A Study on Co-Injection Resin Transfer Molding

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ABSTRACT

In this study the compatibility of Epoxy resin with Phenolic using three different separation layer techniques was investigated; some important process variables such as pressure, flow front and deformation were monitored in order to get a better understanding of the process.

Key Words: CI-RTM (Co Injection Resin Transfer Molding) Resin compatibility, Separation layer

1. Introduction

Co-Injection Resin Transfer Molding (CI-RTM) is a new technique where a multi-step hybrid composite is fabricated in 1 step. In this process two or more resins are simultaneously injected into a mold cavity pre-loaded with fiber reinforcement, reducing the time and cost of the traditional multi-step process. In CI-RTM the mixing of the resins is avoided with a separation layer, some techniques have been developed to separate the resins such as permeability layers, pre-impregnated layers, impermeable layers, in-situ formation layers and the combination of techniques.

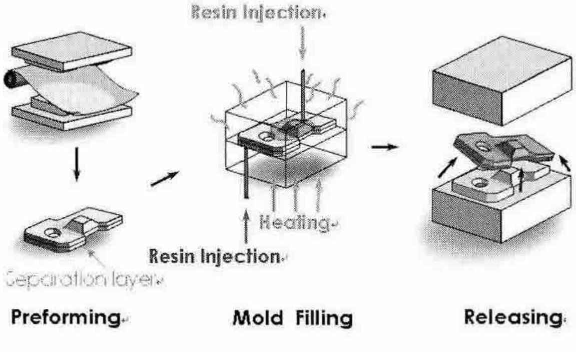

Fig. 1 Schematic of the CI-RTM process

In CI-RTM the preform and separation layer are cut and placed inside the mold, the prepreg is divided in 2 or more zones, (as it can be observed from fig. 2), next the mold is closed and the perform is compressed in order to get the desired fiber volume, two or more resins are co-injected, when the perform is completely impregnated by the resins, the injection is stopped,. After the resins are totally cured, the mold is opened and the part is released. Fig. 1 shows the schematic of the CI-RTM process.

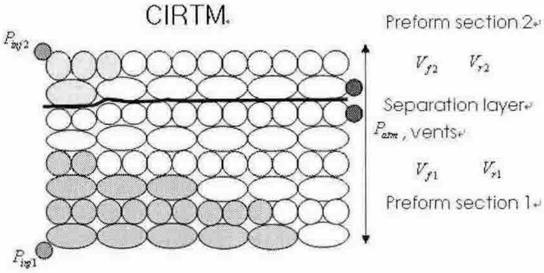

Fig. 2 In CI-RTM deformation occurs when the resin reaches the deformation layer, changing the fiber volumes

In the CI-RTM process, a separation layer is placed to separate the flows. This layer will move when the resin makes contact with it. At the beginning of the process the total load in the separation layer is the atmospheric pressure and it is supported by the dry fibers, therefore there wont be any displacement but as the flow fronts advance and one of the resins make contact with this layer, the total load will be shared between the fiber and the resin, making the separation layer deform, this deformation will be caused by a pressure difference between the resin pressure and the atmosphere pressure, the second resin will make contact with the separation layer and the deformation will be function of the pressure difference between the resins in the separation layer, as it is shown in fig. 3.

In order to get a better understanding of the CI-RTM process, especially the relationship between the deformation and the pressure distribution, the detection

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of the flow fronts, the deformation of the separation layer and the pressure distribution were monitored.

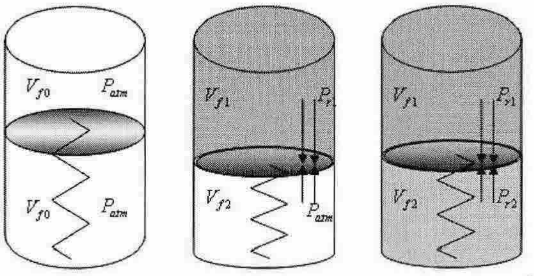


Fig.3 Schematic of the separation layer's deformation.

