

TRANSPARENT COMPOSITES BASED ON A THERMOSET RESIN REINFORCED WITH S-2 GLASS[®] FIBER

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ABSTRACT

Fiber reinforced plastics or composites are known for their exceptional strength to weight ratio, far outperforming engineering plastics exemplified by acrylics and polycarbonates. An interesting feature of these engineering plastics is their transparency, which allows their use in applications where transmittance of light or see-through visibility is important. A transparent composite therefore expands the potential application of composites to uses where both mechanical strength and transmittance of light are desirable.

We hereby present a transparent composite system that achieves such features. This composite consists of thermoset resin reinforced with S-2 Glass[®] fiber. The tensile and flexural properties of unidirectional (UD) laminates produced from drum wound prepreg were determined and compared to a baseline resin system containing the same fiber. Comparable tensile but superior flexural properties were observed in the transparent system. The light transmittance of the composites was assessed relative to engineering plastics as well as baseline thermoset composites. The described composite system therefore offers previously unavailable benefits compared to non-transparent composites, including means of facile non-destructive visual inspection, in-situ process evaluation, as well as applicability of composites for load-bearing applications requiring the transmittance of light.

Keywords: Transparent Composites, S-2 Glass[®] Fiber

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1. INTRODUCTION

Fiber reinforced plastics or composites are known for their exceptional strength to weight ratio, far outperforming engineering plastics exemplified by acrylics and polycarbonates¹. Composites have steadily increased their presence in applications that engineering plastics cannot achieve due to their structural limitations. Due to the presence of fibers, however, composites are not transparent, and currently do not find applications where engineering plastics are used for their see-through visibility, such as in bullet proof glass. A transparent composite therefore offers the opportunity to provide a high structural performance material in applications requiring see-through visibility².

A novel transparent composite based on a thermoset resin reinforced with *S-2 Glass* fiber is introduced in this paper. See-through visibility of the transparent composite is demonstrated through light transmittance measurements and is compared with both a base-line epoxy composite as well as acrylic and polycarbonate plastics. Mechanical properties of the transparent composite

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are further compared with a base-line epoxy resin reinforced with the same *S-2 Glass* fiber to demonstrate retention of structural performance. Collectively, high light transmittance and retention of mechanical properties are demonstrated, opening the avenue for new applications of composite materials that feature transparency as well as mechanical properties, of which high strength, stiffness, and toughness are noteworthy hallmarks of composites reinforced with *S-2 Glass* fiber.³ Finally, examination of failed specimens from mechanical property measurements point to the possibility of facile non-destructive inspection (NDI) in transparent composites that relies only on visual inspection, which may further expand their use in applications where rapid yet accurate NDI is critical.

2. EXPERIMENTATION

2.1 Materials

The *S-2 Glass* roving known as ZenTron9™ (758-AC-1400) and E-Glass assembled roving 517-BA-750 were produced by AGY. The E-Glass roving Hybon 2026 was produced by Nippon Electric Glass. All rovings were surface treated with an epoxy compatible sizing during production. Kaneka proprietary resin 19-04-12 and the associated curative were produced by Kaneka Aerospace. Hexion supplied the epoxy resin Epon 828 and Lindride 6 anhydride curing agent was supplied by Lindau Chemicals Inc.

2.2 Resin Properties of 19-04-12

The viscosity of the mixed resin was measured using a Brookfield Ametek DV-1 viscometer using spindle number 21 at 100rpm in accordance with ASTM D-2393. The glass transition temperature (T_g) was measured through a TA Instruments Q800 dynamic mechanical analyzer in accordance with ASTM D-7028. The flexural strength and flexural modulus of the resin cured in accordance with Table 1 were measured using an Instron test frame with a 50kN (11,000 lb) load capacity in accordance with ASTM D-790. The cure profile used for curing the resin is summarized below in Table 1.

