T300HoneySiC – Problems Solved
NASA Phase II SBIR Contract NNX11CB94C

TREX ENTERPRISES CORPORATION

November 20, 2014
Mirror Technology Days
Albuquerque, New Mexico

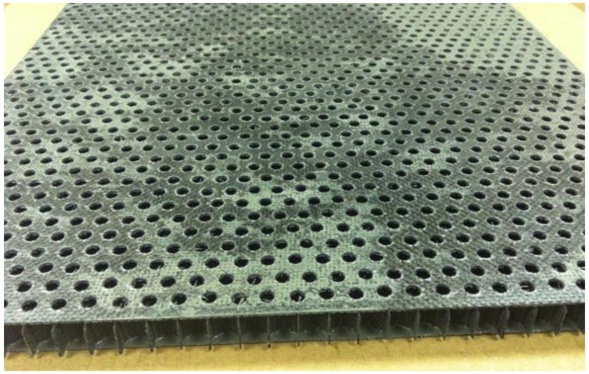
Dr. Bill Goodman
Trex Enterprises Corporation
2071 Pan American Freeway NE
Albuquerque, New Mexico 87107
858-437-3899; bgoodman@trexenterprises.com

Outline

- Honeycomb Delamination Solved
- A New End-To-End Process Flow
- Machining Lesson Learned
- What is the Best Matrix?
- Machining Problem Solved, We Made Parts
- HoneySiC Test Data from Southern Research Institute (SoRI)

Honeycomb Delamination Solved

- **PREMISE →If you can mold Honeycomb in prepreg state, then you can make lightweight structures with minimal machining (e.g., mirrors, optical benches, trusses, structures) very rapidly (<3 weeks), and at low cost.**



- **What problems were encountered?**
  - SiC fibers (any kind) 10X higher $, weigh more.
  - Desire to lightweight with triax or leno weaves, but cannot with Nicalon.
  - Chose plain weave T300 to build up plies.
  - Initial attempts to mold honeycomb failed.

- **Plain weave T300 is *almost* too stiff to mold into honeycomb.**
- **Successfully employed extended hold time procedure (167 deg F to promote cross linking of SMP-730 preceramic polymer, prior to an elevated cure temperature) to produce laminate & honeycomb materials.**
- **Produced a 12-inch x 12-inch plate**

A New End-To-End Process Flow

1) ProcureT300 fiber, ship to weaver. Weave fabric, lock-out edges w/carbon fiber selvage edge. Roll up and ship to Starfire Systems.
2) Starfire Systems inspects, re-rolls and heat treats to burn off sizing.
3) SMP-730 pre-ceramic polymer and fabric to prepregger.
4) Drop ship prepreg roll to Ultracor. Cut $0^o/90^o$ and $+/-45^o$ sheets and layup. The facesheets have 8 plies with a total thickness on the order of 0.04-inches. One facesheet is holed for venting.
5) Honeycomb is two plies thick where two cell walls adjoin, single ply elsewhere.
6) New low temperature extended hold pre-cure (167 ^oF), followed by a higher temperature cure for the honeycomb core and formation of sandwich panel.
7) Starfire Systems fortifies sandwich panel bonds with SMP-10 pre-cure step, then pyrolyzes at 600 degrees C. A 1st infiltration and pyrolysis step with pre-ceramic polymer SMP-10 is performed (600 degrees C).

MAY WE MACHINE NOW ?

Machining Lesson Learned

- Machining by Dean Szwabowski Professional Services (DSPS) in San Diego
- Sample tensile coupon was cut, resulting in fraying and separation of the plies.
- Further noted by DSPS that the laminate panel was very difficult to mount for machining using a vacuum approach – after a single polymer infiltration and pyrolysis step the material is simply too porous. The panel was also too delicate to mount with double-sided sticky tape. Consequently no attempt was made to machine the honeycomb.
- The team concurred that some of the porosity would have to be filled in order to machine.
- SO NO, YOU CANNOT MACHINE YET!

1000 deg C Matrix Densification Selection Study

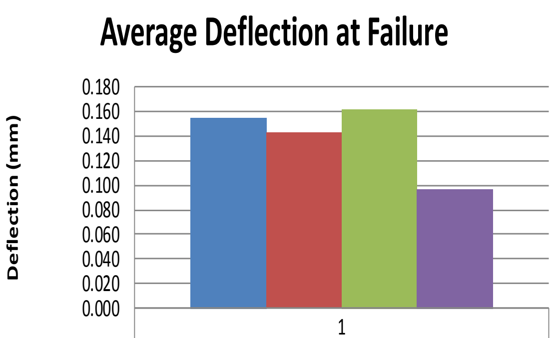
- U of H Studied preceramic polymers pyrolyzed at 1000 °C, four cases:
  1. Pure SMP-10
  2. Pure SPR-212
  3. SMP-10 Hybrid (first infiltration/pyrolysis with SMP-10 and subsequent infiltrations/pyrolyses with SPR-212)
  4. SPR-212 Hybrid (first infiltration/pyrolysis with SPR-212 and subsequent infiltrations/pyrolyses with SMP-10)

- 0.75-mm x 2.8-mm x 44-mm coupons underwent 8-10 PIP Cycles. Vacuum assisted infiltration. Inert vacuum pyrolysis. Convergence criteria weight gain of less than 1%.

