POROSITY INFLUENCE ON ORGANIC COMPOSITE MATERIAL MECHANICAL PROPERTIES

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Abstract

This paper presents the work done to propose a manufacturing procedure to produce high performance composite material with controlled porosity content for research and development activity. Different solutions have been proposed to produce composite with controlled porosity. Micro spherical product "Expansel" have been used to create porosity at the interplies level (inter laminar porosity). Two foaming agent have been used to create porosity distributed in all the composite volume (intra laminar porosity). The first one is DY5054 from Huntsmann, the second one is azobisisobutyronitrile (AIBN). Both create porosity by thermal degradation during polymerisation. Mechanical properties have been determined and the influence of porosity content is clearly seen with compression and shear tests. Future work will be possible to run in order to determine the porosity influence on thick composites as the problem on "how to create on demand porosity" is now solved.

1 Introduction

The utilization of organic composite material for structural application in the aerospace field is more and more important and is now an inescapable fact. To guaranty the mechanical performance of the structure we need to avoid any defect in the composite part during the manufacturing process. For thin structures, quality procedure have been established in the past to define the maximum level of porosity content linked with the manufacturing effort which can be acceptable for required mechanical performance of the parts. Today, with the extension of composite utilization on high loaded structure, the structures are getting thicker. The previous established criteria need to be updated to maintain an acceptable compromise between mechanical properties drop due to defect, and acceptable manufacturing cost. One of the most important and current defect found in composite materials is the porosity. This paper presents the work done to produce composite material with controlled level of inter laminar and intra laminar porosity and first results on their influence on mechanical properties.

2 Work description

2.1 Material definition

To perform this study, we have chosen a unidirectional prepreg made from epoxy 180°C curing system and intermediate modulus carbon fiber. The matrix density is 1.27 g/cm^3 and the carbon fibre density is 1.8 g/cm^3. The nominal cured ply thickness is 0.256 mm. The resin content in the prepreg is 35% by weight. The polymerization cycle is presented figure 1.

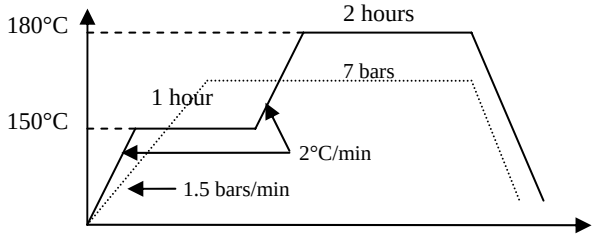


Figure 1 : Composite Curing cycle.

2.2 Artificial porosity creation

In order to create controlled porosity, we have selected 3 solutions to create artificial porosity with

the objective to create intra laminar porosity and inter laminar porosity.

- Expancel. This product is a spherical material made with a PVC membrane full up with isobuthane as presented figure 2. This product has been used to produce inter laminar porosity. Different product quantities have been used, 0.5 mg/cm^2, 1 mg/cm^2 and 1.5 mg/cm^2. A microscopic observation presents the result obtained on composite figure 3.

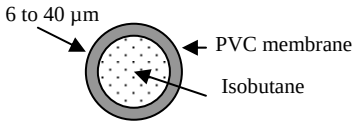


Figure 2 : Expancel micro spherical product.

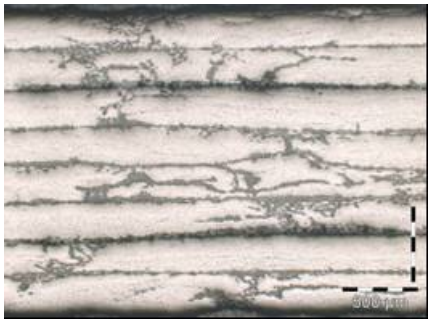


Figure 3 : Inter laminar porosity with 1.5 mg/cm^2 Expancel micro spherical products between 2nd and 3rd plies from each surface of the laminate. Dark area is the porosity, grey area is pure matrix zone. Magnitude x 25.

- A foaming agent DY5054 from Huntsmann has been used to produce inter and intra laminar porosity [1]. Different product quantities have been used to create porosity 5 mg/cm^2, 8 mg/cm^2 and 13 mg/cm^2. A micrographic observation presents the result obtained figure 4.

- The azobisisobutyronitrile (AIBN) is a molecule which degrades with nitrogen production. This product has been selected to produce also inter and intra laminar porosity [2]. Different product quantities have been used to create porosity 3 mg/cm^2, 5 mg/cm^2 and 8 mg/cm^2.
  A micrographic observation presents the result obtained figure 4. The photo figure 4 shows that AIBN product is very efficient to create intra plies porosity in the plies in contact with the surface on which we introduced AIBN. With the same amount of additives, the DY5054 creates lowest porosity content in the laminates.

