New resin film infusion process for manufacture of large composite structures

C. Leone[1], M. Durante[1] and I. Crivelli Visconti[1], I. De Iorio[2]

[1]Department of Materials and Production Engineering, University of Naples "Federico II", Piazzale Tecchio 80, 80125 Naples - Italy
[2]Department of Space Science and Engineering, University of Naples "Federico II", Piazzale Tecchio 80, 80125 Naples - Italy

Abstract

A new process developed for the manufacture of large composite structures was presented. The basic idea is the use of a porous media (sponge) that is impregnated before placing it upon the dry preform with the aim of generating impregnating flow perpendicular to the reinforcement layers only. The major advantage of this process is the drastic reduction of the resin flow path that corresponds to shorter infusion time compared to the classic infusion methods.

In the present work, the process (principle) criteria, the influence of the staking sequence and the sponge type were studied with the aim of understanding the process features. The typical defects were analysed by means of microscopy and the mechanical properties were measure in three-point bending tests and compared with those of specimen obtained by hand lay up and other infusion processes.

The performed test indicate that the new process is able to produce high quality composites if the flow dynamics were well taken into account, however this do not appear as a problem.

The fibre volume fraction, the modulus and the strength measured were sensibly influenced by the stacking sequence, remaining about the same when the plies number was increased maintaining the same sponge thickness.

finally it was found that the developed process is a valid alternative to hand lay up and other infusion processes in terms of mechanical properties, with the advantage of a drastic reduction of the impregnation time.

1. Introduction

Infusion is one of the most advantageous techniques used in polymeric matrix composites production, thanks to its ability to reduce the manufacturing cost and the volatile organic compound emissions, keeping good mechanical properties, surface finishing and higher quality of the products [1-3]. For this reason the infusion process is a candidate for the replacement of the traditional open mould process and the RTM technology.

Compared to the open mould, infusion processes show also the advantage of reducing the dangerous [4] styrene vapour, emitted in workplace atmosphere when unsaturated polyester or vinylester resins are used. So the increasing legislation to limit styrene emission is the principal reason that guided the development of new variety of resin infusion under flexible tool. Furthermore the resin infusion technology is less expensive than the RTM and than the other technologies with closed rigid mould.

One of the drawbacks of this production technique is the difficulty to reduce the infusion time needed by the resin to completely embed the preform. Clearly, these phenomena essentially depend on the preform permeability, the overall dimension and the resin outlet and inlet points position. A possible solution is the use of multiple injection ports, but this results in convergence of resin multiple flow fronts that can produce entrapment of void, gas bubble or production of large dry preforms zones.

Therefore, in the last years in order to reduce the resistance of resin flow and to control flow patterns many efforts have been devoted to study groove channels, high permeability distribution media or networks. So many patents have been realised from 1959 to 1995 [5]. On the other hand new FE codes have been developed to simulate the flow front behaviour for the new vacuum infusion technology with the aim to reduce long term trial and error procedures, while the FE simulation could support the development process and optimise the better locations of outlets and inlets reducing the defects, the infusion time and then the production time, the costs and the risks [6-7].

In this work a new type of vacuum bag has been studied with the aim of generating only impregnating flow perpendicular to the reinforcement layers. The major advantage of this process is the drastic reduction of the resin pattern that corresponds to shorter infusion time compared to the classic infusion methods. Moreover the presence of a unique resin flow can avoid the above mentioned defects due by multiple injection ports.

The basic idea is the use of a porous media (sponge) that is impregnated before placing it upon the dry preform. The porous media have both the function of resin tank and distributor. When the vacuum is applied to the bag the resin squeezes from sponge into the reinforcement. This new process was called resin impregnated sponge infusion (RISI).

In this work GFRP samples were produced using different stacking sequence, with the aim to understand the impregnation dynamics and assessment of the process features. Two different types of reinforcement (unidirectional and plain weave fabric) with an epoxy resin. The optimal porous media were celluloid sponges that have shown a good process applicability.

The main aspects discussed are the sponge selection, the impregnation time, the defect generation, the mechanical properties and the obtainable fibres volume fraction as a function of the stacking sequence.

The experimental data indicate that the new process is able to produce GFRP with very short time, good properties in terms of reinforcement content percentage, mechanical properties and defect equivalent to those observed in the other infusion process (void formation, gas bubble entrapment and dry zone).

The results also showed the possibility to improve the material quality, reducing defect and increasing both V_f and mechanical properties by means of the flow velocity regulation and gas bubble filtering using a suitable procedure in the stacking sequence order.

