Skybond® 700 Technical Bulletin

Industrial Summit Technology Corporation · 250 Cheesequake Rd., Parlin, NJ 07739 · (732) 238-2211

SKYBOND® 700 High Heat Resistant Polyimide Resin

INTRODUCTION

SKYBOND® 700 is a heat reactive aromatic resin which can be thermally cured to a crosslinked polyimide. It is specifically designed for structural and specially applications where extended exposure at temperatures up to 700°F is required. Application of this resin in a glass cloth laminate structure to obtain optimum physical characteristics and heat resistance is described in this technical bulletin.

SOLVENT

The solvent in Skybond 700 is primarily N-methyl-2-pyrrolidone (NMP) with some ethanol present. NMP can be used for dilution. Other solvents having limited dilution capability include ethanol, isopropyl alcohol and xylene. Ketones are not recommended for dilution.

VARNISH PROPERTIES

PROPERTY	RANGE	I.S.T. <u>TEST PROCEDURE</u>
Cured Solids	45.0-48.0%	S-001.0 (1 gram, 1 hour, 288°C)
Viscosity at 25°C	3000-7000 cps	S-004.0 (Brookfield LVF, #3 Spindle at 12 RPM)
Specific Gravity at 25°C	1.15-1.18	S-005.0

STORAGE AND HANDLING

Good practice is to store Skybond 700 below 5°C and use within three months of receipt. Skybond 700 may be stored for longer periods of time below 5°C depending on the application. Lower temperatures extend storage life. Storage above 25°C is not recommended. See Graph 1 for viscosity stability at 5°C and 25°C.

Avoid moisture contamination and contact with iron which will react with the resin. Also avoid contact with materials which will be attacked by the solvent in the product. Stainless steel is suitable for use.

Volatile products given off during drying and curing operations are mainly aromatic acids, aromatic nitrogenous compounds and solvent. When exposing Skybond 700 to high temperature conditions, mechanical exhaust ventilation must be employed to insure good air movement and the avoidance of vapor inhalation.

NOTICE: Refer to the Material Safety Data Sheet for this product for health, safety and toxicity information. To obtain a MSDS please contact Manager of Technology at (413) 730-3578.

PREPREG PROPERTIES

The type of glass cloth recommended for use with Skybond 700 is 181E glass, A-1100 soft finish. It permits good resin penetration and hence the formation of a dense laminate when the prepreg is pressed. The saturated glass cloth is B-staged at temperatures in the range of 200-250°F. Recommended prepreg conditions for processing with platen presses or with a vacuum bag procedure are:

Platen Pressing		Vacuum Bag	
	%	%	
Resin pick-up	42-44	46-5 0	
Volatile content	8.0-8.5	12-16	
Flow	11-17	20-30	

PRESS LAMINATES

Laminates of 1/8" thickness can be prepared by using 12 plies and placing these in a press preheated to 600°F. A kiss contact time of 1½ - 2½ minutes with pressure at approximately 10-25 psi is important. This is necessary to insure that little or no NMP solvent is trapped in the laminate. Otherwise the resin may precipitate as discrete particles during further cure. Application of full pressure of 250 psi for a period of 30 minutes should follow the initial contact pressure. At the end of 30 minutes the press should be cooled to 140°F or lower before pressure is released.

Initial contact conditions cited are based on laboratory work with 2" x 2" twelve-ply laminates. The test is a quick, simple one. The test laminate is ejected hot from the press after a cure cycle of 10 minutes at 600°F and a pressure of 250 psi, cut open and examined microscopically to establish the desired vitreous polymer character in the interior of the laminate. This is done because the resin passes through a progressive change from a vitreous condition in the prepreg to a solid vitreous state when processed with optimum conditions. However, a dry curd-like condition related to resin precipitation and inferior laminate properties can be obtained if optimum contact conditions have not been used. Generally, an average of two minutes contact time is required but this should be determined under the conditions which are present for the application of interest.

VACUUM BAG LAMINATES

Laminates of approximately 1/8° thickness can be prepared by placing 12 plies of vacuum bag prepreg between surface release mats. Bleeder cloth and edge bleeders are then placed on and around the assembly which is placed in a 2-mil vacuum bag of polyester film. Zinc chromate tape is used as a vacuum sealant.

