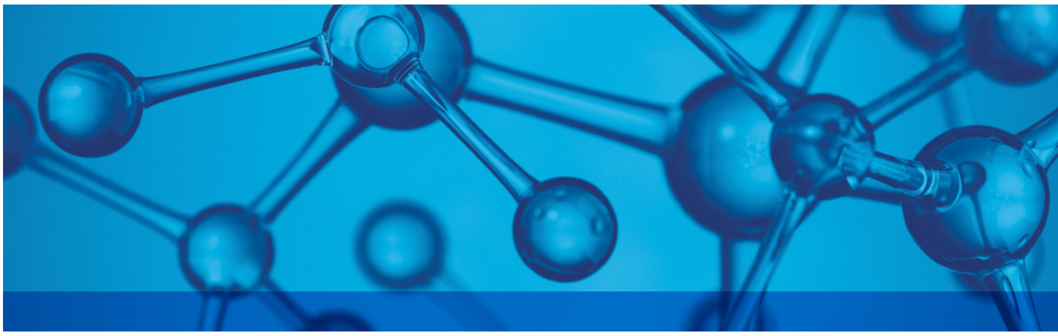

Ryton® PPS High-Temperature, Non-Stick, Corrosion-Resistant Coatings

Introduction

Ryton® PPS (polyphenylene sulfide) is a family of specialty polymers possessing properties desired in many coating applications. These include good electrical properties, excellent chemical resistance, and high heat resistance.

Ryton® PPS resin used for coatings is produced as a finely divided off-white colored powder having a modest molecular weight and high melt flow. This powder may be applied using slurry or electrostatic spray methods and the coatings are then fused and cured (cross-linking and/or chain extension) at temperatures (371°C, 700°F) well above the melting point of the polymer (285°C, 545°F). Heating the modest molecular weight resin below the melting point in the presence of air partially cures the resin to a lower melt flow. Then, depending on the degree of curing, the resulting resins may be selected for powder spraying, electrostatic spraying, flocking, or fluidized bed deposition methods typically used to apply thicker coatings.

Ryton® PPS Coating Resins

There are presently three grades of Ryton® PPS resins most commonly used in coating formulations.

Ryton® PR11 and Ryton® V-1

High melt flow, uncured PPS polymers typically used for thin coatings (0.013 to 0.051 mm, 0.5 to 2 mils). Use of these resins as coatings or components of coatings cured at temperatures above 371°C (700°F) is permitted by the FDA (21 CFR 177.2490) for coatings on articles intended for repeated food contact use.

Ryton® P-6

Moderate melt flow, partially cured PPS polymer typically used for thicker coatings (up to 0.25 to 0.30 mm, 10 to 12 mils).

A number of commercial manufacturers supply ready to use PPS coating formulations for specific purposes. These manufacturers provide their own application instructions, curing recommendations, and technical service.

Typical Slurry Formulations

PPS has an excellent affinity for a variety of fillers. If the filler can withstand the 371°C (700°F) curing temperature, it can probably be blended with PPS. Some typical examples of the many possible Ryton® PPS slurry formulations for specific purposes are listed below.

Industrial Release

100 parts	Ryton® V-1
33 parts	TiO_2
20 parts	PTFE
185 parts	Water
100 parts	Propylene Glycol
4 parts	Wetting agent (such as Triton® X-100)

This formulation is normally ball milled for 24 hours

Pigmented Coatings

100 parts	Ryton® V-1
16 parts	TiO_2
16 parts	Pigment
185 parts	Water
60 parts	Propylene Glycol
3.5 parts	Wetting agent (such as Triton® X-100)

This formulation is normally ball milled for 16 hours

Unpigmented Coatings

100 parts	Ryton® V-1
130 parts	Water
20 parts	Propylene Glycol
2 parts	Wetting agent (such as Triton® X-100)

This formulation is normally ball milled for 12 hours

Cobalt Oxide Primer

100 parts	Ryton® V-1
100 parts	Cobalt Oxide
300 parts	Propylene Glycol

This formulation is normally ball milled for 16 hours

Surface Preparation

The surface preparation is probably the most important factor in obtaining a good coating with PPS. This is true for most coatings that require baking at elevated temperatures. Although in some cases chemical treatment is sufficient preparation, the preferred method is grit blasting with a 60 to 120 grit medium. The grit should be fresh and free of contamination because contaminated blasting medium can deposit foreign material on the part surface that is difficult to remove. Any contaminant that will degrade at the curing temperature will produce an inferior coating.

