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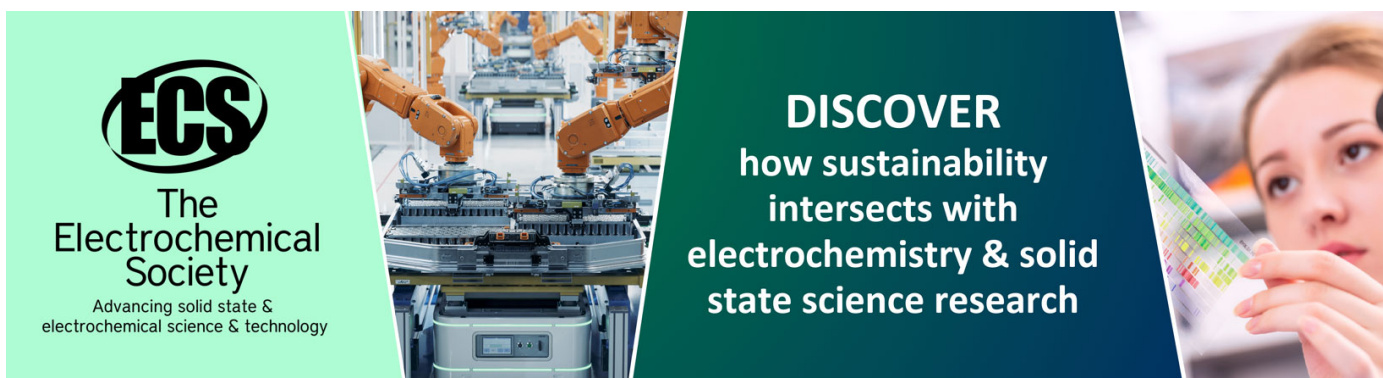
Designing of Hybrid Structured Glass Laminated Transparent Nano Composites through Vacuum infusion Technique

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Designing of Hybrid Structured Glass Laminated Transparent Nano Composites through Vacuum infusion Technique

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Abstract. Glass is the most commonly used transparent material. However, glass is not suitable in applications where low weight, high strength is required. The present invention comprises a method of making a Transparent Glass Laminated Nano composite product. The product contains a Bi-directionally oriented E-Glass Fabric an essentially bidirectional yarn woven fabrics is stretched Bi-directionally by specially fabricated steel frame associated with both co and counter rotating device. These fibers include glass fabrics/cloths or mixtures of any of these. The synthetic fiber may be any synthetic silica based oven waived bi-directional or Uni-directional fabrics. Engaged gear provided in the device develops uniform tension on fabric, in both direction. Nano particle dispersed resin to be used is formulated with their respective curing agents and extenders. The formulated resin contains 0.1-0.5% of Nano additives and the product composed from 5-10 % of Glass fabric, between 10 to 20 % of ordinary glass, and between 60 - 80 % of the product is the Nano particles dispersed formulated resin, all measured by volume.

1. Introduction

Composite structures are increasingly being used in a wide variety of applications due to their high specific strength and modulus. Even though uses of composite structures can range from such fields as marine and automotive to skis and tennis rackets, their great strength-to-weight ratio makes composites ideal for aerospace applications. Glass has been commonly used for windows in airplanes because of its hardness, chemical inertness, abrasion resistance and relatively low costs. However, glass is brittle, heavy, shatters upon impact and difficult to form complex shapes. Light weight and mechanically strong alternatives are highly desirable. Optically transparent composite is a viable solution. Transparent composites can replace traditional heavy glass on the airplanes with light weight windows and windshields. Glass laminates can be used as windows and glazings in architectural and transportation applications, including automobiles, rolling stock and airplanes. As used herein, a glazing is a transparent or semi-transparent part of a wall or other structure. Common types of glazings that are used in architectural and automotive applications include clear and tinted glass, including laminated glass. Laminated glazings comprising opposing glass sheets separated by a plasticized poly(vinyl butyral) (PVB) sheet, for example, can be used as windows, windshields, or sunroofs. In certain applications, glass laminates having high mechanical strength and sound-attenuating properties are desirable in order to provide a safe barrier with reducing sound transmission from external sources. A key function of an interlayer within a laminated glass structure is to prevent or at least minimise dispersion of dangerous glass fragments should the glass break.