It is suggested that full vacuum of 25-29 inches be applied to the assembly during the first stage of heating. Application of heat should be as rapid as possible, 5°F per minute or better, until a temperature of 350°F is obtained. Augmented pressure of 100 psi may be applied five minutes after reaching 350°F.

The assembly should be kept at the 350°F temperature for a total of 30 minutes. As the assembly is cooled, full vacuum and any augmented pressure should be maintained until the temperature is lowered to 150°F.

These processing conditions are suggested for initial evaluation. Some modification may be in order depending upon size and geometry of the laminate and the capabilities of the processing equipment being used. A variety of cycles with different heating rates and hold times have been used in commercial practice to optimize laminate properties.

POST-CURE

Post-curing of laminates made with Skybond 700 resin up to and including the use temperature is essential to achieve maximum heat resistance and retention of properties. For good initial hot strength at 700°F a total post-cure of 16 hours with the following steps is recommended for 1/8" thick laminates. It is likely that thicker laminates should have this post-cure cycle extended.

- 2 hours @ 392°F (200°C)
- 2 hours @ 437°F (225°C)
- 2 hours @ 482°F (250°C)
- 2 hours @ 572°F (300°C)
- 2 hours @ 617°F (325°C)
- 2 hours @ 662°F (350°C)
- 2 hours @ 700°F (372°C)

SKYBOND 700 LAMINATE PROPERTIES

The following results are representative of laboratory data on 1/8" thick laminates. Laminates were made using 181 glass cloth with A-1100 soft finish. The thermal aging was in air. The testing temperatures were the same as the exposure condition temperatures in most cases. Testing temperatures are noted in parentheses.

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,	High Temperature High Pressure	Vaçuum Bag
FLEXURAL STRENGTH, PSI		
Standard conditions (@ 75°F) ½ hour @ 700°F (@ 700°F) 100 hours @ 700°F (@ 700°F)	75-85,000 45-60,000 20-35,000	76-83,500 22-32,000 20-24,000
Weight loss 100 hours (@ 700°F)	3.0%	< 5%
FLEXURAL MODULUS, PSI		
Standard conditions (@ 75°F) 335 hours @ 570°F (@ 570°F) 100 hours @ 700°F (@ 700°F)	3.12 x 10° 3.12 x 10°	2.8 x 10°
ULTIMATE TENSILE STRENGTH, PSI		
Standard conditions (@ 75°F) 335 hours @ 570°F (@ 570°F)	57,000 42,000	50,300
100 hours @ 482°F (@ 75°F) 100 hours @ 572°F (@ 75°F) Barcol hardness Flammability	70 Non-burning	48,800 48,200 60 Non-burning

^{*} The numerical values set out above for all properties are based on samples tested and are not guaranteed for all samples. Such values are intended as guides and do not reflect product specifications for any particular property.

SKYBOND 700* LAMINATE PROPERTIES

	High Temperature High Pressure	Vacuum Bag
ELONGATION		
Standard conditions (@ 75°F) 335 hours @ 570°F (@ 75°F) 100 hours @ 482°F (@ 75°F) 100 hours @ 572°F (@ 75°F)	1.90% 1.40% 	2.0% 1.7% 2.0%
WATER ABSORPTION		
24 hour immersion 24 hour immersion, coated	0.70% 	2.0% <1.0%

^{*} The numerical values set out above for all properties are based on samples tested and are not guaranteed for all samples. Such values are intended as guides and do not reflect product specifications for any particular property.