Cleaning and Degreasing

Cleaning and degreasing of the parts is equally important to surface preparation. Any part to be coated with PPS should be thoroughly degreased. This can be accomplished by various methods such as vapor degreasing, solvent washing, sonic degreasing, and thermal degradation. Parts that have been exposed to cutting oils, rust inhibiting chemicals, and other preservatives should be degreased prior to grit blasting. It is also recommended that the part be degreased after a grit blasting operation. The recommended procedure for castings is to bake the parts at a temperature from 11°C to 28°C (20°F to 50°F) above the maximum expected curing temperature. This will degrade any oils and trapped gases that could cause holidays in the coating. This pre-bake should be done prior to grit blasting. In some cases, castings have some porosity. If this porosity is large, it is recommended that it be filled by welding or with some material that will withstand the baking temperature prior to coating with PPS. Care should be exercised in handling parts after cleaning so that body oils are not deposited on the part. If the parts must be handled, the use of clean cloth gloves is recommended.

Primers for Ryton® PPS Coatings

For most materials that are to be coated with PPS, primers are not required for external coatings. Internal coatings for pipe, fittings, valves, etc., made from steel, cast iron, or stainless steel, require a primer for best adhesion. There may be other primers that will prevent oxidation of the substrate and promote adhesion, but the best materials tested to this point are:

- Corro Therm CT-33
- Sermetel W
- Cobalt Oxide

These primers should be applied and baked according to the manufacturers' instructions. Each of these primers has produced excellent adhesion of Ryton® PPS coatings. In addition, users have noted that Ryton® PPS bonds easily to zinc phosphate converted coatings on carbon steel (Battelle Report No. GRI-90/0219).

Copper is very difficult to coat with materials that must be cured at elevated temperatures or in cases where the part is subjected to elevated temperatures during operation. Copper oxidizes very rapidly and the oxide does not adhere to the substrate. The best results in coating copper have been obtained by either electroless nickel plating or silver plating prior to coating with PPS.

Other materials, such as aluminum, may be coated without primers or treatments other than grit blasting and degreasing.

Slurry Coatings

Water based slurries of PPS can be applied using most standard industrial type spray guns. Air pressures from 1.0 to 3.8 bar (15 to 55 psi) can be used depending on the particular equipment employed. The spray gun should be adjusted to produce a fine mist that will produce a wet film of 0.025 to 0.038 mm (1.0 to 1.5 mils). The slurry coated part may go directly into the oven for curing. Air drying or preheating is not required for Ryton® PPS slurry coatings. Multiple coats, except for formulations containing PTFE, can be applied by repeating the spraying procedure. Coating thickness up to 0.25 to 0.38 mm (10 to 15 mils) can be achieved by slurry spraying. The first coat should not be applied more than 0.038 mm (1.5 mils) thick, but the second and subsequent coats can be applied as thick as 0.064 mm (2.5 mils) without experiencing mud cracking. A thicker first coat will usually produce severe mud cracking. Slurry coated parts should be baked at a temperature of at least 371°C (700°F) for at least 30 minutes. A curing schedule for minimum and maximum time for various temperatures is provided below.

Electrostatic Coating

Electrostatic spraying can be done on either cold or hot surfaces. Listed below are steps for obtaining optimum coatings using both techniques.

Cold Electrostatic Spraying

1. The surface should be prepared by degreasing and grit blasting with clean grit, with care being taken not to contaminate the cleaned surfaces. Small, intricate, or ceramic surfaces should be preheated to 371°C (700°F) and then cooled to room temperature. This will form an oxide on the surface and the first coat will give better coverage.
2. Coat the surface evenly to a 0.051 to 0.076 mm (2 to 3 mils) maximum film thickness per pass.

3. Place the coated part in a circulating air oven at 371°C (700°F) until 5 to 10 minutes after the coating melts. Cool the part to room temperature, and then repeat this procedure until the desired thickness has been achieved.

4. Final cure at 371°C (700°F) should be approximately 45 minutes for coatings up to 0.13 mm (5 mils) thickness and 90 minutes for coatings of 0.13 to 0.25 mm (5 to 10 mils) thickness.

Hot Electrostatic Spraying

1. Use the same surface preparation as for the cold spraying technique specified above.

2. The initial coat (0.051 to 0.076 mm, 2 to 3 mils) should be applied at room temperature, and the part then heated to 371°C (700°F). Subsequent coats may then be applied hot. If the part does not have enough mass to retain sufficient heat to melt the PPS as it is applied, it is not suitable for hot spraying and should be cold sprayed.

