

MECHANICAL PROPERTIES OF POLYMERIC COMPOSITES REINFORCED WITH HIGH STRENGTH GLASS FIBERS

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ABSTRACT

In the early 1960's the first high strength glass fibers, S-glass, were developed in joint work between Owens Corning and the US Air Force. Today high strength glasses with various batch formulations are manufactured commercially by several companies in every major economic region. The utility of high strength glass fiber compositions are compared by physical, mechanical, electrical, thermal, and optical properties. The influence of filament diameter, over the range 9 - 26 microns, and size chemistry on composite mechanical performance is described, in epoxy and vinyl/polyester resins. Data on high strength glass composites reinforced with UD fiber and fabrics are reported from -55°C to 80°C .

KEY WORDS: Fibers, Glass Reinforcements, Glass Fiber Composites, Mechanical Properties, Prepregs, S-glass, ZenTron.

1. INTRODUCTION TO HIGH STRENGTH GLASS FIBER

In the early 1960s high strength glass fibers, S-glass, were first used in the joint work between Owens Corning and the United States Air Force for use in strategic missiles and other military end-uses. In the period 1966-1968 Owens Corning introduced high strength glass reinforcements for commercial applications under the trade name S-2 glass[®]. The two fibers share the same glass composition; the primary difference in the two product families was the sizing. The original S-glass product range has now been entirely replaced by *S-2 glass* rovings.

S-2 glass fibers are a magnesium alumino-silicate produced for textile substrates or reinforcements in composite applications. RH glass was introduced in 1968 by Vetrotex, and remained in production until the early 1990's. More recently yarns and assembled rovings belonging to the S-glass family have been manufactured in Japan under the names T glass and U

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glass. A comparison of the chemical composition and physical, mechanical and other properties of S-glass, E-glass and various other commercially available glass fibers has been published elsewhere[1]. *S-2 glass* fibers are manufactured commercially in slivers of 11 to 66 tex, with filament diameters of 5 to 13 microns. *S-2 glass* rovings are assembled from these slivers, and are available up to 2050 tex. In 1997 a new single ended roving process for high strength glass was introduced commercially, and these S-glass rovings are sold under the trade name *ZenTron*. Unlike the *S-2 glass* assembled rovings, the single ended rovings are made in a one step process, which typically results in rovings with a linear density in the range of 660-2050 tex, and filament diameters in the range of 13 to 25 microns. Whereas *S-2 glass* products are largely used in defense and aerospace, the *ZenTron* products have found applications in the automotive (2) and sporting goods industries.

2. COMERCIAALLY AVAILABLE GLASS FIBER COMPOSITIONS

2.1 Introduction The following glass types account for the vast majority of glass fibers produced commercially for use as reinforcements:

Advantex®. Calcium aluminosilicate glass used as a boron free substitute for E-glass, providing E-CR Glass acid resistance with the reinforcing characteristics of E-glass. Introduced in 1997.

AR Glass. Alkali resistant glasses composed of alkali zirconium silicates, developed for use in use in cement and concrete. Mainly used for GRC (Glass reinforced Cement) roofing and sheeting panels.

C glass. Calcium borosilicate glasses used for their exceptional stability in acidic environments. The resistance to water is poor, and so C glass is rarely used as a reinforcement in North America or Europe, but finds application in surface mats, glass flakes for coatings and acid-resistant surface cloth.

D glass. Borosilicate glasses with low dielectric constant used for radomes and other specialty applications requiring permeability to electromagnetic waves.

E glass. Alumina-calcium-borosilicate glasses with a maximum alkali content of 2% w/w used as a general purpose reinforcement where strength and high electrical resistivity are required. Together with Advantex, accounts for over 90% of all GRP reinforcements produced today. Introduced in 1939.

E-CR Glass. Calcium aluminosilicate glasses with a maximum alkali content of 2% w/w used where acid corrosion resistivity is required in addition to strength and electrical resistivity. Introduced in 1980.

R-glass. Calcium aluminosilicate glasses with higher strength and temperature resistance than E-CR Glass or Advantex.

S-glass. Magnesium aluminosilicate glasses used for textile substrates or reinforcement in composite structural applications, which require high strength, modulus and durability under conditions of extreme temperature of corrosive environments. *S-2 glass*, T glass, U glass and *ZenTron* are examples of current commercial products. Introduced for commercial use in 1966.

TABLE 1-Composition ranges for glass fibers

	Advantex	AR glass	C glass	D glass	E glass [‡]	E-CR glass	R glass	S-2 glass
Oxide								
SiO ₂	59-62	55-75	64-68	72-75	52-56	54-62	56-60	64-66
Al ₂ O ₃	12-15	0-5	3-5	0-1	12-16	9-15	23-28	24-25
B ₂ O ₃	<0.2	0-8	4-6	21-24	5-10		0-0.35	
CaO	20-24	1-10	11-15	0-1	16-25	17-25	8-15	0-0.18
MgO	1-4		2-4		0-5	0-4	4-7	9.5-10.2
ZnO						2-5		
BaO			0-1					
Li ₂ O		0-1.5						
Na ₂ O +K ₂ O		11-21	7-10	0-4	0-2	0-2	0-1	0-0.2
TiO ₂		0-12			0-0.8	0-4		
ZrO ₂		1-18						
Fe ₂ O ₃		0-5	0-0.8	0-0.3	0-0.4	0-0.8	0-0.5	0-0.1
F ₂		0-5					0-0.1	

[‡]E glass as defined by ASTM D578-00 for aerospace and printed circuit board applications, and also by MIL-G-55636 and IPC-EG-14Q. Advantex meets the ASTM D578-00 requirements for a general purpose E-glass.