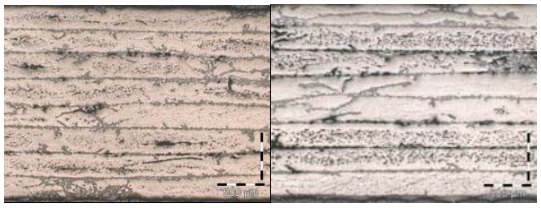


Figure 4 : Inter and intra laminar porosity with 3 mg/cm^2 of DY5054 (left) and 3 mg/cm^2 of AIBN product (right). Magnitude x 25.

2.3 Laminate manufacturing

Laminates with different porosity content from 0% up to 12% in volume have been manufactured with inter laminar and homogeneous distributed porosity.

Two kinds of laminates have been manufactured with the curing cycle presented figure 1 :

- 8 plies +/-45° for shear test.
  The products used to create the porosity are placed between the plies 2 and 3 and between the plies 6 and 7 as presented figure 5.

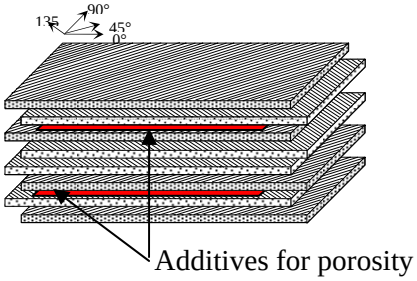


Figure 5 : Distribution of products creating porosity inside 8 plies +/-45° laminates.

- 16 plies quasi isotropic laminates for compression tests.
  The lay up used is :
  45/0/135/90/45/0/135/90/90/135/0/45/90/135/0/45
  The products used to create the porosity are placed between the plies 4 and 5, between the plies 8 and 9 and between the plies 12 and 13, as presented figure 6.

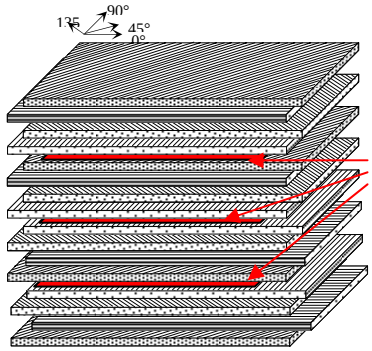
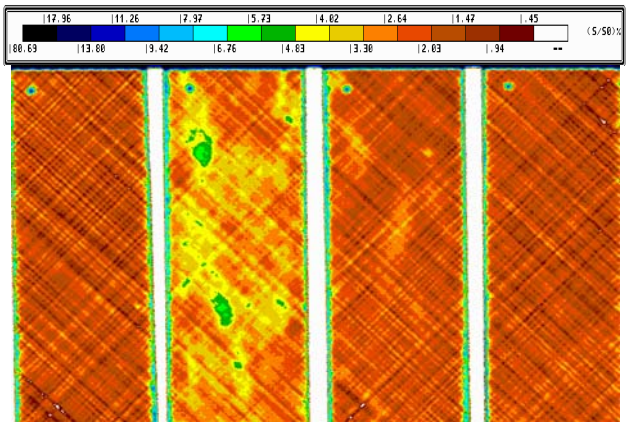


Figure 6 : Distribution of products creating porosity inside 16 plies quasi isotropic laminates.

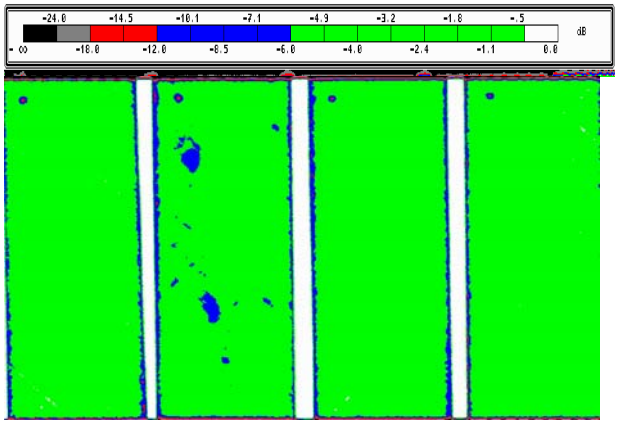
2.4 Quality control of laminates

The quality control of the laminates has been done by Ultrasonic C-Scan to evaluate the surface porosity content [3], [4]



