

WHITE PAPER

DEVELOPMENT OF PRECERAMIC POLYMER (POLYSILOXANE) PRECURSOR MATERIALS THAT WHEN PROLYZED PRODUCE A FIRE SAFE CERAMIC SURFACE

PRESENTED TO:

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INTERNATIONAL AIRCRAFT FIRE AND CABIN SAFETY RESEARCH CONFERENCE

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\*Pub. No.: WO/2009/054995/International Application No. PCT/US2008/012059 "Fire Resistant Flexible Ceramic Resin Blend and Composite Products Formed There From"

#### INTRODUCTION OF NEW RESIN AND PREPREG DEVELOPMENTS

- <u>RESINS</u> DEVELOPED RESIN BLENDS THAT WILL OVERWHELMINGLY PASS THE ASTM E-136-09 VERTICAL TUBE FURNACE AT 750°C FOR 30 MINUTES WITHOUT IGNITION OR RISING ABOVE THE STABILIZED FURNACE TEMPERATURE OF 750°C (1382°F).
- <u>RESINS</u> THESE RESIN BLENDS EXTEND THE ELASTIC THERMAL CAPABILITY OF COMPOSITES INTO THE RED HEAT ZONE (600 - 1000°C)
- <u>RESINS</u> THESE RESIN BLENDS ALSO ENABLE COMPOSITE MECHANICAL PROPERTIES TO BE INCREASED IN THERMAL CAPABILTY THROUGH RESIN DENSIFICATION PROCESSING.
- <u>PREPREG</u> FORMS "FIRE SAFE CERAMIC BARRIER" AT HIGH SURFACE EXPOSURE TEMPERATURES WHILE RETAINING INTERIOR ELASTIC PROPERTIES
- <u>PREPREG</u> CAN INCREASE FIRE PROTECTION OF LOWER TEMPERATURE ORGANIC POLYMER MATRIX COMPOSITES BY COCURING THE COMPOSITES WHEN FABRICATED WITH SURFACE PLIES OF THE "FIRE SAFE CERAMIC BARRIER" FORMING PREPREG
- <u>PREPREG</u> CAN INCREASE FIRE PROTECTION OF LOWER TEMPERATURE ORGANIC POLYMER MATRIX COMPOSITES BY COATING THE EXPOSED EXTERIOR SURFACES WITH THE "FIRE SAFE CERAMIC BARRIER" FORMING RESIN FORMULATED FOR PAINTING.

Flexible Ceramics Kiln Tested at 750 degrees Celsius (For 30 minutes with 98% yield)

## TEST RESULTS PERFORMED BY GOVMARK ORGANIZATION, INC. ASTM E-136 - 09

Standard Test Method for Behavior of Materials in a Vertical Tube Furnace at 750°C

(Passed exposure to  $750^{\circ}C$  ( $1382^{\circ}F$ ) furnace temperature for at least 30 minutes without flaming or rising above stabilized furnace temperature of  $750^{\circ}C$ .)

	Flex	ible Cera	mics, Inc. Pr	eceramic Pol	ysiloxan	e Mater	ial	
	Temperature Rise Beyond 750°C		Flaming: T Occurrence					
			of Test		Weight			
Specimen #	T/C-3 (°C)	T/C-4 (°C)	Begins (seconds)	Ends (seconds)	At Start (gms)	At End (gms)	Loss (gms)	(%)
1	0	0	DNO*	DNO	134.6	131.6	3.0	2.2
2	0	0	DNO	DNO	134.4	131.4	3.0	2.2
3	0	0	DNO	DNO	133.9	130.9	3.0	2,2
4	0	0	DNO	DNO	134.0	131.1	2.9	2.2
							Yield	97.8

#### Flexible Ceramics, Inc. "Elastomeric" Peceramic Polysiloxane Material

	Temperature  Rise Beyond 750°C		Flaming: Time of Occurrence From Start of Test		Weight			
Specimen #	T/C-3 (°C)	T/C-4 (°C)	Begins (seconds)	Ends (seconds)	At Start (gms)	At End (gms)	Loss (gms)	(%)
1	0	0	DNO*	DNO	133.0	131.1	1.9	1.4
2	0	0	DNO	DNO	133.1	131.1	2.0	1.5
3	0	0.	DNO	DNO	134.0	131.5	2.5	1.9
4	0	0	DNO	DNO	133.5	131.5	2.0	1.5
							Yield	98.5

\*DNO = Did Not Occur

### COMPARISON OF FLEXIBLE CERAMICS, INC. FIRE TEST PANEL TENSILE PROPERTIES (ASTM D638) BEFORE AND AFTER FAA POWERPLANT FIRE PENETRATION TESTING AT 1000°C FOR 15 MINUTES

The fire test panels were prepared from 8HS E-Glass 1583 woven fabric reinforced elastic preceramic polysiloxane prepreg laminated in a (0, -60, +60) 3-ply warp aligned architecture.