TABLE 2- Glass fiber properties by composition

	Advantex	AR glass	C glass	D glass	E glass	E-CR glass	R glass	S-2 glass
Density g/cc. ASTM D1505	2.624	2.70	2.52	2.11	2.55-2.62	2.68-2.72	2.54	2.48
Refractive Index	1.561	1.562	1.533	1.465	1.558	1.574-1.576	1.546	1.521-1.525
Softening point °C		773	750	771	846	882	952	1056
Strain point °C	691		522	477	610-630		736	766
Sp. heat cap. J/g°C			0.787	0.733	0.810		0.732	0.737
Thermal exp. coef. ppm°C	5.8	6.5	6.3	2.5	5.4	5.9	3.8	1.6
Dielectric strength kV/mm	10.12				10.4-10.6	9.84	10.8	13.0

2.2 Manufacturing of High Strength Glass Fiber.

Continuous glass fibers were first manufactured in commercial quantities by Owens Corning Textile Products in the 1930's for high temperature electrical applications. High strength glasses differ from E-glass and Advantex products in several aspects, most importantly the higher softening point temperature and the much more modest scale of production. As a consequence it is necessary to use specially built ultra high temperature (UHT) furnaces for continuous commercial production, as shown in [figures 1 and 2](#).

Raw materials such as silicates, limestone or metallic oxides are finely ground and then blended together in a bulk quantity, called the "batch". More than half the mix is silica sand, the basic building block of any glass fiber. The blended mix is then conveyed to a small furnace. The temperature is sufficiently high for the sand and other ingredients to dissolve into molten glass. The walls of the furnace are protected with tailor made liners in order to withstand the very high temperatures necessary to allow the glass to flow and form fibers. The molten glass flows through a platinum/rhodium bushing – a block of metal with precisely drilled holes to allow the glass to run through, and then through individual bushing tips and orifices ranging from 0.75 to 2 mm diameter. High strength glasses are typically processed at about 120°C higher than E-glass in the melter and hundreds of degrees higher at the bushing. The glass is then rapidly quenched and attenuated (drawn down) in air and water to prevent crystallization, forming fine fibers ranging from 5 to 26 μm in diameter. Experimental filaments over 35 μm have been produced in Huntingdon, PA.

Mechanical winders pull the fibers at lineal velocities up to 60 meters/second over an applicator which coats the fiber with an aqueous mixture of chemicals called a "sizing". This protects the filaments from abraiding each other during further handling, aids processing, and provides compatibility and adhesion of the fiber with the matrix or resin.

The greater temperatures involved in producing high strength glass present formidable technical challenges to commercial production, and few companies globally are able to make a consistently high quality product. The platinum/rhodium bushings have a hard, short life. The bushings need to be designed to allow a flat temperature profile across all orifices, such that all filaments of glass have the same diameter. As a consequence, the process is intrinsically more expensive than the larger scale lower temperature E-glass production from conventional ceramic lined furnaces.

In most cases the strands, which are typically 5-13 μm diameter filaments aligned in 11 to 80 tex slivers, are wound onto a "forming cake". The forming package is an intermediate product that is cured in an oven to drive off the moisture in the size, and then typically processed in one of two ways before being shipped to the customer. To produce a twisted yarn, the forming cake is unwound, twisted and wound onto a bobbin. To produce a multi-end roving package, several forming cakes are simultaneously unwound, then wound again onto a new package with typically 5 to 30 ends of glass strand. These "conventional" roving doffs or spools are external take off packages similar to carbon fiber or aramid fiber spools, and are used for prepregging, filament winding or weaving. The conventional roving/yarn process is illustrated in [figure 1](#).

The *ZenTron* roving is unique in the world of high strength glass as the winder collects all the strands into a single end roving package. This process is illustrated in [figure 2](#). The roving has a filament diameter in the range 13 – 26 μm and a tex from 660 to 2400 g/km. This necessitates an