Table 1. Cure Profile for Resin Casting used to measure T_g

Step	Details
1	Ramp to 71°C at 3.3°C/min
2	Dwell at 71°C for 90 min
3	Ramp to 121°C at 3.3°C/min
4	Dwell at 121°C for 90 min
5	Cool slowly to ambient temperature

2.3 Fabrication of Unidirectional (UD) Laminates

Filament winding was completed using a *McClean Anderson* drum winder, Model *WLH-1-2-2M-FLEX*. Wet winding the roving through a bath of liquid resin followed by lay-up, cure and consolidation gave the UD laminates. The winding operation was conducted at a bandwidth setting appropriate for each fiberglass roving based on their associated yield or tex and a winding speed of 15 rpm with 6.7 N (1.5 lb) of tension applied to the strand. All UD laminates were constructed

of 15.2 cm (6 in.) wide plies with the entire composite thickness developed on the drum mandrel surface by laying down from 3 to 8 plies one on top of another in a circumference equal to that of the mandrel 1.8 m (74 in). After completion of the winding operation, UD preforms 30.5 cm (12.0 in.) long were cut from the mandrel and laid-up on an aluminum mold tool. A caul plate was placed on the surface and the entire assembly positioned within the confines of a Wabash platen press and subjected to a hydrostatic pressure [20.7 kPa, (3 psi)] sufficient to consolidate the laminate to a target thickness of 1 mm (0.04 in). The thermoset resins were then subjected to a cure cycle as prescribed by the manufacturer.

2.4 Mechanical Properties

Mechanical properties were determined in the tension and compression mode using an Instron universal test machine with a 133 kN (30,000 lb) load capacity. The test rig includes an Epsilon Technology Corp. clip-on extensometer having a gage length of 25.4 mm (1.0 in.). Tensile and flexural strength and modulus were determined according to ASTM D-3039 and ASTM D-7264; respectively. Interlaminar short-beam shear strength was determined according to ASTM D-2344 at a span-to-thickness ratio of 4:1 on specimens machined from Naval Ordnance Laboratory (NOL) rings that were produced via circumferentially winding the fiberglass rovings through a bath of liquid resin onto a NOL ring fixture. Impregnated roving strand tensile strength and modulus was determined by ASTM D-2343. Composite damage resistance was evaluated via quasi-static indentation using a blunt-ended indenter with a diameter of 7.6 mm (0.30 in.) according to ASTM D-6264.

2.5 Physical Properties

Density was determined according to ASTM D-792. Fiber, resin and void volume fractions were determined according to ASTM D-2584.

2.6 Transmittance Measurements

Optical measurements were measured at a USA solar manufacturer, taken with a PerkinElmer Lambda 1050 spectrophotometer using a PerkinElmer UL270 integrating sphere accessory. To measure optical transmission, the Lambda 1050 spectrophotometer shines a beam of monochromatic (single-wavelength) light at a sample, then uses a detector to measure the amount of energy that passes through the sample. This energy is compared to unblocked and completely blocked baselines to determine the light transmission percentage. This measurement is repeated across a range of wavelengths to form a spectrum of transmission percentage vs. wavelength. For these samples, data was collected from 250-1500nm at intervals of 2nm.

The Lambda 1050 uses one of two light sources, depending on the measurement wavelength. A deuterium lamp is used at wavelengths shorter than 319.20nm, and a tungsten lamp is used at all wavelengths longer than 319.20nm. The instrument also uses one of two monochromator gratings that are switched at 800.8nm. The monochromatic light is passed through a depolarizer before entering the accessory in order to reduce polarization effects due to the internal optics.

The UL270 accessory consists of a 270mm diameter integrating sphere and a pair of detectors. The integrating sphere allows for the accurate collection of diffuse light in both transmission and reflection modes by using a highly reflective material to coat the interior of a sphere where the detectors are located. The sample is placed over an opening into the sphere, and light that passes

through the sample is collected within. For these samples, a 50mm diameter circular opening was used. The two detectors in the sphere are used for different wavelengths: a photomultiplier tube (PMT) for wavelengths shorter than 890.80nm, and a 3-stage Peltier cooled InGaAs detector for wavelengths longer than 890.80nm.