SKYBOND 700* LONG TERM AGING STUDIES AT 600° AND 500°F			
	Flexural Strength PSI	Flexural Modulus PSI	Weight Loss %
600°F AGING		2	
Initial 500 hours 860 hours 1850 hours	75000 (@ 75°F) 29000 (@ 600°F) 20000 (@ 600°F) 10950 (@ 600°F)	2.61 X 10° (@ 600°F) 2.59 X 10° (@ 600°F) 2.08 X 10° (@ 600°F)	2.2 3.4 7.9
550° AGING			
2300 hours 4500 hours 9000 hours	83000 (@ 75°F) 41200 (@ 550°F) 32000 (@ 550°F) 15000 (@ 550°F)	3.02 X 10° (@ 75°F) 2.63 X 10° (@ 550°F) 2.95 X 10° (@ 550°F) 2.00 X 10° (@ 550°F)	3.6 5.0 12.0

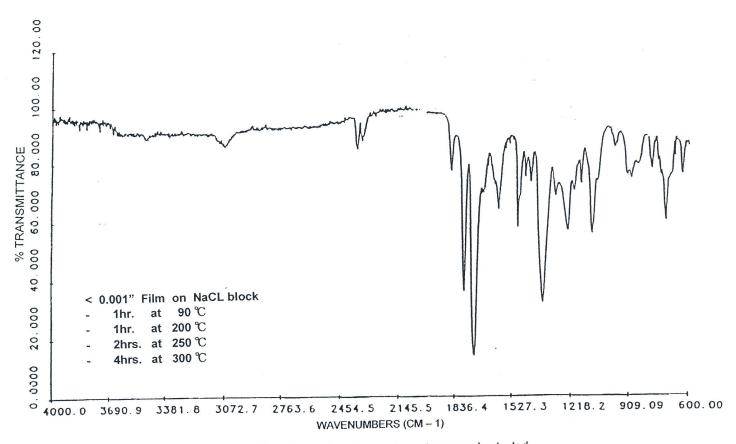
^{*} The numerical values set out above for all properties are based on samples tested and are not guaranteed for all. Such values are intended as guides and do not reflect product specifications for any particular property.

ELECTRICAL PROPERTIES OF LAMINATES UTILIZING SKYBOND 700* (High Temperature-High Pressure Laminates)				
PROPERTY	AS IS	D24/23	D48/50	<u>C96/35/90</u>
Dielectric strength				
-Short time parallel to laminate (volts)	55,000		32,000	*****
-Step-by-step parallel to laminate (volts)	38,000		16,000	
-Short time (volts/mil)	179			
-Stepwise (volts/mil)	140			
Dielectric constant (1 MC)	4.10	4.30	4.81	****
Dissipation factor (1 MC)	.00445	.00639	.01650	****
Insulation resistance (megohms)	1.9x10 ⁷	00000	*****	1.4x10 ²
Volume resistivity (ohm-cms)	2.47x10 ^{LS}	****	****	1.16x10 ¹¹
Surface resistivity	3.35x10 ¹⁴		*****	2.90x10 ¹⁰

X-BAND DATA (8.5 KMC)		
Temperature	Dielectric constant	Dissipation Factor
25°C	3.74	0.016
50°C	3.74	0.015
100°C	3.74	0.014
150°C	3.74	0.018
200°C	3.74	0.013
250°C	3.74	0.010
300°C	3.70	0.015

^{*} The numerical values set out above for all properties are based on samples tested and are not guaranteed for all samples. Such values are intended as guides and do not reflect product specifications for any particular property.

GRAPH III INFRARED ANALYSIS OF CURED SKYBOND 700



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Skybond® 703 Technical Bulletin

Industrial Summit Technology Corporation · 250 Cheesequake Rd., Parlin, NJ 07739 · (732) 238-2211

SKYBOND® 703 High Heat Resistant Polyimide Resin

INTRODUCTION

Skybond® 703 is a heat reactive aromatic resin which can be thermally cured to a crosslinked polyimide. It is specifically designed for structural, and specialty applications where extended high temperature exposure is required. It represents an excellent balance of processing advantages and high thermal stability. Excellent strength retention is maintained up to temperatures of 650°F. Application of Skybond 703 resin in glass cloth laminate structure to obtain optimum physical characteristics and heat resistance is described in this technical bulletin.

SOLVENT

The solvent in Skybond 703 is primarily N-methyl-2-pyrrolidone (NMP) with some ethanol present. NMP can be used for dilution. Other solvents having limited dilution capability include ethanol, isopropyl alcohol and xylene. Ketones are not recommended for dilution.

