hydrolyzes by exposure to moisture in the air, forming a film that bonds securely and uniformly to silicone rubber in dispersion. At normal room temperature and humidity, solvent evaporation and primer hydrolysis are completed within half an hour after dipping. If the fabric cannot be draped and exposed to air for half an hour, warm it while the solvent evaporates, then dry it for 5 minutes at 150°C to complete the hydrolysis. A coating tower can be used for this. In dry areas it may be necessary to introduce moisture so hydrolysis will occur.

CAUTION: Most solvents used in dispersions and primers are flammable or combustible; and chlorinated solvents, though not flammable or combustible, may produce toxic vapours. Accordingly, an exhaust system of ample capacity must be provided for the dip tank and for the heated zones - particularly, the solvent-removal zone -to make sure that the vapour concentration is kept below the maximum allowable for the solvent used.



Principal features of a typical coating tower for dispersion coating.

Reverse-Roll Coating

Coating Procedure

Production coating of fabric with *Silastic* silicone rubber dispersions is usually carried out in a coating tower. The fabric is unrolled and fed continuously through the dispersion in a dip tank, then through a heated vertical tower for vulcanization and onto a takeup roll.

The fabric is wetted best when it enters the dispersion at an angle of about 45 degrees; at this angle, it is not so likely to trap air in its interstices.

In most cases, dispersions containing 15 percent to 25 percent solids are used. The exact solids content will vary with the specific gravity of the rubber, the viscosity of the dispersion, and the coating method.

The following methods are used:

Dip-and-Flow Coating

This method is used where relatively thin coatings such as size coatings are desired. A low concentration of dispersion is used to provide free flow. The cloth is passed through the dip tank, and the excess dispersion flows off the cloth as it leaves the tank. The thickness of coating is controlled by varying the concentration of the dispersion, the speed of the cloth through the dip tank, or both.

Knife or Rod Coating

This method is the same as the dip-and-flow method except that after dipping the fabric is drawn past a knife or rod on one or both sides to strike off the excess dispersion. Ordinarily, higher solids content dispersions are used than in dip-and-flow coating, and heavier coatings can be obtained. Thickness of coating is controlled by the concentration of the dispersion, the distance between the knife or rod and the cloth, and coating speed.

A grooved or threaded rod helps control the uniformity of coating, as well as the thickness (the coarser the thread, the thicker the coating). If the thread tends to leave ridges, this can be remedied by allowing the rod to rotate freely. With a threaded rod, the viscosity of the dispersion is less critical than with a knife or unthreaded rod.

With this coating method, a roll rotating opposite to the movement of the cloth is used in place of rod or knife. Some manufacturers find that more complete penetration is obtained with this method.

Drying and Vulcanization

Usually, the coating tower has three zones or sections:

- a solvent-removal zone,
- an intermediate vulcanizing zone, and
- a final vulcanizing zone.

The temperature of the solvent-removal zone should be held below 80°C, to avoid degrading the vulcanizing agent and to minimize the danger of fire. Note that the temperature and length of this zone must be great enough that the fabric is solvent-free before it enters the hotter zones above.

For glass fabrics, the intermediate vulcanizing zone is generally held at about 200°C, and the final vulcanizing zone is often operated at temperatures as high as 316°C. In general, the temperatures in these two zones should be as high as practicable without producing blisters on the fabric. This is especially true for very thin coatings, to avoid loss of vulcanizing agent before the rubber vulcanizes.

For a typical operation-coating .1mm glass cloth to an overall thickness of .25mm -the time in the solvent-removal zone is about 4 minutes; in the intermediate vulcanizing zone, 2 minutes; and in the final vulcanizing zone, 3 minutes. For very thin coatings, these times can be shortened. For heavy coatings, longer times are required.

In dispersion-coating organic fabrics, make sure that the temperatures do not exceed the decomposition temperature of the fabric.

The rubber should be completely vulcanized before it goes over the top roll, to prevent the coating from picking-off on the roll. If complete vulcanization cannot be obtained, the top roll may be heated to vulcanize the rubber more completely and prevent pick-off.

Where unvulcanized coated fabric is produced, a liner of polyethylene or other suitable material must be inserted before the fabric goes over the top roll -to prevent picking-off of rubber on the top roll and idler roll, and to prevent adhesion between layers on the takeup roll *Silastic* silicone rubber can be bonded to many materials, including metals, glass, ceramics, most plastics, silicone-glass laminates, fabrics, and silicone rubber itself. There are two ways of doing this:

- Using unvulcanized rubber, and obtaining adhesion as the rubber vulcanizes.
- Using vulcanized rubber, with an adhesive to provide the bond.