3. RESULTS

3.1 Resin Properties of Kaneka Composite Resin 19-04-12

Key characteristics of Kaneka 19-04-12 are summarized in Table 1. Kaneka 19-04-12 is among a family of resin systems within Kaneka that enables transparency in composites. The mixed viscosity of 19-04-12 exhibits a value of 480cP at 25°C. The low viscosity makes this resin system amenable to a number of different composite manufacturing processes at ambient temperature, including vacuum assisted resin transfer molding (VaRTM) and filament winding. Resin castings of 19-04-12 were manufactured following the cure profile in Table 1 to make specimens for Tg and flexural property measurements. A thick resin casting after cure is shown in Figure 1 and displays water white clarity with no yellowing. The Tg measured from the DMA plot yielded a value of 144°C, which is in the range of expected values for this system. The flexural modulus, and flexural strength, exhibited values of 3310 MPa and 96 MPa respectively.

Table 2. Resin Characteristics of 19-04-12

Property	Value
Mixed Viscosity, cP	480
Tg, °C	144
Flex Strength, MPa	96
Flex Modulus, MPa	3310



Figure 1. Resin casting of 19-04-12 after cure.

3.2 Composite Laminate Fabrication

The glass fiber resin combinations described within Table 2 were used to fabricate UD composites. Wet filament winding glass fiber rovings through a bath of liquid resin and onto the surface of a drum mandrel followed by lay-up, resin cure and laminate consolidation allows for their convenient conversion into UD composites, Figure 2. This method has the following advantages.

First, conversion of the roving into a UD prepreg or a woven fabric format followed by resin pre-impregnation is unnecessary. Second, any future developmental rovings can be easily and rapidly screened and compared to the composite properties database that is currently being generated using this fabrication technique.

Table 3. The UD composites fabricated and evaluated during this development program.

Glass Fiber and Resin Combinations for UD Composites	Fiber Diameter (μm)	Roving Tex (g/km)	Fiber Density (g/cm ³)	Impregnated Roving Tensile Strength (MPa)	Impregnated Roving Tensile Modulus (GPa)
ZenTron9™ S-2 Glass Roving *758-AC-1400/Kaneka 19-04-12	9	355	2.46	3623	90
ZenTron9™ S-2 Glass Roving *758-AC-1400/Epon828/Lindride 6	9	355	2.46		
E-Glass Roving **517-BA-750/Kaneka 19-04-12	9	662	2.58	2250	76
E-Glass Roving Hybon 2026 250-16/Epon828/Lindride 6 Anhydride	16	2400	2.58	2058	73
*(758-AC-1400) 758 = Epoxy compatible sizing, AC = Single end roving with 9 micron filament diameter, 1400 = yield in yards/pound.					
**(517-BA-750) 517 = Epoxy compatible sizing, BA = Assembled roving with 9 micron filament diameter, 750 = yield in yards/pound.					



Figure 2. Filament winding of S-2 Glass roving and Kaneka 19-04-12 followed by cure and consolidation gives the clear UD composite (right).

The clear composite shown in Figure 1 was achieved by giving special attention to the fabrication strategy in order minimize or eliminate surface and bulk laminate defects.⁴ Moreover, fiber surface treatments need to resist yellowing or darkening over the course of the resin cure cycle. Other demonstration articles were produced (Figure 3) by filament winding the ZenTron9™ roving and 19-04-12 onto the surface of a mirrored #8 finish aluminum alloy mandrel.



Figure 3. Demonstration articles filament wound on an aluminum alloy mandrel. Tube dimensions OD = 35 mm, wall thickness = 1.6 mm.

The importance of the details associated with the fabrication strategy as it relates to the production of a composite that is clear and colorless can be seen in Figure 4. NOL rings were fabricated via wet filament winding and are shown from viewing left to right as *S-2 Glass* roving 758-AC-1400 and 19-04-12, *S-2 Glass*/ Epon 828/Lindride 6 epoxy resin and E-glass/Epon 828/Lindride 6 epoxy resin.