2. MATERIALS AND EXPERIMENTAL PROCEDURES

2.1 Sponge selection

In order to select the best porous medium, the absorbed and released resin quantity of different sponges were evaluated using a laboratory apparatus, showed in figure 1. We have used five kind of sponge, characterised by different materials or thickness; in table I the characteristic of analysed sponges are reported. Every sponge dimension were measured and weighed before and after impregnation. For the impregnation we have used an epoxy resin (mates SX10) without catalyst. After manual impregnation the sponge were introduced in a vacuum bag supported by a grid and the vacuum was kept for five minutes. During the vacuum application the resin flew out from the sponge into a container. After this the resin and the sponge were weighed. In figure 1 a scheme of the equipment used to measure the released resin quantity is reported. In figures 2a-b the absorbed and the released resin normalised by the contact area, a), and by the volume, b), are reported for the different sponges. Data in figure 2a, clearly show that the resin quantities normalised by the surface depend on the sponge material, achieving the lower value for the cellulose sponges, and the maximum for the polyurethane. Looking at the figure 2b, where the resin quantities are normalised by the sponge volume, the polyurethane media show the highest value but no remarkable difference are evident between the cellulose and the polystyrene sponges behaviours, especially for the released quantity.

In figure 3 the transfer efficiency, defined as the adsorbed to the released resin ratio are reported. The major efficiency is for the polystyrene and the polyurethane sponges that show similarly value, about 0.94; on the opposite there is the cellulose that show an efficiency value from 0.4 to 0.7 . There are two different approaches to sponge selection: the efficiency or the required resin for a given number of reinforcement layers. The latter is the used approach;

therefore, considering eight layers of and a desired fibre volume fraction of about 50%, the type A cellulose sponge was selected as the best sponge.

Table I: Characteristics of the analysed sponges.

Sponge	Material	Thickness (mm)	Ra/S (g/cm^2)	Rr/S (g/cm^2)	Ra/V (g/cm^3)	Rr/V (g/cm^3)	Ra/Rr (g/g)
A	cellulose	4	0.55	0.25	1.39	0.62	0.45
B	cellulose	4	0.66	0.39	1.66	1.04	0.59
C	cellulose	10	0.85	0.63	0.85	0.53	0.74
D	polystyrene	10	0.86	0.91	0.86	0.85	0.95
E	polystyrene	10	0.95	0.93	0.95	0.93	0.98
F	polystyrene	19	2.21	1.94	1.16	1.07	0.88
G	polyurethane	10	2.64	2.55	2.65	2.55	0.97

Ra/S Absorbed resin / Contact Surface - Ra/S Released resin / Contact Surface.
Ra/V Absorbed resin / Volume - Ra/V Released resin / Volume.

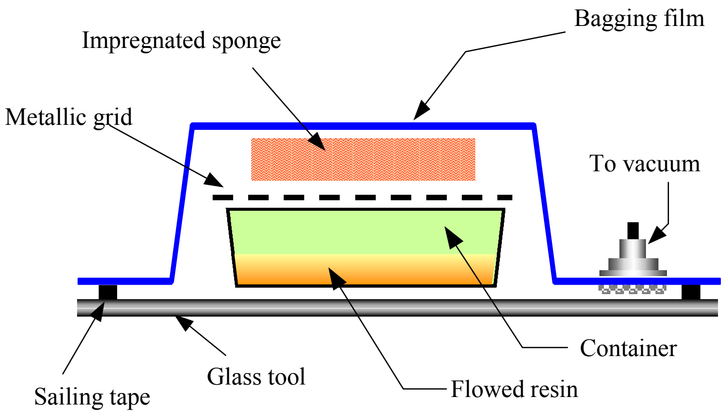
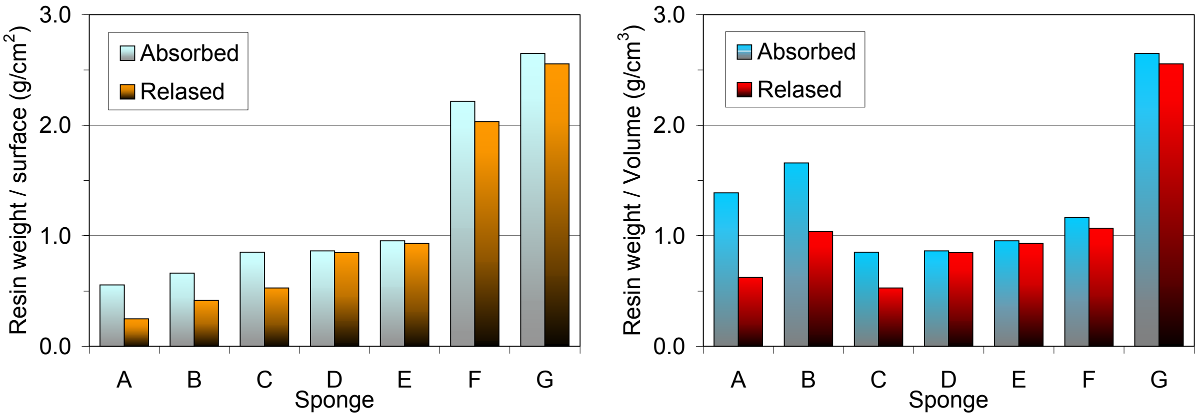


Fig. 1: Basic scheme of the equipment used to measure the released resin quantity.



a)

Fig. 2: Coparision between adimensionalised adsorbed and released resin with respect to: ***a)*** contact surface and ***b)*** Volume.

