



LIGO Laboratory / LIGO Scientific Collaboration

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Advanced LIGO

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Polyimide Hydrocarbon Outgas Rate
and Temperature Limits for the Isolation System Actuator

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This is an internal working note
of the LIGO Project.

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1 Introduction

The hydrocarbon outgas rate for polyimide materials is derived from existing mass spectrum test data. The outgas rate is then used to set a maximum temperature limit on the voice coil actuator for the advanced LIGO Seismic Isolation System (SEI).

2 Polyimide Outgas Rate

In order to set a maximum in-vacuum operating temperature for the voice coil, we would ideally have measurements of the outgas spectrum and rate, including temperature dependence, for the intended SEI voice coil actuator (or at least its materials). The voice coil actuators used to date (BEI/Kimco LA50-62-004Z and LA18-32-006Z) have not been developed for UHV use. It is likely that the epoxies, wire insulation and potting compound outgas at too high of a rate (though this has not been confirmed by measurement). A version of the smaller actuator (LA18-32-006Z) with kapton insulated wire potted with Cycom 3001 (a high temperature polyimide adhesive¹) has been produced. The mass spectrum of this unit, after cleaning and vacuum baking at 200C for 48 hours has been measured. The hydrocarbon (HC) outgassing rate derived from this residual gas assay (RGA) is suspect due to lack of repeatability of measurements on this chamber. The HC rate (sum of AMUs 41, 43, 53, 55 and 57) is 3.8E-10 torr-liter/sec (very likely limited by the background outgassing rate of the chamber). In addition to being suspect, this measurement was only made a room temperature. In order to establish a maximum in-vacuum operating temperature, we must know the doubling temperature (or some other measure of the outgas rate dependence with temperature). A repeat vacuum bake and mass spectrum measurement of the voice coil is planned in March, 2004 in the LHO vacuum bake oven with measurements at ~70C, ~45C and room temperature (~20 C).

In the interim we might use the outgas rate from other measurements of Polyimide materials to set approximate limits on the temperature of the voice coil. The mass spectrum of a number of polyimide materials have been measured. In particular, the Virgo measurements include the mass spectrum at multiple elevated temperatures (a practice that LIGO should adopt). Unfortunately it seems the Virgo group has not performed mass spectrum measurements of Cycom 3001 (confirmation has been requested). Five polyimide measurements were examined:

- 1) Coils with Polyamide-Polyimide Insulation and Polyimide Resin "varnish". The "varnish" is BASG 48816 polyimide/carbon pigment resin from Ball Aerospace.²
- 2) Coils with Polyamide-Polyimide Insulation (without the varnish)³
- 3) Cabling with Pyre-ML Insulation. Pyre-ML is a Dupont aromatic polyimide.⁴
- 4) Kapton ribbon cables (Kapton is a Dupont trademark polyimide). Virgo cables were provide by Axon.⁵

¹ A datasheet for Cycom 3002 (similar to Cycom 3001) has been included in the appendix of this memo.

² Virgo document VIR-TRE-PIS-3400-114, "Outgassing Test of Coils with Polyamide-polyimide Insulation and Varnishing".

³ Virgo document VIR-TRE-PIS-3400-113, "Outgassing Test of Coils with Polyamide-polyimide Insulation".

⁴ Virgo document VIR-TRE-PIS-3400-115, "Outgassing Test of Cabling with Pyre-ML Insulation".

5) Kapton Ribbon cables with PEEK weave and PEEK connectors from MDC Vacuum Products Inc.⁶

The Virgo report gives the total outgassing rate and the mass spectrum (in uncalibrated units) at multiple temperatures. The sequence was to dwell at each temperature and then take a mass spectrum measurement before increasing the temperature. The dwell time at temperature was not always long enough to reach a steady-state outgassing rate. The temperatures were typically 33 C, 50 C, 100 C, 150 C and then back to 40 C to 33 C. The mass spectrum plots were digitized and integrated to get a scale factor to calibrate the mass spectrum. This scale factor was then used with the HC flag AMU sum to get the HC outgassing rate.

