Silicone Coatings

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Although silicone polymers are based on silicon dioxide, the most abundant component of the earth's crust, they are a relatively recent development in the coatings industry compared to polymers of carbon compounds.

Silicone polymers, based on Si-O-Si linkages, have superior heat and ultraviolet resistance (based on bond energy and oxidation resistance) compared to polymers based on carbon-to-carbon linkages.

Silicone polymers generally can be classified as fluids, gums, and resins. The difference between these is related to molecular weight, cross linking, and composition of organic groups attached to the silicon atom.

- Silicone fluids are linear polysiloxanes composed primarily of methyl groups and silicon, although other organic groups such as phenyl can be used. A linear siloxane polymer based on methyl is called polydimethylsiloxane.
- Silicone gums are high molecular weight fluids with some cross linking and functional groups such as vinyl. They are used to make silicone elastomers.
- Silicone resins vary in cross link density and organic substitution as well as molecular weight. Phenyl on silicon is better for heat resistance, whereas methyl allows for better cure and water repellency. The resin used depends on the requirements of the end use application.

This introduction to silicone resin technology is intended as an overview of coating systems for high-temperature applications.

History

The first commercial applications for silicone resins came during World War II, when they were used as damping fluids in aircraft engines to prevent corona discharge at high elevations. Resinous silicones prepared from trifunctional organosilicon intermediates were used with glass tapes as insulation in electric motors. Then, in 1945, both Dow Corning and General Electric announced the development of silicone rubber that was useful at high and low temperatures. When the war ended that year, military demands for silicones ceased, but their use was adapted to a peacetime economy. Applications soon surpassed the earlier requirements for military use, and expansion of facilities became necessary for all producers. As production of silicones increased, so did their uses. Silicone fluids and certain silicone resins are now commodity chemicals with wide ranges of applications.

The range of high temperature service for silicones continues to increase.

Most silicone resins can be classified as low to moderate molecular weight (2,000 to 50,000) phenyl and methyl cross linked silanol polysiloxanes. Silicone resin characteristics depend on the following:

- molecular weight,
- phenyl to methyl ratio,
- degree of cross linking, and
- silanol content.

Silicone resins are available as solids, emulsions, and organic solvent solutions.

Development of silicone resins made possible hundreds of coating formulations that can withstand high temperatures. What began as a few applications has grown to a substantial market for coatings using silicone resins as vehicles, reactive intermediates, and additives. Through expanding silicone chemistry and formulation know-how, the range of high-temperature service continues to increase, while the corrosion resistance, weathering resistance, and color retention of silicones improve in a broad spectrum of medium- to high-temperature coatings.

Aluminum flake and black-pigmented silicone coatings for purely functional uses were the first major commercial applications of these high-temperature products. Exhaust stacks, boilers, engines, heat exchangers, mufflers, stove parts, and aircraft components were all coated with these silicone formulations during the early 1950s to provide protection at temperatures up to 1500 F (816 C). Also during the 1950s, pigments for a variety of colors became available, expanding the utility of

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silicone coatings for applications with special color requirements, such as olive drab space heaters and exhaust pipes for military trucks.

Formulators at that time also began to modify silicones with organic polymers, or modify organic polymers with silicones, to significantly lower the product's cost while still providing quality performance.

Alkyd resins are still the most widely used coating in maintenance painting. This popularity is due to their good weathering properties, ease of application, and low cost, as well as the low toxicity of their aliphatic solvents. Silicone modification of the alkyd resin improves its overall weathering resistance and, to some degree, heat resistance. The inertness of the silicone, coupled with its moisture resistance, produces air-drying silicone alkyd copolymer resins and paints with excellent exterior durability.

Silicone alkyd copolymer paints have the same general physical properties as conventional alkyd paints. These paints can be applied by either brush or spray. They require no unusual surface preparation. It is generally believed that the same types of primers used for conventional alkyds can also be used with silicone alkyds since the main function of the silicone is to provide topcoat protection.

The current applications of silicone alkyd copolymer paints include outdoor storage tanks, ships (above deck line), chemical process equipment, buildings, bridges, and many other steel structures where improved film life and good exterior weathering resistance are required. Heat resistance of these paints depends on the silicone content of the copolymer as well as the type and content of oil in the alkyd.