2. Materials compatibility

To meet the multi-functional requirements such as electromagnetic interference, ballistic, structural, shielding, fire, smoke and toxicity protection of composite materials, designs based on hybrid composite technology are used. New processing methods such as CI-RTM have been developed to fabricate a hybrid composite in a single step.

Marine structures require structural and fire resistance properties, epoxies can give the structural properties required but they are flammable and produce toxic smoke upon combustion, phenolics do not provide high structural properties but they exhibit excellent smoke and fire resistance, a hybrid laminate can solve this problem, manufacturing a thick epoxy layer and a thin phenolic coating.

Submarines and tracked vehicles need excellent structural strength as well as low impact and wear resistance. A hybrid composite of epoxy and polyurethane can solve this problem.

Materials compatibility refers to the capability of any combination of thermoset resins to be cured when they are in contact, the materials ending groups must react together in order to co-cure.

Amine cured epoxy resins are compatibles with phenolics since the epoxide group reacts with the hydroxyl group making the co-cure possible. Epoxies and Polyurethanes are compatible since the isocyanate end group of the polyurethane can react with the epoxy group.

Diffusion Enhanced Adhesion is a bonding process in which a thermoplastic film is co-cured with a thermoset. Materials compatibility is a major issue in this process.

Immodino proved that epoxy–amine thermosets diffuse into polysulfone film forming a strong interpenetrating network, this diffusion takes place in two steps, first the amine diffuses into the polysulfone making it swell and then the epoxy diffuses into the film.

For this study the compatibility of an epoxy with phenolic resin was investigated for a structural part with fire protection using 3 types of separation layer techniques: impermeable layer, a pre-impregnated layer and the combination of techniques. In order to compare the mechanical properties and the interphase toughness of the specimens, some mechanical tests were performed. With these bases the materials used in this experiment were selected.

3. Experiment

3.1 Materials characterization.

An aluminum mold with Teflon coating to facilitate release was fabricated; the mold was divided in 4 parts as it is illustrated in fig. 6. The lower part holds the section 1 of the preform (20 x 20 x 2 cm) and the separation layer together with the middle part of the mold, which also holds the section 2 of the preform (20 x 20 x 1.2 cm), then a glass cover is used to facilitate the flow observation, the upper part of the mold is used to close the mold and hold the glass. In order to measure the pressure at the inlet gates, two pressure gauges were used, the resins were pressurized in 2 aluminum tanks by an air compressor and the inlet pressure was controlled by a pressure regulator, as it is shown in fig. 4

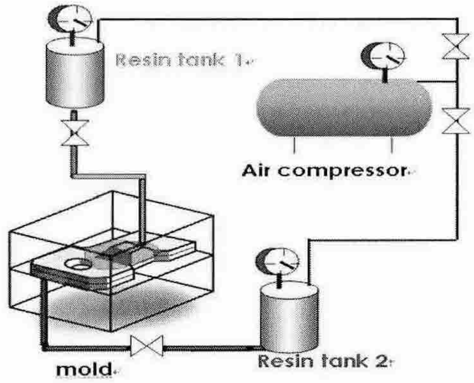


Fig. 4 Schematic of the CI-RTM process

The preform was made using an unidirectional stitched glass fabric (Dong Il Industry L 900-E11-3) alternated layer by layer to obtain isotopic in-plane permeability, Three different separation layer techniques were used, In the first case the impermeable layer technique, where a thin polysulfone film (Good Fellow LS282845)was used to separate the flows, in the second case, the pre impregnated layer technique, where a glass epoxy prepreg (Hankook Fiber T132EPC) was placed to control the flow and in the last case, the combination of techniques, where the polysulfone film was sandwiched between two layers of epoxy-amine adhesive (EPONS AR-16), fig. 5 shows the different techniques used in the experiment.

