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GLASS FIBER REINFORCED

POLYCARBONATE COMPOSITES

by

WEN CHING HU, 1961-

A THESIS

Presented to the Faculty of the Graduate School of the

UNIVERSITY OF MISSOURI-ROLLA

In partial Fulfillment of the Requirements for the Degree

MASTER OF SCIENCE IN CERAMIC ENGINEERING

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Approved by

Delbert (Advisor)

ABSTRACT

The purpose of this work was to develop a transparent glass fiber reinforced polymer composite material which is superior to the transparent unreinforced thermoplastic in mechanical properties but similar in optical clarity. Polycarbonate matrix composite specimens containing 0 to 40 wt% SK-12 glass fiber were studied, with mechanical and optical properties determined as a function of glass fiber content. The mechanical properties - flexural strength and elastic modulus increased with increasing glass fiber content. The optical properties - transmission and transparency decreased with increasing glass fiber content. The factors which influence the mechanical and optical properties were investigated. I would like to express my thanks to Dr. Delbert E. Day for his guidance and support. I would also like to thank Dr. Robert E. Moore and Dr. James O. Stoffer for their instructions.

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I. INTRODUCTION

Glass fiber reinforced plastics¹ have been used as engineering materials for more than thirty years with the advantages of low weight and high strength. Transparent plastics³ also have been commercialized for more than twenty years and have partially replaced glasses in the applications of transparent materials with the advantages of low weight and high toughness. But transparent plastics lack the strong and hard characteristics of glasses which are usually required under critical conditions. One method of making transparent materials which are stronger than plastics and less brittle than glasses is the development of transparent glass fiber reinforced plastics. PMMA, polycarbonate, polyvinyl butyral, cellulose acetate and polystyrene are several commercially available transparent plastics⁴. Polycarbonate was selected as the matrix in this study for its high strength and toughness.

Two methods of producing polycarbonate⁴ are: Bisphenol A reaction with phosgene (COCl₂) in pyridine:



Transesterfication of diphenol carbonate with bisphenol carbonate in acid or basic catalyst at 260°C.





+ 2× <___он

Disadvantages of this process are the need for apparatus permitting the use of high vacuum and the limitation on molecular weight imposed by the high melt viscosity which leads to low mechanical strength.

The more convenient way of making composite specimens is to mix the matrix materials and glass fiber directly. For good uniformity, it's necessary to reduce the viscosity of the polymer by either increasing the temperature or dissolving the polymer in a solvent. Both processes were investigated in this study.

The most important properties to be considered in evaluating composite materials are mechanical properties which are strongly dependent on the interfacial bonding strength. Definite physical adhesion or a true chemical bond between the polycarbonate and glass fiber is desirable for general applications. In the case where no interfacial bonding exists between the matrix and the glass fiber, the fiber will weaken the composite since it functions only as a diluent. Some commercial chopped fiber reinforced polycarbonates are available in pellet form^{3,4}. The most outstanding improvement in reinforced polycarbonate (40 wt% glass fiber) over pure polycarbonate is that flexural and tensile strengths increase 100%.

Optical properties are also important for the purpose of replacing transparent glass. The transparency and optical transmission of a composite specimen are essentially dependent on the refractive index difference⁵ between the polymer and glass fiber, the interfacial bonding between the polymer and the glass fiber, the orientation of the glass fiber, and the interfacial area.

The interface between dissimilar phases can be modified by applying a silane coupling agent^{2,6,7} to improve the interfacial bonding and upgrade the mechanical and optical properties. Good adhesion of the polymer to the fiber is critical to the mechanical properties, since stress is transferred in shear through the interface. Optical properties will be permanently improved by good bonding at the interface which will eliminate the air gaps between matrix and glass fiber.

Composite specimens with glass fiber content varying

from 0 to 40 wt% were studied. The mechanical and optical properties were measured and the interfacial bonding was investigated.

II. EXPERIMENTAL PROCEDURES

A. <u>SPECIMEN PREPARATION</u>

1. <u>Glass fiber preparation</u>: Commercially available Schott SK-12* optical glass which has the same refractive index at 589 nm as polycarbonate was selected for preparing the glass fiber. The optical, mechanical and thermal properties of polycarbonate and SK-12 glass are listed in Table 1.

During the glass fiber pulling procedure, glass fiber was pulled at different glass melting temperatures and at several pulling speeds. The relationship of the glass fiber diameter to the pulling speed was established for different glass melting temperatures.

