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From Pig Fat to Polysiloxane

A Brief History of Elevated Temperature Coatings for Exposed and CUI Service

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If you grew up in the South, your mom — or your granny — had an example of one of the earliest successful elevated temperature coatings. She had a cast-iron skillet which she "seasoned" by coating it with lard and then baking the lard into the cast-iron to keep it from rusting between uses. But why are we starting an article on industrial elevated-temperature coatings by talking about Granny's cast-iron frying pan? Simply because until recently, Granny's frying pan and jar of bacon fat was as good as elevated temperature coatings got. We were well into the age of Teflon-coated pans and microwave ovens before elevated temperature coatings for exposed surfaces and under insulation got past the industrial equivalents of pig fat and Granny's cast-iron skillet.

Although it satisfied the primary requirement for an elevated-temperature coating — since the smoke point of lard is 370 F, higher than vegetable shortening or many liquid cooking oils — there were three problems with Granny's system: it was a thin-film coating, it needed to be baked (or fried) to get past a soft surface and the biggest problem of all, the pig fat was an organic material based on a long chain mostly composed of carbon molecules. Until recently, corrosion engineers and specifiers of protective coatings for high-temperature surfaces — insulated or exposed — were faced with the



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same limitations. Until the 1940s, the only available "elevated-temperature" coatings were oil-based materials highly filled with red lead. For exposed appearance they could be overcoated with an oleoresinous aluminum or black, basically leafing aluminum or carbon black pigment with just enough light oil resin to hold the pigment in place.

These systems were of a very low dry-film thickness, typically 3 or 4 mils dry-film thickness (DFT) for the red lead and as little as a half mil dry for the aluminum. The red-lead primer air dried, but really didn't dry hard, especially at higher red-lead pigment loading, and the aluminum could be rubbed off the surface since there was so little binder holding it in place.

For use under insulation where appearance was not a factor, the red lead was put on as thickly as possible; sometimes (if old painters' stories can be believed) it was actually troweled on rather than being brushed or sprayed. With asbestos insulation over the red lead, the organic oil-based binder burned out, washed out, steamed out or was attacked by chemicals, but the red-lead pigment continued to protect the steel as long as there was enough red lead left on the surface.

In exposed service, ultraviolet from sunlight quickly broke down the organic resins even if elevated temperatures didn't burn them out. The relatively low film thickness could not afford sufficient protection, so the coating system failed and the steel beneath started to rust. Not much better than Granny's pig fat.

LIMITATION OF ORGANIC BINDER RESINS

The problem was the binder resin — a substance that had to be liquid for starters, so it could be spread thinly onto the surface to be protected and would then become hard on the surface to provide the needed protection. Binders range from modified egg yolk (tempera) to polyurethane, but until recently most were organic — that is carbon-based. Organic resins are sensitive to a wide range of chemicals and the ultraviolet in sunlight and have a limited maximum temperature (mostly around 400 F) above which they turn from film to dust and from light color to black. Inorganic resins do not have these limitations, but until recently there were no inorganic resins suitable for protective coatings. Available inorganic resins did not adhere or cohere well, could not be applied in a film thick enough to give meaningful protection, would not accept fillers, did not air dry, became too hard and brittle when they did dry, or all of the above.

But there was one notable exception to the "organic only" paint resins. In the 1830s, King Ludwig I of Bavaria was enamored of the beautiful fresco paintings he had seen in Italy, but the lime-based fresco process would not hold up well back home in the harsh German climate. Ludwig ordered the royal chemists to come up with a process equal to fresco, but suitable for the Bavarian climate. It took years of trial and error for the court chemists to get it right, but once they did, the results were great. They used inorganic liquid potassium silicate — water glass — and inorganic, metal-based pigments and came up with an extremely stable UV-resistant coating which actually became part of the exterior, exposed stonework it was applied over. This was due to the chemical structure almost entirely being composed from an SiO2 matrix, which is also the basic polymeric structure for inorganic polysiloxane coatings.