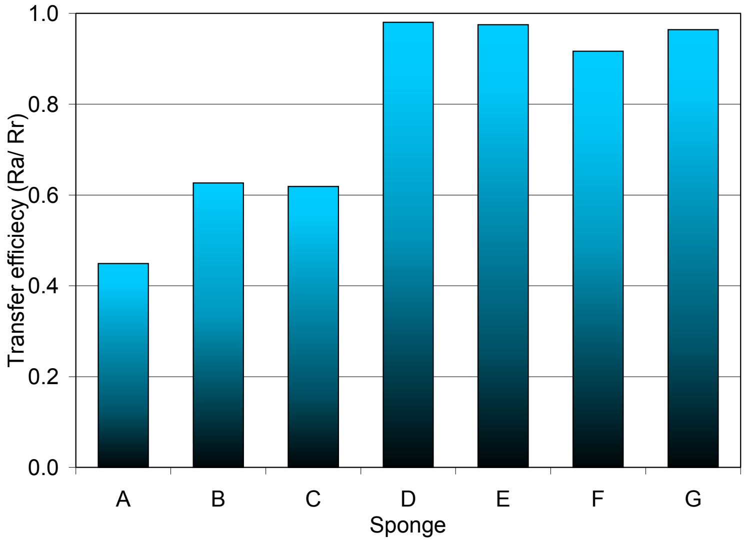


Fig. 3: Transfer efficiency: adsorbed to released resin ratio, for the analysed sponge type.

However some experiment were also done with the polyurethane sponge (type G) with the aim to evaluate the advantage of the availability of high quantity of resin. Unfortunately the test conduced with this sponge did not produce good results; this was because of the great quantity of adsorbed gas during the impregnation and of the high longitudinal deformation showen during the vacuum application, that did not permit a complete preform impregnation .

2.2 Specimen fabrication and testing

Two different types of reinforcement: unidirectional 450 gr/m^2 and plain weave fabric 500 gr/m^2 were used with an epoxy resin (mates SX10) to produce 200x200 mm^2 plates. Different numbers of reinforcement plies were used in the preform preparation to evaluate the impregnation capability and the effect of the layers number on both the composite thickness and the fibre volume fraction. In table II the experimental plan is reported.

The basic schemes of the production sequence are reported in figure 4. The production sequence are: ***a)*** the sponge is completely immersed in the resin and then impregnated by hand rolling; ***b)*** the prefom, the peel ply, the sponge and polymeric bag are placed into the mould, ***c)*** the vacuum is applied and the resin squeezed out of the sponge into the preform; ***d)*** after the cure the bag is removed.

Bending samples were cut from the plate and tested in tree point bending test using a support span to dept ratio value of 32, according to the ASTM D 790- 86 standard. Not less then five samples were tested for all condition.

After bending tests, the samples were sectioned and the surfaces were polished and examined by optical microscopy, to assess the defects.

3. RESULTS & DISCUSSION

3.1 General properties

Figure *5a* shows the sample thickness against the ply number for both the reinforcement formats. For both the materials the thickness increases about linearly increasing the ply numbers. The fibre volume fraction analysis, done by combustion methods, said that it’s not at all right; in fact the fibre percentage increase from a 50% to a 55% when the reinforcement increases from 4 to 6 plies, then keeps approximately constant even for twelve plies.

This means that the resin absorbed depends on the preform fibre content and only when a higher resin quantity (three time more than the necessary) is stored in the sponge a reduction of the fibre volume percentage can be obtainable, figure *5b*.

Table II: Matrix of the experimental tests.

Reinforcement	**Plies number**
Unidirectional (450 gr/m^2)	4 – 8 – 12
Weave fabric (600 gr/m^2)	4 – 6 – 12