3. When melting of the powder starts slowing down, place the part back in the oven to reheat it to 371°C (700°F) and then continue spraying. If the coating should sag or run, the coating is being applied too heavily, and thinner coatings should be applied.

4. Final cure at 371°C (700°F) should be approximately 45 minutes for coatings up to 0.13 mm (5 mils) thickness, 90 minutes for coatings of 0.13 to 0.25 mm (5 to 10 mils) thickness, and 120 minutes for coatings of 0.25 mm to 0.51 mm (10 to 20 mils) thickness.

Fluidized Bed Coating

This method will produce smooth, glossy finishes but is limited to thicker, heavier parts that will retain heat long enough to melt the resin and coat the part with the desired thickness. The part is heated in an oven to 371°C (700°F) then removed and immersed in the fluidized bed as quickly as possible. While in the bed, the part is kept in motion to ensure a uniform coating. One to three seconds of immersion is generally sufficient to yield a 0.13 to 0.25 mm (5 to 10 mils) coating. After removing the part from the fluidized bed, the excess un-melted powder is removed by air or vibration. The coated part is then immediately placed in a circulating air oven before the coating surface cools. The coating should then be baked at 371°C (700°F). Multiple coats are recommended to ensure pinhole free coatings. After the first coat and while the item is hot from baking, the dipping and baking operation should be repeated.

Powder Spraying (Flocking)

Flocking can only be done on a hot part. After the proper surface preparation, degreasing, or priming has been done, the part should be preheated to 371°C to 427°C (700°F to 800°F). The PPS powder is then sprayed with a flocking gun onto the part to a thickness of 0.25 to 0.38 mm (10 to 15 mils). If a thicker coating is desired, it is preferable to apply two or more coats with a 30 to 45 minute cure between coats. Applying the coating too thick in one pass can produce blistering in the coating. To produce a smooth, glossy PPS coating, the powder should be sprayed on the part only as long as it readily melts. The addition of powder that does not melt will result in a rough, gritty coating. The powder coated part should be returned to the curing oven as soon as possible, at least prior to the part cooling to below the melting temperature of PPS (285 °C, 545 °F). Cooling the uncured coated part below the melt point could result in the coating cracking. The coating should be cured according to the curing schedule given below, which depends on the coating thickness and the temperature selected.

Coating Properties

Typical properties of Ryton® PPS coatings are shown in Table 1 and Table 2. Further information on the properties and chemical resistance of Ryton® PPS coatings is provided in the accompanying technical reprint.

Table 1: Properties of polyphenylene sulfide coatings

Property	$PPS:TiO_2$, 100:33	$PPS:TiO_2$:PTFE, 100:33:10
Hardness, pencil	2H	2H
Mandrel bend, 180° 4.85 mm (3/16”)	Pass	Pass
Elongation, %, ASTM D522	32	32
Reverse impact, in·lbs (J)	160 (18.1)	160 (18.1)
Abrasion resistance, Taber, CS-17 wheel, mg/1000 rev.	50	57
Chemical resistance	Excellent	Excellent
Thermal stability	Excellent	Excellent

Table 2: Long term stability of polyphenylene sulfide coatings in air at 260°C (500°F)

	Weight Loss [%]	
Exposure Time [hours]	$PPS:TiO_2$, 100:33	$PPS:TiO_2$:PTFE, 100:33:10
24	0.003	0.02
100	0.06	0.07
500	0.18	0.21
1000	0.50	0.34
1182	0.47 (cracked)	0.31
1686	-----	0.95 (cracked)

Curing Schedule

Curing of PPS coatings is dependent on the coating thickness, temperature and time. The following is the recommended curing schedule:

Thin coatings – 0.025 to 0.05 mm (1 to 2 mils)

Temperature	Minimum Time	Maximum Time
316°C (600°F)	16 hours	72 hours
342°C (650°F)	6 hours	48 hours
371°C (700°F)	30 minutes	12 hours
399°C (750°F)	20 minutes	8 hours
427°C (800°F)	5 minutes	2 hours
454°C (850°F)	3 minutes	30 minutes

Thick coatings – 0.25 mm (10 mils)

Temperature	Minimum Time	Maximum Time
316°C (600°F)	-----	72 hours
342°C (650°F)	12 hours	48 hours
371°C (700°F)	1.5 hours	12 hours
399°C (750°F)	1 hour	6 hours
427°C (800°F)	34 minutes	2 hours