Films made from plasticised PVB are considered to be particularly suited for use as an interlayer in laminated glass products due to their ability to strongly adhere with glass, their transparency and their sound physical and mechanical properties over a broad range of temperatures.

However, there are several following disadvantages to using plasticised PVB films as an interlayer:

- Films are relatively expensive.
- PVB films are also hygroscopic and must be handled under controlled atmospheric conditions to prevent excessive moisture uptake.
- Plasticised PVB film is often subject to blocking problems at the time of windup after film formation due to its very tacky surface. To prevent blocking, plasticised PVB films are typically provided with some form of parting means when stored or transported in the form of stacks of die-cut blanks or in the form of rolls. The use of PVB as an interlayer material therefore adds considerable expense to the final laminated glass product.
- PVB polymer itself is not particularly well suited to pigmentation or decoration. Polyvinylchloride (PVC) has been considered as a potential replacement for PVB as an interlayer material due to its following advantages:
- PVC is a commercial polymer that is readily available and relatively inexpensive.
- PVC can be easily calendared to a range of thicknesses, it can be plasticised to give a range of hardness values, it has suitable physical and mechanical properties for use as an interlayer and it can be pigmented or decorated to give a wide range of colours.
- PVC is not water sensitive and unlike PVB, does not require storage in a special controlled humidity environment.

However, the use of PVC in laminated glass structures has been limited due to the polymer having practically no inherent ability to adhere to glass. Attempts made to promote adhesion between glass and PVC within laminated glass structures

Thus, a transparent composite in which general-purpose glass filler is used and which shows high light transmissivity in a wide wavelength range is desired.

In view of vehicular applications, fuel economy is a function of vehicle weight. It is desirable, therefore, to reduce the weight of glazings for such applications without compromising strength and sound-attenuating properties. Desirable properties of such glazing should have sufficient strength to hold broken glass; sufficient adhesion to the glass to prevent or at least minimise dispersion of the broken glass; acceptable weather ability, etc. Normally thin interlayer polymers glass lamination process adapted so far. Normally production process involves two steps. In the first step the interlayer is placed between two sheets of glass and the resulting sandwich type structure is heated as it passes through nip rollers that squeeze the glass sheets into contact with the softened interlayer. A further step involves the treatment of the laminate so formed in an autoclave to complete the bonding process. US 4,121,014 discloses laminated windows containing adjacent layers of polycarbonate and plasticized polyvinyl acetal using adhesive film as polyurethane or an epoxy resin. But in case of polycarbonate, it's not laminated to glass. Also high temperature and high pressure is required for lamination in such case.

A transparent composite is based on the concept of matching the refractive index of the glass fibre with that of resin. Refractive index of the matrix can be modified to match with that of the glass fibre by changing the degree of polymerization of the polymer matrix. Various processing factors such as the amount of initiator, cross-linker, curing temperature and time must be carefully controlled to obtain a given refractive index. Significant reduction in weight can only be brought in by increasing the volume portion of polymer composite and reducing glass volume portion. But in doing so, one may not have desire strength to replace toughen Glass. Hence to get over from above limitation, moulded laminated reinforced composite glass by using glass fabric and nano composite may be the only solution. In view of transparency, refractive index plays a major role in polymer composites composed of more than two phases. Hence, polymer matrix with its given refractive index has to match the refractive index of the glass fibre. The refractive index of the glass fibre depends on its chemical composition, thermal history and fibre diameter. The diameter of the glass fibres pulled from a crucible of constant geometry depends upon the glass flow rate through the nozzle and the velocity at which the fibres are drawn by a take up wheel. Annealing can also increase the refractive index of chilled glass and could be potentially useful

