Introduction to the characterization of hygrothermal microcracking of composites reinforced by stitched non-woven UD laminas

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ABSTRACT

This article presents a first approach of hygrothermal microcracking in stitched laminates. The presentation includes the definition of the material mesostructure and the selection of a characterization method adapted to the specificity of the study. The hygrothermal loading selected is discussed and the evolution of the crack density in stitched composites during the loading is presented and commented.

1. INTRODUCTION

New high performance composites are presently being developed for advanced aerospace applications. These materials aim to minimize preconditioning to ease processing and decrease storage and raw material costs. In this study, we focus on composites reinforced by stitched non-woven unidirectional carbon fiber laminae. These products are non-crimped, allowing taking full advantage of the high stiffness of the carbon fibers, which represent the main part of the raw material cost. These kinds of reinforcements can be utilized in LCM processes – injection, infusion - aimed to lower manufacturing costs compared to prepregs (easier storage, higher throughput,…). However, the stitching introduces new local parameters into the laminate morphology, which can induce some effects on the durability of these materials. Our objective is to study the impact of those heterogeneities due to the stitching on the behaviour of the composite during hygrothermal aging. This research is of particular interest to the aerospace industry since aircraft materials are often submitted to harsh temperature and moisture conditions. These effects are known to create microcracking. Hence, efficient and reliable techniques must be developed to characterize the microcracking of a complex three-dimensional material mesostructure.

2. MATERIALS

This type of material is composed of several laminas with different orientations stitched together. The stitching has no mechanical property and is simply used to ease the handling and the processing of the reinforcement (figure 1). The stitching yarns introduce ellipsoidal perturbations in the fiber alignment [1]. These perturbations have different size and shape according to the yarn size, the stitching type and the plies orientations. Nevertheless, these elliptical perturbations have some interest since they serve as channels to facilitate resin flow during resin infusion [2]. However, after processing, these perturbations become resin rich inclusions in the composite. In most cases, the final laminate is composed of several layers of stitched plies. For example, the stitching process allows assembling several plies into stitched stacks of two to four plies per stack. So, a twelve plies laminate will be composed of three to six stacks, each having a different resin inclusion distribution. Moreover, the stitched plies are not oriented in the same way. The ellipsoidal inclusion rotates with the fiber orientation [3] (figure 2). So it becomes a periodic three-dimensional inclusion. The spatial period is known for each individual stack, but in an assembly it becomes impossible to control the relative inter-stack offsets.