A low viscosity epoxy resin, 600 cps, (Mas Epoxy # 30-002) with a modified amine catalyst (Mas Slow Hardener # 30-008) was co-injected using two different separation layer techniques. The mixing ratio of the resin and catalyst was 2:1, the resin and catalyst were mixed using a motor -driven stirrer. The impermeable layer and combination of techniques were used, the adhesive was spreading manually, and special care was taken to avoid wrinkles between the film and the adhesive, 10% of extra amine was added in the adhesive in order to complete the cure cycle since it was expected that the amine diffused into the polysulfone film, opening the possibility of leaving not enough amine to complete the reaction,.

The co-injected specimens were cured for 11.15 hours at 140° C in a convection oven.

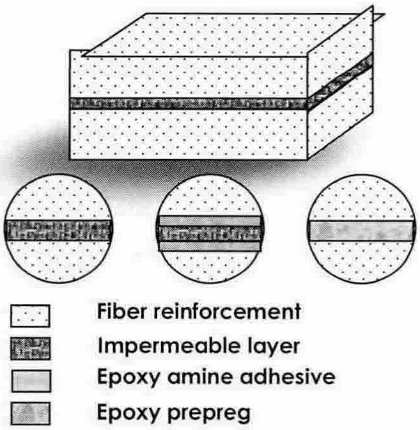


Fig. 5 Types of separation layers used in this study

The same epoxy was co injected with a phenolic resin (Resol type with an Acetic acid catalyst Kang Nam Chemicals SRE-2) with a mixing ratio of 98.5:1.5; the resin and catalyst were mixed using a motor -driven stirrer. To separate the flow, 3 kinds of separation layers techniques were used, impermeable layer, pre impregnated layer and the combination of techniques.

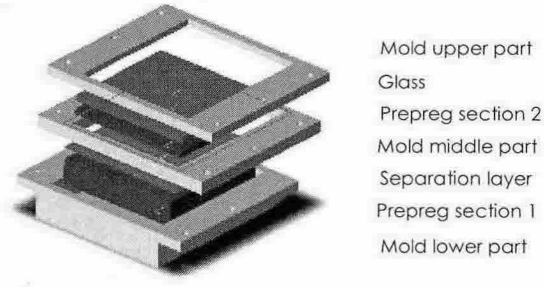


Fig. 6 Mold and prepreg sequence

For this resins system 20% of extra amine was used in the adhesive since it was expected that the amine and the phenolic diffused into the polysulfone film.

The co-injected specimens were cured for 58 hours at 95 ° C to avoid water formation from the phenolic.

In order to compare the compatibility of the materials, the mechanical properties and the toughness of the interphase, two mechanical tests were performed. To evaluate the overall quality of the parts, and to compare the apparent shear stress, the Short Beam Test (SBT) was selected. To tests the stress and the toughness of the interphase, Lap Shear Test (LST) was used. For each specimen 6 parts were cut with a diamond coated saw blade, following the ASTM 2344 and ASTM D5868 specifications for SBT and LST respectively, the mechanical tests were performed.

The epoxy resin was used as a reference to compare the results, as we can observe in fig. 7 and fig. 8, when the polysulfone film sandwiched between epoxy–amine adhesive was used as a separation layer, the apparent shear stress and the shear stress were higher, which indicates that with this technique, the parts have better quality and interphase toughness than the parts fabricated with the polysulfone film without adhesive and the prepreg techniques.

Considering that the phenolic does not posses as good structural properties as the epoxy, the results from the SBT proved the compatibility of the system since the apparent shear stress (SBT) of co-injecting epoxy and epoxy with phenolic with the same type of separation layers were similar, indicating that the epoxy-amine adhesive co-cured with both resins and also it diffused into the polysulfone film. The Phenolic did not diffused into the polysulfone film since observation after failure showed that the entire film was in the epoxy side. When the epoxy was co-injected, the polysulfone film was observed in both sides of the epoxy, which indicates that this diffused.