Some Bavarian building exteriors painted with the new mineral silicate paint in the 1870s, just after Ludwig's time, still have the original paint job after more than a century. Of course, there were limitations. Potassium silicate binds very well to stone, concrete or plaster, but not well at all to steel, and it is very porous and highly alkaline making it all but useless for corrosion control. But the concept of an inorganic binder for paints that last a century in Bavarian exterior exposure was tantalizing to paint formulators.

HYBRIDS AND PURE INORGANIC COATINGS FOR STEELWORK

The first commercially successful hybrid — that is a mixture of organic and inorganic resins in a paint binder — was silicone alkyd enamel, developed to increase gloss retention and reduce the effects of ultraviolet exposure on alkyd enamel. The "silicone alkyd" name was a misnomer, since even the best formulations only had about 25-percent silicone and the performance of the coating was closer to alkyd than to true silicone. Its usefulness as an elevated-temperature coating was limited, because even with the silicone addition, maximum service temperature was well under 400 F and film build was too low to provide meaningful corrosion resistance.



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The 1940s brought inorganic zinc, which raised service temperatures to over 700 F, and silicone coatings, which further raised service temperatures to 1,000 F. Inorganic zinc was a true, pure, inorganic binder and the binder's maximum service temperature was limited only by the zinc dust inclusion, which tended to degrade above 750 F, the melting point of zinc. But inorganic zinc was not originally envisioned as an elevated-temperature coating at all. It was developed by an Australian engineer as a replacement for hot-dip galvanizing on exteriors of large pipeline sections and originally included red lead and a bake cure. The pipeline carried water across the Australian outback and was never in elevated-temperature service except for an occasional brush fire. The primitive inorganic-zinc coating lasted over 50 years. Improved later versions were air-dry, had no red-lead component and some were waterborne.

Fifty years exposed in the Australian bush is a much milder environment than a couple of years under insulation in a U.S. Gulf Coast refinery, but inorganic zinc was a great step forward. It was an air-dry coating, inorganic and suitable for service to 750 F, but it could be applied only up to 3 mils DFT. It was also limited in length of service life by the anodic sacrifice of the incorporated zinc dust. Once the zinc had completely sacrificed, the silicate matrix was permeable and had no corrosion resistance whatsoever.

Inorganic zinc alone under insulation did well as long as there was unsacrificed zinc available, but once the zinc was gone, localized corrosion could spread rapidly. The cost of monitoring inorganic zinc under insulation and jacketing was just too costly in both time and money. After several spectacular events of perforation, explosion or fire directly linked to corrosion of insulated steel piping or vessels (formerly) protected by inorganic zinc, the use of this product under insulation fell out of favor.

Thin-film pure silicone enamels became available at about the same time. These had a maximum service temperature of 1,000 F but could only be applied at a dry-film thickness of 1 mil, with a maximum of two or three coats, not nearly thick enough to provide good corrosion resistance. Later formulations dried at ambient temperatures (and weren't particularly hard when dry) but had a prohibitively high VOC content of nearly five pounds per gallon. The combination of inorganic zinc topcoated with thin-film silicone sounded like an ideal system for exposed service but was too thin to provide good service life at ambient temperatures.

INTRODUCTION OF POLYSILOXANE HYBRID COATINGS

The use of polysiloxane as a description for a coating was as much marketing as a chemical description, since to a chemist, all silicones are polysiloxanes. The first polysiloxane-labeled coating came out in the mid 1990s and just as with inorganic zinc, its original purpose was not as an elevated-temperature coating. At this time, the standard new construction fabricator coating system for petrochemical, oilfield, offshore and heavy industrial equipment was inorganic zinc, epoxy midcoat and a gloss-retaining topcoat. Since all topcoats used an organic binder, "gloss-retaining" was an optimistic and very relative term. The newer acrylic urethanes held gloss well but chalked eventually, even when liberally doctored with acrylic resin.



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Fig. 1: Results from laboratory testing of the hardness of ambient dried polysiloxane coatings, per ASTM D3363/ISO 15184. All figures courtesy of James C. Reynolds



Fig. 2: Thin-film silicone aluminum enamel over inorganic zinc, failing due to excessively thick application of the silicone enamel.