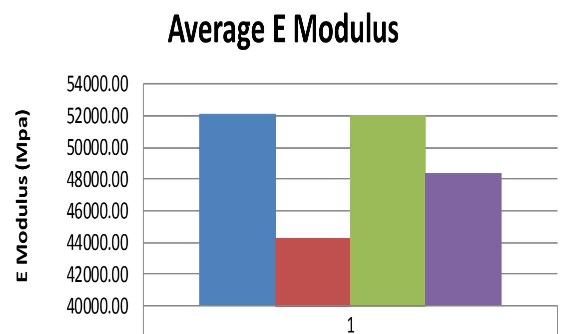
- Performed 4-point bending tests according to ASTM standards, measured Flexural Strength, Strain to Failure, Deflection to Failure, Modulus, and Toughness.

- The SMP-10 Hybrid (Case 3 above) worked best - highest overall Strength, Modulus, and Toughness as the data will show.

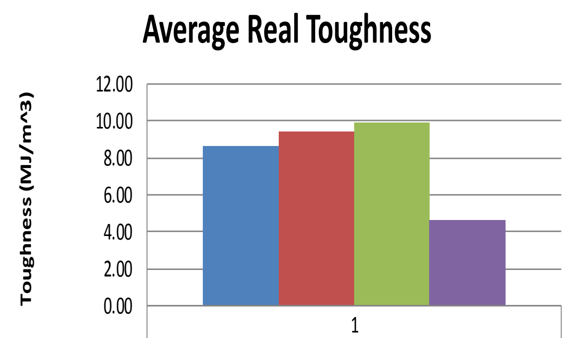
What is the Best Matrix Precursor?



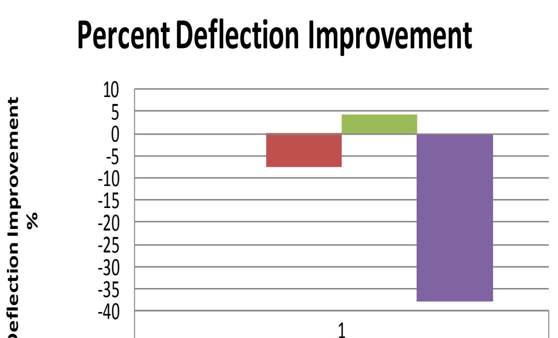
	1
SMP-10	0.155
SPR-212	0.143
SMP-10 Hybrid	0.162
SPR-212 Hybrid	0.096



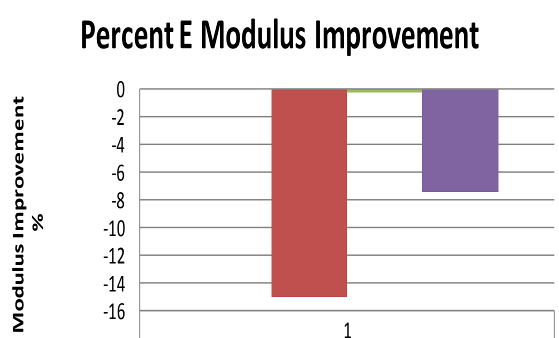
	1
SMP-10	52144.15
SPR-212	44330.17
SMP-10 Hybrid	52034.33
SPR-212 Hybrid	48308.42



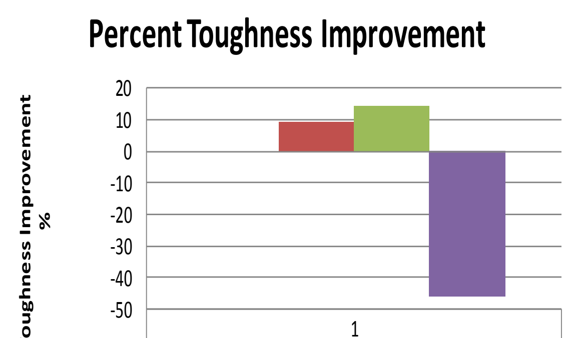
	1
SMP-10	8.65
SPR-212	9.44
SMP-10 Hybrid	9.90
SPR-212 Hybrid	4.67