in fabricating transparent composites. The thickness, TEX of fibre involved and the free voids appeared while fibres are wet in liquid polymer. A proprietary thermoset epoxy and polyester resin has been developed from Atul Pvt. Ltd and Satyen Polymers Pvt. Ltd. The resin has a low viscosity which allows it to be utilized in various liquid composite manufacturing processes. Several concepts of transparent composite were reported in literature. Olson, J. R., Day, D. E., Stoffer, J. O., 1992, "Fabrication and Mechanical Properties of an Optically Transparent Glass Fibre/Polymer Matrix Composite," *Journal of Composite Materials*, (Vol. 26, No. 8, pp. 1181–92) proposed transparent composite with glass reinforcements in thermoplastic Poly(methyl methacrylate) PMMA matrix. Kagawa, Y., Iba, H., Tanaka, M., Sato, H., and Chang, T., 1998, "Fabrication and Optical/Thermal Properties of Glass Particle-Epoxy Optically Transparent Composites," *Acta Materialia*, (Vol. 46, No. 1, pp. 265-271) manufactured mixing glass powder with the epoxy resin. Specialty rectangular glass ribbons were used as reinforcements by Chandrashekhara, K., Schuman, T., Sundararaman, S., Day, D., Velez, M., and Phan, N., 2008, "Manufacturing and Performance Evaluation of Glass-Ribbon Reinforced Transparent Composites," *SAMPE International Symposium*, (53, pp. 1-12) studied multiple density glass fibres in a polymeric matrix.

2. Materials

A clear polyester based one part resin system with refractive index matching the glass fibers has been developed at the SP advanced Materials Pvt. Ltd. Ordinary Floating 1.5 mm thick glass were used This resin system has been tailored to have a low viscosity thus enabling it to be used in a wide range of composite manufacturing processes. Bi-directional (0°/90°) woven glass fabric from Owens Corning Composite Materials, LLC, OH were used as fiber reinforcements. Epoxy from Atul Pvt. Ltd was used to coat the surface of the Glass.

3. Manufacturing

Manufacturing of the transparent composite was accomplished by the use of Under Gravity resin transfer and vacuum assisted resin transfer process. First method is engaged of a mould of desire size, made by thin and thick glass using phenol formaldehyde adhesive backing polyethylene cross-linked foam separated. In both the cases, two type of resins; formulated epoxy and Polyester are used. Respective curatives along with extenders used before transfer in the Mould. All leakage is sealed by liquid silicon glue. Resin transfer is done under gravity through flat hopper and a accumulator attached. The schematic of a typical first process as shown in Fig. 1. Wherein second method is engaged of tool comprises of two steel plates separated four separate steel spacers respectively. The assembly is laid on the station of the isolated steel chamber. Resin is charged thorough vacuum assisted technique. The schematic of a typical VARTM process is shown in Fig. 2. The infusion of resin into the fabric can be influenced by the permeability of the preform, the fiber architecture, and the presence of fabric crimping. The first step in the manufacturing process is to prepare the mold. The mold is thoroughly sanded to remove any scratches and cleaned using solvents and cotton cloths. The glass fabrics are stretched using tool which is then laid on the mold. Glass fabric is used in only vacuum assisted resin transfer method.

Entire assembly was placed in a vacuum chamber which was sealed around its perimeter with a general use tacky tape. The resin inlet and vacuum lines were positioned at opposite end of the vacuum chamber. The vacuum line is connected to a vacuum pump and the mold is checked for leaks. Any leaks which are found must be sealed. Once the mold preparation is complete, the resin is degassed by casting it on the hopper of Flat base. In the Resin formulation, Menthol was added to reduce viscosity. Volatile methanol will escape out leaving behind bubble free resin. The bubble and air free resin was accumulated and discharged into the mould assemble. The degassed resin is now allowed to flow through the inlet line. Added extender maintains the desire viscosity for flow and gets cure over a period in presence of initiator. The resin transfer process can take approximately ten to fifteen minutes to complete. Once the part is fully filled and gelation starts vacuum lines was opened.

Both the inlet and vacuum lines completely shut-off throughout the gelling. This keeps the resin within the part and prevents voids from forming within the part which in turn will affect the transparency of the finished composite. During curing chilled air passed to reduce the exothermic reaction heat. Once cured, the part were tpst cured. The process involves heating the mold to 240 °F for 2 hours. The cure cycle used for the part manufacturing shown in Fig. 2. After the completion of post cure cycle, the mold cooled to room temperature and the part separated from the mold. The part were then be examined for voids, surface finish, and the refractive indices matched.

4. Results & Discussion

Method -I

In this example a flat Glass laminated Transparent Nano composites product made having a cross section 8 x 1000 of length 1000 mm. This product was normally made with 35% Ordinary Glass in two layers, and 65% resin, all measured by volume. Two type of Ordinary and Toughen Glasses of different thickness, and two types of resin with varying chemical formulation used in making the products of the example. Glass inside surfaces treated chemically with different coupling agents.