These times for curing PPS coatings do not include the heating time required to bring the part to the curing temperature. The time required to bring the part to this temperature must be added to the cure time. The following are some representative times required to heat substances to 371°C (700°F):

Pipe
Length: 5 inches (12.7 cm) **30 minutes**
Diameter: 10.2 cm (4 inches)
Weight: 2.04 Kg (4.5 lbs)

Open steel box **1 hour**
Sides: 20.3 cm (8 inches)
Wall Thickness: 2.54 cm (1 inch)
Weight: 10.4 Kg (23 lbs)

Steel Plate
Diameter: 17.8 cm (7 inches) **2.5 hours**
Thickness: 10.2 cm (4 inches)
Weight: 19.5 Kg (43 lbs)

For parts with large variation in thickness, it may be desirable to use a thermocouple on the heaviest section to determine when the part has reached the curing temperature.

Ryton® PPS Applications

The case histories of successful applications of Ryton® PPS coatings are much too numerous to list in this paper. It has been successfully used in many applications (many in high temperature, highly corrosive environments) such as pipe couplings, pumps, valves, tanks, reactors, sucker rods, oil well tubing, fan drive discs, coil insulation in motors, masking insulation on electrodes used for electrowinning metals, and many, many others. In addition, it has been recommended that PPS coatings be more completely evaluated over extended periods in heat exchangers in typical condensing furnaces (Battelle Report No. GRI-90/0219). Another good application for PPS is for cookware non-stick coatings. This is presently the largest single use for PPS coatings.

If there is a problem where high temperature and/or corrosion are encountered, then Ryton® PPS coatings will probably be the answer.

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Ryton® PPS Coating Resins

Polyphenylene Sulfide

Ryton® PPS (polyphenylene sulfide) resins used for coatings are finely divided powders having a modest molecular weight and high melt flow. The powders (neat or mixed with other components) may be applied using slurry or electrostatic spray methods, and then fused and cured (cross-linking and chain extension) at temperatures from 316 °C (600 °F) to 427 °C (800 °F) to form high-temperature, non-stick, corrosion-resistant coatings. For further information on Ryton® PPS coatings, request Ryton® PPS TSM-275 high-temperature, non-stick, corrosion-resistant grades.

Property	Unit	V-1	PR11	P-6	Test Method
Melt flow, 316 °C melt temperature, 5 kg weight	g/10 min	5,000	5,000	380	ASTM D1283, Method B
Ash	wt %	0.8	0.8	0.8	
Particle size					
35 mesh	wt % passing	99.8	99	95	
60 mesh	wt % passing	99.5	95	90	
Color		Off white	Off white	Tan	

Note: Some powders may be available only in limited quantities.
Off-gas products produced during processing can be irritants to mucous membranes, therefore adequate ventilation is recommended.

A number of manufacturers supply ready to use Ryton® PPS coating formulations for specific purposes. Those manufacturers provide their own application instructions, curing recommendations, and technical service.

The nominal properties reported herein are typical of the product but do not reflect normal testing variance and therefore should not be used for specification purposes.

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Characterization Of Polyphenylene Sulfide Coatings

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Polyphenylene sulfide is a unique thermosetting-thermoplastic material characterized by chemical resistance, thermal stability, and low flammability. Coatings of this material can be applied to metallic substrates by a variety of techniques such as slurry, fluidized bed, or electrostatic methods. Each of these application methods involves a thermal crosslinking process to produce an infusible chemical resistant coating of considerably reduced crystallinity. Coatings are tough, hard, glossy, flexible, and ductile as well as impact and abrasion resistant. In combination with small amounts of polytetrafluoroethylene, release coatings are obtained that are suitable for both domestic and industrial applications.

KEY WORDS: Polyphenylene sulfide; Coating application methods; Thermal stability; Chemical resistance; Release coatings; Crystallinity, Crosslinking curing; Mechanical properties.