The outgassing rate, k , is assumed to follow an Arrhenius rate equation:

$$k = Ae^{(-E/T)}$$

where in general the pre-exponential factor, A , and the activation energy, E , are temperature dependent; Here these coefficients are assumed to be temperature independent. The temperature change required to double the outgassing rate is:

$$\Delta T = \frac{-T^2 \ln(2)}{E}$$

Of course a single rate equation may not apply. In addition measurements at significantly higher temperatures than the operating temperature range may bring different kinetics into play. However the required hydrocarbon outgas rate is too low to measure at room temperature.

2.1 Polyimide Resin

For the Polyimide Resin, the outgassing rate (torr-liter/sec/cm²) versus temperature is given in Figure 1. The same data is plotted versus 1/T in Figure 2. The outgassing rate at the two low temperatures (33 C pre-bake and 40 C post-bake) is limited by the mass spectrometer noise floor (or at least by the plot scale in the Virgo report). Also shown is a fit to an Arrhenius equation.

⁵ Virgo document VIR-TRE-PIS-3400-137, "Outgassing Test of an Axon Kapton Ribbon".

⁶ Vacuum Bake Traveler for Kapton Ribbon Cable, LIGO-E980274-00. This vacuum bake load appears to have the lowest outgassing rate measured at Caltech for initial LIGO kapton cables. **Vacuum bake load travelers from LHO should be checked as well.**