S-2 Glass[®] Manufacturing

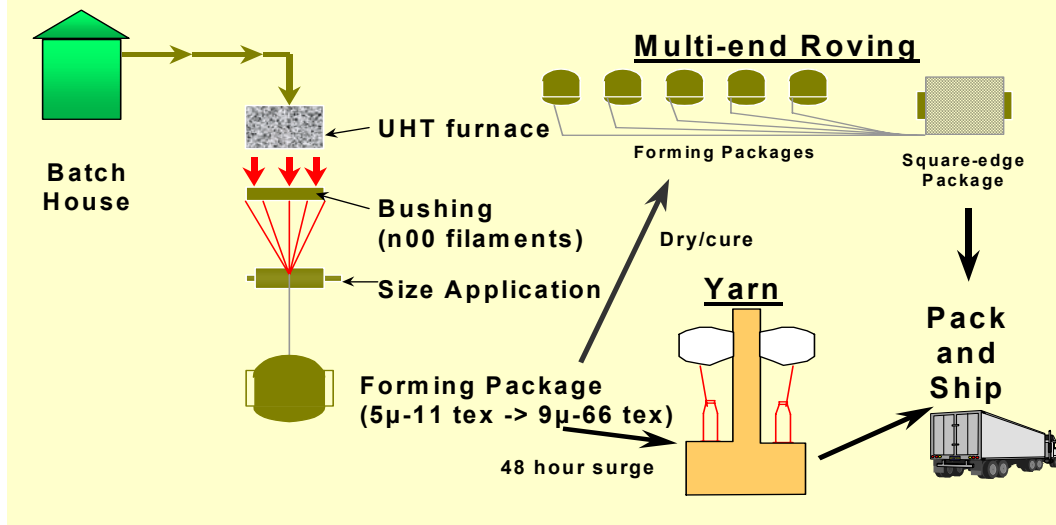


Figure 1 Schematic of conventional roving/yarn manufacturing.

order of magnitude increase in bushing size and design complexity, as thousands rather than hundreds of holes are needed. The packages may be internal take off, which is typical of E-glass rovings, or external take off, which is typical in the advanced composites

ZenTron[®] Roving Manufacturing

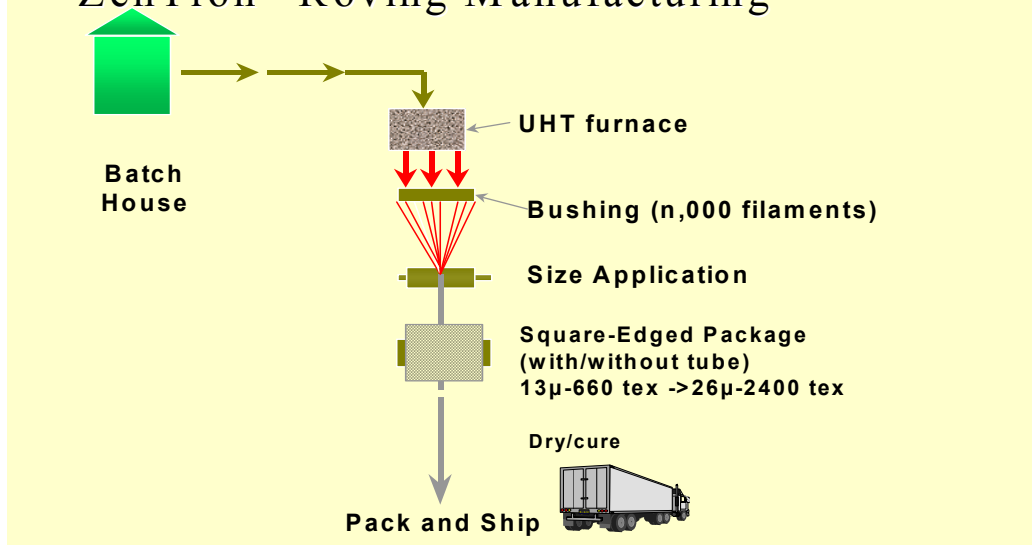


Figure 2 Schematic of single end glass roving manufacturing

industry. The packages are dried in an oven and are then ready to be shipped to the customer without further processing.

2.3 Properties of High Strength Glass Fiber.

These have been described in detail by Hartman, Greenwood and Miller(1). The S-glass family is represented commercially by *S-2 glass* and *ZenTron* from the US, and T glass and U glass from Japan. All three have similar compositions conforming to S glass in [Table 1](#). U glass is available only as a RFL coated cord for use in synchronous (timing) rubber belts.

R glass is produced in Europe, and is intermediate in performance between Advantex or EC-R glass and the S glass family.

3. GLASS FIBER SIZE TREATMENTS

3.1 Introduction As far as composite performance is concerned, the fiber matrix interface is at least as critical as the properties of the reinforcing fiber. An E glass fiber with the appropriate adhesion and interface will outperform an S glass or carbon fiber with an unsuitable fiber-matrix interface. This is especially true of thermosetting resin systems in the presence of water.

3.2 Current Industrial Practice A “size” or coating is applied to glass filaments during the extrusion process and prior to the filaments being gathered into a strand and wound into a package. The sizing provides a number of properties and functions to the glass. One important function is to prevent defects and fracture of the filaments during formation. Bare glass is very abrasive to itself; it takes a coating of size to be able to wind a continuous strand into a package during forming. Size is typically an aqueous solution or emulsion that is applied by passing the filaments over a rolling applicator. The applicator can be made from such materials as a rubber belt or graphite cylinder. By dipping the applicator into a bath of sizing a thin layer is deposited on the applicator surface and then onto each individual glass filament

This surface treatment also plays an important role in downstream functions of the glass fibers. Textile sizes, typically made from starch/oil and polyvinyl alcohol, have low coefficients of friction and provide good protection during subsequent processes of weaving, braiding, or knitting. The weaver will then typically remove the size by heat cleaning and apply a finish compatible with the matrix resin used in the final product.