INTRODUCTION

Polyphenylene sulfide is a unique material characterized by excellent chemical resistance, good thermal stability, and low flammability. While polyphenylene sulfide may be processed as an injection molding resin as a typical thermoplastic material, under more extreme conditions of time and temperature it takes on some of the characteristics of a thermosetting resin. Thus, it has been called a thermosetting-thermoplastic resin. Differential thermal analysis[1] of a sample that has been rapidly cooled from a temperature above its melting point to less than 85°C (i.e., quenched) indicates a second order transition (Tg) of 85°C, a pre-melt crystallization exotherm at 125–135°C and a crystalline melting point of 285°C. X-ray diffraction studies[2] indicate that a fully annealed sample of virgin resin is highly crystalline. These measurements allowed the assignment of crystallinity indexes (Ci) which represent approximate crystallinities. The Ci for virgin resin is about 65%. Typical diffraction patterns for amorphous and crystalline specimens of polyphenylene sulfide are shown in *Figure 1*. The admirable thermal stability of the polymer backbone is exemplified in *Figure 2*, a plot of weight loss temperature for one-hour exposure in air. Below 425°C the weight loss is less than five percent in one hour and less than 10% in four hours. Polyphenylene sulfide is also quite resistant to radiation, being unchanged by exposure to 40.8 megarads of electron irradiation in a 17 MeV linear accelerator.

As produced in the current Phillips polymerization process, Ryton® polyphenylene sulfide is a light tan powder of small particle size (95% through a 65 mesh screen).[3]

(1)

The insolubility of the polymer has precluded determination of exact molecular weight and structure. Laboratory work with model systems that exhibit increased solubility indicate that the molecular weight of the virgin resin produced in this polymerization process is modest. However, when this polymer is heated in air, it undergoes some rather significant changes in properties and molecular weight. The untreated material possesses a moderate degree of mechanical strength and is soluble in solvents such as 1-chloronaphthalene above about 200°C. Polymer that has been properly heat treated is tough and ductile and no longer soluble in any known solvents. In addition, melt flow, as determined by ASTM D-1238 at 316°C with a 5 kg weight, is reduced from a value of ≈ 6000 g/10 min to values of 0–1000 depending upon the degree of treatment.[4] This process is frequently called "curing". In the actual application of coatings, coalescence and curing are generally carried out in the melt phase at about 370–425°. Chemcially, the process is quite complex and involves several simultaneous reactions: (1) chain-extension. (2) oxidative crosslinking, (3) thermal crosslinking, and (4) oxygen uptake followed by loss of SO_2.[5]

APPLICATION OF COATINGS

Coatings of polyphenylene sulfide can be applied to steel, aluminum, and other metals by a variety of techniques. When aqueous slurries are used, a typical formulation consists of a ball-milled mixture of 100 parts of uncured polyphenylene sulfide, 33 parts TiO_2, 3 parts of

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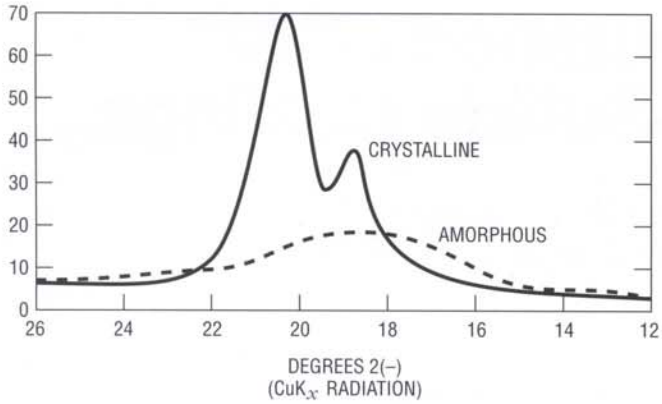

Figure 1 — X-ray diffraction patterns of PPS

Table 1 — Recommended Cure Conditions – Polyphenylene Sulfide Coatings

Temperature, °C	Cure Time Minimum	Cure Time Maximum
Slurry coatings (1–2 mils)		
371°	45 min	12 hr
385°	30	8
399°	20	5
413°	15	1
427°	10	1
Fluid bed coatings (10 mils)		
371°	2 hr	8 hr
385°	2	8
399°	1	4

a wetting agent such as Triton® X-100, and 300 parts of water. These slurries are generally sprayed through conventional paint spray equipment onto a metal substrate which has been degreased and grit blasted. Thin coatings (1–2 mils per coat) are usually applied in this manner. Thicker coatings are generally applied by use of dry powders and either fluidized bed, flocking, or electrostatic spraying techniques. These techniques require the use of a pre-cured (lightly crosslinked) polymer to prevent sagging and dripping during the curing operation. Typically, resin with a melt flow of ≈ 125 g/10 min is preferred for fluid bed coating while a melt flow of about 1000 is preferred for electrostatic spraying (*Figure 3*).