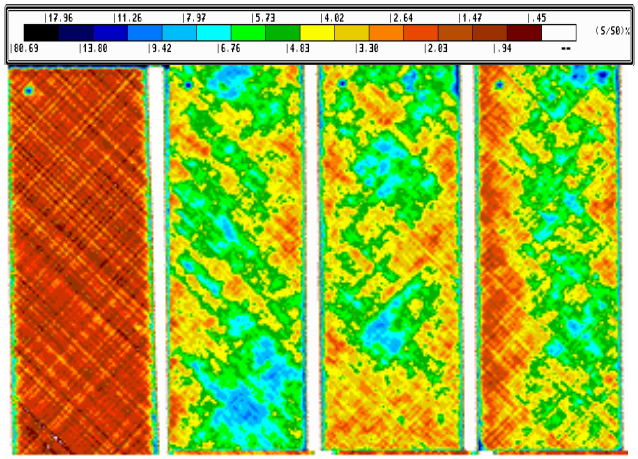
Reference | Expancel 2x1.5 mg/cm^2 | Expancel 2x1 mg/cm^2 | Expancel 2x0.5 mg/cm^2

Figure 7 : Ultrasonic C-Scan on 8 plies laminates with Expancel product.



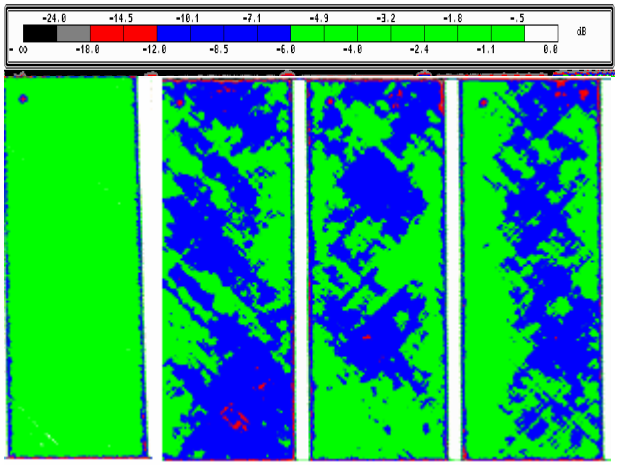
Reference | Expancel 2x1.5 mg/cm^2 | Expancel 2x1 mg/cm^2 | Expancel 2x0.5 mg/cm^2

Figure 8 : Ultrasonic C-Scan on 8 plies laminates with Expancel product. Class exploitation



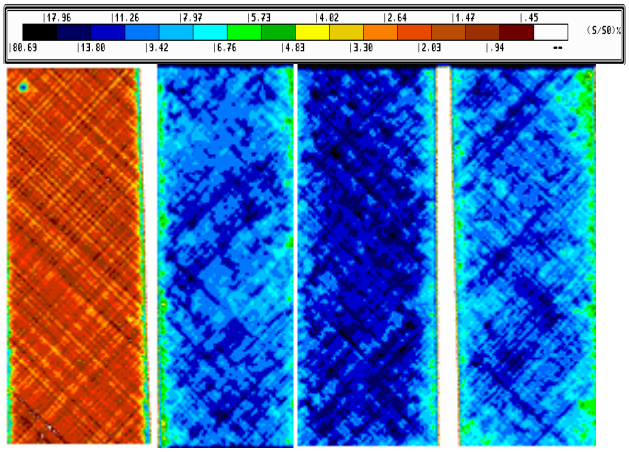
Reference | DY5054 2x13 mg/cm^2 | DY5054 2x 8 mg/cm^2 | DY5054 2x4 mg/cm^2

Figure 9 : Ultrasonic C-Scan on 8 plies laminates with DY5054 product.



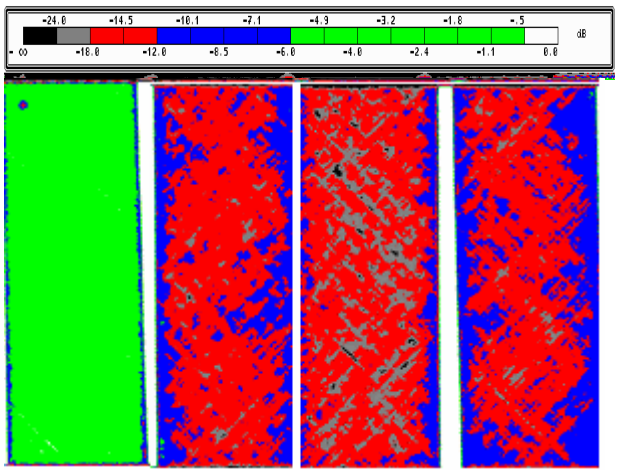
Reference | DY5054 2x13 mg/cm^2 | DY5054 2x 8 mg/cm^2 | DY5054 2x4 mg/cm^2

Figure 10 : Ultrasonic C-Scan on 8 plies laminates with DY5054 product. Class exploitation.