Specifications for silicone alkyd copolymer paints have been written by the U.S. government, states, companies, and nonprofit organizations. Examples include 2 specifications by the SSPC: SSPC-Paint 21, White or Colored Silicone Alkyd Paint, and SSPC-PS 16.01, Silicone Alkyd Painting System for New Steel. MIL-E-24635 (Enamel, Silicone Alkyd Copolymer) is widely used by the U.S. Navy and other government departments. It replaces TT-E-490 (Enamel, Silicone Alkyd Copolymer, Semigloss [For Exterior and Interior Nonresidential Use]), the first specification for silicone alkyd paints. MIL-E-24635 covers 4 classes of gloss and 3 levels of volatile organic compounds (VOC), from 3.5 to 2.8 to 2.3 lbs/gal. (420 to 335 to 275 g/L), as applied. In addition to low VOC, high solids silicone alkyd copolymer paints, several water-reducible paint

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systems provide excellent exterior weathering resistance (minimum 100 percent improvement based on gloss retention, chalk resistance, and color retention) and improved heat resistance (minimum 50 percent improvement based on gloss and color retention).

As with many other technologies, advances in silicones were spurred by the space program and later applied to other industries. A 100 percent silicone coating (providing heat resistance to 2,500 F [1,371 C]) used to protect the space shuttle's orbiter's tiles during reentry to the earth's atmosphere[1] has had subsequent use in the nuclear fabrication industry. Later, a patented process with this coating was developed in the chemical processing industry to protect fused-glass structures from crystal formation at temperatures of 1,800 F (982 C) during the manufacture of titanium dioxide.

General Characteristics and Performance Properties

In this column, high temperature service is defined as 250 F to 1,400 F (121 C to 760 C). Within this broad range, 5 general categories of coatings effectively meet high temperature needs (Table 1). Most use silicone resins by themselves or with organic resins.

As indicated in Table 1, the term "silicone-modified organic" refers to a coating in which the silicone resin solids are typically 15 to 50 percent of the total formulation. The silicone resin acts as a reactive intermediate to upgrade performance of the organic. Its main purpose is to improve heat stability and exterior durability. In contrast, an "organic-modified silicone" is a formulation with more than 50 percent silicone resin solids. The organic modification improves abrasion resistance, hardness, and adhesion. It also provides faster curing and reduces thermoplasticity.

Polyorganosiloxane resins for high-temperature coatings are usually a mixture of organic groups attached to silicon atoms. While many factors contribute to a coating's resistance to high temperatures, the primary reason these resins are effective is their excellent bond energy, or energy required to break down the siloxane bonds.

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Table 1
5 Types of Silicone Coatings

Temperature*	Coating System Type
250-400 F (121-204 C)	Silicone-modified organic (all pigmentation)
400-600 F (204-316 C)	Silicone-modified organic (aluminum) Organic-modified silicone (colored)
600-800 F (316-427 C)	Organic-modified silicone (black and aluminum) Silicone (colored)
800-1,000 F (427-538 C)	Silicone (black) Silicone (aluminum)
1,000-1,400 F (538-760 C)	Silicone (ceramic)

**1,000 hours minimum dry heat (low moisture)*
Silicone-modified organic—15-50 percent silicone resin solids
Organic-modified silicone—50-90 percent silicone resin solids

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Silicon-oxygen bonds, for example, are far superior in strength to carbon-carbon bonds, so they resist heat and ultraviolet light much longer before disintegrating. A silicon-oxygen bond provides an energy of 108 kcal (kilocalories) per mole, while a carbon-carbon bond has only 82 or 83 kcal per mole. The higher the percentage of silicone in a formulation, and, consequently, the more of these bonds in a coating, the more heat resistance it will provide.

Of organic groups, methyls and phenyls are most common in high-temperature coatings. High phenyl-containing resins exhibit good heat and oxidation resistance and good shelf life. High methyl-containing resins are superior in hot hardness (pencil hardness at elevated temperature), flexibility, water repellence, low-temperature properties, chemical resistance, cure rate, and thermal shock resistance. Because formulators seek a combination of these properties, copolymers made with both methyl and phenyl groups have become common.