Regardless of the method of application, it is necessary to coalesce and cure (crosslink) the coating by heating above the melt point in air. It is important that the curing time and temperature be carefully controlled to provide high quality coatings. A properly cured coating will be flexible and sufficiently crosslinked to prevent embrittlement due to extensive crystallization on annealing (e.g., 2 hr at 204°C). An under-cured amorphous (noncrystalline) coating will be flexible initially, but will become brittle on annealing from crystallization of the polymer. An over-cured coating will be brittle under all conditions because of extensive crosslinking. Proper curing conditions naturally vary somewhat with coating thickness. Cure time limits for the proper curing of coatings of various thicknesses are shown in *Table 1*. In general, the minimum cure times are sufficient to reduce the crystallinity (from 65% to less than 30%) of the polymer (through crosslinking) to a level such that the heat of fusion will be below 3.0 cal/g. Coatings which have not been cured to this level became brittle on crystallization. Heats of fusion were determined using a du Pont Thermal Analyzer equipped with a DSC cell operated with a programmed rate of heating of 10°C/min in a nitrogen atmosphere. The maximum cure times were determined by the onset of brittleness due to extensive crosslinking in the coating as indicated by bending the coated coupon 180° around a $^{3}/_{16}$" mandrel or cutting and picking at the coating with a scalpel. Coatings on heavy metal parts will obviously require longer curing because of the time necessary to heat the metal substrate to temperature.

Although unpigmented coatings of polyphenylene sulfide are dark brown in color, thermally stable pigments such as titanium dioxide, phthalocyanines, quinacridones, and carbon black can provide a spectrum of attractively colored coatings.

Triton is a registered trademark of Rohm and Haas Co.