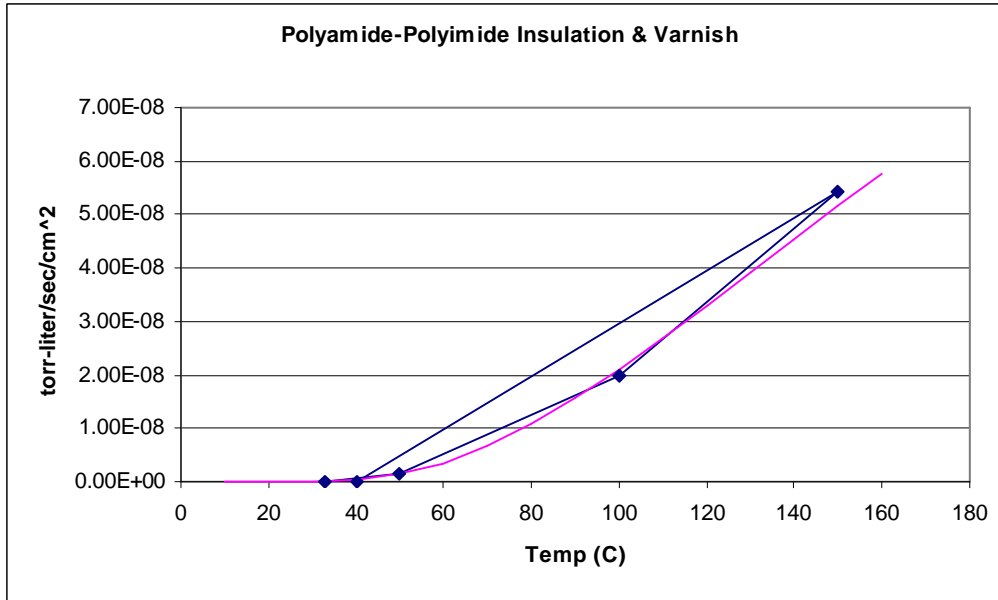


Figure 1: Polyimide Resin Outgas Rate vs Temperature

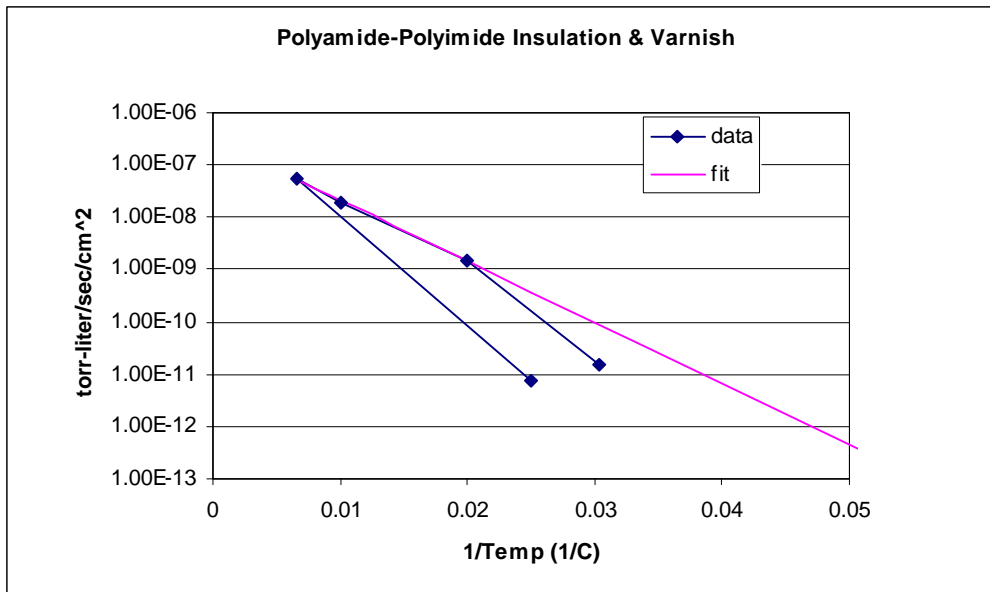


Figure 2: Polyimide Resin Outgas Rate vs Temperature

A least squares fit to the outgas rate at the three temperatures high enough to measure the outgas rate not limited by the noise floor of the spectrometer (or the lower limit of the plot in the Virgo report), yields:

$$A = 3.09e-7 \text{ torr-liter/sec/cm}^2$$

$$E = 268 \text{ C}$$

$$\Delta T = 1.03 \text{ C}$$

2.2 Polyimide Insulation

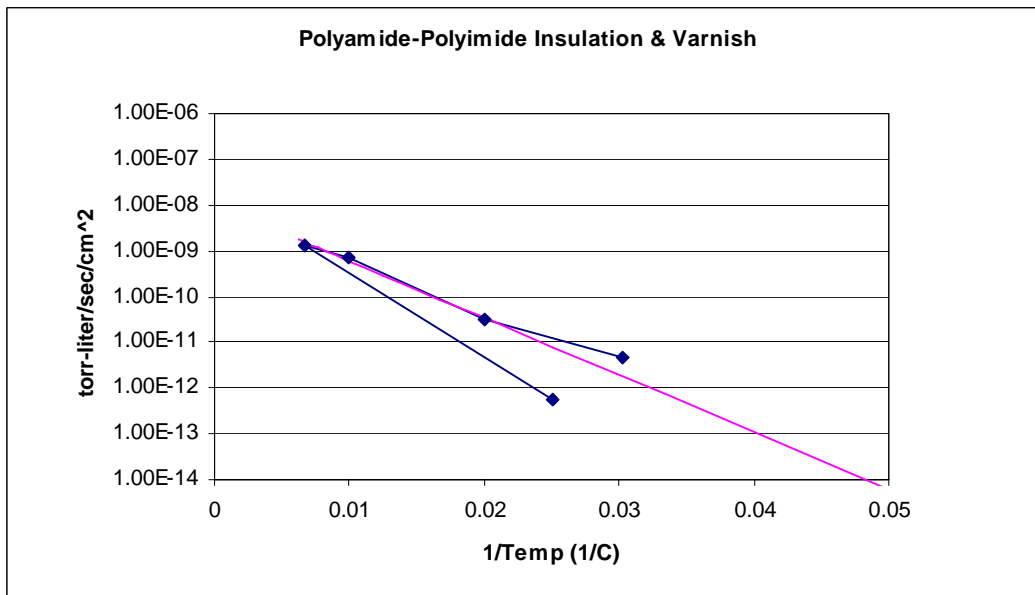


Figure 3: Polyimide Insulation Outgas Rate vs Temperature

$$A = 1.03e-8 \text{ torr-liter/sec/cm}^2$$

$$E = 286 \text{ C}$$

$$\Delta T = 0.97 \text{ C}$$

2.3 Pyre-ML Insulation

The Pyre-ML data is limited by the noise floor (limit of the plot in the Virgo report).