Before Fire	Testing	After Fire Testing			
Ultimate Strength (Ksi)	Modulus (Msi)	Ultimate Strength (Ksi)	Modulus (Msi)		
9,940	2.37	6.650	2.18		
10,680	2.49	10,440	2.23		
9,680	2.18	6,490	2.37		
10,880	2.39	*5,230	0.61		
<u>9,430</u>	<u>2.15</u>	<u>10,150</u>	2.37		
Averages:10,130	2.31	7,790	2.28		
Std. Dev: 620	0.14	2,352	0.09		

\*Test specimen broken and was not included in determination of average ultimate strength or modulus.

Ultimate Strength Retention: 7,790/10,130 x 100% = 77%

Modulus Strength Retention:  $2.28/2.31 \times 100\% = 99\%$ 

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## Flexible Ceramics Inc. Kiln Tested at 1000 degrees Celsius (For 30 minutes with 98% yield)



# Flexible Ceramic Laminates Thickness Recovery from 15% Compression as a



Figure 2. Thermogravimetric Scan of Typical Glass Fiber Filled Polysiloxane Laminate Revealing an 88 wt. % yield.



#### Porosity vs. Post-Cure Temperature

## FLEXIBLE CERAMICS, INC. PRODUCT DEVELOPMENT FEATURES

- CAN FORM HIGH-TEMPERATURE ELASTIC PARTS UP TO 600°C
- FORMS PRE-CERAMIC TO CERAMIC PARTS FROM 600 TO 1000°C
- FORMS "CERAMIC SEALED" "FIRE SAFE END CLOSURES" WHEN COMPOSITES CUT WITH LASER AT 16,500°C RESULTING IN LABOR AND MATERIALS COST SAVINGS WITH 25% HIGHER TENSILE STRENGTH
- 88 TO 98% YIELD WITH NO OUT-GASSING >700°C
- RESIN AND PREPREG PROCESSING IS COST EFFECTIVE:
  - \* COMPATIBLE WITH CURRENT COMPOSITE PROCESSING EQUIPMENT
  - \* SOLVENTLESS
  - \* NON-TOXIC
  - \* AMBIENT TEMPERATURE RESIN PROCESSING
  - \* RECYCLABLE
- PROCESS INNOVATIONS DEVELOPED TO ADDRESS ENVIRONMENTAL CONCERNS, REDUCED PROCESSING TIMES AND COST SAVINGS INCLUDE:
  - \* MULTIPLE PART LASER CUTTING IN STACKS
  - \* MULTIPLE PLATEN PRESSING OF 1000 LAMINATES PER HOUR
  - \* REVERSE ROLL PREPREG PROCESSING AT 400 FT. PER MINUTE
  - \* AMBIENT STORAGE





Top line data points represent densified recovery. Lower line data points represent porous recovery.

#### TENSILE STRENGTH AND MODULUS PROPERTIES OF LAMINATE FABRIC REINFORCEMENTS AFTER DENSIFICATION ASTM D638-08

Cure Temperature (Densified after cure)		Woven 8HS Carbon 94207 AS4-3K		Woven 8 HS Alumina Nextel 610-DF-11		Woven 8HS S-Glass S6580	
۴	(°C)	Ultimate Strength (Ksi)	Modulus (Msi)	Ultimate Strength (Ksi)	Modulus (Msi)	Ultimate Strength (Ksi)	Modulus (Msi)
450	(232)	57.3	7.7			43.7	3.4
575	(302)	56.1	7.6	50.7	14.2	42.9	3.4
650	(343)	57.5	7.3	50.9	13.5		
700	(371)	58.6	7.5	50.6	14.0		

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#### HIGH TEMPERATURE PERFORMANCE PROPERTIES

- FORMS PROTECTIVE CERAMIC BARRIER
- HIGH REBOUND CAPABILITY
- RETAINS ELASTIC RECOVERY AT HIGH TEMPERATURE
- MATERIAL CAN COMPENSATE FOR VIBRATION AND MOVEMENT AND IS SELF-CURING WITH ADDITIONAL HEAT EXPOSURE
- INSTANTLY SELF-EXTINGUISHING
- LASER CUTTING CREATES A CERAMIC SEALED EDGE
- PASSED FAA FIRE PROOF BURN THROUGH AND HEAT RELEASE TESTING AND BOEING TOXICITY AT LOWEST LEVELS
- PASSED FAA POWERPLANT FIRE PENETRATION TESTING AT 1000° C FOR GREATER THAN 15 MINUTES
- PASSED ASTM E-136-09, BEHAVIOR OF MATERIALS IN A VERTICAL TUBE FURNACE AT 750° C AS A PRE-CERAMIC, POLYSILOXANE MATERIAL AND AS A ELASTOMERIC POLYSILOXANE MATERIAL
- PROVEN DURABILITY
  - PASSED OEM EXHAUST (850° C) MANIFOLD GASKET CAB FLEET TEST EXCEEDING 350,000 MILES (150,000 MILES REQUIREMENT).
     STILL RUNNING AFTER FOUR YEARS
  - PASSED OEM TRUCK ENGINE EXHAUST (950° C) MANIFOLD
     INJECTED SEAL RINGS DYNAMOMETER TESTING FOR OVER 6,000
     HOURS EQUIVALENT TO 400,000 MILES