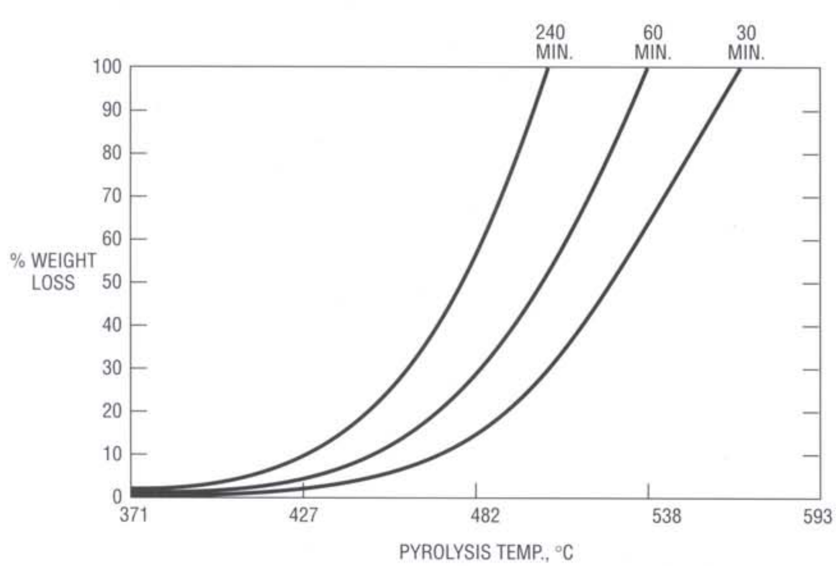

Figure 2 — Weight loss of PPS in air

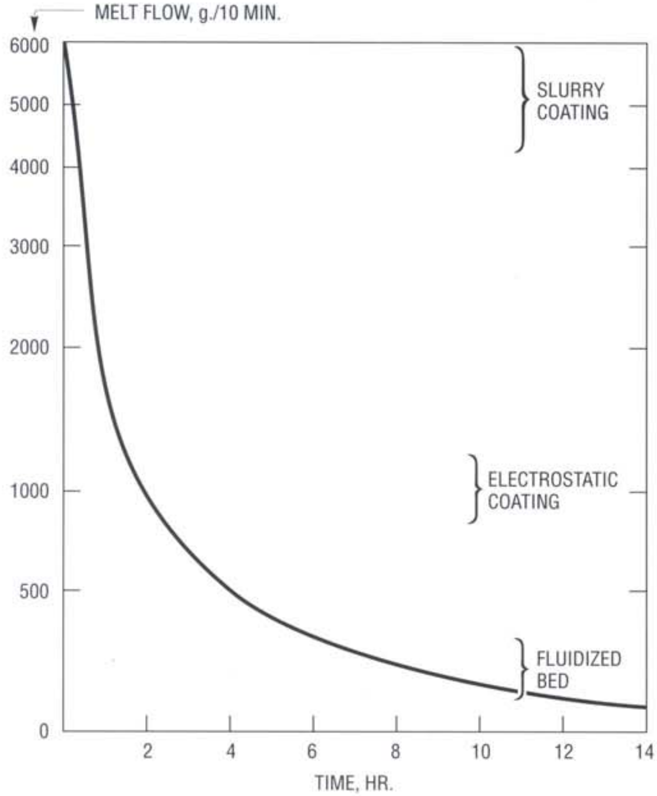

Figure 3 — Preparation of PPS coating resins by precuring at 232°C

PROPERTIES OF COATINGS

In general, polyphenylene sulfide coatings are best described as being tough, hard, glossy, flexible, ductile, impact and abrasion resistant, thermally stable and resistant to a wide variety of chemical environments. General properties are listed in *Table 2*. Two outstanding properties of polyphenylene sulfide coatings are its thermal stability, which allows its use in high temperature applications, and its unusual chemical resistance which suggests its use as a protective coating resin in corrosive environment applications.

Table 2 — Properties of Polyphenylene Sulfide Coatings

Property	Method	Test Rating of PPS/TiO_2 (100/33) Coating
Hardness	Pencil	2H
Flexibility	Mandrel bend. 180°, 3/16"	Pass, no cracks
Elongation	Conical mandrel, ASTM D-522, %	> 32
Impact	Gardner falling dart, in. lbs.	160
Abrasion resistance	Taber CS-17 wheel, mg. loss/1000 rev.	50
Chemical resistance	Direct exposure	Excellent
Thermal stability	Thermal aging	Excellent

Thermal aging tests conducted on thin (1–3 mils) coatings demonstrated that the resin can withstand continuous air exposure at 232°C for long periods (>1300 hr) without appreciable deterioration. Weight losses were small (≈ 0.5%) and some loss in flexibility resulted. Surprisingly, exposure to temperature as high as 316°C for one week or 371°C for 48 hr caused only slight deterioration of mechanical properties.

Chemical exposure tests conducted on 5-mil coatings on aluminum clearly exhibited the unusual resistance of the resin. Excellent resistance was demonstrated to organic acids, esters, amides, aromatic hydrocarbons, alcohols, aqueous inorganic salts, and inorganic base solutions. The coatings suffered some attack by mineral acids, chlorinated solvents, and bromine water (*Table 3*). Testing on a much broader scope has been conducted on polyphenylene sulfide moldings where property changes are more easily monitored[6] and has clearly established the broad chemical resistance of the resin.

Table 3 — Chemical Resistance of Polyphenylene Sulfide Coatings

	Rating	
Chemical	**One Week, 93°C**	**One Year, 24°C**
Conc. H_2SO_4		Slight attack
Dil. H_2SO_4	No attack	No attack
37% HCl	Slight attack	Slight attack
HNO_3	Severe attack	Severe attack
50% NaOH	No attack	No attack
20% NaCl	No attack	No attack
10% $KMnO_4$	No attack	
Br_2 water	Severe attack	
HF (75% aqueous)	No attack	No attack
H_2O	No attack	No attack
Acetic acid		No attack
Toluene	No attack	
Xylene		No attack
n-Heptane	No attack	
2-Butanone	No attack	
Ethyl acetate	No attack	
Chlorobenzene	No attack	No attack
Ethylene dichloride	Slight attack	
Perchloroethylene	Slight attack	
Ethanol	No attack	
N,N-Dimethylformamide		No attack

Coating hardness is not an easy property to define and measure. In our evaluation of polyphenylene sulfide coatings, two hardness testing procedures were used: the Arco Microknife which measures "cut through" hardness, and pencil hardness which meas-ures resistance to indentation. According to both pro-cedures polyphenylene sulfide coatings receive "hard" ratings. Hardness decreases in the temperature range 25-204°C; however, even at the higher temperature, the coatings retain considerable integrity and hardness (*Table 4*).

Table 4 — Hardness Rating of Polyphenylene Sulfide Coatings[a]

Test Method	Temperature, °C 25	93	149	204
Pencil[b]	2H	H	-	HB
Arco Microknife, g.[c]	725	625	425	250

(a) Nominal 3 mil coatings on aluminum panels, slurry sprayed, cured 371°C 30 min.
(b) Hardness index of pencil that leaves permanent mark on coating, order of increasing hardness HB, H, 2H.
(c) Weight on curring point needed to completely cut through coating in two passes.