Table-1:Composites properties

Types of Fabrics	Degree of Inclination		Heat of Reaction	Transmittance in visible light I/I ₀ (a.u)	Surface indolence	Air Bubble
Ordinary 1.5mm Glass (without surface treatment)	(A1)	50	160	92	Low	Low
	(A2)	100	165	90	Moderate	Moderate
	(A3)	150	170	86	Higher Side	Higher
Ordinary 3mm (AIES) Treated Glass	(B1)	50	166	89	Low	Low
	(B2)	100	167	87	Moderate	Moderate
	(B3)	150	170	85	Higher Side	Higher
Ordinary 6 mm (TDI) treated Glass	(C1)	50	162	89	Low	Low
	(C2)	100	165	87	Moderate	Moderate
	(C3)	150	170	85	Higher Side	Higher
Toughened 6 mm (AIES) Treated Glass	(D1)	50	166	89	Low	Low
	(D2)	100	165	87	Moderate	Moderate
	(D3)	150	168	85	Higher Side	Higher

Toughened and non-toughened less expensive glasses of thickness range from 1.5-6 mm are treated with various coupling agents like Amino isopropyl ethoxy silane, isopropyl triisostearoyl titanate (abbreviated as titanate), g - aminopropyl trimethoxy silane (abbreviated as silane), sebacoyl chloride (SC), and toluene diisocyanate (TDI). Treatment done at ambient temperature. More typically, Amino isopropyl ethoxy silane and toluene diisocyanate (TDI) surface treating agents are used in the present example, to improve Glass to resin bonding.

Under Gravity resin is charged from resin chamber to the pre-fabricated mould through a nozzle. The product made is resin transfer without pre-tensioned Glass fabrics. The charging of resin executed by maintaining different degree of inclination. The degree of inclination ranges from 5 to 20°. Degree of Inclination has direct impact on thickness Variation and curing Mechanisms of the resin. More inclined charging of resin results in bulging of resin at the center part of the Mould and uncontrolled process due to leakage problem. Higher inclination leads to faster curing and high exothermic reaction. Degree of inclination and surface treatments imparts effects on various properties of the Glass as showed in Table 1. The products made are represented in Fig1 where Transparent Nano composites indicated at C1,C2,C3 and D1,D2,D3 were made respectively with treated and Untreated Glass at different degree of inclination. Mould positioned approximately as shown in the drawing.

A first set of trials made with the three types of surface treatment Glass used for isolated pre-fabricated Mould wherein polyester resin was discharged manually. A second set of trials were made using a clear cast epoxy resin. Polyester resin employed was developed by Satyen Polymes. Pvt. Ltd. and Epoxy by Atul Chemicals Pvt. Ltd.. Nano particles like carbon tubes/nano clay/nano silica dispersed and exfoliated in the base resins prior to specific formulation. Base resins formulated of minimum shrinkage and higher toughness. The scope of invention includes the use of extender like vinyl Acetate, Epoxy and Methanol in the formulation to reduce the heat of reaction. Type and dose of Nano materials and extenders in formulation plays important role on transparency, heat of reaction and Bonding of Glass to Resin results as showed in Table 2 .

The products made with Nano material and with both resins were excellent in appearance. The discharging clear resin by manual technique has tendency of Bubble formation and bulging effect at the center on higher inclination.