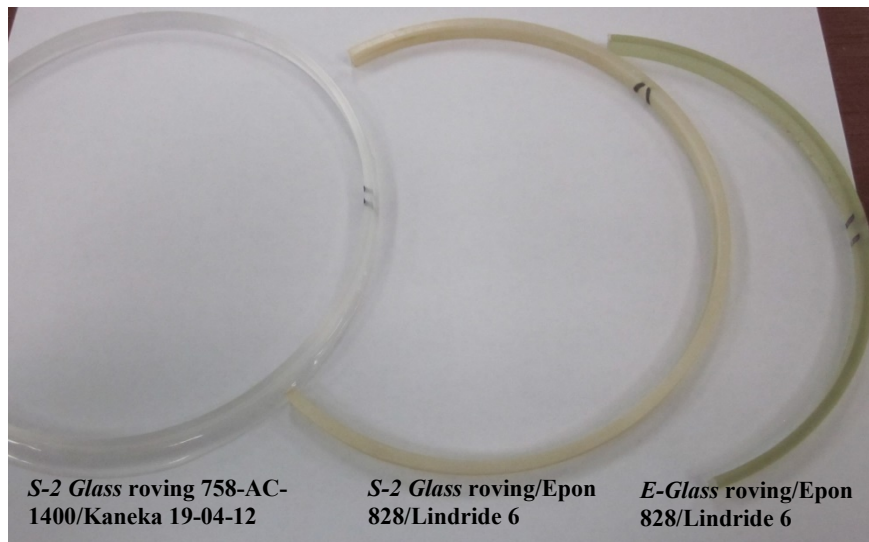


Figure 4. Filament wound NOL rings.

The combination of *S-2 Glass* fiber and Kaneka 19-04-12 resin affords a colorless, clear composite. The *S-2 Glass* fiber and Epon 828 combination results in an opaque composite. The presence of iron oxide in E-Glass gives the composite a green tint.

3.3 Composite Mechanical Properties

Clear and colorless glass fiber reinforced composites afford the advantage of significantly higher mechanical performance relative to traditional transparent engineering thermoplastics such as

polycarbonate or poly(methyl methacrylate), [acrylic] that exhibit a tensile strength of 69 and 72 MPa, respectively.⁵ Glass ribbons² have been developed to overcome the dispersion of light that is inherent to circular fibers but their composites suffer from reduced strength.^{4,6} For example, Velez et al., report a glass ribbon tensile strength of 900 MPa and a composite of thermosetting resin strength of 770 MPa.⁴ The tensile strength as determined on UD *S-2 Glass* fiber reinforced Kaneka 19-04-12 is 1425 MPa; about two times greater than that reported for glass ribbon reinforced plastics, see Figure 4

3.3.1 Tensile Properties

Tensile strength and modulus of the UD composites were determined by standard techniques and are presented in Figure 4. All tensile properties were normalized to a common fiber volume fraction as in equation 1:

$$\text{Normalized Property} = \text{Measured Value} * (\text{Normalizing } V_f / \text{Measured } V_f) \quad \text{Eq. 1}$$

As shown in Figure 4 the tensile properties of the UD composite containing the recently developed thermoset resin 19-04-12 from Kaneka are essentially equivalent to that of a traditional epoxy system (Epon 828/Lindride 6) reinforced with ZenTron9™ *S-2 Glass* roving 758-AC-1400. The tensile properties of two transparent thermoplastic materials are included for comparison. The increased tensile strength and modulus of a transparent *S-2 Glass* fiber reinforced thermoset resin provides a clear competitive advantage over transparent engineering thermoplastics.