The newly-introduced polysiloxane product was an epoxy-siloxane hybrid, about a 50-to-50 mix of the two resins, 80-percent-volume solids giving under one pound per gallon VOC content, 6 mils DFT per coat and an eight-hour "hard dry" time at 77 F. And it dried hard, to a nice low-gloss finish which was UV resistant.

The main market envisioned for this product was as a one-coat replacement for the epoxy midcoat and the gloss topcoat in new construction projects. The hybrid formulation and relatively thick film build were felt to be strong enough to replace the two coats of organic materials used previously — and who could resist the cost savings of replacing a three-coat system with two coats? How that turned out is another story.

As an elevated-temperature coating, exposed or under insulation, two coats of the new polysiloxane gave 12 mils dry-film thickness, it dried hard quickly and was usable to 1,100 F. Unfortunately, the new polysiloxane was a hybrid and still contained a densely cross-linked organic polymer structure. In cyclic ambient-hot-ambient service on small-diameter piping or convoluted small shapes, the full recommended thickness tended to crack and disbond from the substrate due to internal stresses caused by the inherent two-component highly cross-linked hybrid chemistry. In the intervening two decades, first-generation polysiloxane has continued in ambient temperature service but is rarely used on high-heat equipment.

SECOND-GENERATION POLYSILOXANE

The passage of five years elapsed before the arrival of a second generation of polysiloxane coatings, this time designed specifically for elevated-temperature service. There were two versions introduced almost simultaneously; one was a single-component material, highly filled with micaceous iron oxide, the other a two-component with titanium and aluminum. Both could be applied to 8 mils

dry, or more, per coat, both were usable to 1,200 F maximum, both were tightly adhering in cyclic service and both claimed to dry hard overnight at ambient temperatures. All but the last characteristic were undeniably factual for both versions, but as for the "dry hard" claim, it depended on your definition of "hard."

There were already a number of bake-cured polysiloxane coatings available for elevatedtemperature service. The main distinguishing characteristic of these new second-generation polysiloxane products, dubbed "inert multipolymeric matrix" (IMM) coatings to distinguish them from first-generation polysiloxane, was that they would supposedly dry hard, but not as hard as firstgeneration polysiloxane. The changed volume of polysiloxane resin that kept them from embrittling, cracking and disbonding in cyclic service to hot temperatures also kept second-generation polysiloxane coatings from getting hard at ambient temperatures. Now it's true that they were intended for elevated-temperature service and did relatively well once heated, but...

Since these were intended to be elevated-temperature coatings, the most commonly used laboratory test protocol began by heating test panels to 400 F before doing any other testing. Widespread use of this testing protocol masked the fact that the second-generation polysiloxane coatings stayed relatively soft after drying at ambient temperatures and required a short bake to 350 to 375 F to become hard enough for normal transportation, handling and installation.

Equipment fabricated as part of a new construction project is abrasive blasted and coated at the fabricator's shop. Second-generation polysiloxane coatings are specified for what will be elevated-temperature portions once the new unit is put into service. The coating is applied per the manufacturer's product data sheet and allowed to dry at least overnight (as required in the product data sheet). The newly coated unit is then loaded onto a trailer, strapped down with webbing and delivered. When it arrives, there are web imprints from the straps in the supposedly-dry IMM polysiloxane coating.

Even worse, equipment is fabricated somewhere in the Far East for a project in Latin America. Ocean shipment is on a top deck, because the unit is too bulky to store below decks, and when it arrives, it is left in the open in a laydown yard prior to final installation. In many cases, the unbaked second-generation IMM polysiloxane was actually showing pinpoint rust-through after less than a year of weather exposure. Spot repair of such weather-degraded, second-generation, elevatedtemperature IMM polysiloxane coatings is further complicated by the fact that they remain solventsensitive before being baked to hard cure.