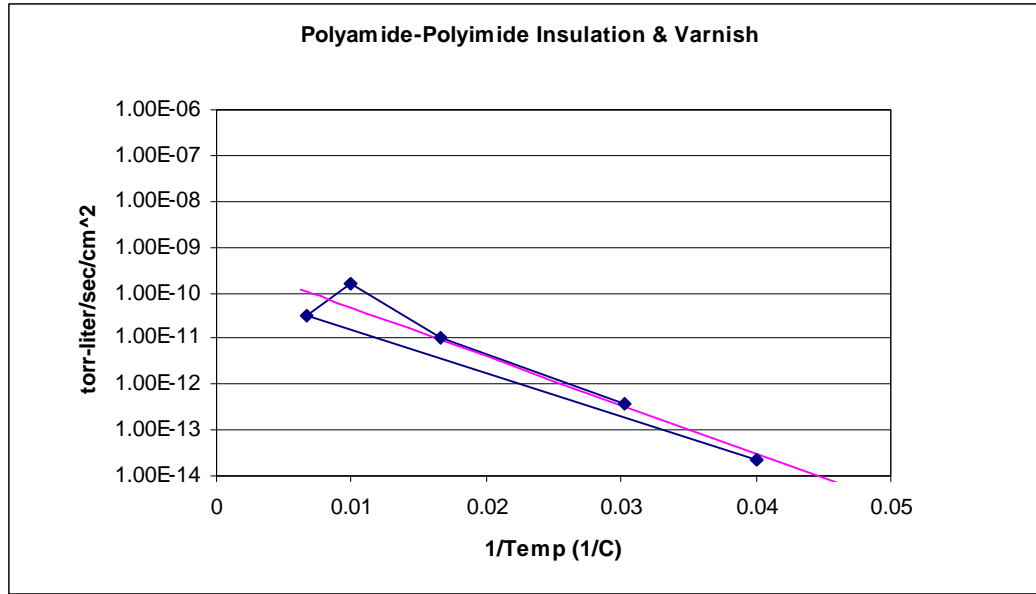


Figure 4: Pyre-ML Insulation Outgas Rate vs Temperature

$$A = 5.46e-10 \text{ torr-liter/sec/cm}^2$$

$$E = 245 \text{ C}$$

$$\Delta T = 1.13 \text{ C}$$

2.4 Axon Kapton Cabling

The Virgo report on Kapton cabling from Axon is still to be further analyzed. Using the 80 C and 150 C data (post-bake), one gets the following Arrhenius constants:

$$A = 1.35e-9 \text{ torr-liter/sec/cm}^2$$

$$E = 753 \text{ C}$$

$$\Delta T = 0.37 \text{ C}$$

With the admittedly uncertain fit, the room temperature (20 C) HC outgas rate is $6e-26$ torr-liter/sec/cm². If the activation energy from the previous fits is used ($E \sim 280 \text{ C}$), then the room temperature HC outgas rate is $1e-15$ torr-liter/sec/cm², which is consistent with the measured MDC kapton cable outgas rate.

2.5 MDC Kapton Cabling

The HC outgas rate reported in E980274-00 is $2.80e-15$ torr-liter/sec/cm² (each cable has 6080 cm² of area). This value does not have the oven or RGA background subtracted. Since empty chamber measurements were extremely infrequent in the Caltech vacuum bake ovens used for initial LIGO bake loads, we do not have a background to subtract. In addition, the RGA was not capable of being valved off from the chamber, so an RGA background can not be subtracted either. Looking at clean load scans (metal) in the same chamber around the same time frame as the E980274-00 load,

it seems that the oven HC background is about at $\frac{1}{2}$ the measured level, so a reasonable estimate of the kapton outgas rate is $1.4\text{e-}15$ torr-liter/sec/cm².

3 Voice Coil Maximum Temperature

The exposed surface area of the larger voice coil is estimated to be 143 cm^2 . With the MDC Kapton outgas level reported above, this means at room temperature (20C), the expected HC outgas rate is $2\text{e-}13$ torr-liter/sec/coil.

The allowed⁷ outgas rate for the SEI voice coil is $3.8\text{e-}12$ torr-liter/sec/coil. Using an activation energy of 245 (the lowest reported above), the allowable temperature is only 26 C, or 6 C above ambient.