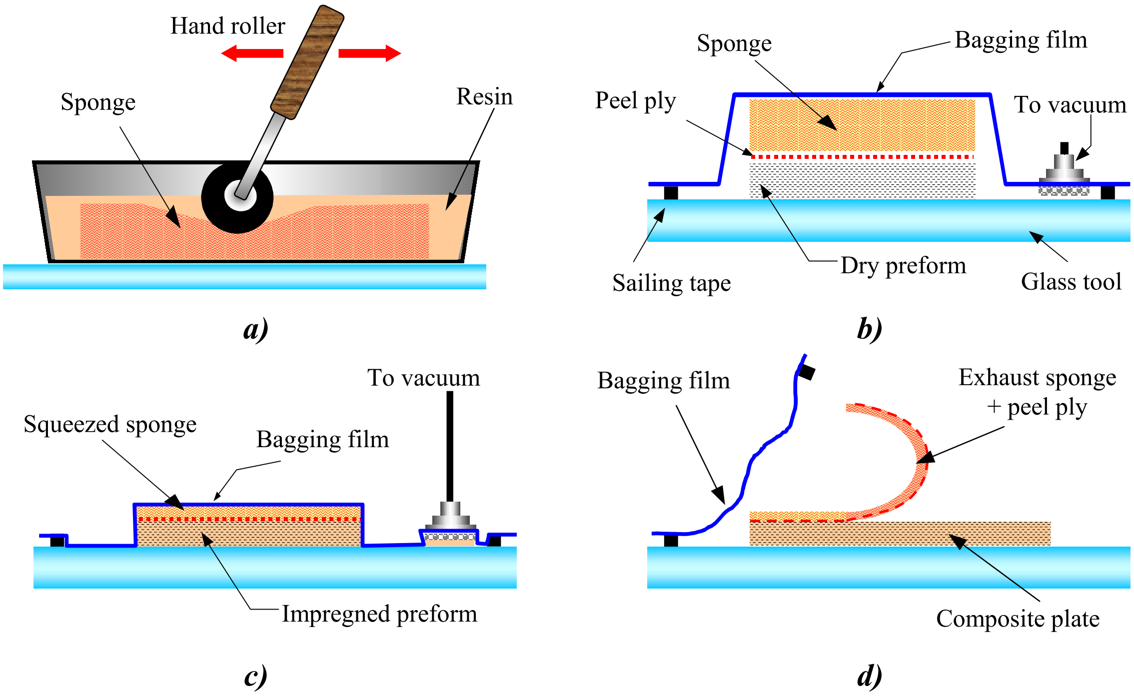


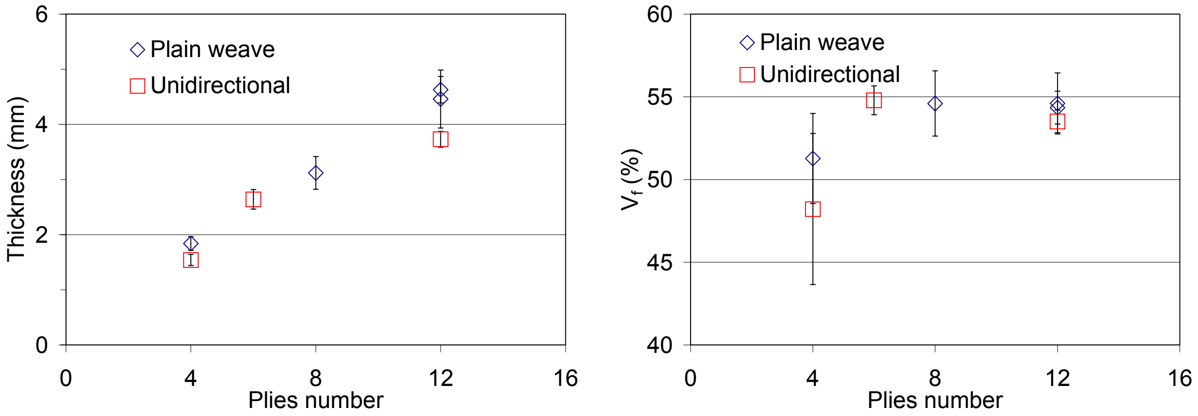
Fig. 4: Basic scheme of the production sequence: ***a)*** sponge impregnation, ***b)*** bag preparation, ***c)*** vacuum application; ***d)*** bag removal.

During the test the times required for the impregnation of the plate were recorded using a cam corded positioned below the glass tools in order to record the flow-time behaviour. The resin flow took about 20 seconds to pass through the preform, 42 to impregnate about 80% of the reinforcement and less than a minute to reach 100%. This was independent from the used reinforcement or it's dimension.

The mean value and standard deviation of the Young's modulus for both the reinforcement formats are reported in figures 6a and 6b, against the plies number and the fibre percentage respectively. As expected the Young's modulus for the unidirectional reinforcement results to be higher than the plain weave and for both materials it increase with the plies number or with the fibre volume fraction percentage.

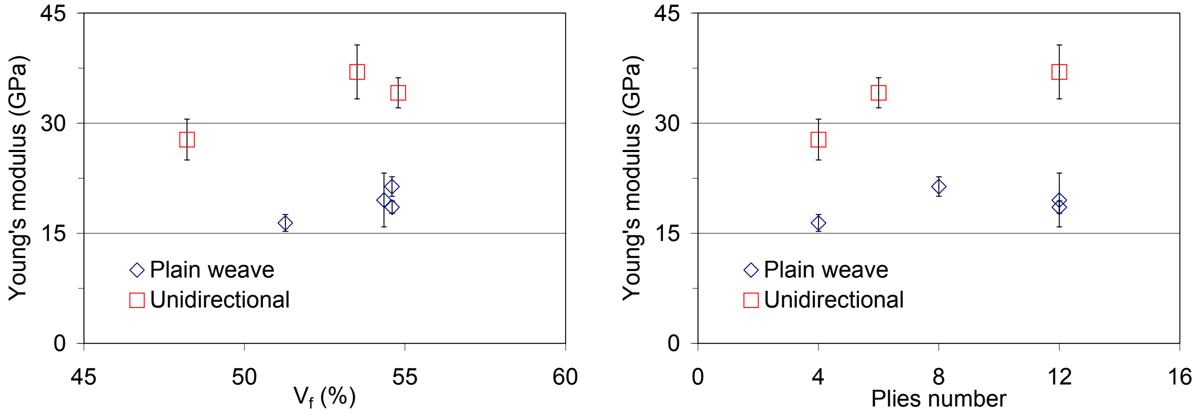
Surprisingly, both the materials exhibit an equivalent flexural strength regardless of the reinforcement format, the ply numbers and the fibre volume fraction. This fact can be justified if we consider that the two materials have two different permeability that produce different flow patterns and then different mechanisms of defect formation, as discussed above.