	VARNISH PROPERTIES*	
PROPERTY	RANGE	I.S.T. <u>TEST PROCEDURE</u>
Cured Solids	48.5-51.5%	S-001.0 (1 gram, 1 hour, 288°C)
Viscosity @ 25°C	3000-7000 cps	S-004-0 (Brookfield LVF, #3 Spindle at 12 RPM)
Specific Gravity @ 25°C	1.15-1.18	S-005.0

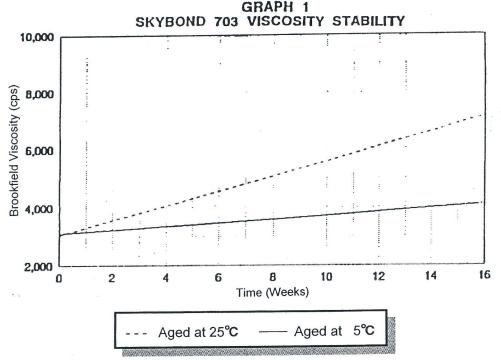
STORAGE AND HANDLING

Good practice is to store Skybond 703 below 5°C and use within three months of receipt. Skybond 703 may be stored for longer periods of time below 5°C depending on the application. Lower temperatures extend storage life. Storage above 25°C is not recommended. See Graph 1 for viscosity stability at 5°C and 25°C.

Avoid moisture contamination and contact with iron which will react with the resin. Also avoid contact with materials which will be attacked by the solvent in the product. Stainless steel is suitable for use.

Volatile products given off during drying and curing operations are mainly aromatic acids, aromatic nitrogenous compounds and solvent. When exposing Skybond 703 to high temperature conditions, mechanical exhaust ventilation must be employed to insure good air movement and the avoidance of vapor inhalation.

NOTICE: Refer to the Material Safety Data Sheet for this product for health, safety and toxicity information. To obtain a MSDS please contact Manager of Technology at (413) 730-3578.



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APPLICATION TEST PROCEDURES

PREPREG FLOW

Flow values recommended should be measured at the curing conditions to be used. Total weight loss due to resin squeeze-out is calculated as percentage flow. Flow is influenced by resin advancement and volatile content.

PREPREG VOLATILE CONTENT

The volatile content is measured by the weight loss of an impregnated sample when it is cured at an oven temperature of 435°F for ten minutes. It is computed by subtracting the cured weight of the sample from its original uncured-impregnated weight and dividing the difference by the uncured-impregnated weight. In some situations it is useful to run volatile content at higher temperatures such as 550°F for 10 minutes. Volatiles consist of solvents used in the manufacture or dilution of the resin and volatiles generated during the condensation reaction as the resin cures.

RESIN PICK-UP OF PREPREG

Resin pick-up is obtained by the weight difference of the cloth before and after resin impregnation. It includes volatile content and is determined by subtracting the dry, unimpregnated weight of the cloth from the treated weight of the sample and then dividing the difference by the impregnated weight.

RESIN CONTENT OF LAMINATE

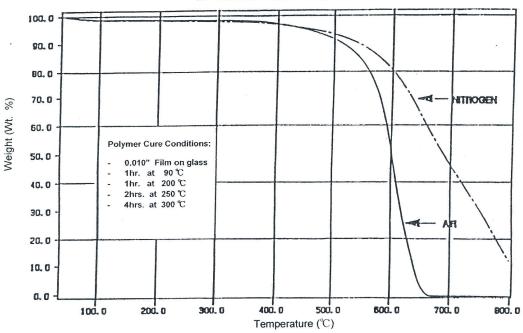
The term "resin content" refers to the value obtained on a cured glass cloth laminate by ignition loss. The weight of the sample after it has been exposed to a minimum of 4 hours in a muffle furnace at 1150°F is subtracted from the original weight of the laminate. This value when divided by the original weight of the laminate is the resin content of the laminate.