4 Appendix: Cycom 3002 Datasheet

⁷ D. Coyne, Vacuum Hydrocarbon Outgassing Requirements", T040001-00.

CYCOM® 3002 High Service Temperature Condensation Polyimide

Description:

CYCOM® 3002 advanced composite prepreg is a condensation polyimide primarily used in applications requiring structural performance in the temperature range of 550-650°F (288-343°C). It is 350°F (177°C) curable with a free standing post cure required for elevated temperature service. CYCOM® 3002 advanced composite prepreg is methylene dianiline (MDA) free which allows ease in layup using ordinary impermeable gloves. CYCOM® 3002 advanced composite prepreg is used widely in antennae and radom applications where low dielectric properties are required.

CYCOM® 3002 advanced composite prepreg is used also in aircraft engine components such as hush kits, wiring harnesses, insulating bulkheads, thrust reversers, and inlet ducts. Parts in excess of 0.3 inches (7.62 ram) thick have been produced successfully having minimum void contents.

CYCOM® 3002 advanced composite prepreg is supplied in woven broadgood and neat resin forms only. Reinforcement types are glass, quartz and carbon fiber. Widths of material are 38 inches (965 ram) standard up to 60 inches (1524 ram) maximum. It is important to follow the prescribed bagging and cure cycle as closely as possible to insure proper polymerization when oven/vacuum bag or autoclave processing.

For more information, contact:
Cytec Engineered Materials
Technical Service
Anaheim, Calif. 92806
714 630-9400

Features and benefits:

- **High strength and dimensional stability at 600°F (316°C)**
- **MDA free: Substantial health risk elimination to manufacturer, end-user, and the environment**
- **350°F (177°C) vacuum bag/oven curable**
- **Free standing post cure**
- **Woven goods up to 60 inches (1524 ram) wide**
- **Excellent tack and drape**
- **Low dielectric**

Typical neat resin properties

Specific gravity	1.36
Tg, TMA rex probe, dry ¹	716°F (380°C)
Gel time @ 350°F (177°C)	6 minutes

¹Dry = ambient temp

Typical prepreg properties

Fiber	E-Glass	E-Glass	Quartz
Reinforcement style	108	7781	581
Finish	A1100	A1100	9288
Fiber area wt., psf (gsm)	0.01 (49)	0.06 (303)	0.06 (285)
Dry resin content, % by wt., nominal	50	30	30
Flow, %:at 350°F (177°C) with 15 psi	—	15-25	15-25
Row, %:at 450°F (232°C) with 15 psi	43-53	—	
Gel time, minutes @ 350°F	3-6	3-6	3-6
Volatile content, % by wt. at 750°F (399°C) for 2.5 minutes	25-35	15-25	15-25

Miscellaneous properties

Shop life	10 days at 80°F (27°C), 65% relative humidity
Shelf life	Six (6) months from date of shipment at recommended storage conditions
Recommended storage	Store at or below 0°F (-18°C)

Mechanical performance

Product description	Glass fabric	Glass fabric	Quartz fabric
Reinforcement style	7781	7781	581
Fiber description	E-Glass	E-Glass	Quartz
Orientation	Warp	Warp	Warp
Cure temperature, °F (°C) ¹	350 (177)	350 (177)	350 (177)
Cure pressure, psi (MPa)	Vacuum 13 (0.09)	Autoclave 45 (0.31)	Vacuum 13 (0.09)
Cure time	2 hours	2 hours	2 hours
Heat-up rate, degrees/min (°F/°C)	1-3 (0.6-1.7)	1-3 (0.6-1.7)	1-3 (0.6-1.7)

Physical properties

Ply thickness, inches (ram)	0.0080 (0.203)	0.0075 (0.191)	0.0105 (0.267)
Specific gravity, g/cc	1.97	1.97	1.62
Resin content, % by wt, nominal	21.0	20.5	24.8

Mechanical properties

Flexural strength, ksi (MPa)³			
RT	70 (483)	75 (517)	70 (483)
450°F (232°C)	—	65 (448)	—
550°F (288°C)	60 (414)	—	54 (372)
600°F (316°C)	—	54 (372)	—
Flexural modulus, msi (GPa)³			
RT	3.8 (26)	3.9 (27)	3.5 (24)
450°F (232°C)	—	3.5 (24)	—
550°F (288°C)	3.4 (23)	—	3.5 (24)
600°F (316°C)	—	3.5 (24)	—

¹ See "Recommended bagging/cure/procedures" section.
² Where testing options for either warp or fill may apply, warp conditions are assumed unless otherwise indicated.
³ Per ASTM D790.