In the case of a flexural test the defects produced have a great influence on the strength because they promote the stess concentration and then the final failure to come early. To ascertain this topic, optical microscopy analyses were done with the aim to characterise the defect produced. Selected images of the sample's sections after polishing are shown in figures 8 and 9. The most abundant defect is bubble presence that occur throughout all the samples; but also matrix rich zone near the sponge side of the samples are visible, figure 9a. Two kind of bubbles are identified: irregular shape bubble, and regular shape bubbles. This is appreciated from a comparison of figures 8 (regular shape) and 9 (irregular shape) concerning the plain weave reinforcement; but similar conclusions were also drawn from the analysis of the unidirectional format.



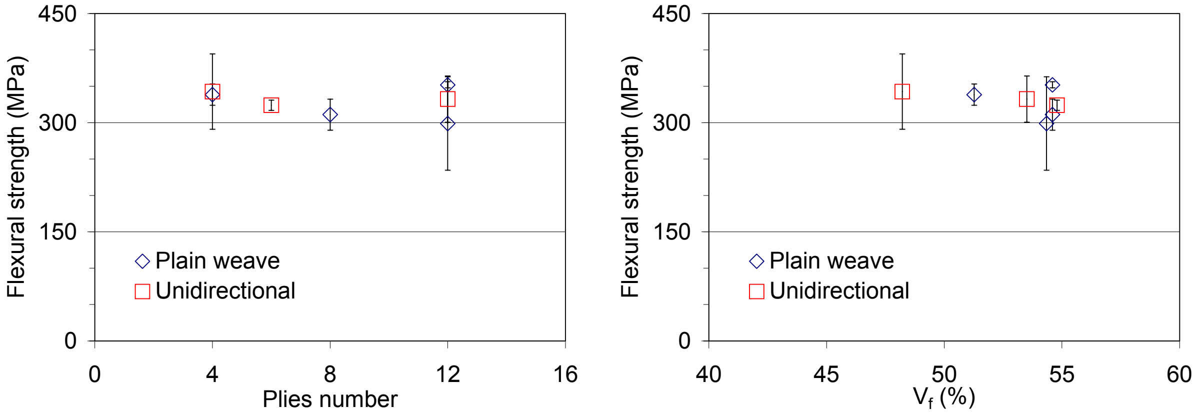
a) ***b)***

Fig. 5: ***a)*** Plate thickness and ***b)*** fibre volume fraction against the plies number.



a) ***b)***

Fig. 6: Young’s modulus against ***a)*** the plies number; and b) the fibre volume fraction.



a) ***b)***

Fig. 7: Flexural strength against ***a)*** the plies number; and b) the fibre volume fraction.

It’s reasonable to suppose that there are two different formation defect mechanisms: the regular shape bubble are produced by gas bubbles entrapped into the sponge that are squeezed together with the resin; these bubbles flow with the resin into the reinforcement and then they are kept between the tows, in a matrix rich zone.
The irregular shape bubbles are produced by the gas entrapping due to the joining of different flow fronts from different sides. The contemporary presence of micro and macro channels, and the variation of capillary pressure [9], produce preferential paths for the resin flow, are probably responsible for the latter defect observed, as reported in [10-11]. A macro phenomenon analogous to the one previously described can be observed on the face opposite to the sponge; this is a large area of dry reinforcement; as visible in picture 10a. This defect is due by the higher flow speed and the inhomogeneous resin flow fronts that do not proceed in parallel this produces a gas bubble entrapping as visible in figure 10b, as reported in [11-12].