Reinforcement sizes are used when the size is not thermally removed from the glass and provides a good chemical bond between the glass and matrix resin. This chemical bonding is typically provided by the addition of silanes. Organofunctional silanes contain a silanol group on one end of the molecule which reacts with the glass surface and a variety of other functional groups on the same molecule which are capable of reacting or interacting with the matrix resin. The only other coupling agents that have found commercial application are methacrylate-chrome complexes such as Volan A®, and although technically effective the current trend is to replace these with organofunctional silanes due to environmental considerations.

Film formers and lubricants are also used in reinforcement size formulations and have a variety of purposes, these include the following: to give a desired level of strand integrity or filament to filament adhesion within a fiber bundle, abrasion resistance to prevent broken filaments and fuzz during processing, and compatibility with the matrix resin to provide desired wet out of resin into the fiber bundle. The various components of sizes as well as their function are given in [table 3](#).

TABLE 3- Glass fiber size formulation

Ingredient Category	Typical Chemistry	Role
Film Formers	Epoxies, polyesters, polyvinyl acetate, polyvinyl alcohol, polyolefins, polyurethanes, starch, etc.	Fiber protection, strand integrity, strand wetting and solubility
Lubricants	Imidazolines, alkanolamides, mineral oil, polyethylene glycol fatty acid esters, vegetable oils, etc	Fiber protection, strand integrity, lower surface friction, improve fiber forming
Emulsifiers	Ethoxylated fatty acids, ethoxylated alkylphenols, other ethylene oxide derivatives, etc.	Make film formers and lubricants water compatible
Coupling Agents	Silanes, titanates, zirconates, chrome complexes.	Provide glass to resin and film former bonding
Other Additives	Metal halides, quaternary ammonium compounds, acids, and bases.	Antistatic agents, pH control, nucleating agents

4. MECHANICAL PROPERTIES OF COMPOSITES

4.1 Introduction The mechanical performance of composites is of more interest to the component designer than are the fiber properties *per se*. We report some typical data of commercially available systems. The data shown below comparing the effect on increasing the fiber filament data was derived from both standard rovings and *ZenTron* rovings custom made for this work, in a commercial prepreg epoxy.

4.2 Filament wound pressure vessels using roving in epoxy resin. This process, either for hand-held anti-tank rocket launchers or metal lined hoop and fully wrapped pressure cylinders (e.g. types NGV2-2, NGV2-3) account for about 15-20% of high strength glass sales worldwide. Safety is of paramount importance to success and the introduction of any cost saving measures in the design or manufacture of the cylinders must not preclude this consideration. Impact performance and resistance to stress corrosion of the composite components are as important as strength in this application. Carbon fibers are poor in the first respect, and excel in the latter; the inverse is true of GRP in general. [Figure 3](#) illustrates data comparing the performance of various glass fibers, courtesy of Powertech in BC, Canada. The % of original ultimate tensile strength (UTS) vs. time after exposure to an acid solution (19% v/v Sulfuric acid). All fiber tows were impregnated with Shell Epon 8132 epoxy/Ancamide 506 hardener cured in ambient conditions for 16 hours followed by a post cure of 2 hours at 100°C.

For practical purposes, the performance of the *S-2 glass*® and RH glass rovings is identical, and as they are both members of the S-glass family as shown in [Table 1](#), any differences in corrosion performance would be unexpected, given suitable sizing. The R-glass and EC-R glass also give very similar performance in terms of % degradation over time. Although not shown as a separate line, Advantex has been measured under the same conditions and found to give the same

performance as E-CR or R glass. E glass is not considered suitable for reinforcement where acid is likely to be encountered.