Thermal-spray aluminum provided solutions to a few of the problems inherent with secondgeneration IMM polysiloxane but introduced a new set of limitations and potential problems, not the least of which was much higher cost than the combined application and material cost of secondgeneration polysiloxane coatings. Field repair of transportation damage to thermal-spray aluminum on new construction and tie-in of new construction to existing equipment was much simpler with a liquid coating, even one which would not dry very hard.



Pull-Off Adhesion Test Single Component Second Generation IMM Ambient Dry Pull-Off Adhesion Test Two Component Second Generation IMM Ambient Dry

Pull-Off Adhesion Test Third Generation Pure Inorganic Polysiloxane Ambient Dry

Fig. 3: Results from laboratory testing of pull-off adhesion of ambient dried polysiloxane coatings, per ASTM D4541/ISO 4624.



Second Generation Polysiloxane Single Component IMM Ambient Dry 4000 Hour Hot Water Immersion

Second Generation Polysiloxane Two Component IMM Ambient Dry 4000 Hour Hot Water Immersion Third Generation Polysiloxane Pure Inorganic Ambient Dry 4000 Hour Hot Water Immersion

Fig. 4: Results from 4,000-hour laboratory testing of hot-water immersion for ambient dried polysiloxane coatings, per ASTM D820/ISO 2812.

THIRD-GENERATION POLYSILOXANE COATINGS

Everyone acknowledged that a "third generation" of elevated-temperature coatings was needed but development took more than a decade. Theoreticians agreed on the required characteristics and even on how to achieve them. Third-generation polysiloxane coatings should include all the good features of first and second generation, as well as a few others, such as very low (or zero) VOC content and water cleanup. And, of course, the new generation should resolve all the problems of its predecessors, the largest of which was the need for hybridization, the blending of organic binder resins with the polysiloxane, providing a pure inorganic ambient cure coating.

As mentioned previously and as experienced in earlier generations of silicone and siloxane coatings, a pure silicone or siloxane polymer backbone usually produces a coating that is resistant to atmospheric attack from UV exposure. Combining the siloxane with an organic resin such as epoxy or acrylic to produce an organic-inorganic hybrid worked, but introduced unwanted characteristics inherent in the organic resin. After additional years of research, a siloxane resin was developed that did not require hybridization but still provided the necessary adhesion, hardness development and flexibility, even in elevated-temperature, cyclic service.



Fig. 5: Acrylic waterborne spray-on insulation failing after less than three years' service in U.S. Gulf Coast exterior exposure.

A small coating development company began to market a non-hybrid, pure inorganic, elevatedtemperature polysiloxane coating in summer of 2015, inaugurating the "third-generation" era. The new third-generation resin allowed the creation of elevated-temperature coatings with features previously unavailable.

The third-generation, elevated-temperature polysiloxane (TGPS) coating released first was specifically designed to mimic the second-generation IMM coatings, but without their limitations. This new coating was a 100-percent inorganic, single component with truly ambient curing properties. It is applied at the same thicknesses as second-generation IMM coatings, but the TGPS gets hard enough without any heating that equipment can be painted in the afternoon and the next morning the newly painted pieces can be handled, strapped down and shipped without damage. Because there is no organic, hybrid component, the same newly coated pieces can be left exposed to sunlight and weather for extended periods, without the chalking and surface breakdown characteristic of organic and organic-hybrid coatings. If a newly fabricated unit is to be shipped overseas or stored prior to installation, no special protective steps need to be taken with the third-generation polysiloxane coatings.

Two additional developments have come from use of the new non-hybrid polysiloxane resins. The first is a zero-VOC elevated-temperature coating. Thin-film silicone enamels and second generation IMM coatings are both at or above current VOC limits; the new TGPS technology allows creation of a solvent-free, zero-VOC coating suitable for use up to the 600 F range with good cyclic service performance. In normal refinery operating temperature ranges, where a 1,000 F tolerance is not needed, this midrange formulation offers the performance of the TGPS and a maximum service temperature 200 F degrees above that of epoxies.