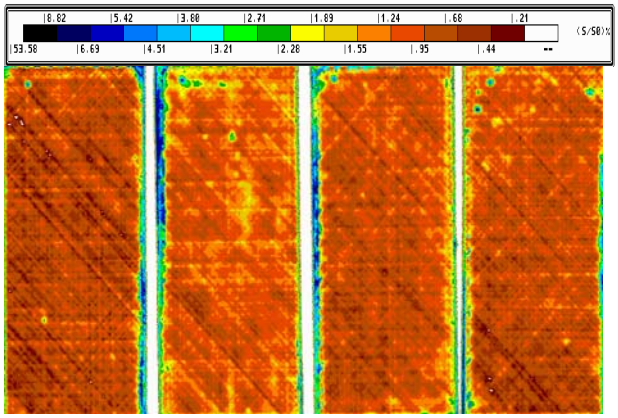
Reference | AIBN 2x8 mg/cm^2 | AIBN 2x5 mg/cm^2 | AIBN 2x3 mg/cm^2

Figure 11 : Ultrasonic C-Scan on 8 plies laminates with AIBN product.



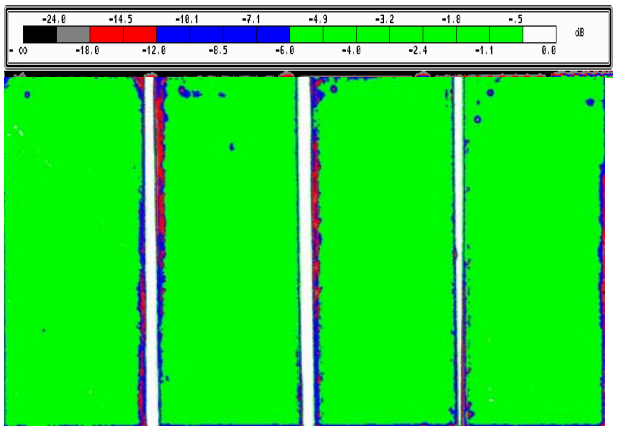
Reference AIBN 2x8 mg/cm^2 AIBN 2x5 mg/cm^2 AIBN 2x3 mg/cm^2

Figure 12 : Ultrasonic C-Scan on 8 plies laminates with AIBN product. Class exploitation



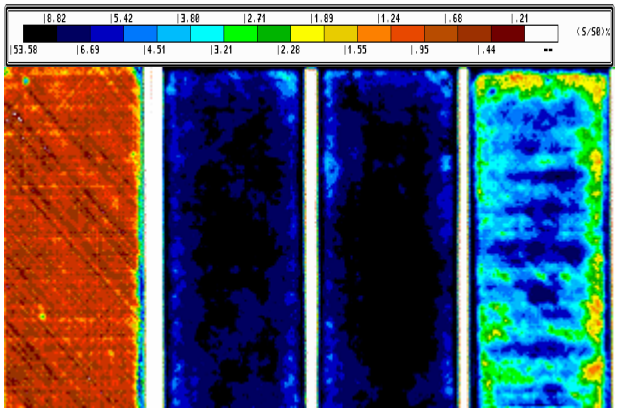
Reference Expancel 3x1.5 mg/cm^2 Expancel 3x1 mg/cm^2 Expancel 3x0.5 mg/cm^2

Figure 13 : Ultrasonic C-Scan on 16 plies laminates with Expancel product.



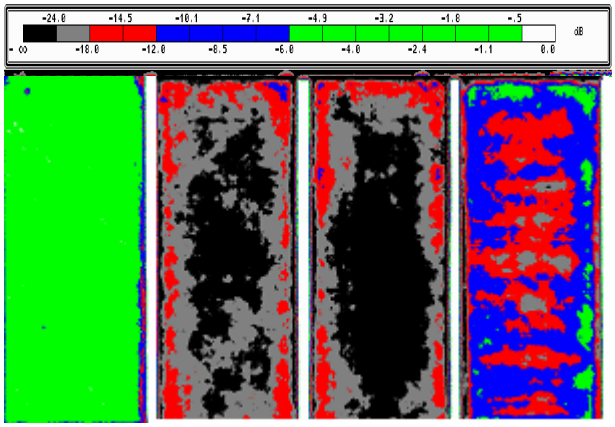
Reference Expancel 3x1.5 mg/cm^2 Expancel 3x1 mg/cm^2 Expancel 3x0.5 mg/cm^2