Fig.1 Schematic 3D image of hydraulically actuated Station Used to charge resin

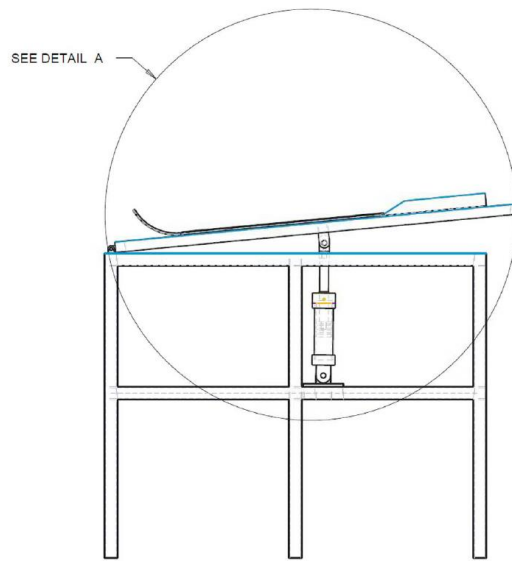


Fig.2 Schematic 2D image of hydraulically actuated Station Used to charge resin

Method - 2

Synthetic fabrics like polyester, Basalt or Glass or more typically E-Glass fabrics having a 80-100 g/m² basis weight more typically 90g/m², a density of about 70 kg/m³ and a thickness between 0.5mm and 1 mm was used. Very thin coating is made on glass by various chemicals like Epoxy, Polyurethane, SBR (Styrene butadiene Rubber) latex; NBR (Nitrile butadiene Rubber), NR (natural rubber), Polybutadine, Solvent based polyvinyl acetate, Polymethyl methacrylate, Polyethyle acrylate, styrene and acrylonitrile and acrylamide in the presence of different redox initiator systems such as vanadium - cyclohexanol, vanadium - cyclohexanone, etc. More typically, Epoxy, Polyurethane and Polyvinyl acetate surface coating used in the present example, to improve Glass to resin bonding. Thick and flexible coating on Glass surface is more vulnerable in holding cure resin on breakage of the product wherein rigid and thin coating holds resin profoundly as showed in Table 2 and 3. Wherein thin flexible coating on top of treated glass surface holds thin Glass broken fragment Glass but has significantly reduce transparency and Mechanical properties.

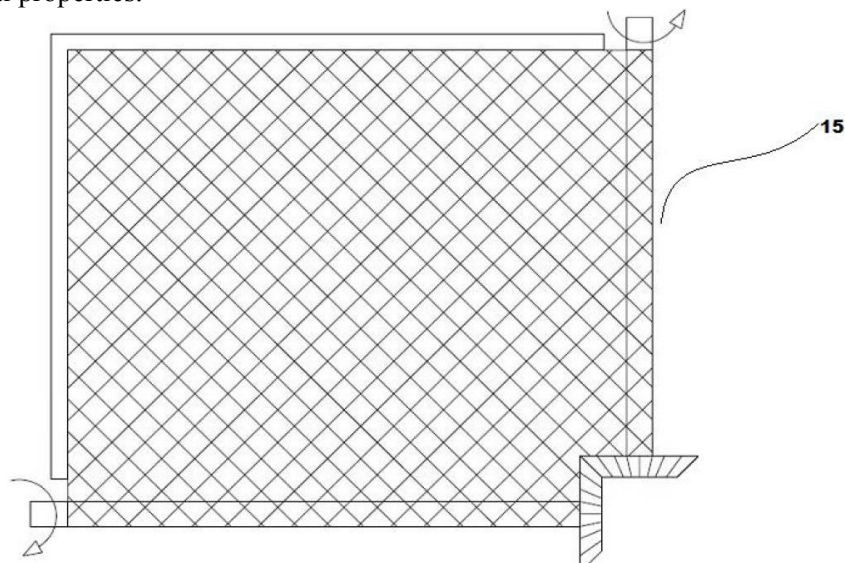


Fig.3 Schematic 2D image of steel Frame of Biaxial stretched Fabrics

Table: 2 Bond features and heat reaction

Formulation: Polyester-100 Phr, cobalt octoate -2%, Methyl Ethyl Ketone peroxide (MEKP)-1.5 %	Weight % used						Heat of Reaction	Bonding Between Glass and Resin
	Nano Materials			Extender			Temperature (0C)	
	Carbon Nano Tube	Clay Nano	vinyl acetate	Methanol	Epoxy			
Ordinary Glass without any coating and treatment	(E)	0.1%	0.2%	2%	0 %	4%	160	Broken into Fragments
Polyurethane Coated ordinary Glass	(F1)	0%	0.5%	2%	0.0%	5.0%	160	Broken into Fragments
	(F2)	0.3%	0%	4%	2%	0.0%	150	Broken into Fragments
Polyvinyl Acetate Coated Toughened Glass	(G1)	0.2%	0%	2%	1.0%	5.0%	150	Broken into Fragments but does not come out
	(G2)	0.0 %	0.7%	4%	2%	0.0%	160	Broken into Fragments but does not come out
Methyl acrylate Coated Toughened Glass	(H1)	0.2%	0.0%	2%	2%	5.0%	145	Broken into Fragments
	(H2)	0.0%	0.7%	4%	2%	0.0%	165	Broken into Fragments