Material	Apparent Shear strength (Mpa)	Failure
Epoxy	13.552 +/- .494	
Epoxies/ Polysulfone	11.763 +/- .391	Epoxy - Polysulfone
Epoxies/ polysulfone + adhesive	11.541 +/- .359	Cohesive
Epoxy-Phenolic / polysulfone	7.211 +/- .452	Phenolic Polysulfone
Epoxy-Phenolic / polysulfone + adhesive	9.391 +/- .484	Cohesive
Epoxy-Phenolic / epoxy prepreg	6.779 +/- .527	Phenolic - prepreg

Fig. 7 Results for the Short Beam Test

3.2 Measurements

In order to observe the process variables and acquire a better understanding of the CI-RTM process the measurement of the pressure distribution, flow front and deformation were made.

The mold was equipped with preparations to position some sensors to measure these variables.

Material	Shear strength (Mpa)	Failure
Epoxy	15.862 +/- 3.12	
Epoxies/ Polysulfone	3.556 +/- .691	
Epoxies/ polysulfone + adhesive	7.501 +/- .958	
Epoxy-Phenolic / polysulfone	2.859 +/- .639	Phenolic Polysulfone
Epoxy-Phenolic / polysulfone + adhesive	6.958 +/- .943	Phenolic cracked
Epoxy-Phenolic / epoxy prepreg	4.965 +/- 856	Phenolic prepreg

Fig.8 Results for the Lap Shear test

Pressure measurements

To measure the pressure distribution in the mold walls, 6 pressure transducers (Green Sensor P-250) were used 3 of them in the lower part of the mold and 3 more in the upper part. The transducers were connected to a 24 volts power supply and then to a data acquisition system (Keithley 2700 Data Acquisition System), where the output voltage was read and the pressure inside the mold could be monitored, as it is shown in fig. 17. Fig. 10 shows the location of the sensors and the injection conditions, fig. 9 shows the pressure distribution at 6 points along the mold wall, the measurements were made using engine oil (L.G. Sigma DX-1), as it was expected the pressure near the injection gates was higher than the pressure near the gates. When the section 2 of the prepreg was totally filled up, at time 578 sec., the vent was closed, at this point the pressures dropped and then they increased until the mold was totally filled up, at time 801 sec. After the first vent was closed it could be observed that the pressure increments were very similar in all the transducers, with a higher tendency for the transducers in section 1, which was still filling.

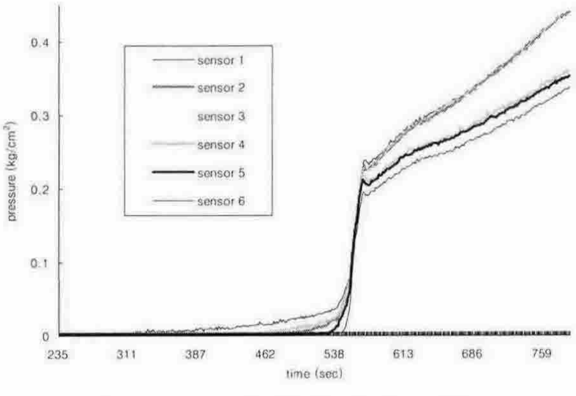


Fig. 9 Pressure distribution in the mold

Flow front measurements

There are many experimental techniques to detect the flow front during injection inside a rigid mold such as visualization, dialectic methods and thermocouples method, In these methods, sensors are basically located in the mold surface so the flow front cant be monitored only along the mold walls.