Mechanical performance

Product description	Glass fabric	Glass fabric	Quartz fabric
Reinforcement style	7781	7781	581
Fiber description	E-Glass	E-Glass	Quartz
Orientation	Warp	Warp	Warp
Cure temperature, °F (°C) ¹	350 (177)	350 (177)	350 (177)
Cure pressure, psi (MPa)	Vacuum 13 (0.09)	Autoclave 45 (0.31)	Vacuum 13 (0.09)
Cure time ¹	2 hours	2 hours	2 hours
Heat-up rate, degrees/min (°F/°C)	1-3 (0.6-1.7)	1-3 (0.6-1.7)	1-3 (0.6-1.7)

Physical properties

Ply thickness, inches (ram)	0.0080 (0.203)	0.0075 (0.191)	0.0105 (0.267)
Specific gravity, g/cc	1.97	1.97	1.62
Resin content, % by wt., nominal	21.0	20.5	24.8

Mechanical properties²

Tensile strength, ksi (MPa)³			
RT	54 (372)	59 (407)	44 (303)
550°F (288°C)	53 (366)	57 (393)	—
Tensile modulus, msi (GPa)³			
RT	3.7 (26)	3.8 (26)	3.3 (23)
550°F (268°C)		—	
Tensile fill strength, ksi (MPa)³			
RT	49 (338)	—	42 (290)
Tensile fill modulus, msi (GPa)³			
RT	3.5 (24)	—	3.2 (22)

¹ See "Recommended bagging/cure/postcure procedures" section.
² Where testing options for either warp or fill may apply, warp conditions are assumed unless otherwise indicated.
³ Glass fabric tested per ASTM D638. Quartz fabric tested per ASTM D3039.



Mechanical performance

Product description	Glass fabric	Glass fabric	Quartz fabric
Reinforcement style	7781	7781	581
Fiber description	E-Glass	E-Glass	Quartz
Orientation	Warp	Warp	Ward
Cure temperature, °F (°C) ¹	350 (177)	350 (177)	350 (177)
Cure pressure, psi (MPa)	Vacuum 13 (0.09)	Autoclave 45 (0.31)	Vacuum 13 (0.09)
Cure time ¹	2 hours	2 hours	2 hours
Heat-up rate, degrees/min (°F/°C)	1-3 (0.6-1.7)	1-3 (0.6-1.7)	1-3 (0.6-1.7)

Physical properties

Ply thickness, inches (ram)	0.0080 (0.203)	0.0075 (0.191)	0.0105 (0.267)
Specific gravity, g/cc	1.97	1.97	1.62
Resin content, % by wt., nominal	21.0	20.5	24.8

Mechanical properties²

Compressive strength, ksi (MPa)³			
RT	70 (483)	—	58 (400)
450°F (232°C)	49 (338)	—	—
550°F (288°C)	42 (290)	—	44 (303)
Compressive modulus, msi (GPa)³			
RT	—	—	3.5 (24)
550°F (288°C)	—	—	3.4 (23)

¹ See "Recommended bagging/cure/postcure procedures" section.

² Where testing options for either warp or fill may apply, warp conditions are assumed unless otherwise indicated.

³ Per ASTM D695.