A third and most intriguing version of the new TGPS coatings is an ultra-high-build (UHB) coating suitable for use as spray-on insulation. The all-inorganic resin can be filled with an insulating medium to provide meaningful insulation value and the resulting coating still has enough flexibility to expand and contract with the differential temperatures encountered between the heated substrate and the cooler exterior surface. Because it is a relatively lightweight material, a very thick film (400 mils, nearly half an inch) can be applied as a single coat over a TGPS primer or direct to prepared steel. Despite its thickness, the TGPS UHB expands, contracts, flexes and vibrates with the steel substrate in service without cracking or disbonding.

TGPS elevated-temperature coatings can be formulated to provide the same dry-film thicknesses as second-generation IMM polysiloxane hybrids (without the drawbacks of underperformance or failure prior to heating) for exposed service or for CUI service under block or fibrous insulation. As an alternative, third-generation polysiloxanes with appropriate 100-percent-inorganic-insulative fillers can be formulated to provide a spray-on insulation material usable to 750 F, far higher than maximum temperatures for acrylic, urethane or epoxy syntactic foam spray-on insulation materials.

SPRAY-ON INSULATION FOR ELEVATED TEMPERATURES

Spray-on, cellular insulation systems have been a preferred alternative whenever temperature limits and other considerations allow. Spray-on materials are much faster and less expensive to apply than block insulations, which must be pre-cut or field-cut to match the surface to be insulated. For most spray-on insulations, metal jacketing is not required, eliminating another pre-cut, piece-up and field-seal process.



Fig. 6: Typical transportation damage on ambient-dried second-generation (IMM) polysiloxane coatings.



Fig. 7: Steelwork protected with red-lead primer after 18 years of CUI service; there is rust scale, but no pitting.

Properly sprayed-on insulation has far fewer seams, joints and potential gaps, reducing the opportunity for water ingress and eventual corrosion under the insulation. Inspection of spray-on insulation is easier and more cost-effective than the three-layer system of CUI coating, pieced-up insulation and pieced-up jacketing, whether metal or plastic. Similarly, when problems are found or mechanical damage has occurred, repair of sprayed-on insulation is simpler than repair of a three-layer insulation system.

Possibly the greatest advantage of seamless sprayed-on insulation is its resistance to water absorption. Fibrous insulation or block materials in their natural state absorb water, sometimes as much as 400 percent of the dry weight of the insulation. Additives can reduce water absorption but may not last the service life of the insulation. Because block or fibrous insulation is not attached to the metal substrate, it may expand and contract at a different rate than the substrate, and may create a gap called an "annular gap" between the insulation and the substrate, allowing water or steam to gather and corrosion to occur. Sprayed-on, cellular insulation is much more resistant to water absorption, regardless of the generic binder used, and since it is adhered to the metal substrate or to the CUI coating, an annular gap cannot form.

Spray-on insulations based on organic resins have always been limited by the maximum service temperature of the resin, usually well under 400 F. The TGPS UHB material can be used in service temperatures, cyclic or stable, up to 750 F, and is ideal for personnel protection service on equipment operating at temperatures above 400 F.

POLYSILOXANE SPRAY-ON INSULATION

The third generation TGPS UHB spray-on insulation has meaningful k value, matching block insulations, but unlike almost all current block or fibrous insulation materials, it does not absorb water. When used in a direct-to-metal (DTM) application, one coat of the TGPS UHB spray-on insulation can replace the traditional three-part CUI system of protective CUI coating, block or fibrous insulation and metal jacketing at service temperatures far above the capabilities of organic spray-on insulation.

If necessary or specified, additional block or fiber insulation can be applied over the TGPS UHB spray-on insulation. Water absorption by the block or fiber insulation is no longer an issue and the TGPS UHB is robust enough not to require metal jacketing in most areas. A one-coat insulation system is far more cost-effective in initial fabrication, inspection and any needed maintenance or repair during the service life. Although current standards and specifications draw a clear line

between elevated-temperature coatings and insulation, the TGPS UHB crosses the line — a thirdgeneration TGPS coating that can replace the traditional three-step CUI system.