Table-3: Bonding and light transmittance properties

Formulation: Epoxy-50% Hardner-50%	Weight % used					Transmittance in visible light I/I ₀ (a.u)	Bonding Between Glass and Resin
	Nano Materials		Extender				
	Carbon Nano Tube	Clay Nano	Polyurethane	Polyvinyl ester			
Polyurethane Coated ordinary Glass	(I1)	0.2%	0%	2%	1.0%	88	Broken into Fragments
	(I2)	0.0 %	0.7%	4%	2%	86	Broken into Fragments
Polyvinyl acetate Coated Toughened Glass	(J1)	0.2%	0%	2%	1.0%	86	Broken into Fragments
	(J2)	0.0 %	0.7%	4%	2%	82	Broken into Fragments
Epoxy Coated Toughened Glass	(K1)	0.2%	0%	2%	1.0%	85	Broken into Fragments
	(K2)	0.0 %	0.7%	4%	2%	88	Broken into Fragments

First set of trial made with two types of fabrics and one UD roving pre-stretched in the steel frame placed in the specific mould wherein polyester resin discharged under vacuum assisted Method. Second set made with formulated clear cast epoxy resin. Mould was kept in a vacuum pump facilitated isolated chamber where in resin discharge process was developed. The fabrics developed and supplied by Woon's Corning. Scope of invention includes Synthetic fibers like Basalt fabrics, Glass fabrics, Aramide, polypropylene and polyester Fabrics. More typically, Basalt fabrics, Glass fabrics and polyester fabrics are used in the present example. Fabrics GSM ranges from 70-400g/m² and more typically 80-200g/m². Higher GSM fabrics results in a significant reduction in transparency but enhancement in resistance to compressive load, higher stiffness and bending strength as well. Pre-stretched fabrics contributes major role in enhancing compressive load bearing property of the product wherein fabrics GSM and text of fibers involved controls transparency of the product as given in Table-4.

The vacuum chamber of 1000-2400mm length, 1200mm width and 800mm of depth used to produce Glass laminated Transparent Nano Composites of 10-20 mm thick, 1000 mm width and of 1500 mm length. The Chamber is Vacuumed to a level of 0.12 mm Mercury. The trials were run after achieving a vacuum level and positioning the Mould assemble at 0 o to Base. Hydraulic actuation of the base station where Mould assemble is placed, started clamping as against top of the vacuum chamber before opening valve to discharge resin. The mould assemble includes the steel spacer separating two halves of the mould. Two sets of independent steel spacer attached separately to respective Mould halves holds the pre-tensioned fabrics. Pre-tensioned fabrics placed at the center of the Mould assemble separates Mould halves equally. Removing air from the isolated chamber through Vacuum Pump attached. This process significantly reduced air bubbles, thickness variation and has controlled cure mechanism. The resin was standard clear cast grade polyester obtained from Satyen Polymers. The resin is charged from resin

chamber through a valve attached nozzle. The product made by Vacuum assisted resin transfer with and without pre-tensioned Glass fabrics. Vacuum level is maintained between 0.8-0.3 mm as are shown in the Table 5. In the present example Glass fabrics between 80-200 GSM are also replaced with 70 - 200 GSM polyester and Basalt fabrics.

The products made are represented in Fig. 3, 4 and 5 where transparent Nano composites indicated at A1, A2, A3 and B1, B2, B3 were made respectively with Glass fabrics, Basalt fabric and Polyester Fabrics with polyester and Epoxy as cast resin. The thin glass surface is also treated with different surface treating agents to have bonding with rein as indicated in Table 4. Surface treatment of Glass surface and use of Glass fabrics have significant impact on Transparency and mechanical Property of the Product.