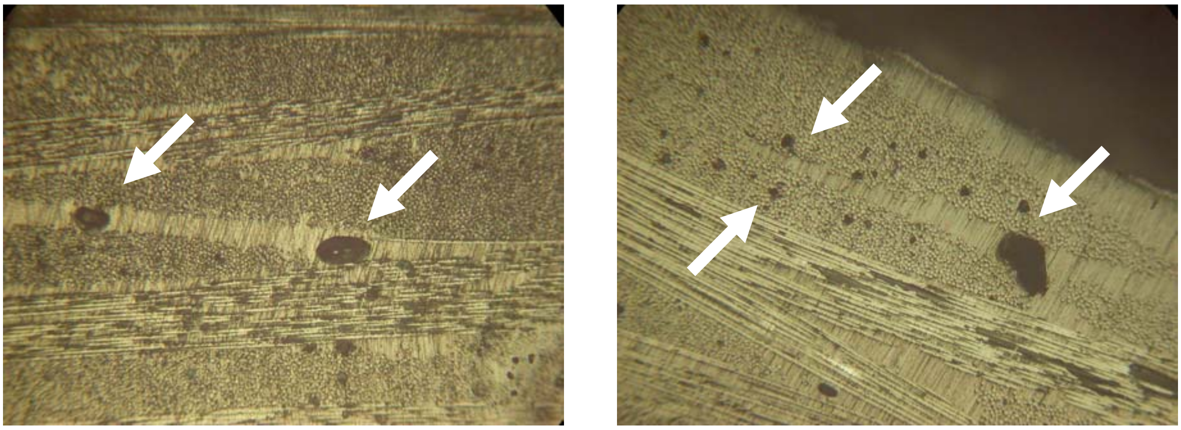
3.2 Impregnating flow control

The defect revealed by the microscopy analyses raise some questions on possible methods to control the bubble flow that have detrimental effect the composite’s mechanical properties.
An easy method to control both the bubble flow and the impregnation velocity is the use of a lower permeability film placed between the reinforcement and the sponge. For this reason new samples of eight plies, for both the reinforcement formats, were done using a polyethylene porous film placed between the peel ply and the sponge, as visible in the scheme of figure 10. The presence of the polyethylene does not reduce the advantage of high flow velocity, increasing the impregnation time from 55 to 70 seconds, an increase of about 27% in time; moreover it is very little compared to the time typically required in infusion process.
The new samples were examined by microscopy analysis and tested in three point bending tests to assess the new procedure effect. From the optical microscopy the visual effect of the flow controller layer is a decrease of both the bubble number and their dimensions; arriving, in some case, to a complete absence. Also the large zones of dry preform were completely absent.
As previously stated, among the most promising applications of the RISI technology are the large composite structures, which are usually built by hand lay up or infusion. Therefore, it is interesting to compare the mechanical properties of specimens obtained using the three methods, in order to evaluate the advantages and drawbacks of the new technology presented here.
To this aim, picture 12 and 13 collects the typical mechanical properties of RISI technology samples obtained with and without the flow controller layer, compared to similar ones (8 plies of plain weave) obtained using hand lay up and infusion methods.
From the pictures 12 and 13 we can observe that for the samples obtained by RISI method the use of a flow controller layer improve both the mechanical properties, red and light blue bars, especially the flexural strength; this is a clear effect of the defect reduction. The mechanical properties of the composite obtained by RISI technology are comparable or better than the ones obtained using the other methods (yellow bars).

3.3 Discussion

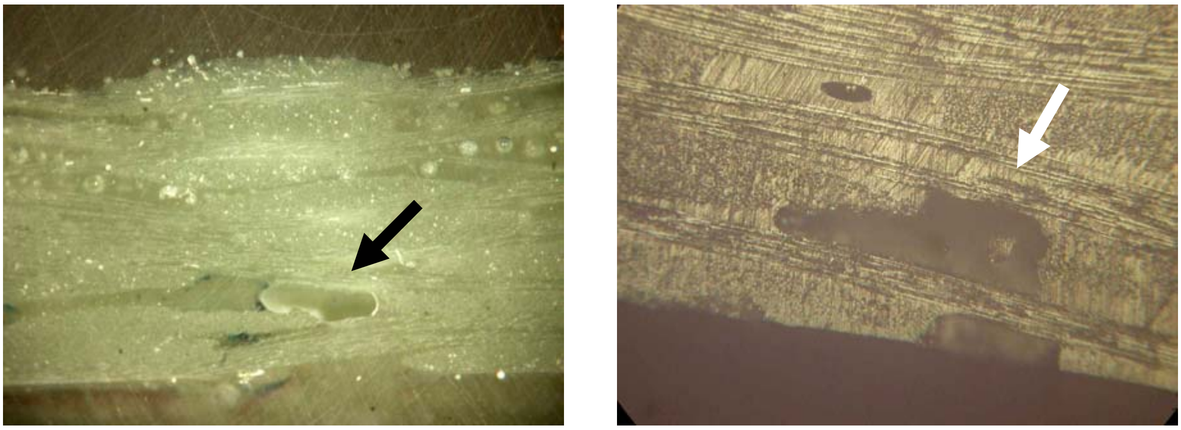
From the results we can deduce that the RISI technology is suitable to produce composites with good mechanical properties compared to the other normal processes in low impregnation time independently on the plane dimension. The thickness and the fibre volume fraction do not depend on the disposable resin quantity and only a little variation (10 %) can occur when the numbers of plies are tripled. It’s reasonable to suppose that the fibre volume fraction depends on the applied pressure and the preform porosity; when the sponge is squeezed by the vacuum application only the resins absorbable by the preform leaves the sponge and this determines an about constant fibre volume fraction value.