In most cases, a primer is also needed to assure the best bond between the surface and the adhesive.

BONDING *SILASTIC* SILICONE RUBBER

Preparing the Surface

In all cases, the surface to which a bond is desired must be thoroughly clean. In addition, some materials require further preparation, as follows:

Metals:

- Degrease with white spirit or other solvent.
- Clear with acetone+ or other grease-free solvent.
- Let the surface dry completely.
- Coat with a recommended primer.

Glass Ceramics and Etched Teflon:

- Clear with acetone+, or other grease-free solvent.
- Let the surface dry completely.
- Coat with recommended primer.

Organic and Silicone Resin Mouldings and Laminates; Vulcanized Silicone Rubber:

- Roughen the entire bonding area with abrasive.
- Clean with acetone+ or other suitable solvent.
- Let the surface dry completely. Warming will assure complete removal of the acetone.
- Coat with a recommended primer.

Fabrics:

Coat the fabric with a primer or dispersion of *Silastic* silicone rubber in solvent.

Primers

Primers are surface treatments used to promote a strong, uniform bond between unvulcanized silicone rubber or unvulcanized silicone adhesive and the surface to which a bond is desired. They are used on all surfaces -before applying the adhesive or unvulcanized silicone rubber.

Supplied in quick-drying solvents, the primers are applied by wiping, brushing, spraying or dipping. As the solvent evaporates, the active ingredient in the primer hydrolyzes by exposure to moisture in the air. The film thus formed promotes a uniform bond between the primed surface and the unvulcanized silicone rubber or adhesive. In some applications better results may be obtained by diluting the primer with moisture-free solvent.

At normal room temperature and greater than 30 percent relative humidity, solvent evaporation and primer hydrolysis are completed within half an hour after primer is applied.

Hydrolysis and solvent removal can be hastened by oven-drying for 5 minutes at 150°C after a few minutes at room temperature to allow the solvent to evaporate.

CAUTION: Solvents used in primers are flammable or combustible. Provide ample ventilation, and keep away from sparks and open flame.

Adhesives

An adhesive is normally used when vulcanized *Silastic* silicone rubber is bonded to any surface, including vulcanized silicone rubber. Several suitable silicone adhesives are available from Dow Corning.

Bonding

Bonding unvulcanized Silicone Rubber

Prepare the cleaned surface and proceed as follows:

- Place the unvulcanized rubber in contact with the prepared surface.
- Vulcanize under moderate pressure, at the temperature recommended for the vulcanizing agent in the rubber being bonded.
- If oven-cure is recommended for the vulcanized rubber bonded on one side to metal, glass, ceramic and other impermeable material, use time and temperature data for a thickness twice as great as the actual thickness of the bonded rubber.

Bonding vulcanized Silicone Rubber

Prepare the surface of the rubber and the surface to which it is to be bonded and proceed as follows:

Place a .25 to .75mm layer of catalyzed *Silastic* silicone rubber of the same stock on the vulcanized silicone rubber surface.

- Press-vulcanize the uncured silicone rubber to the vulcanized silicone rubber at the temperature recommended for the vulcanizing agent and at a pressure to give firm contact. Best results are obtained with benzoyl peroxide followed by Varox or dicumyl peroxide.
- Oven cure as recommended for vulcanized rubber. For rubber bonded on one side to metal, glass, ceramic or other impermeable material, use time and temperature data for a thickness twice as great as the actual thickness of the bonded rubber. The maximum curing temperature should be at least 10°C higher than the expected maximum service temperature of the bonded assembly.

For the adhesive, an oven cure of 4 hours at 250°C is adequate for most service conditions.

Bonding vulcanized *SILASTIC* Silicone Rubber with one-part Room-Temperature-vulcanizing (RTV) Silicone Rubber Adhesives

One-part adhesives vulcanised silicone rubber to itself and to a wide variety of other materials. *Dow Corning*® RTV silicone rubber adhesive is recommended for bonding conventional silicone rubber. *Silastic*® RTV fluorosilicone rubber adhesive should be used in bonding fluorosilicone rubber.