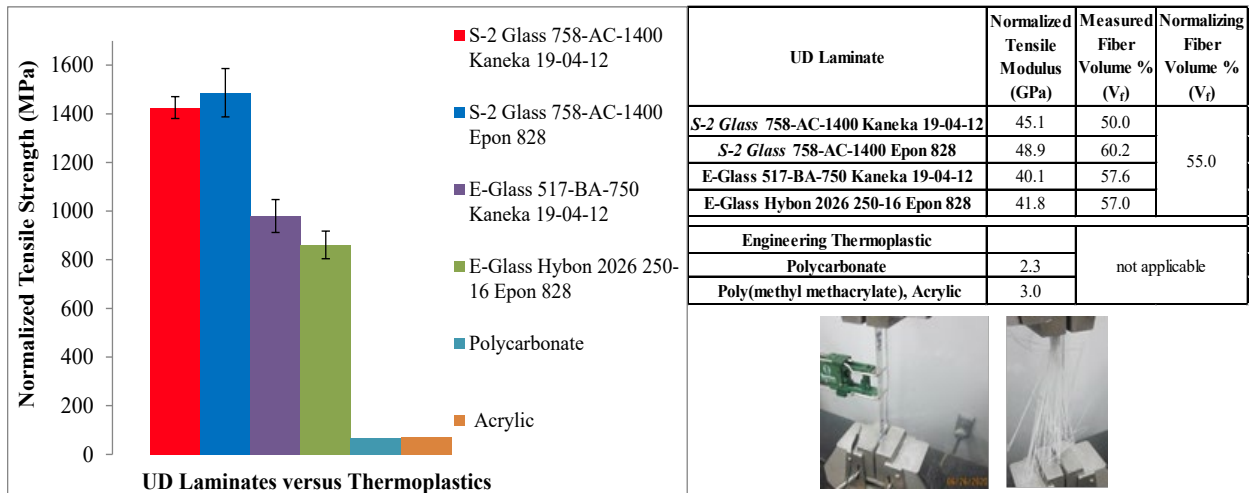


Figure 5. Tensile properties of UD laminates versus polycarbonate and acrylic

3.3.2 Flexural Properties

Flexural strength and modulus of the UD composites were determined by standard techniques in the 3-point bending mode at a span-to-thickness ratio of 32:1 and are shown in Figure 5.

The positive influence of silane coupling agents on the flexural strength of glass fiber reinforced plastics has been known since the 1940's⁷ when researchers found that certain silanes significantly increased the composite initial strength. The flexural strength of the *S-2 Glass* fiber reinforced Kaneka 19-04-12 shown in Figure 6 is indicative of good fiber to resin compatibility and is at the very least equivalent to the strength shown for the traditional epoxy resin system. The flexural

properties of two transparent thermoplastic materials are included for comparison and reveal a potential benefit associated with transparent *S-2 Glass* fiber reinforced Kaneka 19-04-12. Observation of the specimens after break in Figure 6 depicts a white band generated in the region of the failed section as a consequence of resin/fiber failure at the breakage point. This characteristic of the clear composite to whiten at local points of failure or degradation could potentially be used to the advantage of the end-user or manufacturer in assessing the quality and performance of the composite in a non-destructive visual manner during usage and manufacturer, and would be of interest to follow with further investigations.