The high flow velocity and the different kind of flow channels (micro and macro flow) can determine defect formation as bubbles and dry preform zones. The flow control is possible applying a filter that has the dual aim to reduce the flow velocity and to avoid the gas bubble.



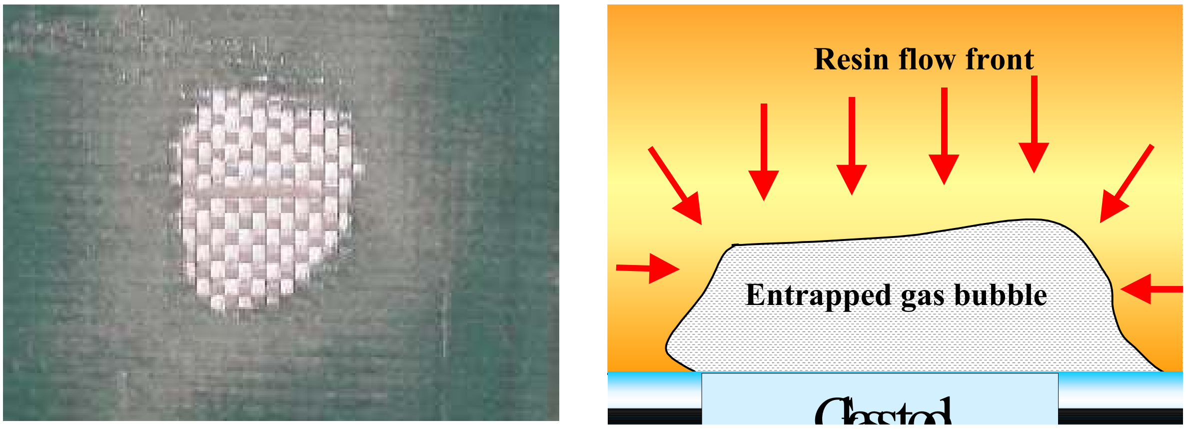
a) ***b)***

Fig. 8: Typical regular defect due to the gas bubbles dragged by the resin.



a) ***b)***

Fig. 9: Irregular shape bubbles due to the fast simultaneous flow fronts from different side.



a) ***b)***

Fig. 10: ***a)*** A typical defect observed as a large zone of dry preform; ***b)*** Mechanism of defect production.

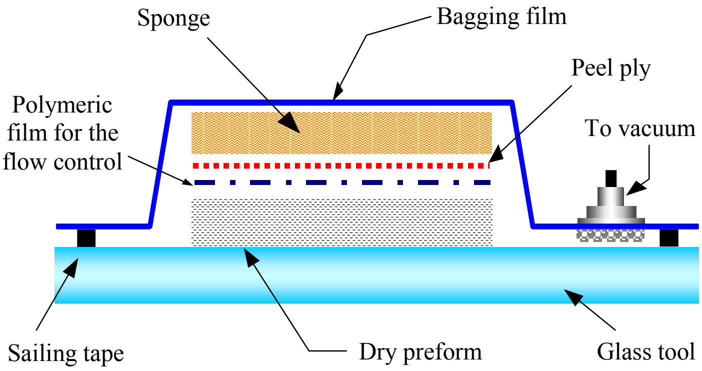


Fig. 11: Variation to the basic scheme of the stacking sequence introducing the flow controller.

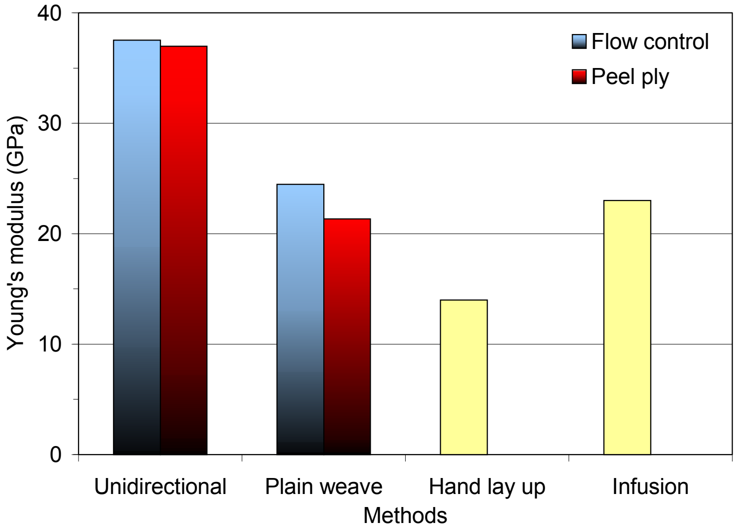


Fig. 12: Young's modulus of RISI method samples compared to the ones obtained using hand lay up and traditional infusion technology.