The diameter of glass fiber used in the following specimen preparation was kept in the range of 20 to 25 um for good insistance in mechanical and optical properties.

2. <u>Glass fiber coating</u> : Chopped SK-12 glass fiber was stirred for one hour at room temperature in a 1% solution of 3-aminopropyltriethoxysilane in methanol containing 1% 2M hydrochloric acid. The glass fiber was allowed to dry in air for one hour and then was cured for one hour at 100^oC under vacuum.

3. <u>Specimen casting</u>: Two different casting procedures were investigated.

^{*} Schott Glass Technologies, Inc., 400 York Ave., Duryea, PA 18642.

a. <u>Hot mixing and pressing</u>: Polycarbonate pellets were put into a 6.35 cm steel die. The die was heated by an external heating coil to about 250°C. The polycarbonate melted and glass fiber was added. The glass fiber mixture was stirred until good mixing and uniformity was achieved. Specimens with high glass fiber content required a higher processing temperature. The mixture was then pressed to eliminate bubbles from the specimen.

b. <u>Solvent casting and hot pressing</u>: An alternative casting procedure used a solvent to dissolve the polycarbonate to reduce its viscosity. Methylene chloride was selected as the solvent for its advantage of good solubility, low boiling point and high evaporation rate.

Polycarbonate pellets were completely dissolved in methylene chloride, glass fiber was added to the solution and the mixture was stirred to improve the uniformity. The mixture was put in a vacuum oven to evaporate the solvent. After the methylene chloride had completely evaporated, the mixture was heated to 260°C which is the melting temperature of crystalline polycarbonate. Pressure (28 MPa) was then applied to the melted polycarbonate-glass fiber mixture. After cooling, the polycarbonate was glassy instead of crystalline.

B. GLASS FIBER CONTENT DETERMINATION

The glass fiber content of each individual specimen was determined by measuring its weight, volume and density. For specimens of regular shape, the volume was easily determined by measuring the dimensions of the specimen with a micrometer. The density of a specimen is equal to its weight divided by its volume and the fiber percentage can be expressed by the following equation:

$$V_{f} = (D_{c} - D_{p}) / (D_{f} - D_{p})$$
 (1)

$$W_{f} = (D_{f}/D_{c}) * (D_{c}-D_{p}) / (D_{f}-D_{p})$$
(2)

Where
$$V_f$$
: Glass fiber volume percentage
 W_f : Glass fiber weight percentage
 D_c : Density of composite specimen
 D_p : Density of polycarbonate
 D_f : Density of glass fiber

C. MECHANICAL PROPERTY MEASUREMENTS

Since flexural properties are more important than tensile properties in the practical application of these transparent materials, a flexural test was adopted to evaluate the mechanical properties. According to ASTM #D790 flexural test standard, a four point bending test instead of three point bending was selected to measure the flexural properties with the advantage of better accuracy.

1. <u>Specimen specification</u>: All the test specimens were cut from the 6.35 cm molded discs. The glass fiber content of the test specimens ranged from 0 to 40 wt%. Three specimens with the same fiber content were tested to avoid variations due to the nonuniform distribution of glass fiber in the specimen. The test results were represented by the average value.

Based on the recommended dimensions for test specimens given in ASTM #D790 for four point bending test, the following values were used,

| Nominal specimen depth | - | 0.254 | CM |
|---|---|-------|--------|
| Specimen width | - | 2.54 | CM |
| Specimen length | - | 6.35 | Cm |
| Support span | - | 3.81 | Cm |
| Load span | - | 1.27 | Cm |
| Rate of crosshead motion | - | 0.127 | cm/min |
| Ratio of support span to depth of beam | _ | 16 | |

2. <u>Test procedure</u>: The whole test arrangement is shown in Fig. 1. The load and support span were aligned such that the axes of all cylindral surfaces were parallel. The load was applied to the specimen at the specified crosshead rate and the load - deflection data were taken simultaneously.

3. <u>Calculation</u>: The flexural strength, maximum strain and modulus of elasticity were calculated from the load -deflection data.

a. <u>Flexural strength</u>: When a specimen is loaded in flexure at two central points and supported at two outer points, the flexural strength can be calculated from the following equation :

$$S = 0.098 PL/bd^2$$
 (3)

Where S : Flexural strength of specimen, MPa.