In most cases, coatings made from a 100 percent silicone resin will work best for the needs of extremely high temperature service (600 F [316 C] or higher). This does not imply, however, that a 100 percent silicone will serve all coating needs. A coating also must meet other requirements: adaptability to its substrate, corrosion resistance, flexibility or hardness, stability during temperature cycling, color stability, water resistance, and desirable economics. Therefore, for each temperature category and the coating's other service requirements, the formulator must develop a product that takes best advantage of various silicones, organic polymers, and inorganic pigments, often using a precise combination of all three. One common error in selecting coatings for high-temperature service is assuming that a single high-temperature coating will be right for all applications above 250 F (121 C).

Temperature Resistance of Coatings

The words "long-term" and "short-term" sometimes are used to describe the temperature resistance of silicone coatings. "Long-term" generally refers to a prolonged, consistent heating cycle of at least 1,000 hours, such as might be required for a utility stack or heat exchanger. "Short-term" heating cycles are less than 1,000 hours—typically less than 10 hours—but they are repeated many times. An example is a grill or gas-fired stove that undergoes repeated short heat flashes. A coating that can

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withstand a certain temperature for a long-term heating cycle also might withstand an even higher temperature for repeated short-term cycles.

Long-term heat resistance of a typical phenylmethyl polysiloxane resin is limited to about 500 F (260 C), based on half-life studies.[2] This means half of the weight of the polymer will be degraded at this temperature in 1,000 hours. The phenyl and methyl groups will slowly oxidize from the siloxane backbone, resulting in a more brittle coating and, ultimately, film fracture and failure.

The addition of heat-resistant pigments increases heat resistance of silicone as well as organic paint systems. The major factors determining heat resistance are silicone or siloxane content of the resin binder and pigmentation type. The most heat-resistant pigments are inorganic and include ceramic frits, aluminum, zinc, and metal oxides.

Advances in formulation have occurred across the temperature range. By temperature category, from lowest to highest, coatings have been developed from the following materials.

250-400 F (121-204 C)

The silicone resin content in this coating is typically 15 to 50 percent of the total resin formulation. The main purpose of the silicone resins is to improve heat stability and exterior durability.

This formulation is commonly achieved by cold-blending the silicone and organic resins before or after pigment dispersion. This method offers an economic advantage because copolymerization facilities are not necessary. However, the types of silicone resins adaptable to coldblending with non-silicone resins are more expensive on a solids basis than those that can be copolymerized. Further, cold-blending does not chemically combine the 2 resins, leaving the reactive groups on the organic and silicone resins susceptible to hydrolysis. Hydrolysis, in turn, limits the coating's ability to resist chemicals and solvents.

Many organic resins can be cold-blended with suitable silicone resins, provided there is a sufficient number of carbonyl groups on the organic resins. Organic systems used to make silicone-modified organic coatings include alkyds, phenolics, epoxies, epoxy esters, acrylics, and saturated polyesters.

If copolymerization is the desired formulation method and the formulator is willing to make a long-term investment in required equipment, the benefits include lower resin cost, proprietary formulation, and coatings with greater chemical and solvent resistance. The 2 primary silicones available for copolymers are the methoxy-functional and silanol-functional types.

400-600 F (204-316 C)

The addition of leafing aluminum pigment will increase the heat resistance of all paints, including organics. For optimum results, the silicone content required for this intermediate temperature range is 15 to 50 percent for aluminum finishes and 50 to 90 percent for colored finishes. Most colored pigments are less heat stable than aluminum, so they require higher levels of silicone. Organic-modified silicones are best for temperatures up to 600 F (316 C).

600-800 F (316-427 C)

Increasing the temperature limit for aluminum and black coatings requires higher silicone content of the resin. Silicone content is 30 to 70 percent for aluminum finishes and 70 to 100 percent for colored finishes. Increasing silicone content usually is accomplished either

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by adding silicone resin to a silicone organic copolymer or blending an organic resin with a silicone resin. Colored coatings other than black require metal oxide pigments for best results. Black coatings can use metal oxides or a blend of metal oxides, graphite, or carbon black.