To use either of these adhesives, prepare the surface of the rubber and the surface to which it is to be bonded and proceed as follows:

- Apply 0.25 to 0.75mm layer of the adhesive to one of the surfaces.
- Place the two parts in contact and hold them together, using only enough pressure to assure good contact between the adhesive and the bonding surfaces.
- Before subjecting the bond to stress, allow about 24 to 72 hours vulcanizing time at room temperature. The length of time depends on the width and thickness of the rubber and the permeability of the material to which it is bonded.

Bonding vulcanized Fluorosilicone Rubber

Vulcanized fluorosilicone rubber can be bonded to itself, to metals, and to most other materials, by using *Silastic*® fluorosilicone rubber adhesive. Follow the procedure given under "Bonding Vulcanized Silicone Rubber with One-Part Room-Temperature-Vulcanizing (RTV) Silicone Rubber Adhesives".

Bonding unvulcanized Fluorosilicone Rubber

Prepare the cleaned surface. Prime with *Dow Corning*® A-4040 primer* and proceed as follows:

- Place the unvulcanized rubber in contact with the prepared surface (or vice versa).
- Vulcanize under moderate pressure, at the temperature recommended for the vulcanizing agent in the stock being bonded.+
- Oven-cure is recommended for the vulcanized rubber. For rubber bonded on one side to metal, glass, ceramic or other impermeable material, use time and temperature data for a thickness twice as great as the actual thickness of the bonded rubber. The maximum curing temperature should be at least 10°C higher than the expected maximum service temperature of the bonded assembly.

***For best results, dilute 50/50 with V&P Naphta.**

+ Use Varox catalyst for maximum adhesion of unvulcanized fluorosilicone rubber

FABRICATING HOSE WITH *SILASTIC* SILICONE RUBBER

Silicone rubber hose, duct and large-diameter tubing are commonly made by mandrel-wrapping, using sheets of unvulcanized unsupported rubber or sheets or tapes of fabric covered with unvulcanized or semi-vulcanized rubber.*

Tapes may be cut parallel to the selvage edge of the fabric or on the bias (at a 45 degree angle). Hose and duct made of bias-cut tape are much more flexible than those made of straight-cut tape.

In most cases, the mandrel is a polished tube or pipe, made of non-corroding metal such as stainless steel or aluminium. For forming irregular shapes, mandrels may be made in several parts to permit removal.

Wrapping the Mandrel

For making a straight section of duct, hose or tube, proceed as follows:

- Spray the mandrel with a light coating of 2 percent to 5 percent solution of detergent in water or isopropyl alcohol, or a 2 percent to 5 percent solution of household detergent in water. Allow the mandrel to dry.
- With sheet material, straight-wrap the mandrel to the desired thickness. With tape, spiral-wrap to the desired thickness by overlapping.
- If a smooth outer finish is desired wrap tightly with high-temperature cellophane or Mylar tape -otherwise, with cotton or nylon tape, soaked with a 2 percent to 5 percent solution of detergent in water for free release. To remove entrapped air, start the wrap at the middle of the mandrel and half-lap to one end, then to the other end and back to the middle.

Extruded Hose

Dow Coming's introduction of high "green" strength (HGS) silicone rubber has enabled hose manufacturers to process silicone rubber on equipment normally used only for organic rubber.

The rubber is first softened on a two-roll mill and stripped from the mill in a standard size. These strips are conveyed to an extruder which forms the rubber into a standard-sized tube. This uncured tube is wound on a roll as it comes out of the extruder. This roll of uncured hose is fed through a knitter where a coating of fabric is applied. During this knitting process, air pressure is used to expand the uncured tube and keep it in the desired size.

*Methods of preparing such fabrics are described in the sections 'Calandering' and 'Fabric Coating' of this guide. Suppliers of fabric manufacturers can be obtained from Dow Corning AETS on request

This covered tube is then processed through a crosshead extruder to apply a coat of silicone rubber over the fabric. The uncured hose can now be cut into desired lengths and placed on straight or curved mandrels. This is wrapped and placed in a steam autoclave for curing.