- P: Load at a given point on the load deflection curve, kg.
- L: Length of support span, cm.
- b: Width of specimen. cm.
- d: Depth of specimen, cm.

b. <u>Maximum strain</u>: The maximum strain was calculated as follows :

$$r = 1.85 \text{ bdD/L}^2$$
 (4)

Where r : maximum strain, cm/cm

D : maximum deflection of the center of the specimen, cm.

c. <u>Modulus of elasticity</u>: The modulus of elasticity is the ratio of stress to corresponding strain and is expressed in MPa (10^6 N/m^2).

$$E_{\rm b} = 1.45 \ {\rm L}^3 {\rm m/bd}^3$$
 (5)

Where E_b : Modulus of elasticity in bending, MPa.

m : Slope of the tangent to the initial straight line portion of the load -deflection curve.

Flexural strength and modulus of elasticity with increasing glass fiber content was investigated.

D. OPTICAL PROPERTY MEASUREMENTS

1. Transmission

a. <u>Specimen specification</u>: The thickness of each test specimen was kept identical (0.254 cm) to avoid transmission variation due to different specimen thickness. The surface condition of each test specimen was also kept as smooth as possible to maximize optical transmission. All the specimens were polished with SiC abrasive paper in the order 120, 240, 360, 400 grit and 0.1 um alumina powder for a final treatment.

b. <u>Measurement procedure</u>: The test specimen was placed in a Beckman 26 spectrophotometer. The optical transmission of each specimen was measured over the visible spectrum (350 nm to 750 nm). For each individual specimen, three measurements were made at different locations and the optical transmission was represented by the average value.

2. <u>Transparency of specimen</u>: Qualitatively visual inspection was selected to describe the transparency of each composite specimen. If possible, the traditional method of measuring the resolution power of transparent materials with bar chart was also used.

E. MICROSTRUCTURE OF INTERFACE

The fractured surfaces of several composite samples were examined by scanning electron microscopy (SEM) to illustrate the interfacial microstructure associated with different coatings.

III. RESULTS AND DISCUSSION

A. SPECIMEN PREPARATION

The relationship of glass fiber diameter and pulling speed at a given glass melting temperature is shown in Fig. 2. For a given glass melting temperature, fiber of smaller diameter was formed at higher pulling speeds; for a fixed glass fiber pulling speed, fiber with larger diameter was formed at high temperature which corresponds to a low glass melt viscosity.

Some defects exist inside the glass fiber which permanently influence the optical properties of the The defects are due to air bubbles composite specimens. entrapped during glass melting, which can be partially eliminated by either physical or chemical fining. Chemical fining would change the refractive index of the glass fiber and was not adopted. Two methods of physical fining -bubble injection and increasing the glass melting temperature were investigated. A large air bubble injected from the bottom of the crucible can absorb smaller bubbles inside glass, but many small bubbles were formed again during the initial bubble injection. From Stokes law, increasing the glass melting temperature will decrease the viscosity of the melt and thus increase the upward velocity of air bubbles inside the glass. Small bubbles take much longer to rise out of the molten glass. Because of the temperature limitation of the glass fiber pulling machine at UMR, premelting the glass

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in a high temperature furnace was an alternative method of high temperature fining.

Glass fiber was coated with 3-aminopropyltriethoxysilane, which acts as coupling agent, to improve the bonding strength of the polycarbonate to the glass fiber. As shown by SEM photography in Figs. 3, 4, 5, and 6, specimens with 3-aminopropyltriethoxysilane coating on the fiber exhibit more adhesion between the polycarbonate and the fiber than specimens without the coating.

The mechanisms² of glass fiber coated with 3-aminopropyltriethoxysilane are:

(a) Hydrolysis of 3-aminopropyltriethoxysilane yields3-aminopropyltrihydrosilane in acid solution.





(b) Polymerization of 3-aminopropyltrihydrosilane yields a crosslinked structure between the glass fiber surface and the polymer of 3-aminopropyltrihydrosilane.



The bonding structure of amino functional group reaction with the polycarbonate matrix during the mixing procedure is¹⁰:



Several kinds of solvent, such as methylene chloride, chloroform, n-butyl chloride and ethylene chloride, were used to dissolve polycarbonate for pressing composite specimens. For samples formed from solvent casting, polycarbonate crystallization was detected by the DTA with a melting point at 260°C as shown in Fig. 7. Specimens prepared from solvent casting and hot pressing were poor transparent, thus the mechanical and optical properties were measured just for specimens formed from hot mixing and pressing.