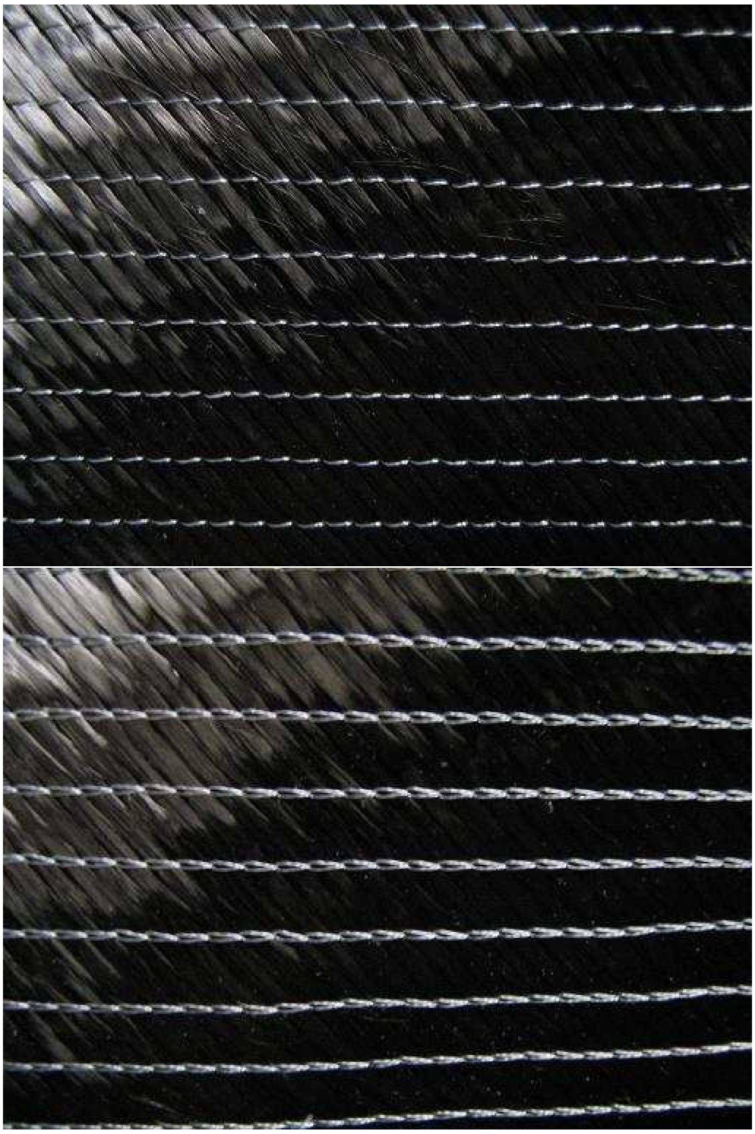

Figure 1 : Picture of the two side of a stitched stack : the I side (up) and the loop side (down)

3. HYGROTHERMAL LOADING

The aging of such materials has not been yet studied. We managed to study a damaging loading related to extreme value of service conditions. The temperature –55°C is considered as representative of the lowest temperature during a subsonic flight. Such a low temperature must be included in the loading because it is probably the most critical parameter leading to residual stresses. To ease the modeling of the sample state, we decided to split the humidification and thermal periods instead of controlling humidity cycles which induce transient humidity profile at each cycle. We selected for this study the hygrothermal loading defined by Klug [4]. This aging loading, presented in figure 3, is divided into 5 blocks with a humidification period at 50°C and 95%RH and 400 thermal cycles of an hour between 80°C and –55°C with a 15 min step at these two temperatures. Five samples of the same material are tested. Between each blocks, one sample is selected to analyze several stitching lines.