Mechanical performance

Product description	Glass fabric	Glass fabric	Quartz fabric
Reinforcement style	7781	7781	581
Fiber description	E-Glass	E-Glass	Quartz
Orientation	Warp	Warp	Warp
Cure temperature, °F (°C) ¹	350 (177)	350 (177)	350 (177)
Cure pressure, psi (MPa)	Vacuum 13 (0.09)	Autoclave 45 (0.31)	Vacuum 13 (0.09)
Cure time ¹	2 hours	2 hours	2 hours
Heat-up rate, degrees/min (°F/°C)	1-3 (0.6-1.3)	1-3 (0.6-1.3)	1-3 (0.6-1.3)

Physical properties

Ply thickness, inches (mm)	0.0080 (0.203)	0.0075 (0.191)	0.0105 (0.267)
Specific gravity, g/cc	1.97	1.97	1.62
Resin content, % by wt, nominal	21.0	20.5	24.8

Mechanical properties²

Compressive interlaminar shear strength, ksi (MPa)³			
RT	9.0 (62)	—	—
550°F (288°C)	6.0 (41)	—	—
Tensile interlaminar shear strength, ksi (MPa)⁴			
RT	4.0 (26)	—	—
550°F (288°C)	3.0 (21)	—	—
Short beam shear strength, ksi (MPa)⁵			
RT	—	7.0 (48)	6.0 (41)
450°F (232°C)	—	5.0 (34)	—
550°F (288°C)	—	—	4.3 (30)
600°F (316°C)	—	3.4 (23)	—

¹ See "Recommended bagging/cure/postcure procedures" section.

² Where testing options for either warp or fill may apply, warp conditions are assumed unless otherwise indicated.

³ Per ASTM D3846

⁴ Per FED-STD-406 method 1042.

⁵ Per ASTM D2344.

Dielectric performance

Product description

	Glass fabric	Quartz fabric
Reinforcement style	7781	581
Fiber description	E-Glass	Quartz
Orientation	Warp	(0°/45°)
Cure temperature, °F (°C) ¹	350 (177)	350 (177)
Cure pressure, psi (MPa)	Vacuum 13 (0.09)	Autoclave 45 (0.31)
Cure time ¹	2 hours	2 hours
Heat-up rate, degrees/rain (°F/°C)	1-3 (0.6-1.7)	1-3 (0.6-1.7)

Physical properties

	Glass fabric	Quartz fabric
Thickness, inches (mm)	0.105 (2.67)	0.105 (2.67)
Resin content, % by wt., nominal	21.6	21.6

Dielectric data²

	Glass fabric	Quartz fabric
Dielectric constant @ 10 GHz	4.8052	3.4000
Dielectric loss tangent @ 10 GHz	0.0006	0.0004

¹ See "Recommended bagging/cure/postcure procedures" section.

² Per "X" band frequency waveguide method.

Recommended bagging/cure/postcure procedures

Layup procedure-vacuum bag/autoclave

1. Cover tool plate with non-porous armalon, or release treat.
2. Laminate.
3. One ply nylon bleeder or porous armalon.
4. 7781 edge bleed touching laminate plies - see "note".
5. 7500 glass bleeder (bleeder plies shall extend 2" beyond edge of laminate) - see "note".
6. Bag and seal.

Note: Use only ply of 7781 and 7500 glass bleeder for every two piles of prepreg. Based on style 7781 prepreg or equivalent.

Cure cycle-vacuum bag/autoclave

1. Apply vacuum of 27" Hg minimum at room temperature. Vacuum lines should be 3/8" minimum diameter and equipped with cold caps for collecting low boiling volatiles.
2. Heat 1°F per minute maximum to 160°F - hold 1 hour.
3. Heat 1°F per minute maximum to 230°F - hold 2 hours.

Cure cycle-vacuum bag only

4. Heat 1°F per minute maximum to 270°F - hold 1 hour.
5. Heat 3°F per minute to 350°F -hold 1 hour.
6. Cool to 150°F or below under vacuum at 3-5°F per minute before removing part.

Cure cycle-autoclave only

4. Heat 1°F per minute maximum to 270°F. Once part temperature reaches 270°F, apply 45 psi and hold 2 hours.
5. Heat 3°F per minute to 350°F hold 1 hour.
6. Cool to 150°F or below under pressure and vacuum at 3-5°F per minute before removing part.

Preform procedure-press

1. Prepare laminate to required ply count.
2. Preform over tool. Preform tool should be heavy wire mesh or screen for allowing maximum breathing.
3. Cover preform layup with porous armalon.
4. Restrain the preform to designed shape with suitable porous/breathable webbed material.
5. Place restrained preform in oven at room temperature and heat at 3°F maximum per minute to 230°F plus zero (0) minus five (5) °F. Hold at 230°F for 2 hours minimum.