As illustrated in Fig. 8, the DTA diagram of polycarbonate in air atmosphere shows an exothermic reaction at temperatures higher than 290°C due to the oxidation of polycarbonate which weakens the mechanical properties and causes the specimen to yellow. A feasible method of avoiding oxidation of polycarbonate at high temperatures is to process the material in nitrogen instead of an air atmosphere.

B. MECHANICAL PROPERTIES

The minimum length of a chopped glass fiber which can be considered as a continuous fiber in terms of mechanical properties can be calculated from the following equation¹:

 $l_{c} = R_{f} \{2 * [E_{f} - E_{m}] / [G * ln(R_{o} / R_{f})] \}^{1/2} * \cosh^{-1} [E_{f} / (E_{f} - E_{m})]$ (6)

Where l_C: Critical length of glass fiber considered as continuous fiber in mechanical behavior.

R_f : Radius of glass fiber.

- R_o: Mean distance of separation of fiber normal to their lengths.
- G : Shear modulus of the matrix.
- Ef : Young's modulus of glass fiber.
- E_m : Young's modulus of matrix.

For a typical 10 vol% composite specimen, the critical length of 30 um diameter glass fiber is calculated to be 0.6 mm. Since the critical length of glass fiber is much shorter than the 2 cm length used in this study, the composite specimens can be treated as continuous glass fiber composites in evaluating mechanical behavior. From the mechanical theory for composites, this type of composite shows the effect of fiber reinforcement, if the interfacial bonding is strong enough.

The load - deflection curves of samples having increasing glass fiber content are shown in Fig. 9. For specimens with higher glass fiber content, the maximum load is larger and the maximum deflection is smaller. The flexural strength and modulus of elasticity calculated from the load - deflection data for increasing glass fiber content are plotted in Figs. 10 and 11.

The flexural strength of composite specimen having no fiber silane coating was also measured and is shown in Fig. 10. The results demonstrate that composite specimens with glass fiber having a silane coating is stronger than one which has the same glass fiber content but without a coating. Therefore, it's confirmed that bonding strength factor in the mechanical strength of the composite specimen. The mechanical strength can be further increased by improving the interfacial bonding strength.

For the same interfacial condition, mechanical strength shows an additive relationship with increasing glass fiber content. Since the mechanical strength of a composite specimen depends on the individual strength of the matrix and the glass fiber, increasing the strength of polymer (matrix) can also improve the mechanical strength of composite.

C. OPTICAL PROPERTIES

The refractive index of polycarbonate³ and SK-12 optical glass⁸ through the visible wavelengths is illustrated in Fig. 12. The refractive index steadily decreases with increasing wavelength for both materials and they have the same value at the wavelength of 590 nm. The difference in refractive index between these materials increases near the short end of the visible spectrum. The Abbe number⁸, which defines the slope of refractive index v.s. wavelength is an important parameter in selecting a refractive index match for transparent materials. The Abbe number, v, is calculated from the equation:

$$v = (N_D - 1) / (N_F - N_C)$$
 (7)

Where N_D : Refractive index measured at D (588.9 nm).

 $N_{\rm F}$: Refractive index measured at F (486.1 nm).

 $N_{\rm C}$: Refractive index measured at C (656.3 nm). The Abbe number of polycarbonate is 30, but is 60 for SK-12 glass. Polycarbonate has an $N_{\rm D}$ value close to SK-12 glass, but the refractive index of polycarbonate over the whole visible spectrum is closer to Schott TiF-4^{*} optical glass⁸ whose $N_{\rm D}$ is 1.583 and Abbe number is 30 as shown in Fig. 12. Unfortunately, TiF-4 glass is not currently available and could not be incorporated into composite specimens for comparison of optical properties.

The refractive index match between polycarbonate and SK-12 glass can be improved by changing the refractive index of either one. The refractive index of the glass can be changed by blending a small amount of glass powder with a known refractive index at high temperature. Since SK-12 glass has a low refractive index and high Abbe number, it should be blended with glass which has high refractive index and low Abbe number. It can be applied in the same way for polycarbonate mixing with another polymer.

The refractive index is also influenced by temperature. The different refractive index temperature coefficients for the matrix and glass fiber will cause the optical properties of the composite specimen to change under different temperature conditions. The refractive index temperature coefficient is related to the thermal expansion coefficient

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^{*} Schott Glass Technologies, Inc., 400 York Ave., Duryea, PA 18642

by the Lorentz-Lorenz equation⁹.