	1
SMP-10	0
SPR-212	-7.588218462
SMP-10 Hybrid	4.391193384
SPR-212 Hybrid	-37.81417587



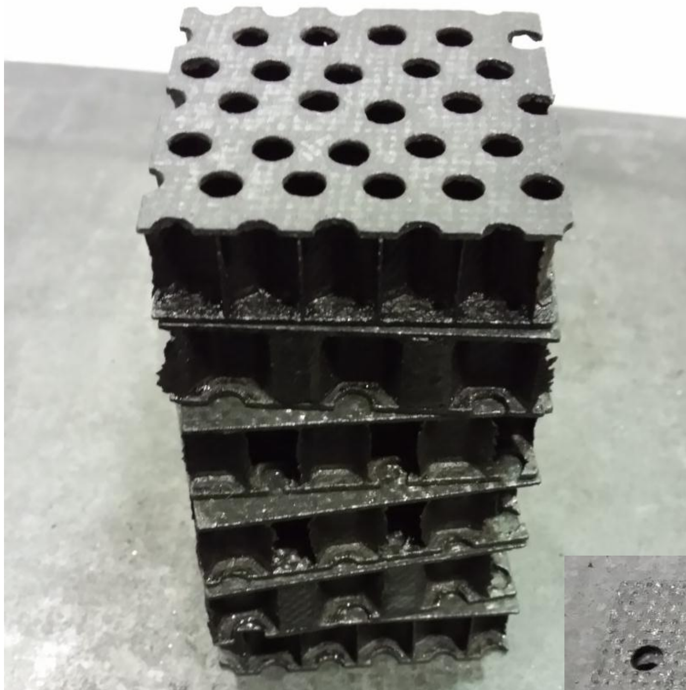
	1
SMP-10	0
SPR-212	-14.98533548
SMP-10 Hybrid	-0.210596857
SPR-212 Hybrid	-7.35600135

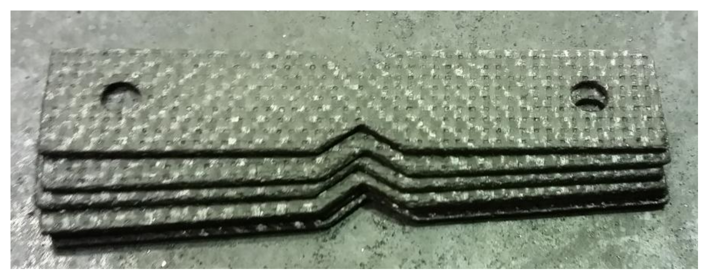


	1
SMP-10	0
SPR-212	9.098001854
SMP-10 Hybrid	14.43703629
SPR-212 Hybrid	-46.07468208

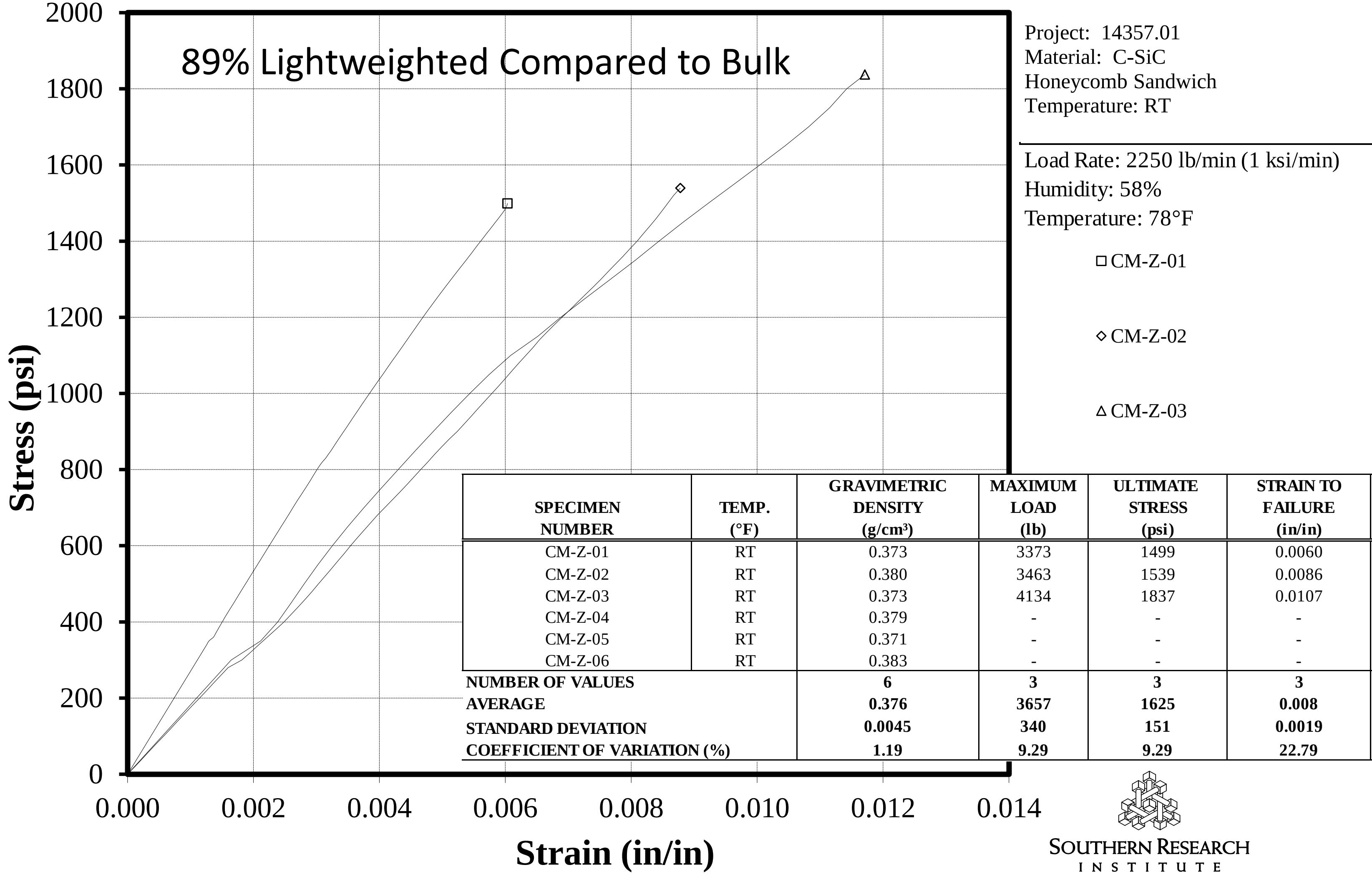
Machining Problem Solved – Voila!