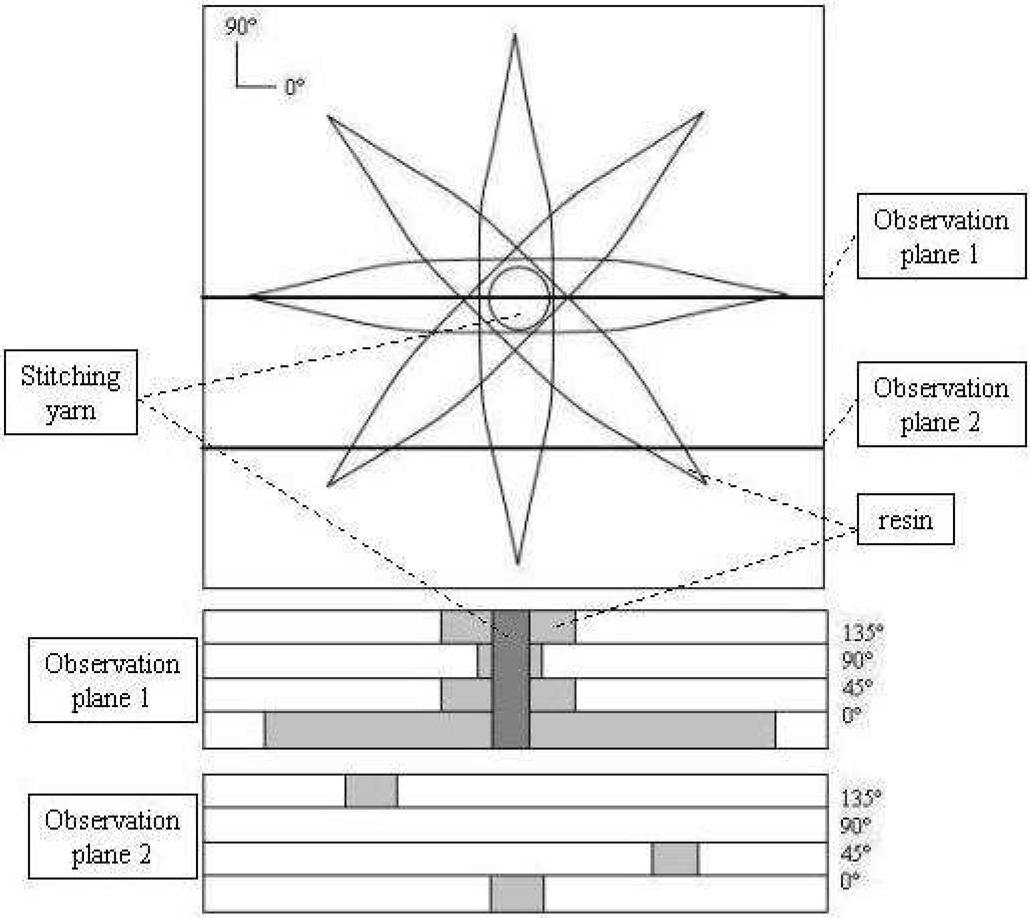

Figure 2 : Resin inclusion representation

4. CHARACTERIZATION

4.1 Analysis method

To study and characterize the stitching area, we select optical micrography since it is the only direct method to look inside the material [5]. Most of the studies on composite cracking limit their observation to the edge of the specimen [5-8]. In this study due to the presence of stitching, the specimen edges are not necessary representative of what occurs inside the material. Hence, it is necessary to observe the specimen in different locations, which means that a careful analysis is required to select relevant areas previously. To solve this problem, we have two possibilities, we can cut the specimen in order to force the stitching to be on the edge of the sample or we can investigate internal stitching yarns. In the first approach, the results could not be representative because the stress field will be perturbed at the free edge. This approach requires identical stress fields for all specimens, which implies a perfect control of the edge position with respect to the resin inclusions.

To avoid these problems, we have selected the observation of the internal stitching yarn. A method has been developed to control the location of the observation plane.

First we set up a protocol to achieve a suitable surface state for optical micrography measurements minimizing the preparation time. Then we cut the sample at a distance equal to or higher than the region consumed by the initial abrasion process. The cut is performed with a circular SiC blade. The cut is also done by abrasion as to not introduce extra-damage. During polishing, we estimate the volume consumed by the process and we continue to polish the surface until we reach the observation plane. This method allows for an accuracy of plane localization up to 20 µm and a surface status suitable for optical observations (figure 4).