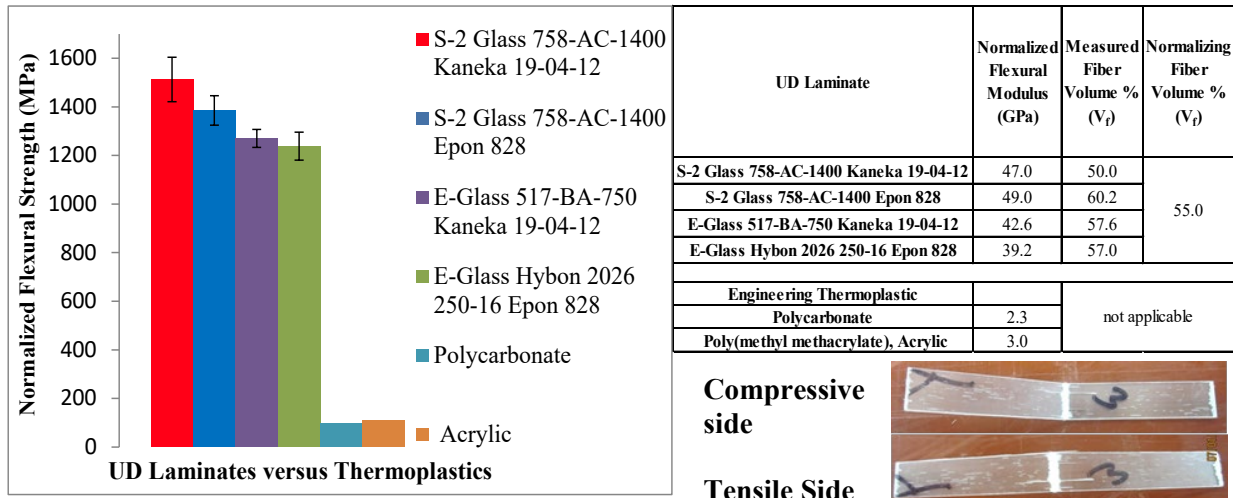


Figure 6. Flexural properties of UD laminates versus polycarbonate and acrylic

3.3.3 Interlaminar Short-Beam Shear Strength

The interlaminar shear strength, evaluated by the short-beam method is routinely used to verify the compatibility of a glass fiber surface treatment with a new resin system. For many years the Epon 828/Lindride 6 resin has been utilized to evaluate epoxy compatible sizings at AGY. Figure 7 presents short-beam shear strength data that show the Kaneka 19-04-12 is comparable to that of other fiber surface treatments evaluated in the Epon 828/Lindride 6 epoxy resin.

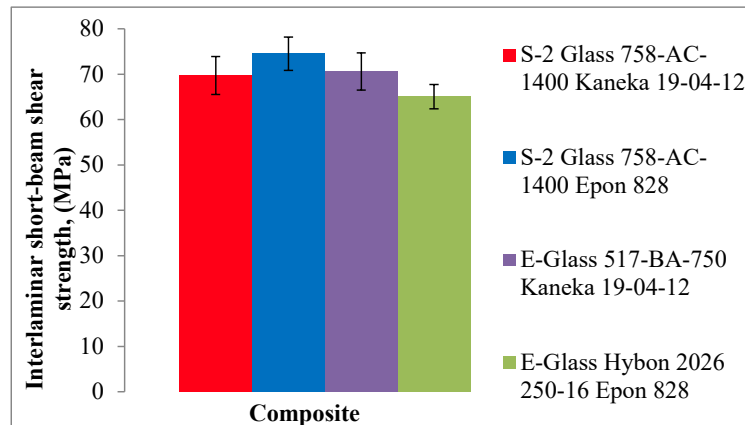


Figure 7. Interlaminar short-beam shear strength.

3.3.4 Damage Resistance by Quasi-Static Indentation

Damage resistance was evaluated by ASTM D-6264⁸ by pressing a flat ended indenter with a diameter of 7.6 mm through the thickness of the specimen, Figure 8.⁹ The support span is a circular cutout with a diameter of 25 mm.