- University of Hawaii infiltrated with SMP-10 resin and cured, but did not pyrolize.
- Coupons are then CNC machined by DSPS.
- Cut parts then pyrolized. Next infiltrate and pyrolyze the samples with SPR-212, as many PIP cycles as required to converge to <1% weight gain, as per 1000 deg C study.





SoRI Data – Honeycomb Sandwich Compressive Strength

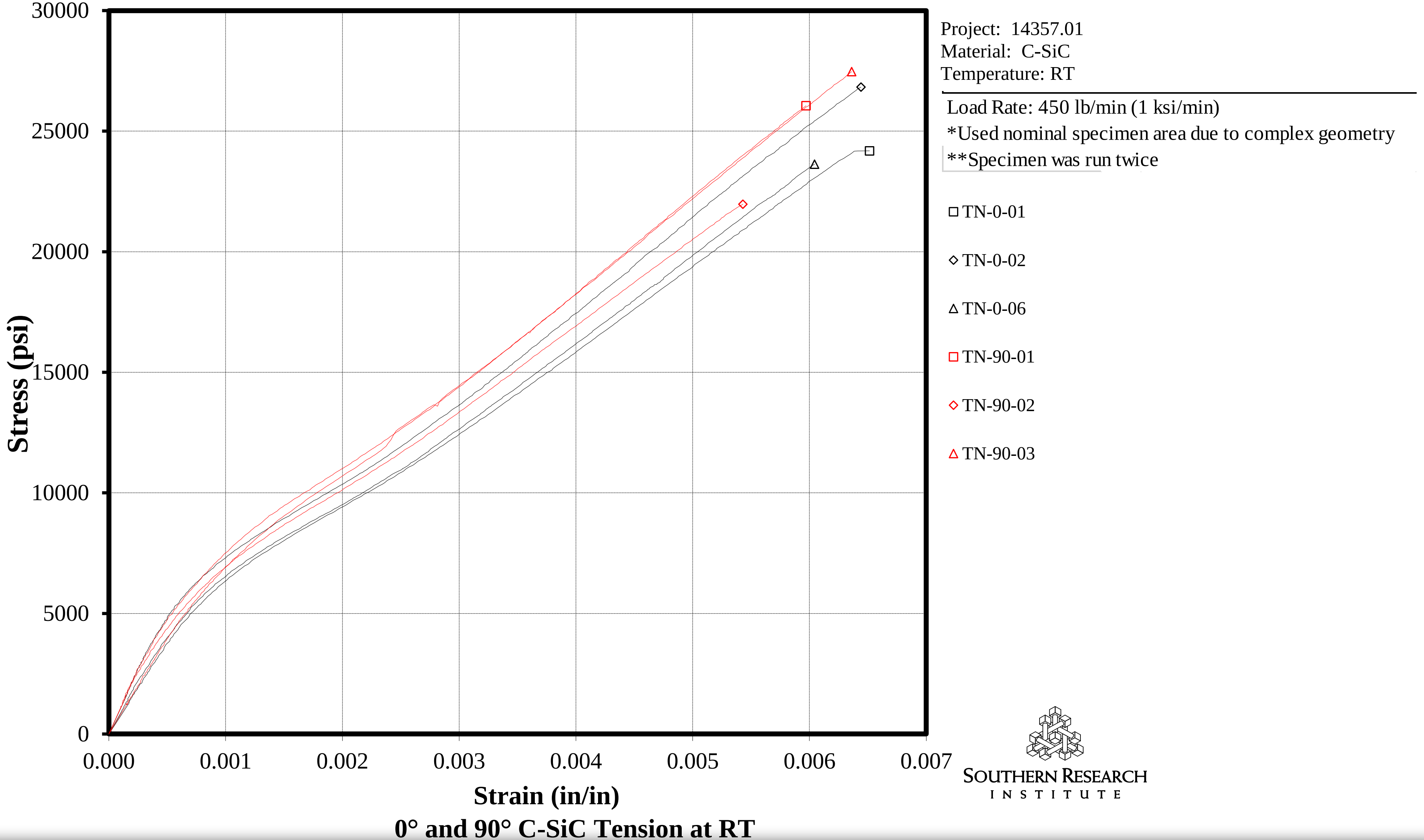


SPECIMEN NUMBER	TEMP. (°F)	GRAVIMETRIC DENSITY (g/cm³)	MAXIMUM LOAD (lb)	ULTIMATE STRESS (psi)	STRAIN TO FAILURE (in/in)	INI. ELASTIC MODULUS (ksi)
CM-Z-01	RT	0.373	3373	1499	0.0060	271
CM-Z-02	RT	0.380	3463	1539	0.0086	175
CM-Z-03	RT	0.373	4134	1837	0.0107	182
CM-Z-04	RT	0.379	-	-	-	-
CM-Z-05	RT	0.371	-	-	-	-
CM-Z-06	RT	0.383	-	-	-	-
NUMBER OF VALUES		**6**	**3**	**3**	**3**	**3**
AVERAGE		**0.376**	**3657**	**1625**	**0.008**	**209.5**
STANDARD DEVIATION		**0.0045**	**340**	**151**	**0.0019**	**43.4**
COEFFICIENT OF VARIATION (%)		**1.19**	**9.29**	**9.29**	**22.79**	**20.74**