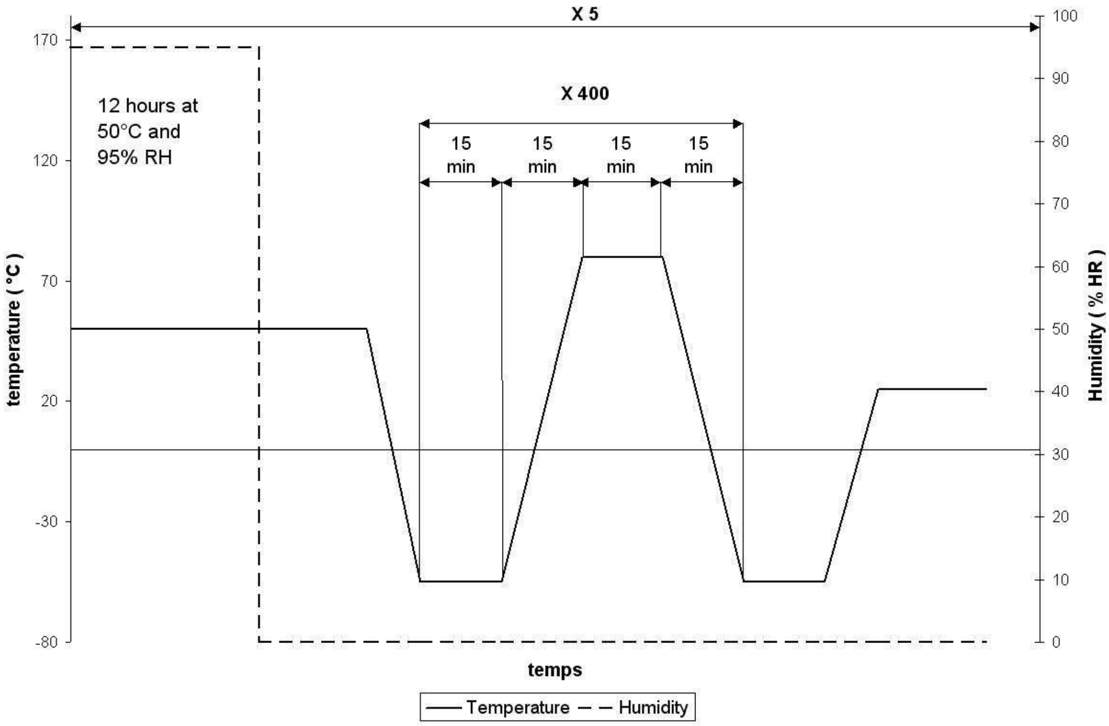

Figure 3 : Hygrothermal loading

4.2 Microcracking quantification

We are able to follow the evolution of the microcracking state near the stitching with the method previously described. To quantify the microcracking, we define a parameter, d, characterizing the crack density of the laminate, suited to our protocol :

$$d = \frac{\sum_i N_i}{L \times p}, \tag{1}$$

where N_i is the total number of cracks in ply i [9], L is the sample length and p is the number of plies observable in the considerate observation plane.

As mentioned previously, the stitching will create inclusions of hydrophilic resin; hence the stitching may influence the water concentration profile of a specimen. The inclusion could be a preferential diffusion path for the transverse direction. In addition it is possible that the residual stress field within the laminate is perturbed by the inclusions, which are unreinforced areas.