Vulcanizing

Put the taped mandrel in a steam vulcanizer or autoclave, supported by its ends or by a pipe through the mandrel -and expose it to steam at the temperature required by the vulcanizing agent in the rubber. Usually, the required vulcanizing time is 10 minutes for the first 3mm of thickness plus 3 minutes for each additional 3mm. With a solid or thick-walled mandrel, allow added time for heating the mandrel. The exact time required depends on the *Silastic* Silicone rubber used, thickness of total wrapping, wall thickness of mandrel, and temperature. Thin duct on a thin-walled mandrel may be vulcanized in a circulating-hot-air oven. A 3.2mm thick duct on a 3.2mm-in-wall mandrel will require approximately 15 minutes at 177 to 200°C. Heavier duct, or duct wrapped on a thick-walled mandrel, will not vulcanize well in hot air, due to the slow rate of heat transfer.

Stripping

First let the wrapped mandrel cool to 100°C or less. Then strip off the outer wrapping. For easiest removal of the vulcanized product, pull it off the mandrel while it is still warm. Then rinse it with water to remove all traces of release agent (detergent), and blow off the water.

Curing

Oven-cure the product as described in the section on oven curing. If the *Silastic* silicone rubber is supported (reinforced) with an organic fabric, curing temperatures above 150°C cannot be used in most cases. With inorganic fabrics, use the time and temperature recommended on the data sheet for the rubber used.

FABRICATING ROLLS WITH *SILASTIC* SILICONE RUBBER

A silicone rubber-covered roll has release from sticky materials, and temperatures from -56 to 260°C. The excellent chemical resistance, good stays resilient and serviceable at covering is applied as follows:

Preparing the Core

- If the core was previously covered with organic rubber, a lathe cut is recommended before cleaning to remove all traces of the rubber. Extremely dirty or scaly surfaces may have to be sandblasted. In any case, the core must be thoroughly cleaned.
- Degrease the core with white spirit, or other strong solvent.
- Wipe the core with an acetone-soaked cloth, and let it dry completely.*
- Apply *Dow Corning*® S-2260 primer to the core. Instructions are given in the section on bonding. Be careful not to touch the core with bare hands, dirty gloves, or oily rags before or after primer application.
- To prevent the cover rubber from squeezing out at the ends during wrapping and vulcanizing, attach end-plates to the core. These should be flat circular pieces of metal, large enough in diameter to cover the thickness of rubber and wrapping that will next be applied.

Covering and Wrapping

- Freshen the cover rubber by thoroughly milling until soft, adding the proper amount of vulcanizing agent to rubbers that have no vulcanizing agent.
- Sheet off the mill or calender and apply this sheet to the prepared core, building it up to the desired thickness. This should be somewhat greater than the finished thickness, since the roll will be ground to size later.
- Tightly wrap the covered roll with cotton or nylon tape soaked with a 2 percent to 5 percent solution of detergent in water for free release -or with high-temperature cellophane or Mylar tape to retain the more volatile vulcanizing agents.

To ensure removal of entrapped air and voids, start wrapping at the middle of the roll, and half-lap to one end, then to the other end and back to the middle. The roll is now ready for vulcanizing.

*Acetone is extremely flammable. Provide ample ventilation, and keep away from sparks and open flame.

Vulcanizing

Place the covered roll in a steam vulcanizer or autoclave supported by its core (not resting on the covering) and expose it to steam of the required pressure for the required length of time, as determined by the vulcanizing agent and core.

Stripping

After vulcanizing, let the roll cool to 100°C or lower. Then remove the outer wrapping.

If an oven cure is recommended, do this before grinding the roll to size. Rolls covered with methyl-vinyl compounds other than *Silastic* no-post-cure-series silicone rubber usually require an oven cure for use at elevated temperatures.

Curing

Place the roll in a curing oven supported by its core (not resting on the covering) using the equipment, and procedures presented in the section on Oven Curing. Because the rubber is on a metal core and therefore has only one exposed surface, use time and temperatures for a thickness twice as great as the thickness of the roll cover.

The highest curing temperature should be at least 10°C greater than the highest temperature at which the roll will be used.

Rolls covered with *Silastic* no-post-cure-series silicone rubber vulcanized with Varox or Dicup can be used at temperatures to 260°C without oven curing, with substantial savings in roll-making time.

Grinding

Grind the covered roll to size with a medium-grit (No. 40-60) power-driven abrasive wheel. The roll should be driven by lathe or grinder at 10 to 25 surface metres per minute and a 150mm wheel should turn at 3500 to 4500 rpm, counter to the roll rotation. The roll is now ready for use.

NOTE: To avoid flat spots, the roll should be supported by its core at all times.