## <u>Key Features of Flexible Ceramics, Inc</u> <u>High Temperature Circuit Board Development</u>

- THE HIGH TEMPERATURE CIRCUIT CONSISTS OF AN ETCHED METTALIC FOIL THAT IS ELECTRICAL GRADE
- THE PREPREG RESIN IS FORMULATED TO PRODUCE A HIGH TEMPERATURE ELECTRICAL GRADE LAMINATE IN WHICH ELECTRONIC WIRING AND CIRCUITS CAN BE EMBEDDED
- THE RESIN FORMULATION IS MADE FROM HIGH PURITY PRE-CERAMIC PRECURSORS AND FILLERS
- THE BONDING OF THE FOIL AND PREPREG MATERIAL OCCURS AT THE PERFORMANCE TEMPERATURE
- THE BONDED FOIL LAMINATE CAN BE TWISTED TO FORM A <sup>1</sup>/2" DIAMETER FLEX CABLE





## THE KEY FEATURES OF FLEXIBLE CERAMICS, INC. FIRE RESISTANT HONEYCOMB DEVELOPMENT

- THE HONEYCOMB PREPREG WILL FORM HONEYCOMB CELLS WITH CO-CURE CAPABILITY
- THE HONEYCOMB PREPREG IS THE SAME USED TO PASS THE FURNACE TEST AT 750° C
- THE S-GLASS OPTION HAS HIGH IMPACT PROPERTIES AND LOW COST Advantages
- THE HONEYCOMB CAN BE MADE IN COMBINATION WITH LOWER TEMPERATURE OPTIONS
- THE HONEYCOMB CAN BE CO-CURED WITH LOWER TEMPERATURE OPTIONS



#### ADVANTAGES TO AEROSPACE MANUFACTURERS

- WE OFFER TRANSFER OF TECHNOLOGY ALLOWING YOU TO DEVELOP NEW PRODUCTS OR TO IMPROVE AN EXISTING PRODUCT
- POTENTIAL LICENSE OR PURCHASE OF EXCLUSIVE RIGHTS AS RELATED TO IDENTIFIED PATENT IS AVAILABLE
- CREATE NEW PRODUCTS FROM HIGHER ELASTIC PERFORMANCE TEMPERATURE MATERIAL
- CREATE HIGH TEMPERATURE CIRCUIT BOARDS AND FLEXIBLE CABLE PRODUCTS
- CREATE FIREPROOF HONEYCOMB STRUCTURES
- CREATE HIGH TEMPERATURE ADHESIVES, COATINGS AND SEALANTS
- **PRODUCTS READY FOR MARKET INCLUDE:** 
  - RESIN
    PREPREG MATERIALS
  - LAMINATE MATERIALS
- HIGH TEMPERATURE
  - **FLEXIBLE CIRCUITS / SENSORS**

- **Flexible Cables**
- HONEYCOMB MATERIALS AND FACE SHEETS
- HIGH TEMPERATURE CIRCUIT BOARDS
- LIQUID GASKETS
- **WIRE INSULATION COATING**
- **FIREPROOF BARRIER**

## Flexible Ceramics, Inc. Patent Abstract

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Pub. No.: Publication	WO/2009/054995 Date: 30.04.2009	International Application No.: International Filing Date:	PCT/US2008/012059 22.10.2008
Applicants:	C08G 77/00 (2006.01), C08I FLEXIBLE CERAMICS, INC CA 92262 (US) (All Except L CLARKE, William, A. IUS/U	<i>L 83/00</i> (2006.01) C. [US/US]; 1060 Paseo El Mi <i>JS</i> ). JS]: (US) <i>(US Onlv).</i>	rador, Palm Springs,
Inventor:	CLARKE, William, A.; (US).		
Agent:	CASCIO, Anthony, Thomas 94030-1905 (US).	s; 423 Broadway Ave., Suite	314, Millbrae, CA
Priority Data	60/999,918 22.10.2007 US	6	
Title:	FIRE RESISTANT FLEXIBL	E CERAMIC RESIN BLEND	AND COMPOSITE
Abstract:	High heat resistant elastic laminates, sealants, adhesiv developed from a resin blend blend is made up of methyl a phenyl silsequioxane resins produce silanol-silanol conde polymers formed in a slowly reaction mass containing sul fillers. The required ratio of s discovered for assuring the f composite blend that will forr 600 degC, then continue to f from 600 to 1000 degC. The greater than 90 wt. % at 100 of heat transformation can be upon the thickness of the lay	composite res, and coatings d. The resin and optionally selected to ensation silicone evolving bmicron boron nitride, silica a submicron boron nitride to silic formation of a high temperatur m intermediate flexible ceram form preceramic then dense of thermal yield of the composi 00 degC. Composite products e fabricated within the same yers of reinforcement.	Heat 10 20 18 FIG. 1A FIG. 1A THE IN FIG. 1A THE IN THE INFORMATION IN THE INFORMATI
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