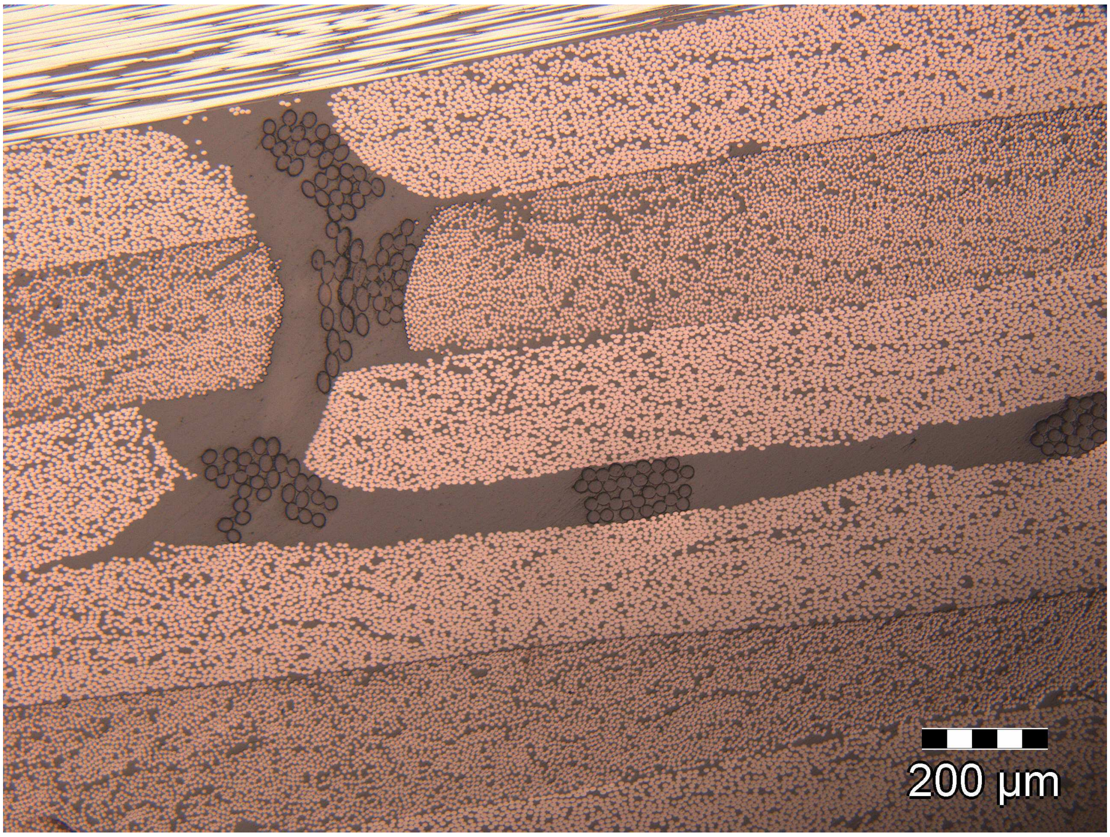

Figure 4 : Optical micrography example

5. RESULTS

5.1 Results on stitched crossply laminates

For the first test on stitched laminate, we decided to emphasize the specific properties of this material. We managed to have very large inclusions in the tested sample. Moreover, to simplify the characterization of the cracking paths, we choose two crossply laminates: $[90/0/0/90]_S$ and $[135/45/45/135]_S$ laminates, where the stitching line is oriented at 0° and the plies are linked four by four. The crack density at different loading times is shown in figures 5 and 6 for the two laminates respectively. We have conditioned two series of samples: the first one is submitted to the complete loading history and indexed *wet* and the second subjected to the thermal cycles only and indexed *dry* (figure 5-6).

It seems that no significant difference can be detected between the crack densities of the dry and wet specimens, meaning that moisture conditioning is not so critical. This conclusion is even more obvious in figure 6.

These results also prove the importance of the p parameter in equation 1. Indeed, the comparison between two laminates with different stacking sequences becomes possible.

If we compare the two materials, we can see that the $[135/45/45/135]_S$ laminate tends to a lower crack density than the $[90/0/0/90]_S$ laminate. The second one reaches a value around 3,5 cracks/cm although the first seems to converge to 2,5 cracks/cm.

This is due to the inner material morphology. $[90/0/0/90]_S$ laminate presents resin inclusions close to the channels linking the stitching points. The resin inclusions of the second laminate are smaller and not linked to one another (figure 7). The difference of behaviour to hygrothermal loading can be attributed to this morphological difference. Thus the internal stress profile and the crack propagation properties are very different in these two materials and could lead to some difference in the crack density evolution during hygrothermal damaging.

To confirm the influence of the morphology on hygrothermal microcrack density, we studied quadriaxial laminates.

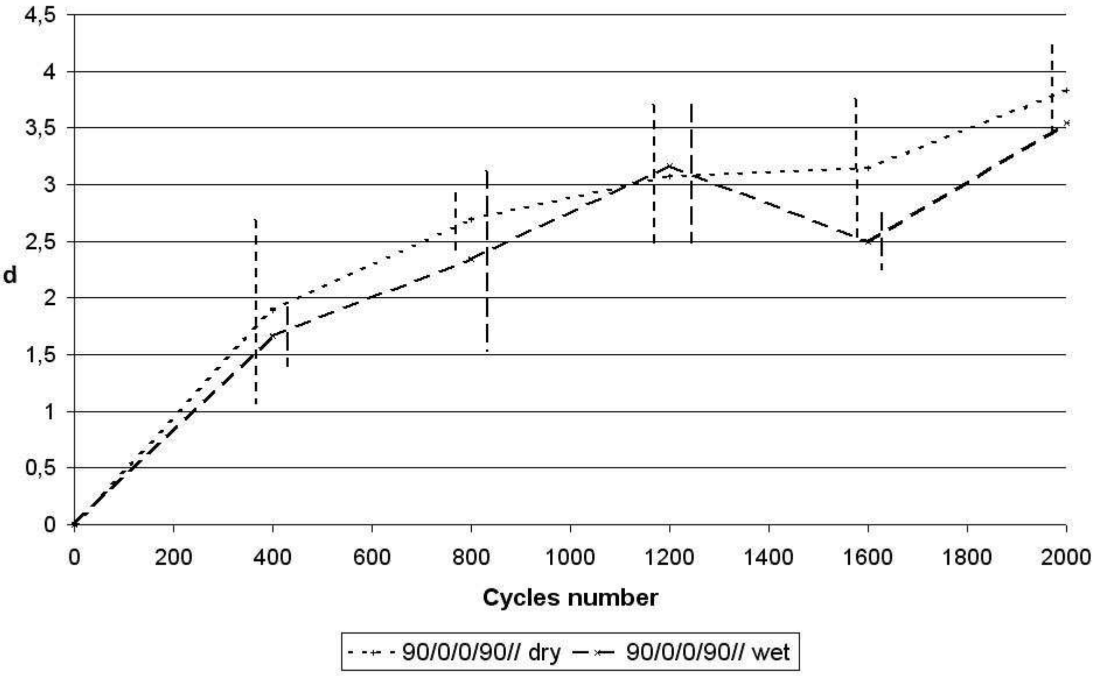

Figure 5 : Crack density evolution during cycles on crossply 90/0/0/90// laminates