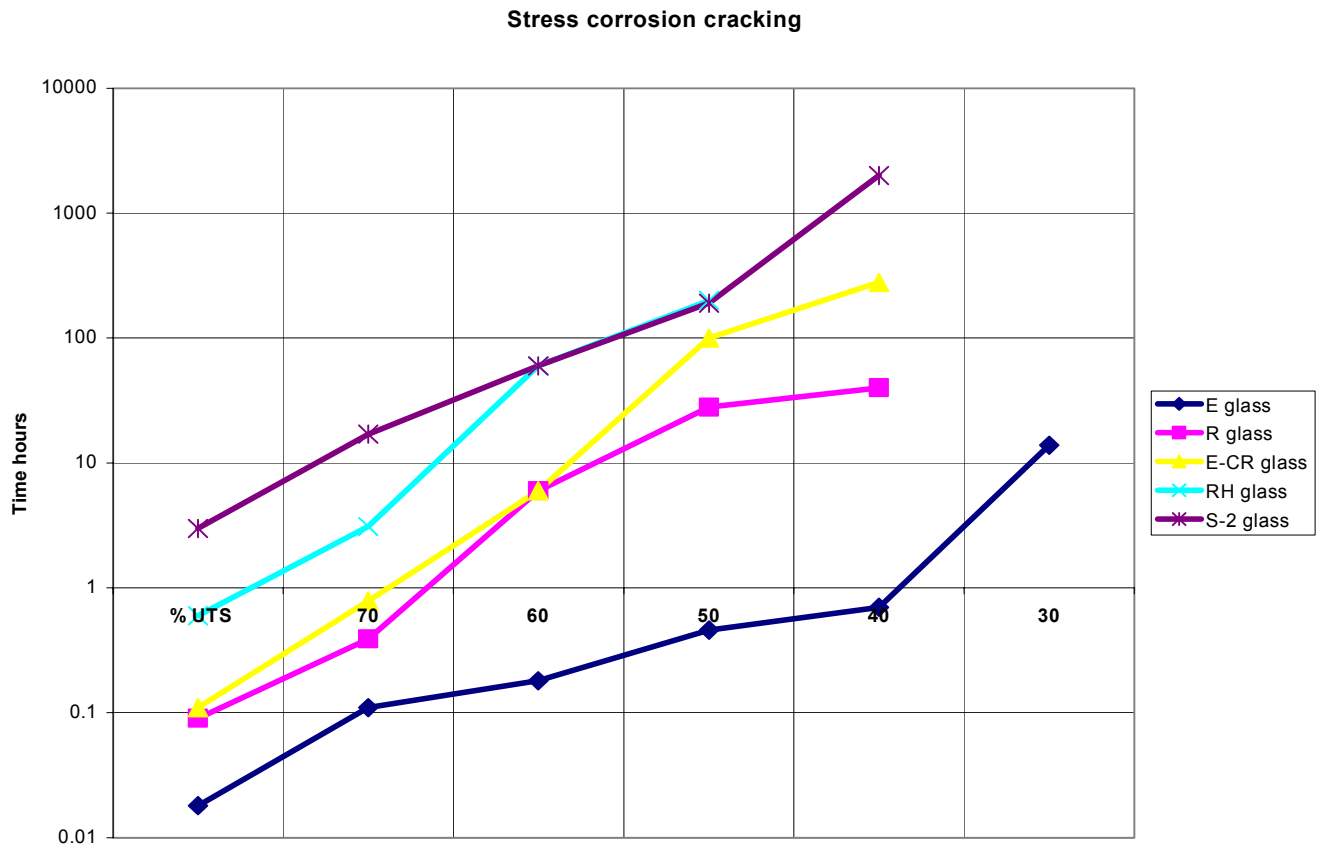


Figure 3 Plot of Stress Corrosion Degradation of GRP vs. Time

4.3 Unidirectional tape and fabrics prepregged in epoxy resin

Probably about 40% of all high strength glass produced globally in 2000 was used in UD tape, mainly epoxy resin, though some phenolic is used for aircraft in flooring and interiors in Europe.

4.3.1 933-AA-750 in Hexcel 913 epoxy resin. A screening program based on Airbus Industrie test methods was performed with *S-2 glass* and Hexcel 913 epoxy resin. This resin cures at 125°C and is used for primary aircraft structures and helicopter rotor blades. 913 has been well established since the 1970's, and a large database exists with many types of fiber. The results are summarized in [table 4](#). These data were generated in the laboratories of Tenax Fibers GmbH & Co. KG in Wuppertal, Germany.

TABLE 4- Summary of screening program results, 125°C cure epoxy UD tape

S-2 glass 933-AA-750 in Fibredux 913G-S2-5-30% UD Tape
Nominal thickness per layer (cured) 0.125mm, Fv 60%, 192 gsm glass

Test Method	Conditioning Temp/Humidity °C/ % R.H	Test Temp °C	Strength MPa	Modulus GPa
Tensile 0° prEN 2561 iss P1 Spec Type B	23/50	-55	2501	57.42
	23/50	23	2297	55.61
	23/50	80	1976	56.15
	24 hours boiling water	23	1414	
	70/85	80	1231	55.54
Compression 0° prEN 2850 A-4	23/50	-55	1912	52.78
	23/50	23	1666	54.24
	23/50	80	1227	56.52
	70/85	80	822	55.05
Compression 90° prEN 2850-B1	23/50	23	245	19.1
Flexural 0° prEN 2562 Spec Type A	23/50	23	1379	47.77
	23/50	80	1287	47.33
	100 hour boiling water	23	915	
	70/85	23	1365	46.79
	70/85	80	1056	46.64
	23°C Jet Fuel A	80	1427	47.47
70°C/Skydrol	80	1445	50.13	
Tensile ± 45° AITM 1.0001 Iss. 2	23/50 Type 1	23	119	
	Phenolic Stripper Type 2	23	97	
	Phenolic Stripper Type 3	23	102	
	23/50 Type 1	23	121	
	Non Phenolic Stripper Type 2	23	110	
	Non Phenolic Stripper Type 3	23	116	
Tensile ± 45° AITM 1.0002 Iss. 2	23/50	-55	139	7.4
	23/50	23	127	6.2
	23/50	80	98	4.1
	70/85	80	56	2.7
QI OHT	23/50	23	357	
QI Tensile	23/50	23	678	
AITM 1.0007-B		KT Factor	1.90	
Test Method	Conditioning Temp/Humidity °C/ % R.H.	Test Temp °C	Strength MPa	Modulus GPa
Bolt Bearing strength AITM 1.0009-A1 Iss. 2	23/50	23	1078	
	23/50 yield load 2%	23	865	
	70/85	80	666	
	70/85 yield load 2%	80		
AITM 1.0009-A2	23/50	23	1027	
	23/50 yield load 2%	23	827	