SOUTHERN RESEARCH INSTITUTE

Through Thickness Honeycomb Sandwich Compression at RT

SoRI Data – Tensile Strength of Facesheets

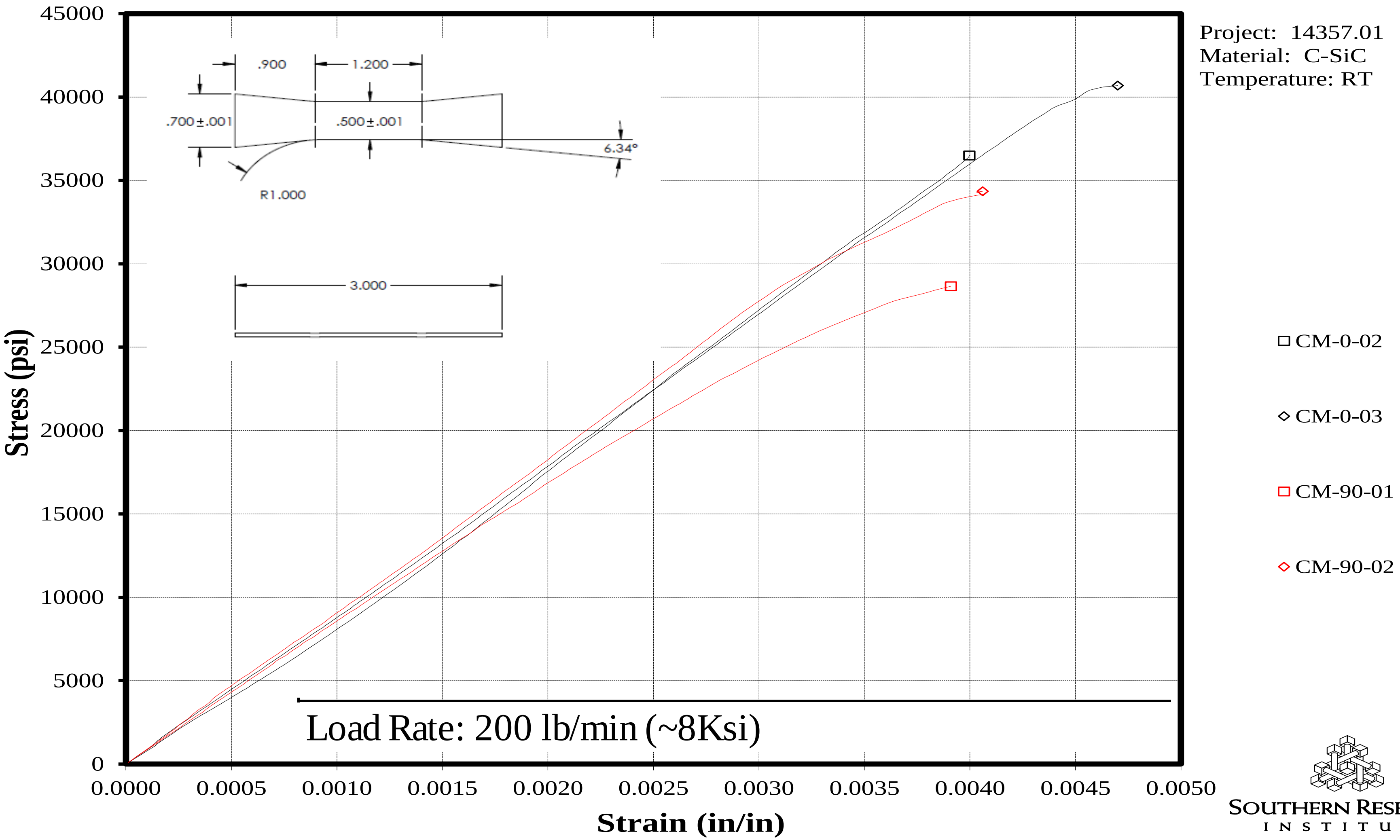


SOUTHERN RESEARCH INSTITUTE

Skins have 50% Strength of Bulk at 58% Relative Density

SPECIMEN NUMBER	TEMP. (°F)	THICKNESS (in)	WEIGHT (g)	VOLUME* (cm^3)	GRAVIMETRIC DENSITY (g/cm^3)	MAXIMUM LOAD (lb)	ULTIMATE STRESS (psi)	STRAIN TO FAILURE (in/in)	INI. ELASTIC MODULUS (ksi)
TN-0-01	RT	0.053	4.823	2.650	1.820	385	24185	0.0065	7.43
TN-0-02	RT	0.049	4.764	2.465	1.933	397	26831	0.0064	10.58
TN-0-03	RT	0.052	4.869	2.610	1.866	-	-	-	-
TN-0-04	RT	0.050	4.757	2.505	1.899	-	-	-	-
TN-0-05	RT	0.052	4.716	2.620	1.800	-	-	-	-
TN-0-06	RT	0.054	4.822	2.680	1.799	382	23633	0.0060	8.55
NUMBER OF VALUES		**6**	**6**	**6**	**6**	**3**	**3**	**3**	**3**
AVERAGE		**0.052**	**4.792**	**2.588**	**1.853**	**388**	**24883**	**0.0063**	**8.85**
STANDARD DEVIATION		**0.0015**	**0.0511**	**0.0773**	**0.0507**	**6.394**	**1395.59**	**0.0002**	**1.304**
COEFFICIENT OF VARIATION (%)		**2.99**	**1.07**	**2.99**	**2.74**	**1.65**	**5.61**	**3.27**	**14.73**
TN-90-01	RT	0.046	4.354	2.300	1.893	360	26052	0.0060	10.80
TN-90-02	RT	0.048	4.446	2.410	1.845	318	21968	0.0054	10.62
TN-90-03	RT	0.046	4.262	2.305	1.849	381	27473	0.0064	8.00
TN-90-04	RT	0.045	4.478	2.265	1.977	-	-	-	-
TN-90-05	RT	0.044	4.284	2.205	1.943	-	-	-	-
TN-90-06	RT	0.054	4.930	2.675	1.843	-	-	-	-
NUMBER OF VALUES		**6**	**6**	**6**	**6**	**3**	**3**	**3**	**3**
AVERAGE		**0.047**	**4.459**	**2.360**	**1.892**	**353**	**25165**	**0.0059**	**9.80**
STANDARD DEVIATION		**0.0031**	**0.2246**	**0.1535**	**0.0520**	**26.483**	**2333.29**	**0.0004**	**1.279**
COEFFICIENT OF VARIATION (%)		**6.50**	**5.04**	**6.50**	**2.75**	**7.50**	**9.27**	**6.45**	**13.05**