Figure 14 : Ultrasonic C-Scan on 16 plies laminates with Expancel product. Class exploitation



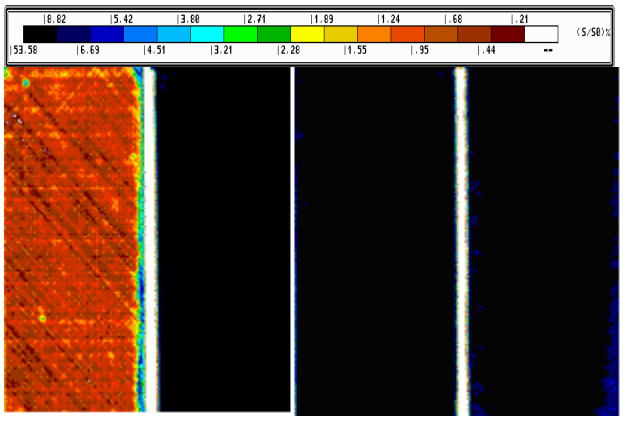
Reference DY5054 3x13 mg/cm^2 DY5054 3x8 mg/cm^2 DY5054 3x4 mg/cm^2

Figure 15 : Ultrasonic C-Scan on 16 plies laminates with DY5054 product.



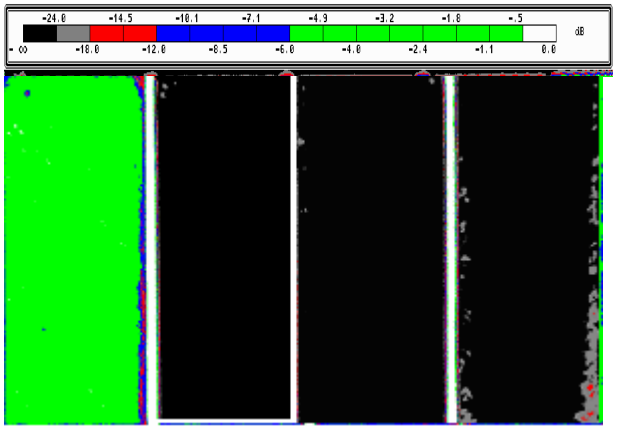
Reference DY5054 3x13 mg/cm^2 DY5054 3x8 mg/cm^2 DY5054 3x4 mg/cm^2

Figure 16 : Ultrasonic C-Scan on 16 plies laminates with DY5054 product. Class exploitation



Reference AIBN 3x8 mg/cm^2 AIBN 3x5 mg/cm^2 AIBN 3x3 mg/cm^2

Figure 17 : Ultrasonic C-Scan on 16 plies laminates with AIBN product.



Reference | AIBN 3x8 mg/cm^2 | AIBN 3x5 mg/cm^2 | AIBN 3x3 mg/cm^2

Figure 18 : Ultrasonic C-Scan on 16 plies laminates with AIBN product. Class exploitation

The C-Scan have been exploited in term of percentage of surface porosity (S/So) as presented figure 7, 9, 11, 13, 15 and 17 or in term of class of quality as presented figures 8, 10, 12, 14, 16 and 18. The quality classes are defined with ultrasonic signal absorption as follow :

* class A : green colour. Absorption 0 to -6 dB
* class B : blue colour. Absorption -6 to -12 dB
* class C : red colour. Absorption -12 to -18 dB
* class D : grey colour. Absorption > to -18 dB

2.5 Porosity determination on laminate

The porosity content by volume has been determined on laminate by density measurement and acid ingestion (figure 19). The results obtained for the evolution of porosity content by volume versus composite density fits perfectly with a linear law. The correlation coefficients are superiors to 0.99.

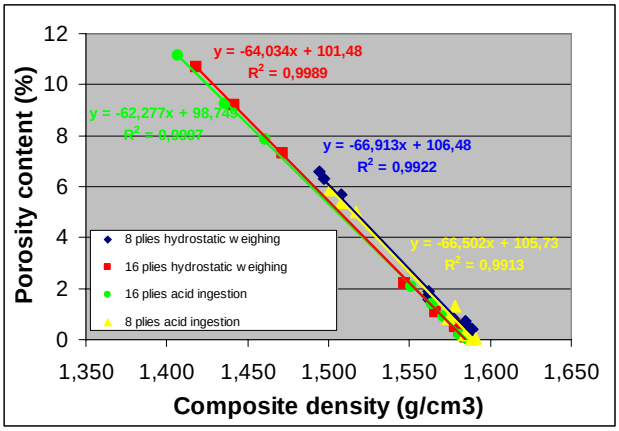


Figure 19 : Porosity content by volume on composite versus composite density.