From the Lorentz-Lorenz equation:

$$dN/dT = (N^2+2) * (N^2-1)/6N * (1/\rho) * d\rho/dT$$
 (8)

$$= (N^{2}+2) * (N^{2}-1)/6N * ^{\alpha}$$
(9)

Where N : Refractive index.

- T : Temperature.
- $_{\rm O}$: Density.
- α : Thermal expansion coefficient.

For polycarbonate with a thermal expansion coefficient of 6.75×10^{-5} °C⁻¹, the coefficient of temperature dependence calculated from the above equation is 4.854×10^{-5} °C⁻¹. The coefficient of temperature dependence of SK-12 glass having a thermal expansion coefficient of 7.5×10^{-6} °C⁻¹ is 5.7×10^{-6} °C⁻¹. When the temperature variation is 25° C, the refractive index difference is 0.001 due to temperature difference.

The transmission curves for composite specimens with increasing glass fiber content are illustrated in Fig. 13. The transmission of specimens with low glass fiber content is superior to those with high glass fiber content. The reduction in transmission with increasing glass fiber content is severe in the short wavelength region where the refractive index difference between polycarbonate and the glass fiber is a maximum.

The theoretical model of transmission related to refractive index difference across one interface was established previously¹¹. This model shows the transmission at normal incidence, but does not illustrate the relationship between the transmission and changing angle of incidence which exists in circular specimen such as a glass fiber. As Fig. 14 shows, the incident angle varies from 0 to 90 degrees around the half circular interface between glass fiber and polycarbonate. From Fresnel's equations⁵, the transmission at different incident angles is expressed by the following:

 $T = 2 \sin^2 A * \cos^2 B / \sin^2 (A+B) * [1 + 1 / \cos^2 (A-B)]$ (10)

- T : Transmission after light goes through the interface.
- A : Angle of incidence
- B : Angle of refraction

For a given angle of incidence, A, the angle of refraction, B, can be calculated from Snell's law:

$$N_1 Sin A = N_2 Sin B$$
(11)

N1 : Refractive index of medium 1

N₂ : Refractive index of medium 2

The transmission T calculated from equation (10) for just one interface is plotted in Fig. 15 for various refractive index differences. The transmission loss through one interface may seem insignificant, but when several hundred interfaces are involved, the transmission loss is substantial.

The transparency of a composite specimen can not be quantitatively measured because they were not without distortion. Visually, the composite specimens became less transparent with increasing glass fiber content. The factors which influence the transparency are interfacial bonding and refractive index difference. If poor interfacial bonding exists, the light wave will severely refract across the interface due to the low refractive index of the air gap between polycarbonate and glass fiber.

If the refractive index of glass fiber is larger than polycarbonate, the glass fiber acts as convex lens. The image through the specimen will be distorted due to the effect of negative focal length.

IV. CONCLUSIONS

1. The flexural strength increases from 97 MPa to 170 MPa as the glass fiber content increases from 0 to 40 wt%.

2. Elastic modulus increases from 2100 MPa to 4500 MPa as the glass fiber content varies from 0 to 40 wt%.

3. Optical transmission decreases from 88% to 28% as the glass fiber content varies from 0 to 40 wt%. Optical transparency decreases as the glass fiber content varies from 0 to 40 wt%.

Overall mechanical and optical properties can be further improved by strengthening the interfacial bonding and minimizing the difference in refractive index between the polycarbonate matrix and glass fiber over the visible spectrum .

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Figure 2. SK-12 glass fiber diameter v.s. pulling speed for glass melting temperatures.



Figure 3. SEM photographs of fractured surface of polycarbonate composite specimens contained 10 wt% SK-12 glass fiber. No silane coating was applied to the fiber.



Figure 4. SEM photographs of fractured surface of polycarbonate composite specimens contained 40 wt% SK-12 glass fiber. No silane coating was applied to the fiber.



Figure 5. SEM photographs of fractured surface of polycarbonate composite specimens contained 10 wt% SK-12 glass fiber, glass fiber coated with 3-aminopropyltriethoxysilane.



Figure 6. SEM photographs of fractured surface of polycarbonate composite specimens contained 40 wt% SK-12 glass fiber, glass fiber coated with 3-aminopropyltriethoxysilane.



Figure 7. DTA diagram of crystalline polycarbonate. " A " denotes the melting temperature.