SoRi Data – Compressive Strength of Facesheets



0° and 90° C-SiC Compression at RT

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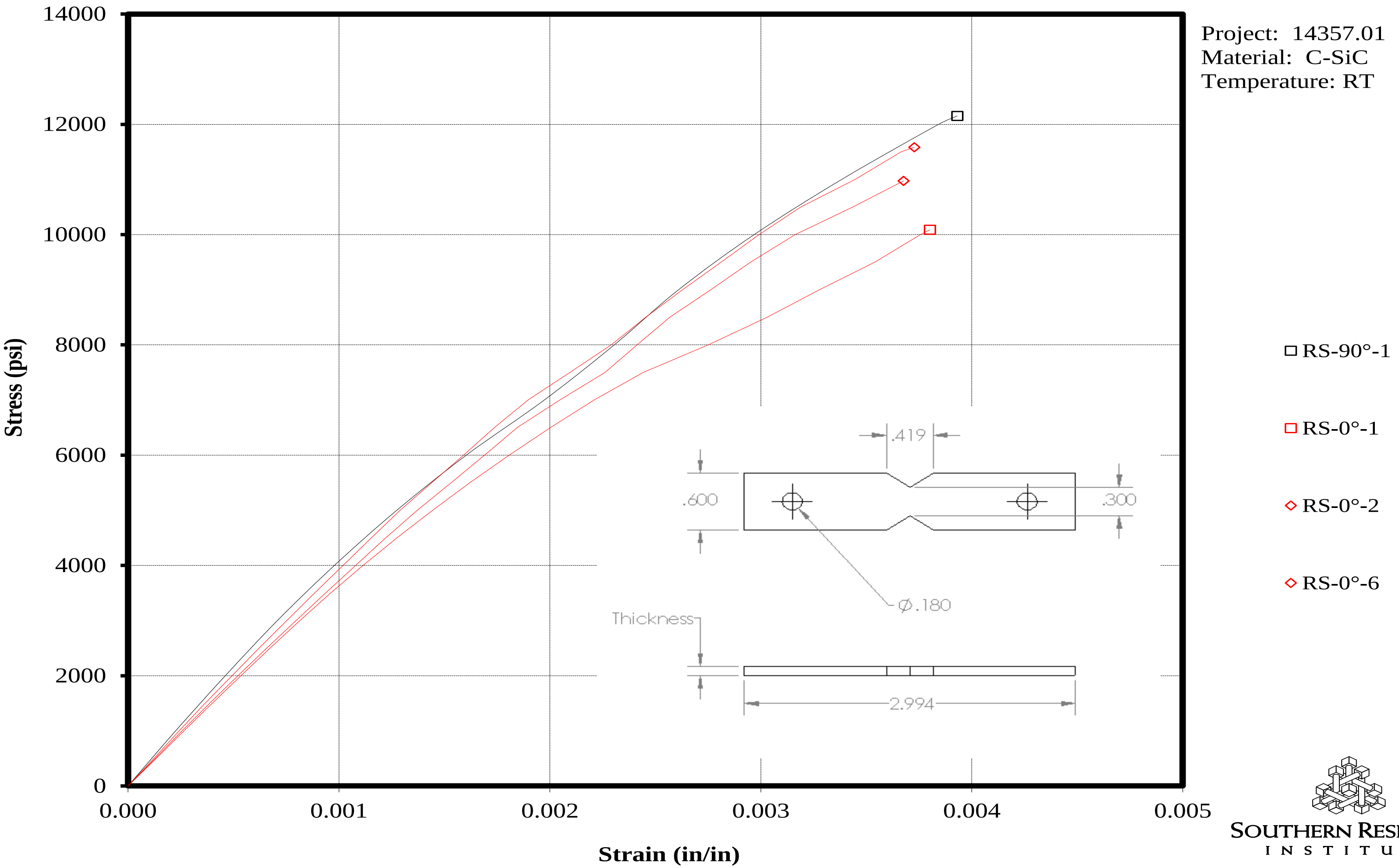
Facesheets Are Strong in Compression

SPECIMEN NUMBER	TEMP. (°F)	GAUGE WIDTH (in)	GAUGE THICKNESS (in)	WEIGHT (g)	VOLUME* (cm³)	GRAVIMETRIC DENSITY (g/cm³)	MAXIMUM LOAD (lb)	ULTIMATE STRESS (psi)	STRAIN TO FAILURE (in/in)	INI. ELASTIC MODULUS (ksi)	SECONDARY MODULUS (ksi)
CM-0-01	RT	0.500	0.048	2.179	1.315	1.657	-	-	-	-	
CM-0-02	RT	0.500	0.051	2.361	1.386	1.704	925	36493	0.0040	7.89	10.45
CM-0-03	RT	0.500	0.051	2.293	1.380	1.661	1027	40682	0.0047	8.67	9.16
CM-0-04	RT	0.500	0.054	2.473	1.476	1.675	-	-	-	-	
CM-0-05	RT	0.500	0.054	2.488	1.487	1.673	-	-	-	-	
CM-0-06	RT	0.500	0.053	2.331	1.460	1.597	-	-	-	-	
NUMBER OF VALUES			**6**	**6**	**6**	**6**	**2**	**2**	**2**	**2**	
AVERAGE			**0.052**	**2.354**	**1.417**	**1.661**	**976**	**38588**	**0.0044**	**8.28**	