Salt fog exposure has been conducted on both thin (3–10 mils) and thick (15–20 mils) coatings on steel panels. Exposure times under conventional salt fog conditions were 1500-2000 hr. Some loss of adhesion and blistering occurred in areas where the coatings had been cut through to expose the substrate. In other areas no detectable signs of deterioration were observed.

Gas transmission experiments conducted on 5-mil polyphenylene sulfide film revealed that the films are not very permeable to gaseous materials with the possible exception of hydrogen. Examples of permeability in cc/mil/100 in.^{2}/24 hr (ASTM D1434, Method M) are: O_2, 30; CO_2, 75; NH_3, 15; H_2S, 3; and air, 20–30.

Electrical and thermal testing show that polyphenylene sulfide is a good insulator. Thermal conductivity is low, dielectric constant is high, dissipation factor is low, volume resistivity is high, and UL Tracking Index is high.

Cursory weatherability and Weather-Ometer® testing conducted on coated aluminum and steel panels revealed that unmodified polyphenylene sulfide coatings are subject to erosion. These tests have shown that both ultraviolet light and water rinsing contribute to the surface erosion.

RELEASE COATINGS

Incorporation of small amounts of polytetrafluoroethylene into a polyphenylene sulfide slurry formulation provides a coating with excellent release characteristics.[7] The use of release coatings of this type is permitted by Section 121.2621 of the Food Additives Regulations. Such release coatings are being used in nonstick cookware and also in industrial operations. For example, tire molds coated in this manner release tires much more effectively than conventional silicone mold release agents. Contact angles for a drop of liquid on the coating surface are 68° for Wesson oil and 110° for water. The hardness of these coatings is somewhat greater than that of conventional polytetrafluoroethylene coating). Some properties of these release coatings are compared with those of polyphenylene sulfide coatings which do not contain any polytetrafluoroethylene in *Table 5*.

Weather-Ometer is a registered trademark of Atlas Electric Devices Co.

Table 5 — Properties of Polyphenylene Sulfide Coatings

Property	PPS/TiO_2 Coating (100/33)	PPS/TiO_2/PTFE Coating (100/33/10)
Hardness, pencil	2H	2H
Hardness, Arco Microknife, g[a]	500	350
Adhesion, Arco Microknife, mils[b]	5	4-6
Mandrel bend, 180°, 3/16"	Pass	Pass
Elongation (ASTM D-522), %	>32	>32
Reverse impact, (in./lb)	160	160
Abrasion resistance, Taber mg loss/1000 rev., CS-17 wheel	50	57
Contact angle, [c]water, degrees	82	110
Contact angle, [c]Wesson oil, degrees	41	68
Chemical resistance	Excellent	Excellent
Thermal stability	Excellent	Excellent
Color	Light tan	Light tan

(a) ASTM 2197; measured on 1-mil coatings.
(b) ASTM 2197; measured on aluminum coupons that had been grit-blasted before coating (coating thickness, 1 mil).
(c) Measured with a Rame-Hart Model A-100 goniometer.

CONCLUSIONS

Polyphenylene sulfide is a unique thermosetting-thermoplastic material characterized by excellent chemical resistance, good thermal stability, and low flammability. Differential thermal analysis of a quenched polymer sample indicates a second order transition temperature of 85°C, a pre-melt crystallization exotherm at 125–135°C, and a crystalline melting point of 285°C. X-ray diffraction studies indicate that a fully annealed specimen is about 65% crystalline. The material can be crosslinked by appropriate thermal treatment in air to produce an infusible material of considerably reduced crystallinity. Coatings of polyphenylene sulfide can be applied by slurry, fluidized bed, or electrostatic techniques in conjunction with a thermal crosslinking treatment. These coatings are tough, hard, glossy, flexible, and ductile as well as impact and abrasion resistant. They can withstand continuous exposure to air at 232°C for at least 1300 hr. Chemical exposure tests of coatings to a variety of materials indicate excellent resistance to organic acids, esters, amides, aromatic hydrocarbons, alcohols, aqueous salt solutions, and base solutions. Some attack is caused by mineral acids, chlorinated solvents, and bromine water. The incorporation of small amounts of polytetrafluoroethylene into a polyphenylene sulfide coating increases the contact angle for water from 82° to 110° and that of Wesson oil from 41° to 68°. These coatings provide excellent release behavior for cookware and industrial applications.

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