FABRICATING SPONGE WITH *SILASTIC* SILICONE RUBBER

Silicone rubber sponge is made by thoroughly mixing a chemical blowing agent into unvulcanized rubber, then heating to decompose the blowing agent and vulcanizing the rubber. Free-blowing and press-blowing (described below) are the methods used. As the blowing agent decomposes, it produces bubbles of gas, which form the cells of the sponge. This increases the length, breadth and thickness of the part by 2 to 3 times -depending on the quantity of blowing agent, temperature, and method of processing.

While it is possible to make sponge from almost any silicone rubber, specific stocks give better results.

Blowing Agents

Note that the blowing agent and vulcanizing agent must be selected to suit the entire part-making process. This includes the forming and vulcanizing method, as well as the sponging method.

To provide the thorough mixing required, the blowing agent is best added on the mill as a masterbatch after the rubber is softened. To prepare the masterbatch, place a quantity of raw rubber on the mill, slowly add an equal weight of blowing agent, mill and cross-blend thoroughly.

Ready-to-use sponge compounds are available from Dow Corning.

Making free blown Sponge

As the name implies, free-blown sponge is made with the rubber unconfined during blowing. Extrusions and calendered sheets can be free-blown and vulcanized in a hot-air-vulcanizing (HAV) unit, in an autoclave or in an oven.

In making sponge from extrusions in an HAV unit maintain a slight tension on the extrusion at the exit end of the unit, to prevent the part from kinking due to expansion during sponging. Sponge made by this method will have some variation in cell structure.

Large, thin-calendered sheets (1,5mm thick or less before sponging), if placed loose in an oven or press, will warp badly during sponging. To prevent this, sandwich the sheet between two pieces of fabric such as cotton, glass or Dacron - first coating the fabric with mica dust, or with a release agent such as a 5 percent to 10 percent solution of detergent in water or isopropyl alcohol. Press the fabric into the rubber to remove all air -and to embed the fabric enough to hold the rubber from expanding sidewise and lengthwise. When this is properly done, the rubber blows in the vertical direction only, producing a flat sheet of sponge. When the above technique (using fabric) is not practical, the sheet must be turned over in the oven after about 1/3 of the sponging time has elapsed.

This provides more uniform heating -and thus, more uniform blowing. If the sheet warps, turn it over an additional time or two as required to obtain the desired flatness.

To produce thick free-blown sponge sheet, ply-up the required thickness of milled or calendered sheet. Then cold-press the rubber between sheets of holland cloth to remove all air from the rubber; strip off the holland cloth; and blow the rubber in an autoclave or oven.

When using an oven, turn the sheet over after 1/3 of the sponging time has elapsed to obtain more uniform heating. lf the sheet warps, turn it over an additional time or two as required to obtain the desired flatness.

Oven Curing

After sponging is completed by either method, the part must be oven cured to remove volatile by-products and enhance compression set. In general, the curing time and temperature should be about the same as for solid silicone rubber 3/4 the thickness of the sponge. Final curing temperature should be at least 14°C higher than the maximum service temperature of the sponge.

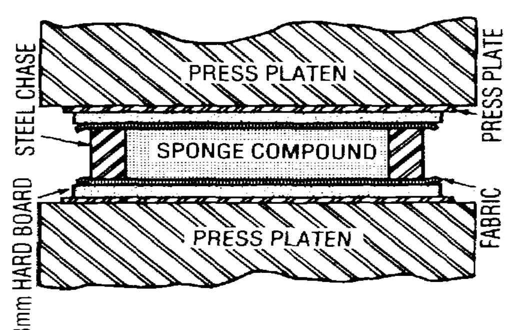
Making press blown Sponge

This method is used for making parts to close dimensional tolerances, and parts of higher density than can be produced by free-blowing. It also produces parts with a thicker and, therefore, stronger skin than is possible with free blowing.

There are two methods of press-blowing: 1-step, and 2-step. Both methods start with a preform of rubber confined in a form, frame or chase in a hot press. For making flat preforms thicker than 6mm sheet the softened rubber off the mill in a thickness of 6mm or less, and ply-up the required thickness.

1-Step-Method

With this method, the preform is blown to finished size (approximately twice its thickness) and is completely vulcanized in the press. Length of press time depends on the temperature, vulcanizing agent, and thickness of part.



Sectional view of press setup for making silicone rubber sponge, using hardboard liners covered with fabric.