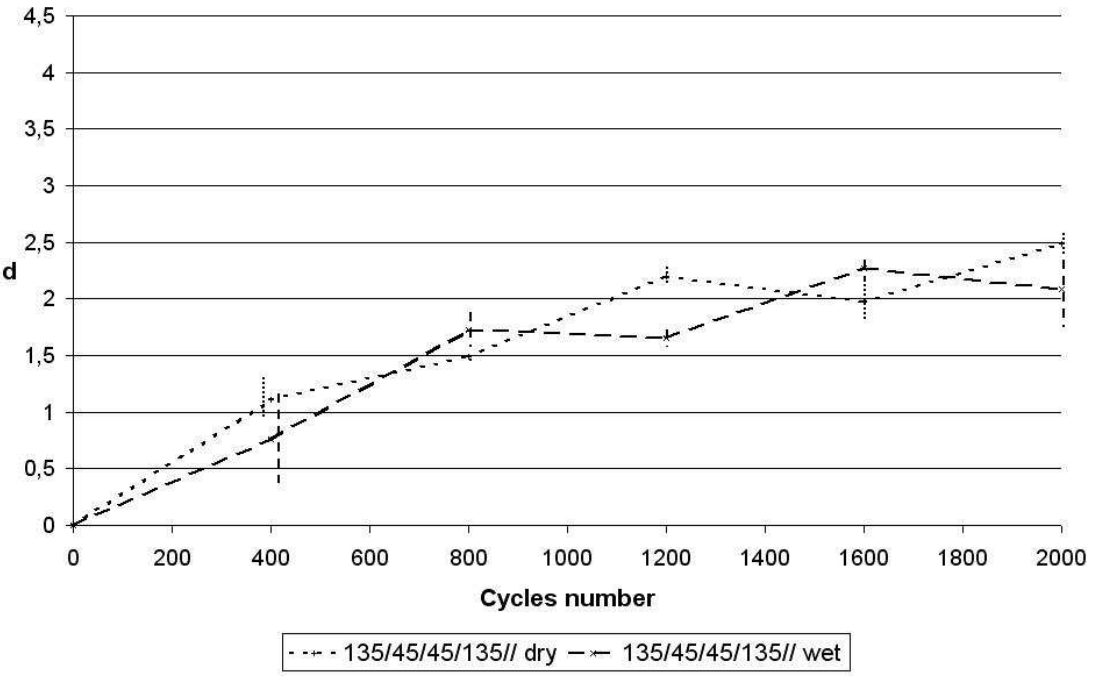

Figure 6 : Crack density evolution during cycles on crossply 135/45/45/135// laminates