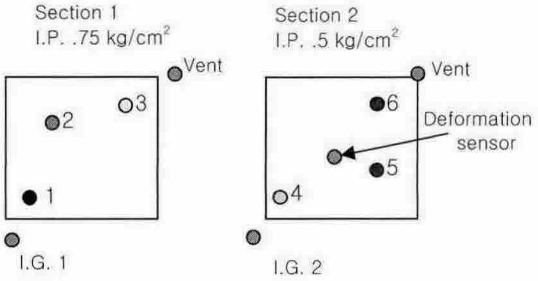


Fig. 10 Location of pressure transducers and deformation sensor

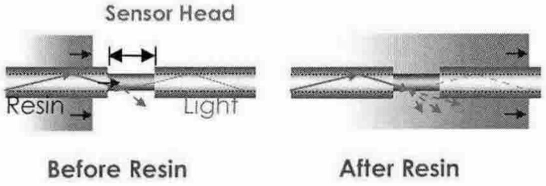


Fig. 11 Optical fiber behavior before and after resin contact

To determine the Flow front in a 3-D mold, optical fiber sensors can be used. Optical fibers consist of cladding jacket and silica core, the core is the path for the light and the cladding gives mechanical protection. In this experiment a fluid flow sensor based on the measurement of light intensity is developed by replacing a certain portion of the original cladding by a fluid. A laser light was connected to a power supply, a line of optical fiber (Ceramic Optec HWF 200/230 T) is cleaved and sensors are made by removing the polymer jacket and cladding, the optical fiber line and the laser light (wave length 633nm) are aligned with an ultra aligner and the light intensity is read on the opposite end with a CCD camera connected to a multi functional optical meter (New Port 1815) as it is shown in fig. 15. The optical fiber is positioned inside the fiber preform and the intensity of the laser light can be monitored. When the fluid makes contact with the sensor head, the intensity of the light is changed as illustrated in fig. 11 making possible the detection of the flow front in that point.

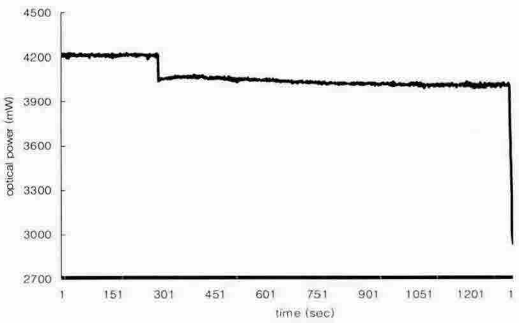


Fig. 12 Results for the flow front detection

\ Location Sensor	X (mm)	Y	Z
1 (2mm)	10	10	1
2 (4mm)	18	18	20

Fig. 13 Location of the optical fiber sensors

To detect the flow front in CI-RTM with a single laser source, different fluids and sensor sizes were tested with the purpose of distinguish the fluid that makes contact with the sensor. Fig. 14 shows that when different fluids are used to detect the flow front, the optical power lost is different. The epoxy and phenolic, which have a similar refractive index to the polymer cladding, have a lower optical power lost than the oil, whose refractive index is much lower; thereby its optical power lost is higher.

Fig. 12 shows the flow front detection results for the zone 1 of the CI-RTM preform using epoxy resin, the location of the optical fiber sensors is shown in fig. 13, the size of the sensors was 2 and 4 mm, making the optical power lost different. The small sensor had smaller optical power lost than the big sensor.

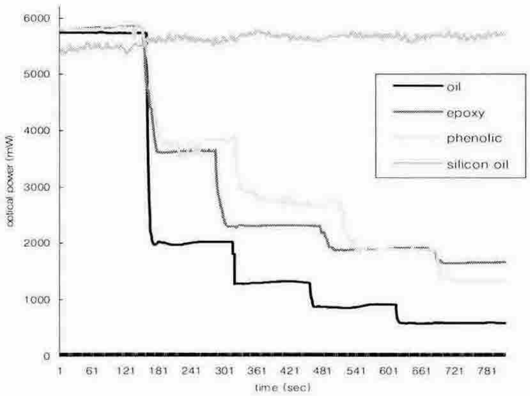


Fig. 14 Variation of optical power with different fluids

As we can see from fig. 12 and 14 the optical power lost depends on the fluid that is sensed and the size of the senor, using these facts, the detection of two different fluids necessary for the CI-RTM process can be made with a single laser source.