2-Step-Method

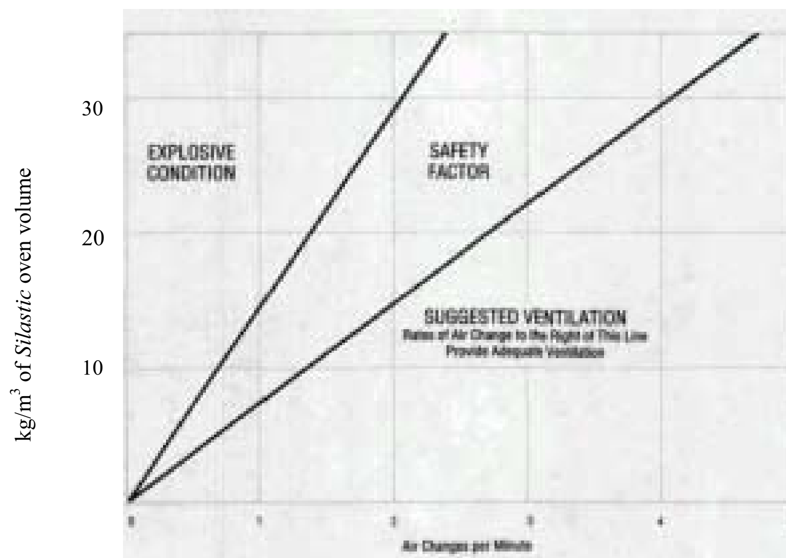
This method consists of hot-pressing the preform just long enough to partially vulcanize it, then free blowing it in a hot-air-circulating oven, or blowing in a press that has shims to maintain the desired thickness.In moulding, press the preform between 3mm hardboard liners covered with glass cloth, cotton, nylon cloth, or heavy Dacron fabric. This provides a tough, uniform skin and uniform cell structure and, in 2-step moulding, it provides constant density over a wide range of thickness. In addition, the fabric prevents trapping air on the surface, thus preventing the formation of surface holes. The fabrics listed must be dusted or otherwise treated for release. and, in 2-step moulding, it provides constant density over a wide range of thickness. In addition, the fabric prevents trapping air on the surface, thus preventing the formation of surface holes. The fabrics listed must be dusted or otherwise treated for Specific gravity ranges from 0.20 to 0.50. Oven blowing at somewhat higher temperatures will result in lower specific gravity. Manufacturing conditions (including rubber, dimensions of part, and processing equipment) may differ widely. However, in any case

- Before oven blowing, trim off the vulcanized edges of the slab.
- During the blowing process, if dishing or warpage occurs, turn the slab over in the oven occasionally to correct or prevent this.

POSTCURING OF *SILASTIC* SILICONE RUBBER

Many silicone rubber parts require a postcure in addition to vulcanization, for two reasons:

- To drive off volatile materials from the rubber. Otherwise, if the part is heated in service, these volatiles might make the inside soft or porous -especially if the part is largely confined.
- To stabilize the properties of the rubber for high-temperature service. Curing is done by heating the parts in a circulating-hot-air oven to the required temperature for the required length of time.



Graph for determining safe load limit for any oven used for curing *Silastic* rubber.

Postcuring Ovens

To assure both safety and proper cure, any oven used for curing or postcuring silicone rubber must have the following features:

- It must maintain the required temperature within a few degrees.
- It must have adequate internal circulation of air.
- It must have an exhaust system of sufficient capacity to prevent volatiles from reaching an explosive level. The exhaust system must be vented to the outside.

These features and others are covered in the following paragraphs.

Heat Source

Either electric resistance elements or gas burners may be used to heat the oven. These should be in a separate compartment - to eliminate hot spots due to radiant heat, and to provide for heating fresh air from the room before it is admitted to the curing compartment. When gas burners are used, the fresh-air intake should be located so that the flame is supplied with air from the room rather than with air drawn from the oven. If air containing volatile silicone materials is used in combustion, a fine silica powder will be formed and may be deposited on oven surfaces and the parts being cured.

Temperature Controls

The oven should have an automatic controller that will maintain the oven temperature within ±5 °C. A temperature recorder is convenient, but not essential.