STANDARD DEVIATION			**0.0023**	**0.1058**	**0.0619**	**0.0323**	**51.064**	**2094.52**	**0.0003**	**0.388**	
COEFFICIENT OF VARIATION (%)			**4.37**	**4.49**	**4.37**	**1.94**	**5.23**	**5.43**	**8.03**	**4.68**	
CM-90-01	RT	0.500	0.046	2.140	1.246	1.717	-	-	-	-	
CM-90-02	RT	0.500	0.047	2.129	1.285	1.657	674	28648	0.0039	8.58	-
CM-90-03	RT	0.500	0.047	2.196	1.293	1.698	808	34155	0.0041	8.96	9.57
CM-90-04	RT	0.500	0.045	2.098	1.216	1.725	-	-	-	-	
CM-90-05	RT	0.500	0.046	2.109	1.246	1.692	-	-	-	-	
CM-90-06	RT	0.500	0.046	2.135	1.266	1.687	-	-	-	-	
NUMBER OF VALUES			**6**	**6**	**6**	**6**	**2**	**2**	**2**	**2**	
AVERAGE			**0.046**	**2.134**	**1.259**	**1.696**	**741**	**31401**	**0.0040**	**8.77**	
STANDARD DEVIATION			**0.0009**	**0.0310**	**0.0258**	**0.0220**	**67.059**	**2753.18**	**0.0001**	**0.192**	
COEFFICIENT OF VARIATION (%)			**2.05**	**1.45**	**2.05**	**1.30**	**9.05**	**8.77**	**1.89**	**2.19**	