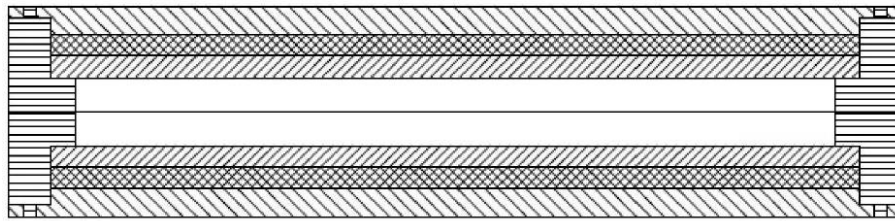


Fig.4 Schematic Sectional View of Mould used in Glass composites manufacturing

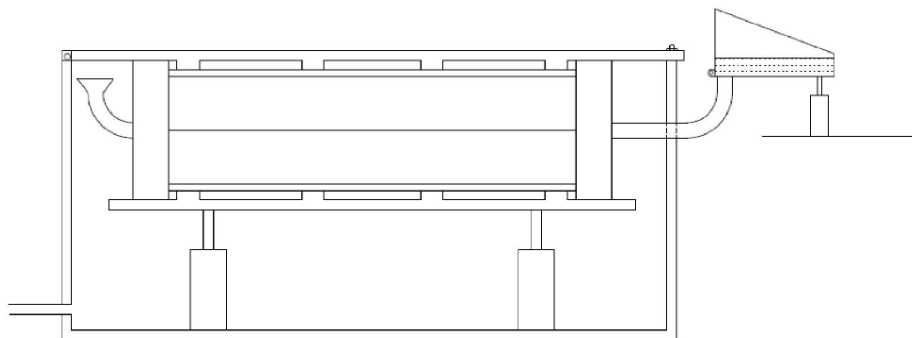


Fig.5- Schematic 2D image of Hydraulically actuated Station Used to charge resin Under Vacuum Infusion Technique.

The present invention relates to an improved glass laminated nano-composite which is very durable, higher mechanical properties compared to conventional glass and heat insulative yet very light weight. The present invention also provides a method of producing the same in very simple and economic manner by using very less expensive glass as a starting material and various easily available resins. The basic resins are used for this purpose are suitable mixtures of unsaturated polyester, Vinyl ester and clear cast Epoxy and their blends. The resins formulated before using to minimize shrinkage and higher toughened properties.

Table 4 Impact performance

Epoxy coated 1.5 mm Ordinary Glass at Vacuum 200							
Types of Fabrics	GSM		Bending Strength N/mm ²	Transmittance in visible light I/I ₀ (a.u)	Surface indolence +/- mm	Ballistic 80 m/s speed 3J	Air Bubble
E-Glass Fabrics	(L1)	80	70	90		1.2 au	95% Micro voids free
	(L2)	120	73	86	+/-2	1.03 au	90% Micro voids free
	(L3)	200	80	84	+/-2	1.04 au	87 Micro voids free
E-Glass UD (A)	(M1)	200	40	82	+/-2	2.5 au	80 Micro voids free
Basalt Fabrics	(N1)	80	70	88	+/-2	1.02 au	90% Micro voids free
	(N2)	120	78	85	+/-2	1.5 au	94% Micro voids free
	(N3)	200	87	83	+/-2	1.3 au	90% Micro voids free
Polyester	(O1)	180	40	72	+/-2	2.5 au	60% Micro voids free
	(O2)	200	40	74	+/-2	3.5 au	65% Micro voids free

Table-5: Mechanical properties

Types of Fabrics	Vacuum Level		Compressive Strength	Transmittance in visible light I/I0 (a.u)	Surface indolence +/- mm	Drop Impact (50GM) At 20 ft Height level at 10 sites	Air Bubble
E-Glass Fabrics of 80 GSM under pre-tensioned condition	(P1)	80	70	89	+/-1.05	Damage quantification (2 au)	Blister free
	(P2)	120	73	90	+/-1.02	(1.5au)	Blister free
	(P3)	200	80	92	+/-0.5	(1 au)	Blister free

5. Conclusion

The cured moulded laminated reinforced composite glass were tested for density, Glass to resin bond strength, tensile, drop impact and flexural properties. These tests showed that the composite glass possess good physical and mechanical properties. These new composite glass has applications in windshields, windows and other components where a strong, lightweight transparent material is desirable. These moulded laminated reinforced composite glass will also have potential applications as backing material in transparent armor systems. These cured composite glass are low density product as compared to normal Glass. Also, the laminated composite glass of the invention has improved glass to resin bond strength which helps hold the glass fragments on breakage. The laminated composite glass has excellent weather ability or environment resistance and has excellent laceration property as compared to normal composite glass. Thus the present invention provided an moulded laminated reinforced composite glass which is very durable, higher mechanical properties compared to conventional glass and heat insulative yet very light in weight. The present invention also provides a process of producing the same in very simple and economic manner by using very less expensive glass as a starting material and various easily available resins.

6. References

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