Porosity content by volume determined by hydrostatic weighing is comparable to porosity content by volume determined by acid ingestion (figure 20). Material parameters such as resin density, fibre density, are very important on the obtained results and these values need to be known with a high degree of confidence. If we determine the porosity content by volume with density measurement only, we also need to know with precision the dry fibre area weight of the prepreg to minimize the error.

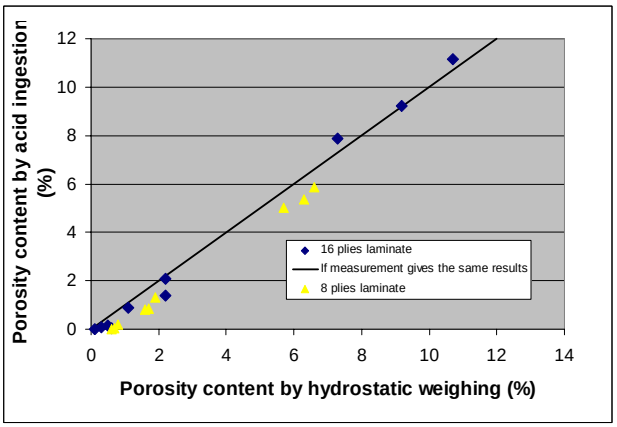


Figure 20 : Porosity content by volume on 8 and 16 plies laminates

The evolution of porosity content by volume on laminate with the global Expancel content in the laminate is presented figure 21. One point with the 16 plies laminate determined by hydrostatic weighing is not correlated with the other measured points. An explanation could be that in this range of low porosity content by volume, the results strongly depend on material data as explained previously. The porosity content by volume created with Expancel is very low (inferior to 1%) but concentrated in the inter plies area. We do not have any diffusion of the micro spheres inside the plies as we can see on figure 3.

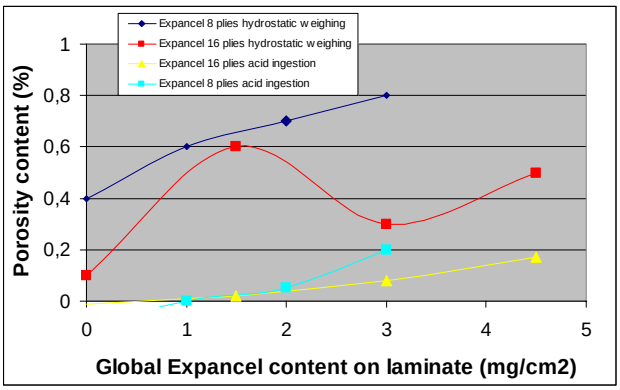


Figure 21 : Evolution of porosity content by volume versus Expancel content on laminate.

The evolution of porosity content by volume on laminate with DY5054 content is presented figure 22. On the 16 plies laminates the obtained results between hydrostatic weighing and acid ingestion are very closed except one point on the laminate with 24 mg/cm^2 of DY5054. On the 8 plies laminates the obtained results gives the same evolution with hydrostatic weighing and acid ingestion, but with a recurrent difference of about 0.6% in the porosity content by volume. For the porosity content by volume determined by hydrostatic measurement, if we modify the dry fiber area weight by 6g/m^2 for a mean value of 268g/m^2, we could explain such a difference. The porosity content by volume evolution is approximately linear with the quantity of DY5054 added in the laminate. This product has been used to create porosity distributed in all the composite with a porosity content by volume between 1% and 3%.

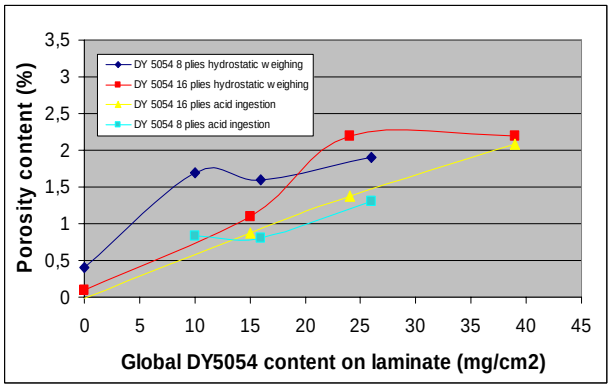


Figure 22 : Evolution of porosity content by volume versus DY5054 content on laminate.

The evolution of porosity content by volume on laminate with AIBN content is presented figure 23. The obtained values by hydrostatic weighing and acid ingestion are very closed. The AIBN product has been used to create porosity in all the composite with a porosity content by volume between 6% and 11%. For the global AIBN content in laminates between 5mg/cm^2 and 25 mg/cm^2, the evolution of porosity content by volume is proportional to the AIBN content.