PREPREG PROPERTIES

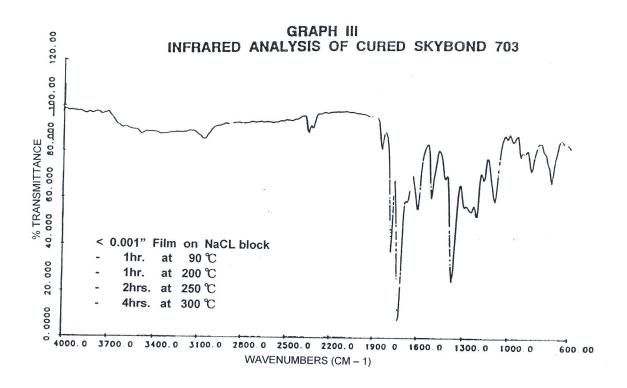
The type of glass cloth recommended for use with Skybond 703 is 181 E glass, A-1100 soft finish. It permits good resin penetration and hence the formation of a dense laminate when the prepreg is pressed. The saturated glass cloth is B-staged at temperatures in the range of 200-250°F. Recommended prepreg conditions for processing with platen presses or with a vacuum bag procedure are:

PLA	PLATEN PRESSING	
	%	%
Resin pick-up	40-45	40-45
Volatile content	8.5-10.5	10-13
Flow	10-20	15-23

GRAPH II THERMOGRAVIMETRIC ANALYSIS OF SKYBOND 703 IN NITROGEN AND IN AIR



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Skybond® 705 Technical Bulletin

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SKYBOND® 705 High Heat Resistant Polyimide Resin

INTRODUCTION

Skybond® 705 is a heat reactive aromatic resin which can be thermally cured to a crosslinked polyimide with high heat resistance. It has good film forming properties and it is designed for coating applications where extended high temperature exposure is required.

SOLVENT

The solvent in Skybond 705 is N-methyl-2-pyrrolidone (NMP) with some xylene present. NMP can be used for dilution. Other solvents having limited dilution capability include xylene, toluene and n-butanol.

VARNISH PROPERTIES*

RANGE	I.S.T. TEST PROCEDURE
16.5-19.5%	S-002.0 (2 gr./2 hr. 200°C)
1100-2600 cps	S-004.0 (Brookfield LVF, #3 Spindle @ 30 RPM)
1.05-1.08	S-005.0
	16.5-19.5% 1100-2600 cps

STORAGE AND HANDLING

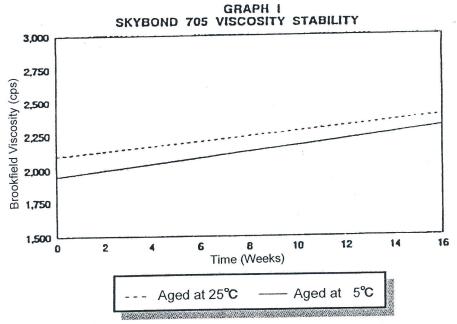
Good practice is to store Skybond 705 below 5°C and use within three months of receipt. Skybond 705 may be stored for longer periods of time below 5°C depending on the application. Lower temperatures extend storage life. Storage above 25°C is not recommended. See Graph 1 for viscosity stability at 5°C and 25°C.

Avoid moisture contamination and contact with iron which will react with the resin. Also avoid contact with materials which will be attacked by the solvent in the product. Stainless steel is suitable for use.

Volatile products given off during drying and curing operations are mainly aromatic acids, aromatic nitrogenous compounds and solvent. When exposing Skybond 705 to high temperature conditions, mechanical exhaust ventilation must be employed to insure good air movement and the avoidance of vapor inhalation.

NOTICE:

Refer to the Material Safety Data Sheet for this product for health, safety and toxicity information. To obtain a MSDS please contact Manager of Technology at (413) 730-3578.



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DRYING AND CURING

Skybond 705 is a low solids solution in NMP of a polyamic acid polymer which can be converted to a crosslinked polyimide by curing at elevated temperatures. It produces smooth, glossy, pale yellow coatings which adhere well to metallic substrates. The coatings are resistant to abrasion, impact, solvents and high temperatures.

Initial drying of wet coatings of Skybond 705 to remove solvent should be done at a low temperature to prevent polymerization in the presence of solvent which can result in poor film formation. Solvent removal at 60°C for 20 minutes is a good starting point.

After initial low temperature drying to remove solvent and accomplish film formation, the coating should be cured at a temperature above 200°C. Cure at 225°C for 20 minutes is recommended for initial studies.