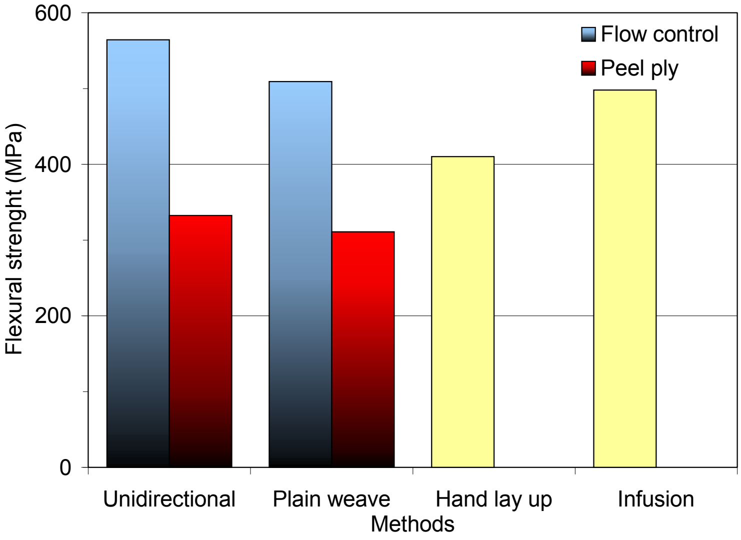


Fig. 13: Flexural strength of RISI method samples compared to the ones obtained using hand lay up and traditional infusion technology.

5. CONCLUSION

From the results presented and discussed in this work, the main conclusions are as follows:

A new vacuum bag method has been developed and studied with the aim of generating only impregnating flow perpendicular to the reinforcement layers.

The major advantage of this process is the drastic reduction of the resin flow path length that correspond to shorter infusion time compared to the classic infusion methods. In our case the impregnation time was of about a minute.

The obtainable fibre volume fraction is about 50%; and it's independent on the disposable resin.

The microscopy analysis showed the presence of bubbles due to the gas transportation phenomena or to the gas entrapping. Both these defects can be reduced by reducing the flow velocity by means of a semi-permeable ply placed on the preform that don't affect the impregnation time significantly.

From the three-point bending tests results, the measured mechanical properties are comparable or better than the ones obtained using the hand lay up or classic infusion methods.

References

1) **M. K. Kang, W. I. Lee, H. T. Hahn,** "Analysis of vacuum bag resin transfer moulding process" Composites, Part. A, 32, 2001, pp 1553-1560.
2) **M. Durante, A. Langella, S. Covito, M. Ussorio,** "Properties of laminates manufactured by infusion process", Proc. of Int. Conf. Advancing with Composites 2003, may 7-9 2003, Milan, Italy.
3) **Vistap M. Karbhari, Dongqing Wang, Yanqiang Gao**, "Processing and performance of bridge deck subcomponents using two schemes of resin infusion" Composite structure, 51, 2001, pp. 257-271.
4) **G. Groth – Marnat,** "Neuropsychological effects of styrene exposure: review of current literature" J. perceptual end motor skills, 1993, Vol. 77, pp. 1139-1149.
5) **C. Williams J. Summerskales and S. Grove, "Resin infusion under flexible tooling (RIFT): a review "** Composites, Part. A, 27, 1996, pp 517-524**.**
6) **Kerang Han, Shumhang Jang, Chuck Zhang, Ben Wang,** "Flow modelling and simulation of scrimp for composites manufacturing" Composites, Part. A, 31, 2000, pp 79-86**.**
7) **G. A. Barandun, U. M. Fasel, J. S. U. Schell and P. Ernanni,** "Development of an Optimisation and Simulation Tool for LCM processes using Evolutionary Algorithms" Proc. of International Meeting on Composite Materials, Plast 2003, Milan, Italy, May 7-9, 2003.
8) **U. Huber, M. Maier,** "Methodical applications of liquid molding simulation" Proc. Of Int. Conf. High Performance Structures and Composites - HPSC2002, 2002, Seville, Spain.
9) **M. Connor, S. Toll and J. A. E. Manson**, "On surface energy efefcts in composite impregnation and consolidation, Composite Manufacturing, 6, 1995, pp. 289-295.
10) **J. Varna, R. Joffe, L. A. Berglund, T. S. Lundström,** "Effect of void on failure mechanism in RTM laminates", Composites Science and Technology, 53, 1995, pp. 241-249.
11) **N. Pearce, F. Guild, J. Summerscales** "A study of the effects of convergente flow fronts on the properties of fibre reinforced composites produced by RTM", Composites Part A, 29A, 1998, pp. 141-152.
12) **M. K. Kang, J. J. Jung, W.I. Lee**, " Analysis of resin transfer moulding process with controlled multiple gates resin injection" Composites, Part A 31 (2000) 407–422