800-1,000 F (427-538 C)

Aluminum-pigmented coatings provide best performance at the high end of this temperature range because Si-O-Al and Si-O-Si bonds result in a very stable oxide mixture. Low gloss black oxide silicone coatings are heat resistant at the low end of this temperature range (800 F [427 C]). Silicone content is usually 100 percent for these finishes.

1,000-1, 400 F (538- 760 C)

Coatings made from 100 percent silicone resins and ceramic frits can provide prolonged service at very high temperatures. When exposed to these temperatures, the silicone resin thermally decomposes and the frits fuse into a substrate-Si-O-Si bond. This results in a finish that is both durable and heat stable.

Formulations and Properties

While the formulator has a choice of many material combinations, the 3 formulations in Tables 2, 3, and 4 are common examples of coatings for hightemperature service. They illustrate 3 heat-resistant levels based on pigment selection and the variation in silicone resin selection for high and low VOC content.

A VOC is any organic compound involved in atmospheric photochemical reactions. High VOC resins are typically 50 or 60 percent solids by weight with molecular weight of 50,000 to 100,000. Low VOC resins are usually 70 percent solids by weight with molecular weight below 10,000.

Silicone aluminum paint (Table 2)

Table 2
Silicone Formulation (Aluminum)

Paint Formulation	**Parts**	
Aluminum Paste (65%)	50	
Silicone Resin Solids	50	
	100	
Paint Properties		
Paint Preparation	Low Speed Blade	
Pigment/Binder Ratio	65/100	
Solvent	Aromatic	
Viscosity, #4 Zahn Cup, Seconds	30	
	Lower VOC Resin	**Higher VOC Resin**
Pounds per Gallon (g/l)	9.1 (1090)	8.7 (1040)
% Solids, ASTM D 2369*	58	39
VOC, lbs/gal. (g/L) (in can, as formulated)	3.8 (456)	5.3 (635)
Maximum Service	**Temperature**	
Short Term (< 1,000 hours)	1200 F (649 C)	
Long Term (> 1,000 hours)	650 F (343 C)	

**ASTM D2369, Test Method for Volatile Content of Coatings*

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is very heat resistant because oxides of aluminum and silicon form a hard, ceramic-like protective coating at high temperatures. This coating, as well as the white coating (Table 4), has excellent heat resistance regardless of the resin sources.[3]

Colored heat-resistant coatings must contain heat-resistant metal oxide pigments for optimum results. Low gloss black paint (Table 3) can be used in applications such as vehicle mufflers and grills.

Silicone resin emulsions were introduced to the coatings market about the same time high solids silicone resins were being promoted for high-temperature applications.[4] Like any material, the emulsions have their advantages as well as their limitations.

Paint formulations for silicone resin emulsions are more difficult than solvent-borne systems and, like organic emulsion paints, require additives such as surfactants, wetting agents, defoamers, and pH adjusters.[5] Freeze-thaw stability is a concern with any emulsion paint. The advantages of these coatings are low VOC levels, high flash point, and easy cleanup.

Heat resistance of silicone resin emulsion coatings is comparable to conventional and high solids silicone coating systems. A wide variety of pigments can be used with silicone resin emulsions. Aluminum paints must be made with treated aluminum flake to prevent reaction of water with aluminum metal.

Applications

By themselves or in combination with organic film formers, silicone resin-based coatings have reduced the maintenance and increased the longevity of processing equipment and appliances. Some of the more common applications for 100 percent silicone coatings include

- high-temperature stacks;
- mufflers and manifolds;
- boilers, ovens, and furnaces;
- steam lines;
- heat exchangers;
- cooking utensils;
- combustion chambers;
- incinerators;
- wood-burning stoves; and
- barbecue equipment.

When the silicone resins are cold-blended or copolymerized with organic resins such as polyesters, alkyds, epoxies, and acrylics, the list of applications expands to include

- space heaters;
- camp stoves;
- ranges and dryers;
- lanterns;
- light bulbs;
- generators; and
- processing equipment.

Solvents, Extender Pigments, and Catalysts

Many silicone resins for high-temperature service can be thinned with aromatic hydrocarbon solvents and hydrocarbon blends, including most ketones and esters. Consult the manufacturer's application instructions for the proper thinner to use.