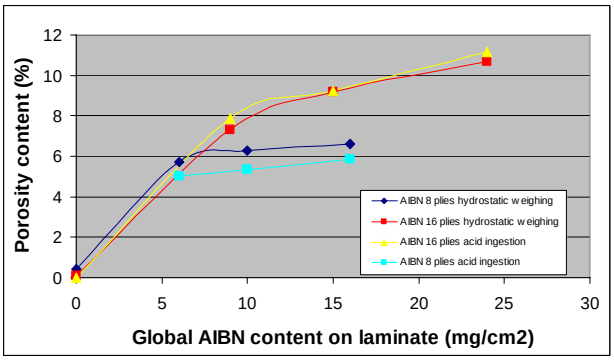


Figure 23 : Evolution of porosity content by volume versus AIBN content on laminate.

The obtained results on porosity content are in good correlation with the ultrasonic C-Scan results presented in 2.4.

2.6 Mechanical properties measurement

The influence of the porosity content by volume on the mechanical properties has been evaluated with shear tests and compression tests. These tests have been selected because the obtained results are strongly dependent on the material quality (porosity content by volume) [5], [6], [7], [8], [9]

2.6.1 Shear test (tensile+/-45°)

The shear tests have been performed according to AITM 1.0002. The sample geometry used is presented figure 24. The coupons have been machined in 8 plies laminates.

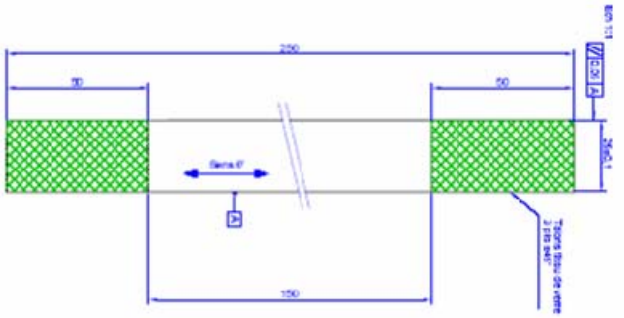


Figure 24 : Sample geometry for tensile +/-45° test according to AITM1.0002.

The results obtained are presented figure 25 for the evolution of stress to failure versus porosity content by volume and figure 26 for the evolution of modulus versus porosity content by volume.

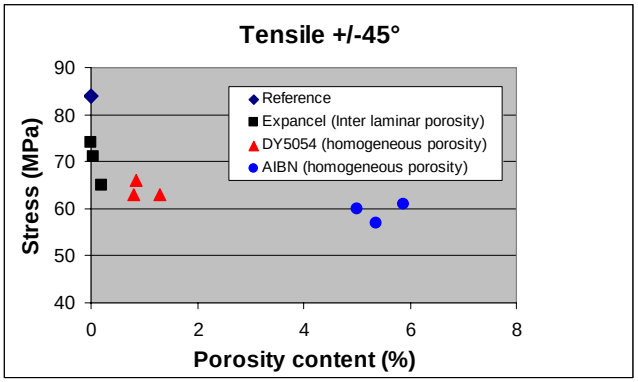


Figure 25 : Evolution of shear property versus porosity content by volume on composite laminate.

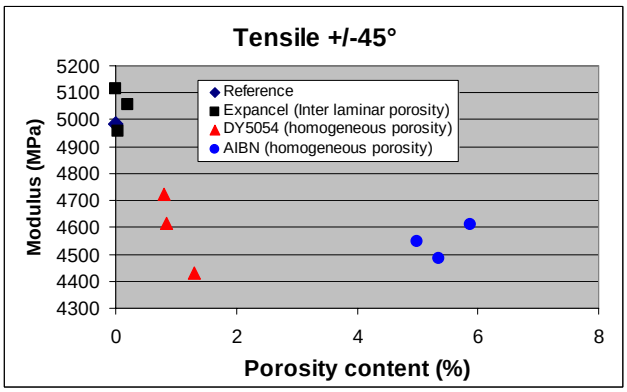


Figure 26 : Evolution of shear property versus porosity content by volume on composite laminate.

The mechanical properties degradation with porosity content by volume is very effective from 0 to 2% of porosity content by volume for tensile +/-45° test for stress at failure and modulus. Over 2% of porosity content by volume the property reduction is stable and is about 25% reduction for stress at failure and about 10% reduction for modulus.