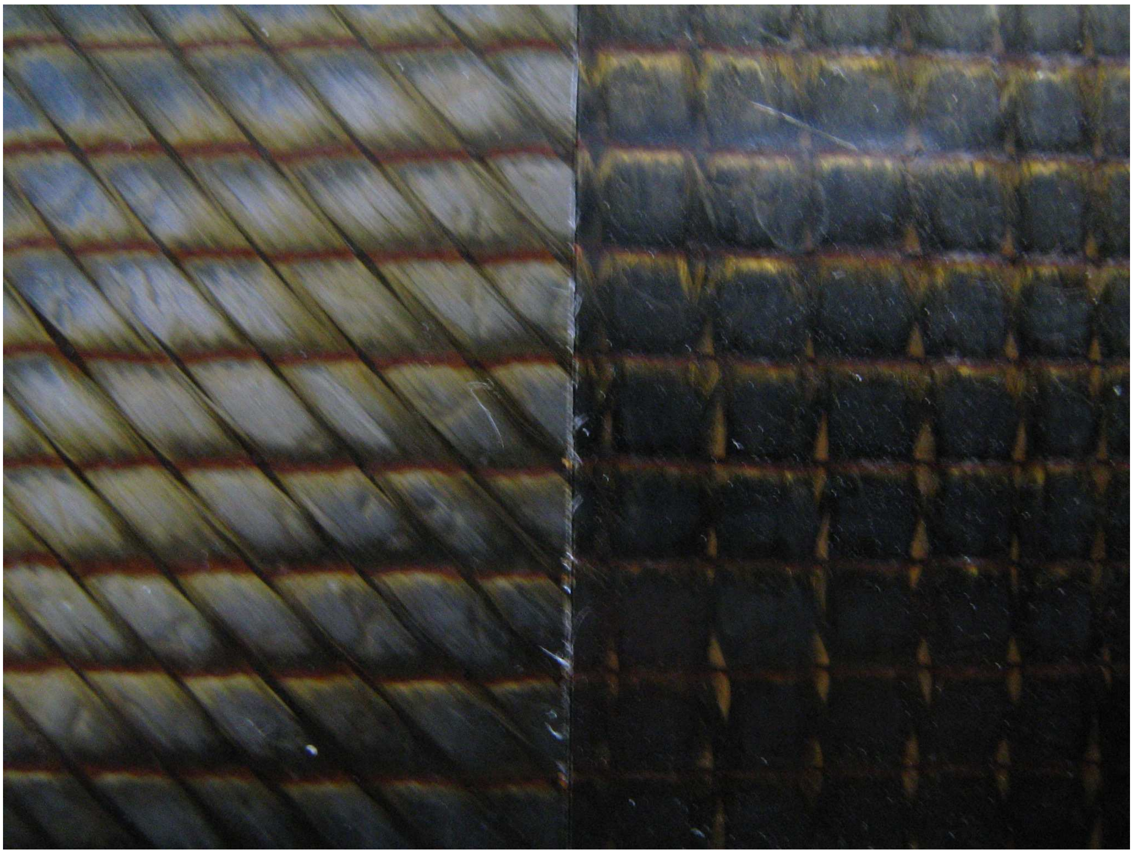

Figure 7 : Comparison of resin inclusions between [135/45/45/135]$_S$ laminate(left) and [90/0/0/90]$_S$ laminate (right)

5.2 Results on stitched quadriaxial laminates

In quadriaxial stitched laminate, the resin inclusion reveals its most complex shape. The main originality associated to this morphology is that the inclusions lead to crack reorientation. The crack propagation in quadriaxial quasi-isotropic composite is modeled and is known to cause delamination before crack jump of one ply to another with different orientation. The small angle between two consecutive plies and the isotropic properties of resin inside the inclusions make easier the creation of three dimensional crack networks.

The effect of this originality is revealed by an erratic crack density evolution. As we can see in figure 8, the d parameter decreases when the number of cycles increases. This results from the averaging of the parameter on several samples. The main causes of the scatter could be the uncertainty on the observation plane position or the non-repeatability of the crack propagation in complex three-dimensional material.

The first hypothesis has been invalidated by a 3D investigation by using an X-ray microtomography. We studied several plies close to the stitching area and find that the uncertainty of observation plane position cannot explain the complete variation of the crack density evolution. The study of the repeatability of the microcracking on several sample is at the moment in progress. We already demonstrate that the scatter is more significant near the sample edges. These results confirm our choice to work far from edge effects.