Extenders also can be included to supply bulk or fill in the formulation. Typical extenders include water-ground mica, micronized mica, magnesium silicate, and aluminum silicate.

Additives, likewise, are often included to achieve a special effect, such as thickening or wetting.

Coatings based on silicone resins will cure with heat without driers. A typical cure schedule for a silicone resin is 1 hour at 400 F (204 C) or 30 minutes at 450 F (232 C). If the coating contains less than 50 percent silicone, it will cure through the organic resin cross linking mechanism. If the silicone content is greater than 50 percent, adding metal catalysts

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will help cross link the system. Metallic driers such as iron octoate or zinc naphthenate cam be used to reduce curing time or temperature. Some of the common metal naphthenates, the percentages added to formulations (metal based on resin solids), and their effects are listed below.

- zinc, 0.1-0.5 percent, best all-around catalyst;
- iron, 0.01-0.1 percent, fastest catalyst, can affect color and shelf stability; and
- manganese, cobalt, 0.1-0.5 percent, give good top hardness but affect color.

Other driers based on compounds of metals such as calcium, potassium, titanium, and tin tend to reduce the heat resistance of silicone coatings. Iron driers are very effective but reduce the shelf life and heat resistance of the formulation.

Driers for silicone-modified resins should be selected according to the organic constituent. Cobalt, manganese, calcium, and zirconium driers can be used in formulations containing drying oils.

Application Guidelines

For the most part, the application methods for silicone-based coatings are the same as those for coatings based on organic resins except that silicones may require heat cure. However, the application of silicone coatings often requires more care and attention to details of surface preparation and film thickness. This is not because of the nature of the resin, but rather because of the high performance required from the coating and the severe conditions to which it may be exposed.

Silicone-based coatings occasionally are used where the primary function is decorative. Usually, though, the chief function is protection against high temperatures.

Consequently, all phases of the application process are important. These steps include preparing the surface, priming the surface, applying the coating, and curing the coating.

Surface Preparation

Poor or inadequate surface preparation is one of the most common reasons a silicone resin-based coating may not fulfill its potential. The coating must be applied to a clean, dry surface.

Cold or hot temperatures may require specially formulated thinners or solvent blend in the manufactured coating to control the rate of solvent evaporation and proper film formation.

Oil, mill scale, rust, and other surface contaminants should be removed thoroughly by chemical or mechanical means. Any contaminant that prevents complete contact between the surface and the coating may result in poor adhesion and premature film failure. This is particularly true if the contaminant is organic and the coating is subjected to high temperatures during service.

Abrasive blasting is recommended for preparing steel surfaces wherever practical. Adhesion of silicone resinbased coatings to smooth steel, even when thoroughly cleaned, is only fair. However, when the steel has been cleaned as well as profiled with abrasive blasting, adhesion is excellent. For best results, clean the surface to Near-White Blast Cleaning (SSPC-SP 10).

For non-ferrous metallic surfaces such as aluminum, copper and brass, wire brushing and solvent cleaning usually provide satisfactory surface preparation. Care should be taken to remove factory-applied protective lacquers or other coatings from new metal, particularly aluminum.

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Table 3
Silicone Formulation (Black)

Paint Formulation	Parts
Black Metal Oxide	16.3
Mica C-3000 (mesh mica)	22.7
Silica	7.2
Silicone Resin Solids	53.8
	100.0

Paint Properties		
Paint Preparation	High Speed Blade	
Pigment/Binder Ratio	86/100	
Solvent	Aromatic	
Viscosity, #4 Zahn Cup, Seconds	30	
	Lower VOC Resin	**Higher VOC Resin**
Pounds per Gallon (g/l)	11.9 (1400)	10.1 (1200)
% Solids, ASTM D 2369	70.1	59.3
VOC, lbs/gal. (g/L) (in can, as formulated)	3.6 (431)	4.1 (491)

Maximum Service	Temperature
Short Term (< 1,000 hours)	1000 F (538 C)
Long Term (> 1,000 hours)	500 F (260 C)

Table 4
Silicone Formulation (White)

Paint Formulation	Parts
Rutile TiO_{2+}	45.0
Mica C-1000 (mesh mica)	10.0
Silicone Resin Solids	45.0
	100.0