4.3.2 E glass and S-2 glass fabrics in Hexcel 913 epoxy resin. In order to provide a back to back comparison, a smaller program was also conducted in the same resin using both 6781 and 7581 style fabrics. Both are 8 harness satin weaves, 295 gsm, using 9µ 66 tex twisted glass

yarns. The former uses SCG75 1/0 1.0z 636 *S-2 glass*[®] yarn, the latter ECG75 1/0 1.0z E glass yarn. The strength results are shown in [figure 4](#). The improvements in flexural and tensile strength obtained in using S-glass are a more modest 30-35%, notably less than in the case of UD tape. There was only about 7-10% increase in moduli, and no change in compressive performance. The most plausible explanation is that the “crimp” effect or the non-alignment of filaments in the fabric negates the higher intrinsic performance of the S-glass. Improved flame and heat resistance, and impact performance would be more compelling drivers for using *S-2 glass*[®] bi-axial fabrics.

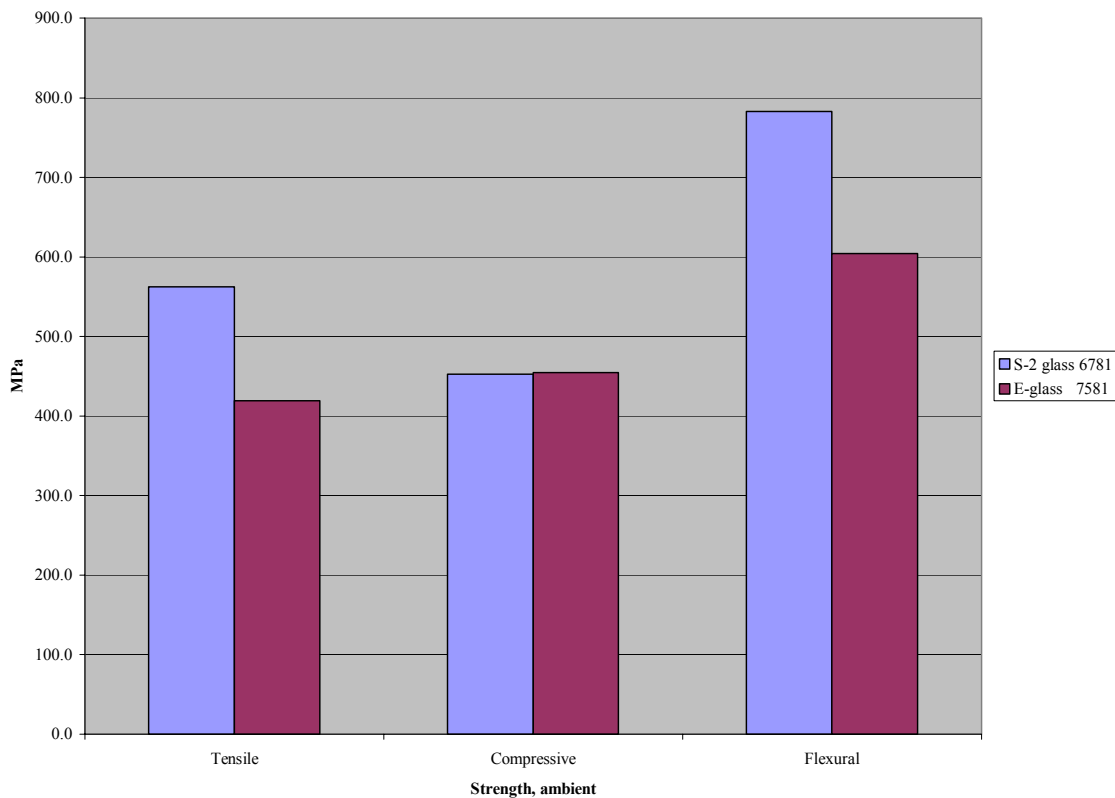


Figure 4 Comparison of *S-2 glass*[®] and E glass 8HS fabric reinforcements in epoxy resin.

4.3 Polyester based SMC. Although thousands of tonnes per year of polyester based sheet molding compound are used by the automotive industry and others, high strength glass has never been used in commercial volumes. [Figure 5](#) shows a comparison of the performance of E-glass and S-2 glass structural SMC panels, based on 65% w/w glass content, using randomly orientated fibers of either 25mm or 50mm length.

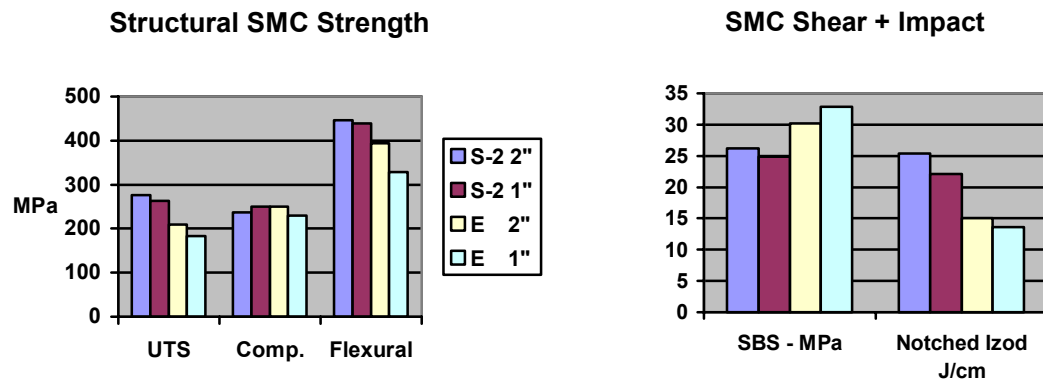


Figure 5 Structural SMC (Sheet Molding Compound) properties.