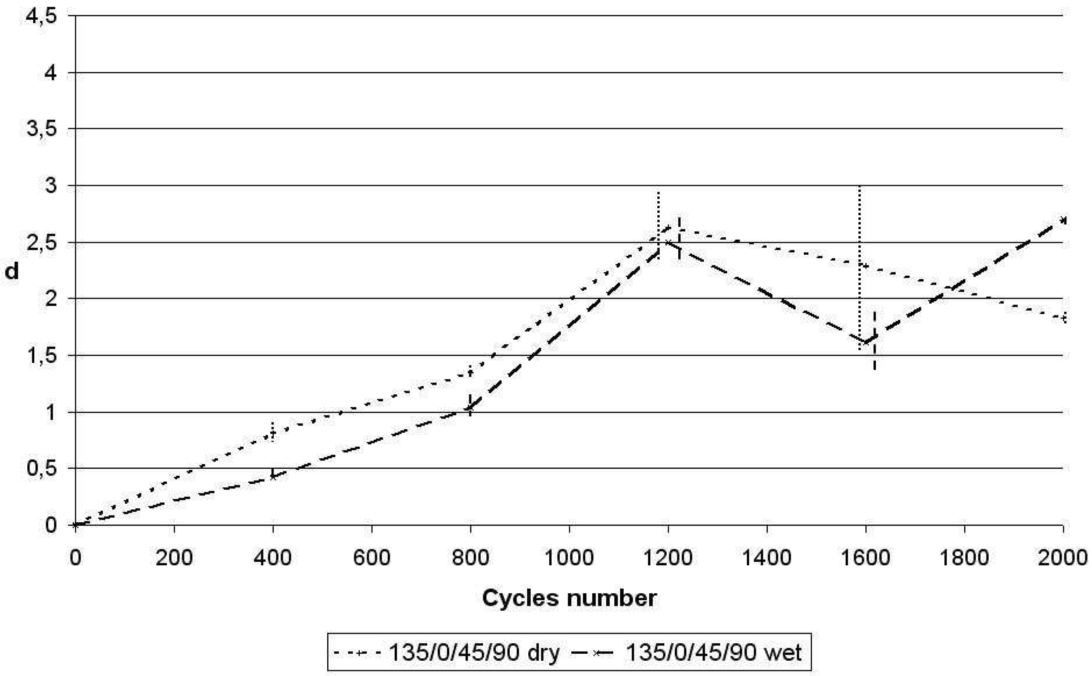

Figure 8 : Crack density evolution during cycles on quadriaxial quasi-isotropic laminates

6. CONCLUSIONS

This article proposes a protocol to investigate the resin inclusion morphologies in stitched laminate and an extension of existing methods of crack characterization. The principal result is that some specific microcracking occurs in this material and that it is possible to characterize it independently of the stacking sequence or cutting plane orientation. This could lead to a new approach in composite microcracking analysis.

The tests with and without pre-humidification period show that the influence of moisture on the crack density cycle is not significant for this material for this particular cycle.

We are now extending the study to different types of stitching yarns which could have a complex effect on crack initiation and material morphology which influence crack propagation. A better knowledge of crack initiation will be derived from the mechanical modeling and interfaces investigation and crack propagation by X-ray microtomography imaging.

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REFERENCES

1- S.V. Lomov et al. “Carbon composites based on multiaxial multiply stitched preforms. Part 1 : Geometry of the perform”, Composite : Part A, vol 33, 2002, p.1171-1183.
2- S. Drapier, A. Pagot, A. Vautrin, P. Henrat, “Influence of the stitching density on transverse permeability of non-crimped new concept (NC2) multiaxial

reinforcements : measurements and predictions”, Composite science and tech., vol 62, 2002, p.1971-1991.

3- Heoung-Jae Chun, Hyung-Woo Kim, Joon-Hyung Byun, “Effects of through-the-thickness stitches on the elastic behavior of multi-axial warp knit fabric composites”, Composite Structures, vol. 74, 2006, p.484-494.

4- J.H. Klug, 1999. “High-performance adhesive systems for polymer composite bonding application”, PhD thesis, University of Washington.

5- Hayes, B.S.and Gammon L.M. , “Composites”, volume 21, ASM Handbook, 2001, p.964-972.

6- F. Gao et al., “Damage accumulation in woven-fabric CFRP laminates under tensile loading : Part 1. observations of damage accumulation”, Composite science and tech., vol 59, 1999, p.123-136

7- C.H. Park, H.L. McManus, “Thermally induced damage in composite laminates : predictive methodology and experimental investigation”, Composite science and tech., vol 56, 1996, p.1209-1219

8- Y.J. Weitsman, Ya-Jun Guo, “A correlation beetween fluid-induced damage and anormalous fluid sorption in polymeric composites”, Composite science and tech., vol 62, 2002, p.889-908.

9- Reynolds, T.G. and McManus, H.L. “Accelerated Tests of Environmental Degradation in Composites Materials”, Composites Structures : Theory and practice, ASTM STP 1383, P. Grand and C.Q. Rousseau, Eds, American society for Testing and Materials, West Conshohocken, PA, 2000, pp.513-525.