To reduce the possibility of fire and loss of parts due to overheating, a separate temperature-limit switch should be provided to turn off the heat if the automatic control fails. This is a must when curing fluorosilicone rubber, for such materials may give off toxic vapours at temperatures above 288°C. Thermocouples are satisfactory for measuring temperatures up to 250°C, which is normally the highest temperature used in curing. To show the true oven temperature, they must be in the circulating air stream and away from hot or cold spots.

Air Circulation

Good curing requires good circulation of air in the oven to ensure that all of the parts being cured receive the required heat and ventilation. To obtain this:

- The circulation should be horizontal rather than vertical. This calls for forced-air circulation, at an adequate rate as described under "Load Capacity".
- The oven shelves should be of expanded metal, lined with open-weave glass cloth to prevent marking the parts. A light dispersion coating of silicone rubber on the cloth will increase its life.
- The parts should not be stacked or otherwise in contact with each other.
- Make sure that the parts are not distorted, as they may take a permanent set while curing.
- When long lengths of tubing with a wall thickness greater than 1.5mm are being cured it is advisable to provide air circulation through the tubing to remove volatiles. A very slight flow of air is sufficient. A piece of copper tubing can be led into the oven and coiled or run for several feet inside the oven to preheat the air before it enters the rubber tubing. If this is not done there is a good possibility that the silicone rubber tubing will revert and degrade throughout much of its length.

A safety switch must be provided to turn off the heat if the blower stops.

Load Capacity

The load capacity of any curing oven depends in part on the capacity of the blower used for air circulation. For a given weight of rubber per cubic foot of oven space, the blower must produce the number of air changes per minute shown. This graph is based on explosion tests made while curing silicone rubber at 250°C in a fixed-volume chamber.
As a safety measure, determine the safe load limit for each oven, and post it conspicuously near the oven.

Precautions

Before heating the oven for curing or postcuring *Silastic* silicone rubber, remove any flammable material such as rags, paper, wood, plastics or organic rubber.
The volatiles given off by silicone rubber in curing or postcuring are irritating, combustible, and detrimental to the parts being postcured. Accordingly, the oven should be vented to a point outside the building.
No fluorosilicone rubber should be heated above 288°C, as it may give off toxic vapours at higher temperatures. Always use a temperature-limit switch (see Temperature Controls) when curing fluorosilicone rubber. Never overload the oven beyond the capacity of the blower (see Load Capacity). Vent stacks should be cleaned periodically to ensure adequate removal of volatiles. Stacks can become clogged with silica.

Schedules for Oven Curing

In general, the time required for curing at a given temperature depends on the rate at which the volatiles can escape from the rubber. This, in turn, depends on the thickness of the part. It also depends on the amount of exposed surface. For instance, a 12mm slab bonded on one side to metal or other impermeable material will require about the same cure schedule as a 25mm slab with both surfaces exposed.

Thin Sections

For sections up to 3mm thickness an oven cure of 4 hours at 200°C is usually sufficient for most applications.

Thick Sections

Thicker sections may require a step wise post cure with an initial cure for 1 hours at 160°C and steps of 20°C for 1 hour by allowing controlled evolution of volatiles to prevent blowing, internal rupture or reversion phenomina. This can be followed by a cure at 200°C.

Final Curing Temperature

As a general rule, parts must be cured through a temperature at least 10°C above the highest service temperature.

EXCEPTIONS: Where maximum resistance to compression set is wanted, and when parts are to be completely or almost completely confined in service, give the full cure recommended in the data sheet for the stock being used.

Checking the Cure

When curing parts with thick sections, it is good practice to run a control piece of the same thickness that may be cut in half to check the internal cure.

In determining the proper curing schedule for your particular conditions, run several such control pieces at the same time. You will find that, as curing progresses at any given temperature, the interior of the piece is first softened somewhat, then gradually hardened. When a control piece shows that the interior hardness is at the proper value, raise the oven temperature to the next-higher temperature and continue the cure.

Vulcanizing Agent

For sections thicker than 12mm, vulcanizing agent should be for a vinyl-specific peroxide such as Varox or Dicumyl - peroxide. These vulcanizing agents can be used with most *Silastic* silicone rubber products supplied without a vulcanizing agent.

Your Global Connection

Dow Corning has sales offices, manufacturing sites, as well as science and technology laboratories around the globe. Telephone numbers of locations near you are available on the world wide web at **www.dowcorning.com**, or by calling one of our primary locations listed below.

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Cover photo - AV01066, inside photo's - AV1065, AV01066, AV01203.

Form no. 45-0111B-01

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