With the obtained results, we note a strong dependence with porosity of the shear properties stress at failure if the porosity is located at the plies interface (Expancel additive) and for a low porosity content level (figure 25). Less than 1% of porosity content by volume located at the inter plies area induce about 25% property reduction for the stress at failure with tensile +/-45° test. For the tensile +/-45° modulus, the presence of 1% of porosity at the inter plies area do not modify the modulus value. But as we obtained with stress at failure, the presence of 2% to 6% of distributed porosity in the laminate induce a constant decrease of the modulus of about 10%.

2.6.2 Compression test on quasi isotropic laminate

The compression tests have been performed according to AITM 1.0008. The sample geometry used is presented figure 27. The coupons have been machined in 16 plies laminates.

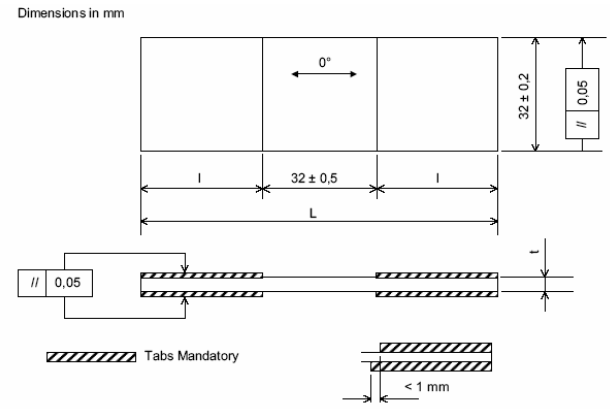


Figure 27 : Sample geometry for compression test according to AITM1.0008.

The results obtained are presented figure 28 for the evolution of stress to failure versus porosity content by volume and figure 29 for the evolution of modulus versus porosity content by volume.

With the compression test, the evolution of mechanical properties is more related to the evolution of porosity content than with tensile +/-45° test. In the range of 0 to 10% of porosity, we may propose a linear evolution of the compression modulus versus porosity content by volume with a coefficient correlation of 0.915. The modulus decreases is about 0.56 GPa per percent of porosity content by volume.

For the compression stress to failure, the property evolution is not so linear.

To compare with the tensile +/-45° test we can estimate that for 6% of porosity content by volume the modulus decreases is about 9% (-8% for tensile +/-45° modulus)

We can also estimate that for 6% of porosity content by volume, the stress at failure deceases is about 16% (-25% for tensile +/-45° stress at failure.

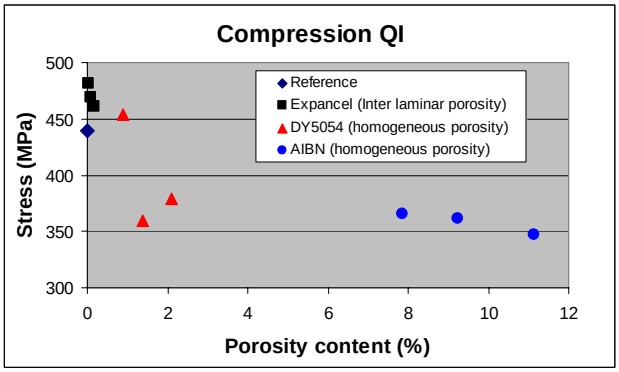


Figure 28 : Evolution of compression property versus porosity content by volume on composite laminate.

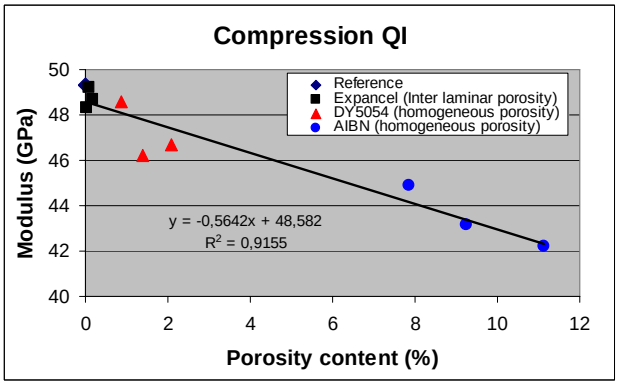


Figure 29 : Evolution of compression property versus porosity content by volume on composite laminate.

3 Conclusion and perspectives

We have validated experimental procedure to create artificial controlled porosity in composite laminates, (inter laminar porosity or homogeneous distributed porosity in the laminate). We have established existing correlation between porosity content and mechanical properties. The porosity distribution inside the laminate (porosity at the plies interface or homogeneous distributed porosity) plays an important role on the final mechanical properties. These first results will help to understand the influence of porosity content and distribution on thick composite structure to relax if possible quality requirement and manufacturing procedures [3], [4] [12], [13] that have been established for thin composite structures.

References

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