Paint Properties		
Paint Preparation	High Speed Blade	
Pigment/Binder Ratio	122/100	
Solvent	Aromatic	
Viscosity, #4 Zahn Cup, Seconds	30	
	Lower VOC Resin	**Higher VOC Resin**
Pounds per Gallon (g/l)	12.3 (1500)	10.5 (1300)
% Solids, ASTM D 2369	74.0	56.0
VOC, lbs/ga.l (g/L) (in can, as formulated)	3.2 (383)	4.6 (554)

Maximum Service	Temperature
Short Term (< 1,000 hours)	700 F (371 C)
Long Term (> 1,000 hours)	450 F (232 C)

Silicone resin-based coatings have excellent adhesion on cleaned aluminum surfaces. These coatings also adhere well to magnesium and tin plate. However, adhesion to copper, zinc plate, and galvanized surfaces is generally only fair. Adhesion is enhanced by the physical abrasion of the substrate, either by wire brushing or abrasive blasting.

Anticipated service conditions determine not only whether a primer should be used under a silicone resin-based coating but also the type of primer to be selected. The type of surface also may have a minor effect on the choice of primer.

Primers are used with silicone resin-based coatings for the same reasons they are used with other types of coatings—to promote better adhesion and protection under certain environmental conditions. A primer may retard the spread of rust or corrosion in case of unexpected breakdown of the surface film.

Generally, silicone resin-based maintenance coatings adhere well when applied directly to metal surfaces. This is true of nearly all coatings applied to equipment used indoors or outdoors under dry conditions. However, if the coating will be exposed to high temperature, moisture, or corrosive conditions, a primer is usually beneficial. Best results are obtained with primers that are based on either silicone resins or silicone-modified resins and that contain zinc dust. Primers used with silicone resin-based maintenance and industrial finishes for metal promote corrosion resistance and adhesion.

Application

Silicone-based coatings usually can be applied by conventional methods. Most are designed for spraying, but formulations may be adjusted for brushing, dipping, silk-screening, or roller-coating. If more than 1 coat of

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a baked paint is to he applied, and if it is impractical to cure the first coat before applying the second—such as on a stack or similar piece of equipment—then the second coat must be spray applied.

Application must also be controlled. A film that is applied thicker than the manufacturer recommends––for example, 2 coats at 3 mils (75 microns) rather than 2 coats at 1.5 mils (38 microns)—can lead to early failure when the coating is subjected to high temperatures. Failure is often in the form of cracking.

Curing

Coatings based on silicone resins generally require baking or curing at elevated temperatures to achieve optimum film properties. This is especially true if the coating will be exposed to extreme temperatures or to thermal cycling and shock.

There is more danger of undercuring than overcuring silicone-baking enamels. Undercuring causes films to be relatively soft and have poor adhesion.

Many coatings based on silicone-modified resins will develop good film properties by curing at ordinary atmospheric temperatures. This is particularly true if the organic resin used is a long-oil alkyd. Special organometallic driers sometimes are used with silicone-modified resins to promote curing at ambient temperatures.

A coating sometimes can be formulated to air dry tack-free, then complete its cure when the unit is put into service. This approach to curing is used commonly on equipment such as stacks, boilers, and furnaces where coatings cannot be baked.

Success of this method requires the following conditions:

- no abrasion until the coating is fully cured;
- no prolonged delay (more than 24 hours) or exposure to wet weather before curing is complete; and
- a gradual rise to maximum temperature, preferably over several hours or even days.

Curing cycles are determined primarily by the silicone content of the resin vehicle. A typical cure for a coating based on 100 percent silicone resin would be 30 minutes at 480 F (249 C). For a 50 to 80 percent silicone, a satisfactory cure would be 15 to 30 minutes at approximately 425 F (218 C). Cures range from air dry to 30 minutes at 350 F (177 C) for silicone-modified organic copolymers containing 25 to 30 percent silicone.

Summary

The main use of silicone or silicone-

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modified coatings is for high-temperature service. Formulations differand must be chosen based on expected exposure temperature. Silicone coatings can be applied usingconventional methods. However, cure of the coating may require baking or exposure to high temperatures. Curing conditions and requirements are given on the manufacturer's product data sheet. **JPCL**

Notes

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