SoRI Data – Romanian Shear Test



0° and 90° C-SiC Iosipescu Shear at RT

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TREX ENTERPRISES HAWAIIAN OPERATIONS

Facesheet Shear Strength Over 11 ksi

SPECIMEN NUMBER	TEMP. (°F)	GAUGE WIDTH (in)	GAUGE THICKNESS (in)	WEIGHT (g)	VOLUME* (cm³)	GRAVIMETRIC DENSITY (g/cm³)	MAXIMUM LOAD (lb)	ULTIMATE STRESS (psi)	STRAIN TO FAILURE (in/in)	INI. ELASTIC MODULUS (ksi)
RS-0°-1	RT	0.314	0.045	2.073	1.249	1.660	143	10088		3.72
RS-0°-2	RT	0.313	0.045	2.101	1.241	1.693	163			
RS-0°-3	RT	0.311	0.046	2.069	1.277	1.620	174	12068		
RS-0°-4	RT	0.315	0.046	2.094	1.268	1.650				
RS-0°-5	RT	0.316	0.046	2.098	1.274	1.647				
RS-0°-6	RT	0.315	0.047	2.113	1.302	1.623	163	10974		3.78
NUMBER OF VALUES			**6**	**6**	**6**	**6**	**4**	**3**		**2**
AVERAGE			**0.046**	**2.091**	**1.268**	**1.649**	**161**	**11043**		**3.75**
STANDARD DEVIATION			**0.0007**	**0.0156**	**0.0197**	**0.0242**	**10.942**	**809.78**		**0.031**
COEFFICIENT OF VARIATION (%)			**1.55**	**0.75**	**1.55**	**1.47**	**6.80**	**7.33**		**0.82**
RS-90°-1	RT	0.320	0.049	2.348	1.351	1.738	175	11159		4.40
RS-90°-2	RT	0.317	0.049	2.291	1.348	1.699				
RS-90°-3	RT	0.317	0.048	2.255	1.315	1.714				
RS-90°-4	RT	0.318	0.049	2.311	1.351	1.710				
RS-90°-5	RT	0.316	0.047	2.202	1.290	1.706				
RS-90°-6	RT	0.315	0.047	2.236	1.299	1.721				
NUMBER OF VALUES			**6**	**6**	**6**	**6**	**1**	**1**		**1**
AVERAGE			**0.048**	**2.274**	**1.326**	**1.715**	**175**	**11159**		**4.40**
STANDARD DEVIATION			**0.0009**	**0.0486**	**0.0254**	**0.0124**	**0.000**	**0.00**		**0.000**
COEFFICIENT OF VARIATION (%)			**1.92**	**2.14**	**1.92**	**0.72**	**0.00**	**0.00**		**0.00**

SoRI Data – Can this be? Near Zero CTE!

Temperature (°F)	Unit Thermal Expansion (x0.001 in/in)	Temperature (°F)	Unit Thermal Expansion (x0.001 in/in)	Temperature (°F)	Unit Thermal Expansion (x0.001 in/in)	Temperature (°F)	Unit Thermal Expansion (x0.001 in/in)	CTE ppm/C
-320	0.04	-320	0.04	-320	0.064	-320	0.054	
-300	0.03	-300	0.033	-300	0.053	-300	0.044	-0.9
-275	0.026	-275	0.026	-275	0.05	-275	0.037	-0.288
-250	0.02	-250	0.03	-250	0.044	-250	0.034	-0.432
-225	0.033	-225	0.023	-225	0.037	-225	0.027	0.936
-200	0.01	-200	0.02	-200	0.03	-200	0.024	-1.656
-175	0.009	-175	0.019	-175	0.026	-175	0.02	-0.072
-150	0.008	-150	0.008	-150	0.022	-150	0.015	-0.072
-125	0.008	-125	0.008	-125	0.018	-125	0.012	0
-100	-0.001	-100	0.009	-100	0.013	-100	0.01	-0.648
-75	0	-75	0	-75	0.01	-75	0	0.072
-50	0.004	-50	-0.006	-50	0.008	-50	0.001	0.288
-25	0	-25	0	-25	0.01	-25	0	-0.288
0	0.004	0	-0.006	0	0.004	0	0.001	0.288
25	0	25	0	25	0.003	25	0.007	-0.288
50	0.006	50	-0.004	50	0.006	50	0.013	0.432
75	0	75	0	75	0.003	75	0	-0.432

Conclusions

We pulled this one out.

There is a process that is amenable to current facilities and practice.

With optimized process and equipment, large, lightweight CMC parts could be turned out in weeks.
CMC with Near-Zero CTE
C/SiC is also High Temperature Material

Several $M Investment would lead to a Big Payoff.

Would like to discuss licensing.