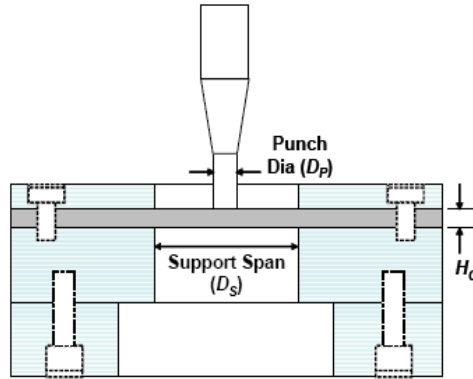


Figure 8. Schematic of quasi-static indentation test set-up.⁹

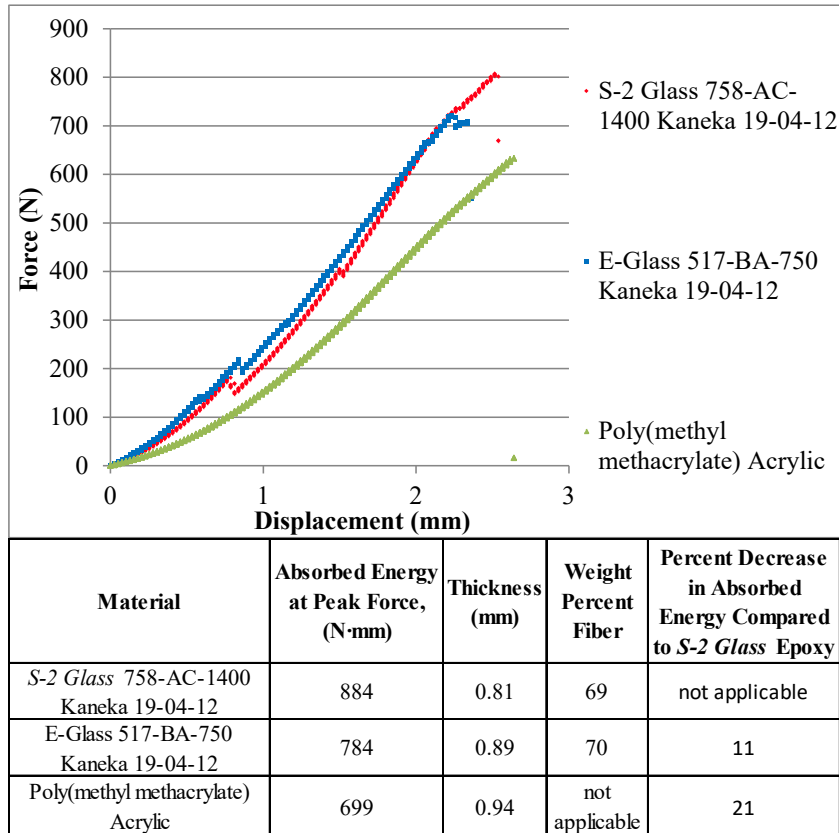


Figure 9. Quasi-static indentation test results.

Figure 9 presents the force-displacement curves for UD composites of *S-2 Glass* fiber and E-glass reinforced Kaneka 19-04-12 as compared to a sheet of poly(methyl methacrylate), acrylic. The total energy absorbed for the *S-2 Glass* fiber reinforced 19-04-12 is about 11 percent higher than the E-Glass/epoxy and 21 percent greater than the acrylic sheet under these test conditions.

3.4 Light Transmittance Studies

Light transmittance measurements over the wavelength range 250-1500 nm were completed on the thermoplastic and composite specimens shown in Table 4. All composites were 100 percent UD and produced as previously described in section 2.3.

Figure 10 presents the percent light transmission as a function of wavelength for all specimens shown in Table 4.

Table 4. Thermoplastic and composite specimens subjected to light transmission measurements.

Thermoplastic and Composite Specimens	Thickness (mm)	Volume % Fiber
Acrylic	1.02	na*
Polycarbonate	0.76	na
<i>S-2 Glass</i> 758-AC-1400 Kaneka 19-04-12	0.76	44
<i>S-2 Glass</i> 758-AC-1400 Kaneka 19-04-12	1.02	31
<i>S-2 Glass</i> 758-AC-1400 Kaneka 19-04-12	0.89	43
E-Glass 517-BA-750 Epon 828/Lindride 6 Epoxy	0.76	50
E-Glass 517-BA-750 Kaneka 19-04-12	0.89	53

*na = not applicable

All specimens were found to be mostly opaque in the ultraviolet (UV) region, 250-400 nm. The *S-2 Glass* fiber reinforced Kaneka 19-04-12 specimens were found to transmit nearly the same amount of visible light over the range from 400 to 700 nm as polycarbonate with two of the *S-2 glass* fiber 19-04-12 specimens displaying an average total light transmission of greater than 90 percent over the range from 450 to 1050 nm. The reduced light transmission of E-Glass reinforced specimens is likely due to the total iron [Fe_{tot}] concentration present in these fibers.¹⁰