Figure 8. DTA diagram of amorphous polycarbonate in (a) air atmosphere, (b) nitrogen atmosphere.



Figure 9. Load - deflection curves for composite specimens made by hot mixing and pressing. Glass fiber had been treated with 3-aminopropyltriethoxysilane.



Figure 10. The flexural strength for composite specimens. (a) Glass fiber treated with 3-aminopropyltriethoxysilane. (b) No silane coating applied to glass fiber.



Figure 11. The modulus of elasticity for composite specimens. (a) Glass fiber treated with 3-aminopropyltriethoxysilane. (b) No silane coating applied to glass fiber.



Figure 12. Refractive index of SK-12, TiF-4 glass and polycarbonate over visible wavelengths and the N_D of SK-12 glass fiber with diameter of 20 um.



Figure 13. Visual appearance and optical transmission curves for composite ω specimens made by hot mixing and pressing. Glass fiber had been treated with 3-aminopropyltriethoxysilane.



Figure 14. The incident angle for various locations on the interface of a circular glass fiber.



Figure 15. The transmission v.s. incident angle for refractive index differences calculated from equation (10). Calculated for only one interface.

| | polycarbonate (ref. 4) | SK-12 GLASS (ref. 8) |
|---------------------------------|-----------------------------|------------------------|
| Specific Gravity | 1.2 | 3.27 |
| Refractive index N _d | 1.586 | 1.584 |
| Abbe number | 30 | 60 |

99.9%

 7.5×10^{-6}

630⁰C

89%

 6.75×10^{-5}

150⁰C

62.1

96.6

2346

Transmission for 0.125" thickness

expansion, °C⁻¹

Coefficient of thermal

Glass transition temp.

Tensile strength, MPa

Flexural strength, MPa

Flexural modulus, MPa

| sample # | glass fiber content (wt%) | fiber treated or untreated with silane ? | flexural strength (MPa) | maximum strain (cm/cm) | elastic modulus (MPa) |
|-------------|-----------------------------------|--|---------------------------------|--------------------------------|-------------------------------|
| 1 | 0 | _ | 96 | 0.043 | 2100 |
| 2 | 0 | - | 99 | 0.039 | 2300 |
| 3 | 0 | - | 100 | 0.047 | 1950 |
| 4 | 9.7 | untreated | 103 | 0.035 | 2250 |
| 5 | 9.5 | untreated | 98 | 0.031 | 2550 |
| 6 | 10.2 | untreated | 109 | 0.039 | 2150 |
| 7 | 20.3 | untreated | 105 | 0.031 | 2700 |
| 8 | 19.4 | untreated | 115 | 0.034 | 2300 |
| 9 | 19.2 | untreated | 106 | 0.027 | 2750 |
| 10 | 29.1 | untreated | 114 | 0.028 | 2850 |
| 11 | 30.5 | untreated | 120 | 0.026 | 2600 |
| 12 | 30.4 | untreated | 101 | 0.030 | 3150 |
| 13 | 39.6 | untreated | 121 | 0.026 | 2900 |

Table II. Flexural strength, maximum strain, and modulus of elasticity for composite specimens made by hot mixing and pressing.

| sample # | glass fiber content (wt%) | fiber treated or uneated with silane ? | flexural strength (MPa) | maximum strain (cm/cm) | elastic modulus (MPa) |
|-------------|-----------------------------------|--|---------------------------------|--------------------------------|-------------------------------|
| 14 | 38.9 | untreated | 129 | 0.029 | 3150 |
| 15 | 40.8 | untreated | 103 | 0.022 | 2750 |
| 16 | 9.1 | treated | 117 | 0.033 | 2900 |
| 17 | 10.2 | treated | 101 | 0.032 | 3350 |
| 18 | 9.4 | treated | 130 | 0.034 | 2750 |
| 19 | 18.9 | treated | 131 | 0.025 | 3600 |
| 20 | 20.5 | treated | 140 | 0.023 | 3250 |
| 21 | 20.3 | treated | 120 | 0.027 | 3700 |
| 22 | 30.7 | treated | 150 | 0.016 | 4200 |
| 23 | 29.8 | treated | 144 | 0.014 | 4400 |
| 24 | 29.5 | treated | 159 | 0.018 | 4050 |
| 25 | 40.1 | treated | 161 | 0.012 | 4700 |
| 26 | 39.2 | treated | 176 | 0.011 | 5050 |
| 27 | 40.9 | treated | 155 | 0.014 | 4300 |

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