Molding procedure-press

1. Remove preform from oven.
2. Remove restraining material and porous armalon.
3. Place laid up preform in release coated press preheated to 230°F. Close press and hold at 230°F for 1 hour.
4. Heat 1°F per minute to 270°F. Apply 200 psi minimum and hold for 1 hour.
5. Heat 3°F per minute to 350°F. Hold for 1 hour.
6. Cool under pressure to 150°F or below at 3-5°F per minute before removing part.

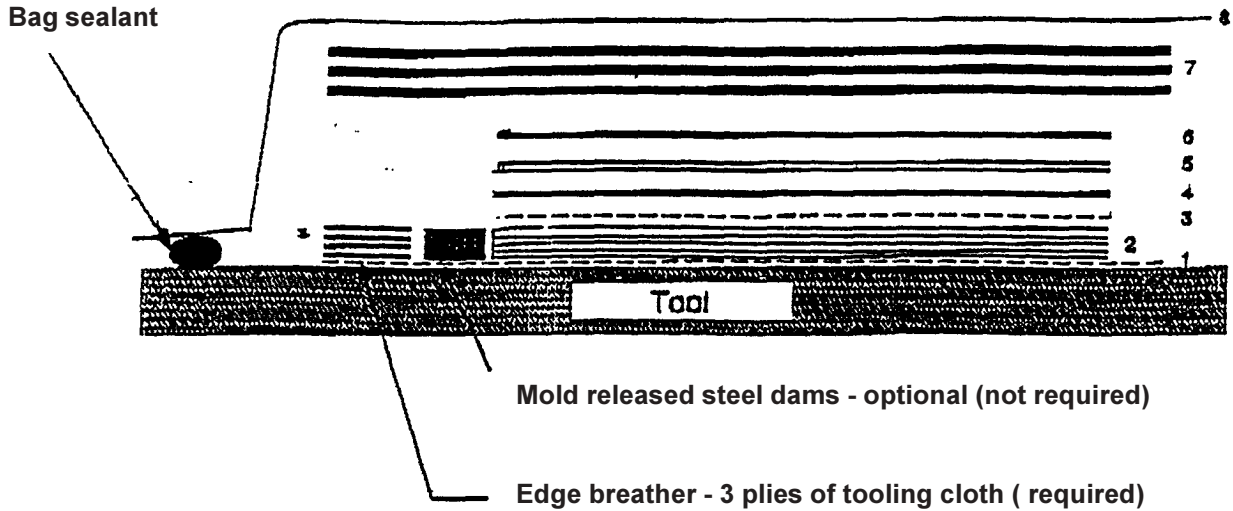
Note: Bump press once temperature reaches 350°F SEVERAL INTERVALS CRUCIAL 230-250-300-350

Postcure-vacuum bag/autoclave press

1. 1 hour @ 350°F
2. 2 hours @ 400°F
3. 2 hours @ 450°F
4. 2 hours @ 500°F
5. 2 hours @ 550°F
6. 2 hours @ 600°F

Bagging scheme for CYCOM® 3002 e-glass/polyimide

Bagging scheme for CYCOM® 3002
E-Glass/polyimide



Number	Material description
1	Porous teflon glass = 1" larger than part
2	Giass/polyimide
3	Porous teflon glass net part area
4	Separator perforated 3" centers net
5	Glass bleeder/ breather net 7500 PREFERRED
6	Separator perforatad 3" centers net
7	Tooling cloth (3 plies) breather for vacuum distribution
8	Vacuum bag

Health and safety information

Warning: CYCOM® 3002 advanced composite prepreg contains polyimide resin. This resin may cause eye and skin irritation. Avoid prolonged or re-peated contact with skin or eyes and overexposure to vapor.

First aid: In case of skin contact, wash affected areas with soap and water. In case of eye contact, immediately irrigate with plenty of water for 15 minutes. Ventilation required: Use mechanical exhaust ventilation when heat-curing resin system.

Detailed handling instructions: Refer to material safety data sheets and product labels.

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