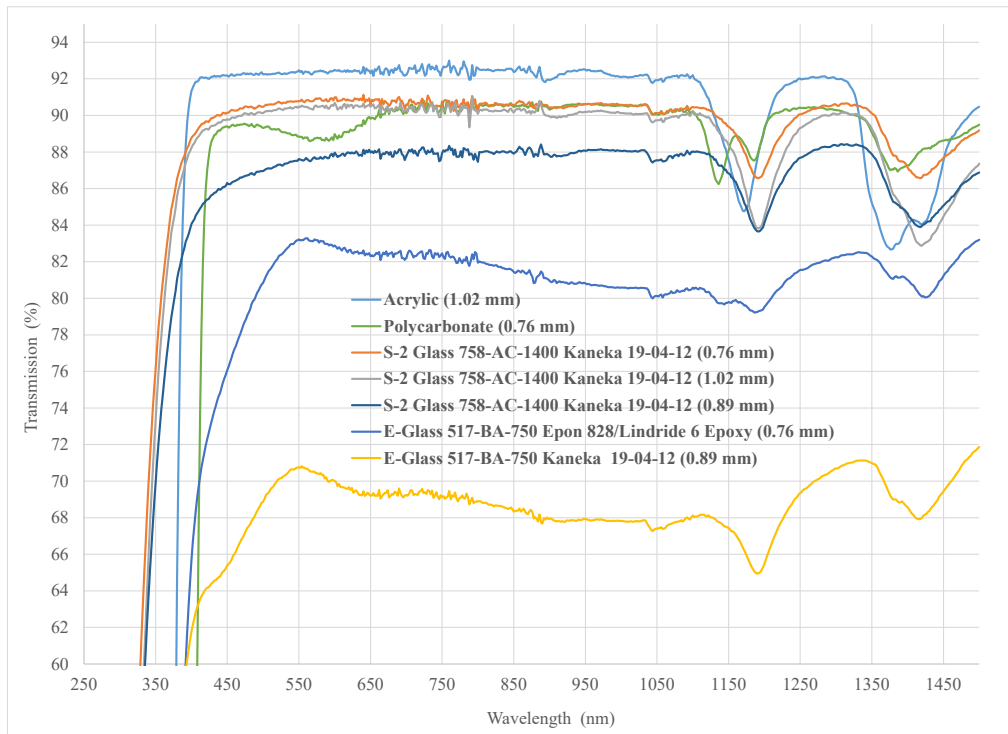


Figure 10. Light transmission spectra.

4. CONCLUSIONS

In conclusion, a novel transparent composite based on a thermoset resin reinforced with *S-2 Glass* fiber was introduced in this paper. Unidirectional laminates produced from *S-2 Glass* fiber reinforced Kaneka 19-04-12 provided a clear laminate that exhibited comparable tensile and flexural strengths to the baseline *S-2 Glass* fiber reinforced Epon828/Lindride 6 system. Short beam shear strengths measured on NOL rings also showed similar values for the two thermoset systems. Damage resistance was also measured and found to exhibit a 21% greater total energy absorption relative to polycarbonate. Light transmission studies illustrated the clarity of the composite relative to E-glass reinforced systems, where an average light transmittance of approximately 90% over the visible spectrum was shown to be comparable to polycarbonate of the same thickness.

While exhibiting comparable light transmittance performance to acrylic and polycarbonate, the *S-2 Glass* fiber reinforced 19-04-12 composite displays an order of magnitude higher tensile and flexural strengths. The *S-2 Glass* fiber reinforced 19-04-12 composite, therefore, presents an interesting system that can offer vastly superior structural load capabilities in applications where thermoplastics are used for their see-through visibility. In addition, the clarity of the composite also presents the potential for facile non-destructive analysis while in use, as well as an additional, simple means of quality control of the composite during manufacture. The damage resistance of the *S-2 Glass* reinforced 19-04-12 may also present the possibility of use in impact resistant applications, where *S-2 Glass* fiber reinforced thermoset composites are used today.

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