BACK TO GRANNY

It has been a long trip from Granny's pig-fat-coated frying pan to pure inorganic third-generation polysiloxane coatings and insulation. The names have gotten longer, too. Pig fat was much shorter and much easier to describe, but as Granny slides the cast-iron frying pan back under the kitchen sink, takes a polypropylene container of gumbo out of the freezer and pops it into the microwave, remember that complexity is sometimes the cost of progress. Somewhere out there is a paint formulator working on a fourth-generation pure inorganic polysiloxane coating for elevated-temperature cyclic service — perhaps an elastomeric?

Chemistry	Inorganic Zinc	Thin-Film Silicone	First Gen. Polysiloxane	Second Gen. IMM Polysiloxane	Third Gen. TGPS Polysiloxane A	Third Gen. TGPS Polysiloxane B	Third Gen. TGPS Polysiloxane UHB
Composition	Inorganic	Inorganic	Hybrid	Hybrid/Inorganic	Inorganic	Inorganic	Inorganic
Max. Service Temp.	750 F	1,000 F	1,100 F	1,200 F	1,200 F	570 F	750 F
Suitable for Cyclic Service	Yes	Yes	No	Yes	Yes	Yes	Yes
Resistance to Extended Sunlight Exposure	No*	Yes	No	No	Yes	Yes	Yes
Single Component Material	No	Yes	No	Yes	Yes	Yes	Yes
VOC Rating	>420 g/l (high)	>420 g/l (high)	<200 g/l (low)	>420 g/l (high)	<340 g/l (medium to high)	o (Zero)	<1 g/l (low)
Suitable for In-Service Application to Hot Substrates	No	No	No	Yes	Yes	Yes	Yes
Max DFT per Coat (mils) / Max Number of Coats	3.0/1	1.5/3	6.0/2	8.0/2	8.0/2	8.0/2	400.0 / 2
Dries Hard at Ambient Temperatures (ASTM D3363)	Yes	No	Yes	No	Yes	Yes	Yes
Corrosion Resistant After Ambient Temp. Application	Yes*	No	Yes	No	Yes	Yes	Yes
Impermeable Before Heat Cure / Exposure	No	No	Yes	No	Yes	Yes	Yes
Temperature Required to Gain Full Corrosion, Mechanical & UV Resistant Properties	Ambient approx. 50 - 74 F	>300 F	Ambient approx. 50 - 74 F	>300 F	Ambient approx. 50 - 74 F	Ambient approx. 50 - 74 F	Ambient approx.x 50 - 74 F

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* Inorganic zinc - before anodic zinc content has sacrificed completely.

ABOUT THE AUTHORS



Peter Bock, North American operations Manager for Performance Polymers Americas, is a petrochemical coatings, insulation and CUI specialist based in Houston. He is a U.S. Air Force veteran and holds degrees from Tulane University and the University of Northern Colorado. Bock has 40 years of corrosion-control experience worldwide and teaches annual courses on corrosion under insulation. Bock is a NACE-certified Coating Inspector (Level 3), is active in industry organizations, has been widely published and has presented numerous papers. He is past chairman of the NACE Central Area Board of Trustees, is a *JPCL* contributing editor and was a *JPCL* Editor's Award recipient at SSPC 2016 featuring GreenCOAT.

James C. Reynolds, Research and Innovation Manager for Performance Polymers Global Operations, is an inorganic coatings chemist originating from the United Kingdom. He holds a degree in chemistry from the University of Kingston, London. Reynolds has headed research and development for Performance Polymers BV in the Netherlands for five years specializing in polysiloxane polymer chemistry for coating applications including high-temperature-resistant, corrosion-under-insulation (CUI) mitigation and liquid-applied insulation. He has formulated numerous novel inorganic coating technologies including single-component, ambient-cure, solventfree siloxanes and ultra-high-build-polysiloxane-insulative coatings for high-temperature applications. Reynolds has also overseen technical support and the global product introduction to end-user (field) applications within the hydrocarbon processing industry.





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