30cm x 46cm panels were molded using a constant resin formulation based on AOC E987 polyester resin and constant molding conditions. The *S-2 glass* was 365-AA-250 9 μ 2000tex roving. The E glass was Owens Corning 973C roving. Testing was to ASTM D639, D695, D790, D5379 and D256 for the tensile, compressive strength, flexural, shear and impact strength respectively. The conclusions drawn are as follows:

- The longer fibers improve tensile, flexural, and impact but not compressive strengths.
- The S-glass gave about 32-45% greater tensile strength than E-glass, and about 13-30% greater flexural performance. In the case of flexural performance it is clearly more cost effective to increase the fiber length than to change the glass chemistry.
- The S-glass improved the impact performance by over 60%, as measured by notched Izod.
- The SBS data indicate that the 365 size may not give such good bonding to Polyester as the 973C, and further optimisation may be required.

4.4 Influence of filament diameter. There are two ways in which this may influence the performance of a composite. The strength of the filament itself may be affected. Hillermeier (3) found that a decrease in fiber diameter increased the tensile strength and in some cases the moduli of polyester, PAN, rayon and E glass fibers. In the case of glass, the tensile strength increased by a factor of 1.5 as the diameter was reduced from 13 to 3.8 μ m. Other early work on E glass (4) also indicated that the tensile strength of glass fibers varies inversely to the diameter. On the other hand there are reports (5-7) that under certain circumstances at least the tensile strength of E-glass fibers may be independent of filament diameter. Pabler and Bruckner (6) found that several drawing factors in the fiber manufacturing process could influence the strength of fibers, and Thomas (6) reports no change in tensile strength of E-glass over the filament diameter range of 5 to 20 microns, under laboratory conditions. The most likely explanation is that the strength is dependent on surface flaws, and under industrial manufacturing conditions finer filament diameters will lead to greater strength.

The second way in which filament diameter will influence the mechanical properties of the composite is by altering the geometry, such that the surface to volume ratio increases as the fiber diameter decreases, assuming constant fiber volume fraction. Thus a greater surface area is available for bonding at the interface. The superior compression performance of Boron fibers

(typically 100 μm diameter) compared to both glass and carbon is often attributed to the greater buckling resistance of a fatter filament.

This section of the present study was initiated as part of a program to increase compression strength in epoxy composites used in rotorcraft prop-rotor retention yokes and prop-rotor blades. The baseline system used 449-AA-250 *S-2 glass* roving in Cytec-Fiberite E773 epoxy resin. The roving is a conventional roving assembled from 30 ends of 66 tex slivers, sized with a standard epoxy compatible silane blend. Standard *ZenTron* 721B-AB-750 roving of 13 μm filament diameter was plied 3 times to give the same overall yield as the *S-2 glass*, and special production runs of 18 μm and 25 μm *ZenTron*® were made using the same 721B size chemistry.

The results are summarized in [table 5](#), and [figures 6 and 7](#).

TABLE 5- Cytec-Fiberite and BHTI mechanical data, normalized to 60% F_v

Roving	Units	9 μ 2000tex 30 end		13 μ 2000 tex 3 end		18 μ 1320 tex 1 end		25 μ 2000 tex 1 end	
			CV %		CV %		CV %		CV %
0° Tensile Strength	MPa	1517	10.8	1207	5.4	1662	4.0	1531	5.0
0° Tensile Modulus	GPa	49	1.6	47	1.1	49	1.7	49	1.2
0° Tens. Strain	%	3.35	16.1	2.6	5.0	3.37	4.9	3.11	5.9
0° Comp. Strength	MPa	1154	12.7	1455	9.2	1503	5.6	1641	4.5
0° Comp. Modulus	GPa	49	4.4	47	2.5	50	1.9	48	2.9
ILSS	MPa	72	1.9	66	1.6	65	2.3	68	2.9
Fiber volume	%			60.63		58.77		59.51	

There appears to be a useful increase in compression strength with filament diameter. The compression and tensile moduli are little affected, as one would expect. The surprisingly good tensile performance of the 18 μm and 25 μm filaments is more difficult to explain. It is possible that the 721B size gives better results than 449 in the E773 epoxy resin although this was not obvious from the short beam ILSS; also single end rovings may allow better filament alignment than multi end rovings. More work is needed to build on these preliminary results.