Extended cure for up to 3 hours at 225°C should be evaluated depending on the application requirements. Temperatures up to 300°C can be used for cure. For multiple thin coats, dry at a temperature no higher than 150°C between coats prior to the higher temperature final cure.

FORMULATION

Coating formulations can be prepared by diluting Skybond 705 with solvents that include toluene, xylene, ethylene glycol, hexylene glycol and n-butanol. Films containing up to 35% of these diluents can be subjected to temperatures of 225°C immediately after coating at a 3 mil wet film thickness without blistering.

Aluminum, zinc, carbon black and titanium dioxide can be used to pigment Skybond 705. Carbon black at 10% on resin is all that is required to give a black coating but 3/1 titanium dioxide to resin still gives a light yellow coating instead of white. Aluminum pastes and powders have been used at 1/1 on the resin. Powdered pigments appear to give maximum resistance to high temperature blistering although their coatings are not quite as smooth and shiny as those made with pastes. Certain diluents may not be compatible with the solvents used to prepare the aluminum pastes.

PERFORMANCE

Extended aging of coatings on aluminum panels was carried out at 300°C with the following results:

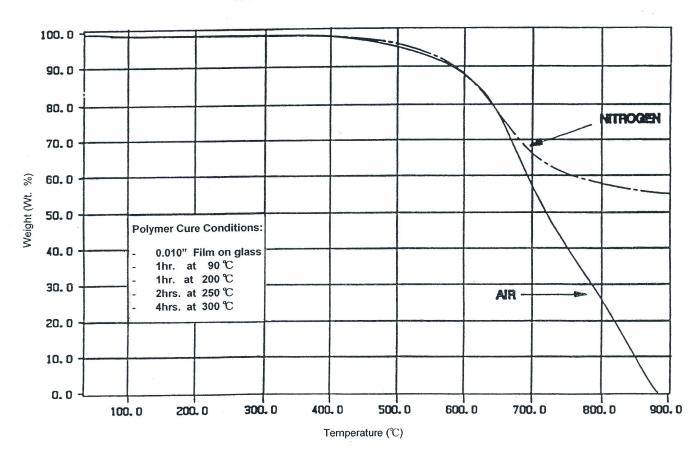
HOURS	% WEIGHT LOSS
90	2 14
1170 2170	29
2110	

Skybond 705 pigmented with aluminum powder, coated at 3 mil wet film thickness on mild steel and cured 20 minutes at 225°C will withstand 3 minute exposure to a direct Bunsen burner flame. The coating will not lose adhesion even when quenched in cold water.

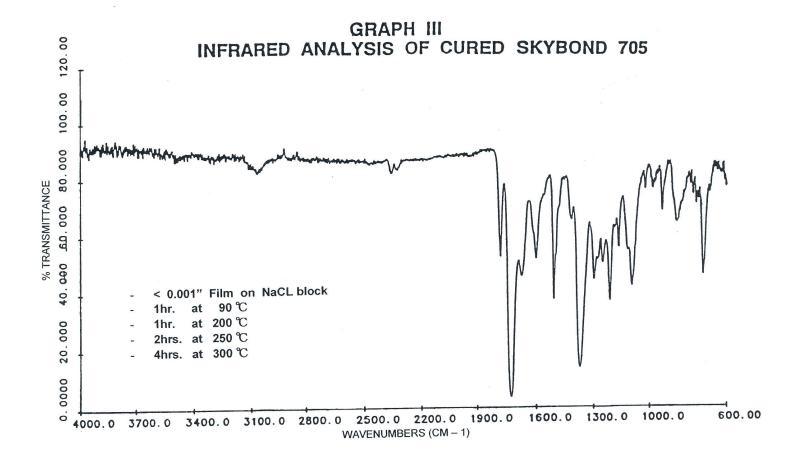
Clear, unpigmented coatings also will withstand 3 minutes of exposure to the Bunsen flame. The film slowly will turn a dark brown color but will remain intact. Longer exposures eventually will char and destroy the film but it will protect its substrate for short times under very severe conditions.

Cured films show excellent solvent resistance although softening by a strong base will occur.

GRAPH II
THERMOGRAVIMETRIC ANALYSIS OF SKYBOND 705
IN NITROGEN AND IN AIR



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