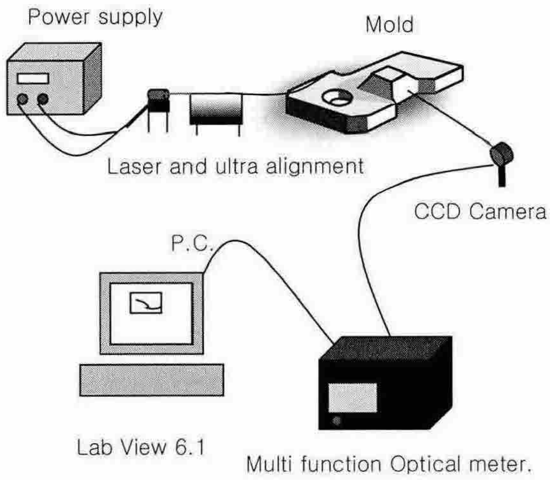


Fig. 15 Schematic of the flow front sensors

Deformation measurements

In CI-RTM, the reinforcement moves together with the flexible separation layer, this deformation will affect the thickness of the co-injected layers; this deformation has to be understood and control in order to have constant thickness part.

There are many techniques to measure displacement or deformation; these techniques can be divided in contact and no contact sensors. No contact sensors such as laser triangular sensors, ultrasonic transducers, electromagnetic transducers or other methods such as infrared camera can be used to measure displacement between two points, in CI-RTM; these techniques can not be used due to the noise made by the preform, the resin and the mold. Since these techniques can't be used a contact method was developed to estimate the order of magnitude of the deformation.

A rigid wire was glued to the separation layer as shown in fig. 16, the displacement of this wire was recorded with a camera, which is connected to a computer and the deformation can be monitored, the precision of this sensor is 100 μm.

Using the rigid wire sensor, the deformation for different inlet pressures was compared, as it was expected, when the pressure difference was increased the deformation was also increased. In CI-RTM this deformation has to be minimized by controlling the inlet pressures.

To validate the results and get more accuracy, one LVDT sensor will be used, the range of this sensor was calculated using the wire sensor;

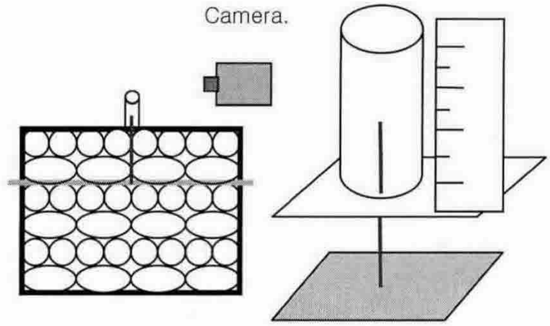
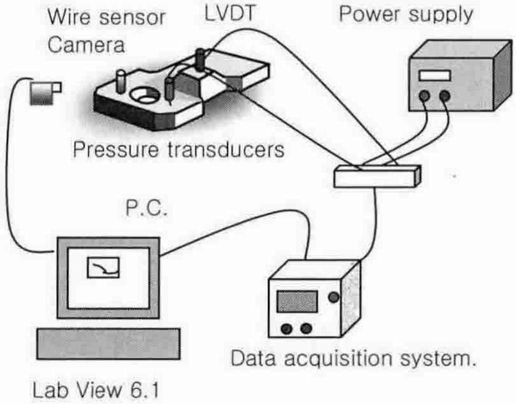


Fig. 16 Wire sensor

I. Gate 1 pressure (kg/m2)	I. Gate 2 pressure (kg/m2)	Maximum deformation (mm)
.4	.75	2.5
.5	.75	2.1
.6	.75	1.6

Fig. 17 Maximum deformation according to injection pressures



. Fig. 18 Schematic of the pressure transducer and deformation sensors

4. Conclusions

In this study, the characterization of some materials was tested and the monitoring of some variables was made. It was proved experimentally that the co injection of epoxy and phenolic has better mechanical properties and bonding when the polysulfone film is sandwiched between epoxy adhesive, The deformation of the separation layer was monitored, when the pressure difference between the resins in the separation layer is small, the deformation is small and the part has better tolerances .A flow front detection method for CI-RTM is proposed using optical fiber sensors with different fluids, and sensors sizes, the compatibility of epoxy and polyurethane is under investigation at this point.

Acknowledgement

This work was supported by the 21C Frontier R&D program.

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