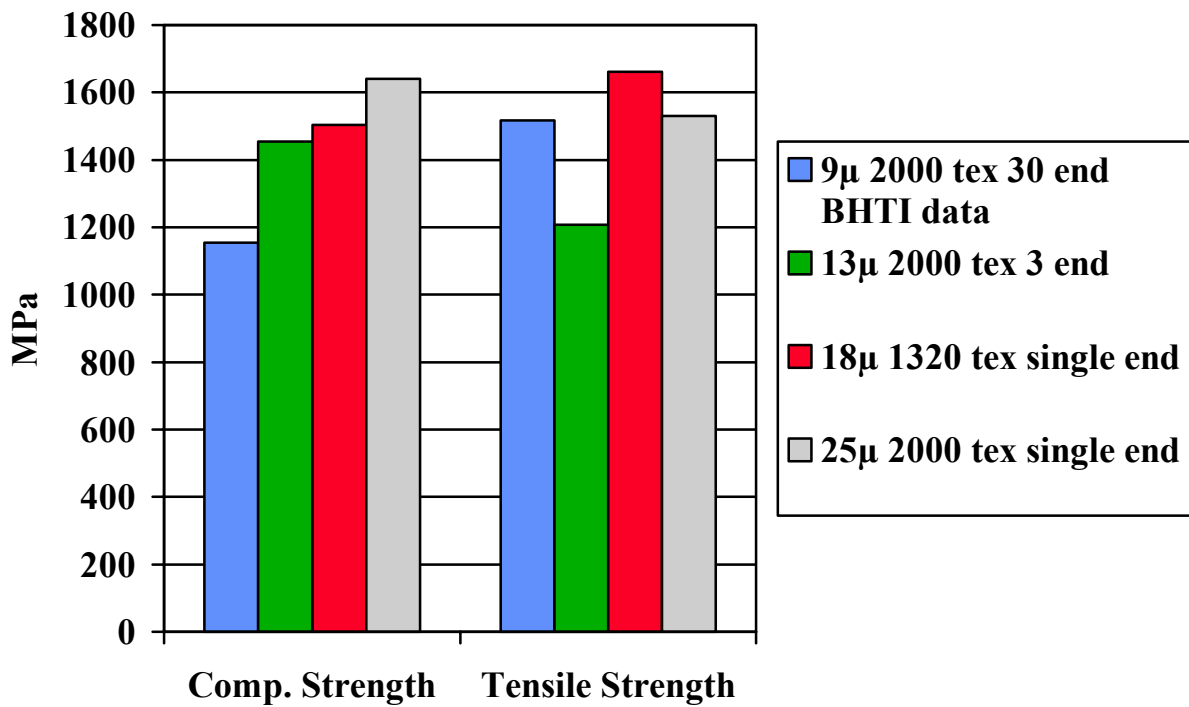


Figure 6 Effect of roving filament diameter on UD tape strength, normalized to 60% Fv.

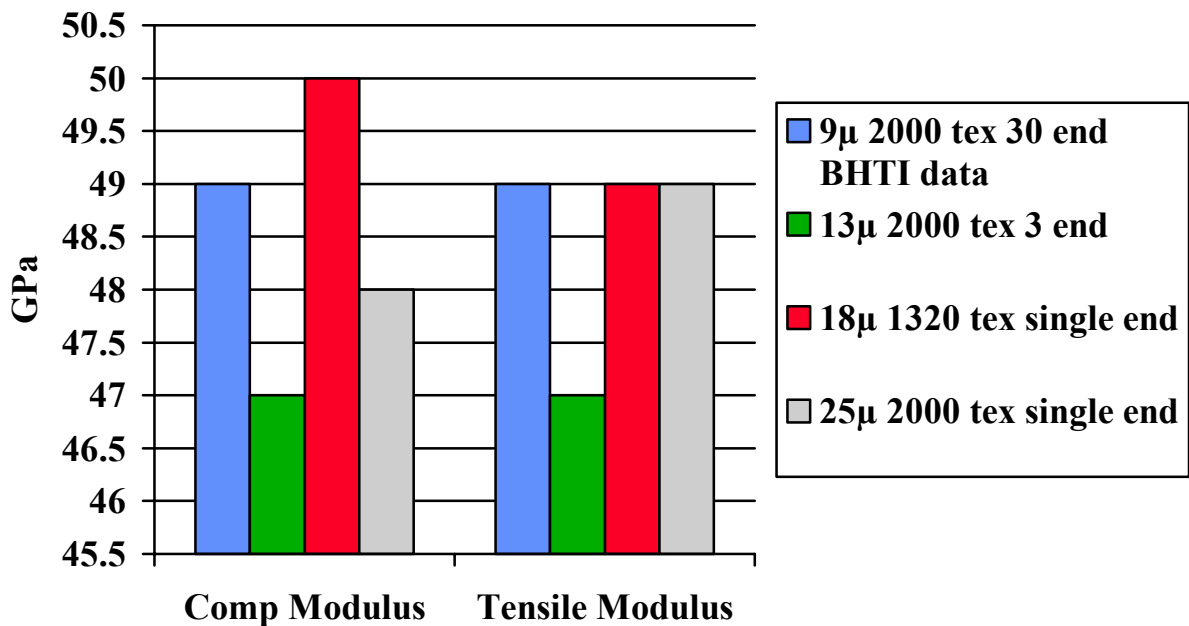


Figure 7 Effect of roving filament diameter on UD tape modulus, normalized to 60% Fv.

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BIOGRAPHIES

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