

Effect of out-time aging in composite prepreg material

Towards a testing methodology for material properties characterization

João Pedro Martins de Silva Luis

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In the aerospace industry, values as sustainability and social impact have become nowadays as relevant as the product direct cost, demonstrating the companies' operation footprint. Within this framework, two main challenges - operational and environment costs - have a common root cause: fossil fuel consumption. One way to address this is through aircraft weight reduction. In this regard, CFRPs, due to their high strength to weight ratio, have found a growing presence in aerostructures. This increased use has brought new challenges to the industry, namely those of properties degradation during out-time exposure affecting the pre-impregnated variety of composites (prepregs).

With the aim to improve resources usage regarding the use of prepregs in an industrial scenery, the purpose of this project is to characterize prepreg material during out-time aging and propose an optimized suitable testing methodology to evaluate it in this condition. This will be accomplished through evaluation of mechanical, physical-chemical and processability tests.

The testing and specimens production were addressed following standardized methods to ensure data reliability and repeatability. Epoxy impregnated prepreg samples were aged to a maximum of 60 days and, with a periodicity of 6 days, they were submitted to tack, drape, solubility, flow, void, ILSS, tensile, impact, CAI and DMA tests.

The DMA and flow proved to be the most advantageous tests and a proposal for an optimized evaluation methodology was made. Although processability degradation was noticed, the material withstood the aging with minor changes in other properties, raising the possibility of future re-utilization.

keywords: *Prepreg, Epoxy, Out-time, Aging, Testing methodology, Resources optimization.*

1 Literature review

Nowadays, mainly due to globalization and international regulatory authorities together with popular organizations, additional factors have compounded the ways companies ascertain where the "value for

money" is when making decisions. Among others, sustainability and associated cost reduction issues have become huge concerns. Within this scope, one of the biggest challenges tackled by the industry is the reduction of fuel consumption. This, besides environment considerations, also represents a substan-

tial part of the airliners operating cost. Apart from new, more efficient engines or energy sources, another way to lower fuel consumption is by aircraft weight reduction. In this regard, the use of composite materials, especially carbon based reinforced plastics (CFRP), is one of the most promising because of their higher strength-to-weight ratio.

Composites consist, usually, in a reinforcement, fiber, which is the main responsible for the material mechanical properties, and a matrix, resin, which has poorer mechanical properties but joins the fibers into a solid material and transfers possible applied loads to the fibers. According to the matrix nature, fiber reinforced composites can be classified as thermoplastics or thermosets. Thermoplastics can be molded under high temperature and solidify at ambient temperature. Thermosets are moldable at ambient condition and become solid when cured under temperature. Besides the opposite relation with the temperature, another major difference is that thermoplastics can be reprocessed several times while thermosets, once cured, never return to their original state, i.e., the cure reaction is irreversible. F. Lucas and B.G.S.E.M. [12] This disadvantage is, however, balanced by their superior properties which lead to their dominance in aerostructures. [4]

Under this context, the present project will address thermoset composites pertaining their cure irreversibility and respective consequences in the usage of epoxy prepreg material. In the prepregs the fiber reinforcement is pre-impregnated into the resin matrix becoming ready to use in the lay-up of parts. However, this type of material is no longer in a stabilized condition, known as stage A, but that the cure reactions are already in progress, i.e., stage B. When completely cured, the material attains again a stabilized stage known as stage C. This creates what is called a perishable condition, has the material cannot be further processed in the stage C. To extend the

prepreg life and safeguard its short usable time period, stage B, for the production processes, prepregs are normally stored under freezing conditions, -18°C , where the resin cure reaction is slowed down to residual levels. The time that the material stays at exposure above -18°C and, therefore, accelerating cure reactions, is known as out-time. e. This period of exposure is known as out-time. If the cure reactions run their full course the material can no longer be used for production and it is scraped. [15, 1] The cure reactions development is highly dependent on the exposure time and temperature. The out-time exposure could be understood as a pre-cure at room-temperature before the material be properly cured in the autoclave. The exposure at room temperature during long periods of time will promote polymerization and, at the same time but with much lower rates, cross-linking, affecting molecular mobility. Depending on the out-time period, cross-linking can increase beyond limits that will hamper the molecular mobility during the final cure process and isolate active groups without any connection. In this case, the degree of cure will decay and the material properties will be affected. Ratna [16], Bilyeu et al. [2, 3]

The limits on out-time for CFRP prepreg material frames the motivation for this work with a twofold purpose: first, to contribute towards an evaluation methodology to characterize different out of validity prepreg samples regarding their mechanical, physical-chemical and processability properties. Second, to add information on the out-time behavior and characteristics of the material tested during the execution of the work.

A solid understanding about the relation between the material out-time and its specific properties is a key aspect in the optimization of use and minimization of waste. Thus, several tests will be applied to material samples with different out-time exposures and its effectiveness in detecting material properties modifi-

cations will be evaluated leading to a suggestion of a group of tests to be carried out.

The battery of tests to be studied includes not only those covering mechanical and physical-chemical properties but also those of processability, an equally critical aspect for the successful use of the material.

2 Material sample

The material used in this project was prepreg carbon fiber tape with an 8552 epoxy matrix. An artificial and controlled aging plan was developed that on one hand, had sufficient resolution to detect a change in the material but on the other hand, allowed a sufficient aging level for that change to occur. All the aging process was promoted in the clean room environment with a temperature average within 20.4°C - 23.4°C and 25% - 67% of relative humidity.

Not knowing in advance the material behavior, it was decided to define an equal time interval between the samples of six days. This interval was chosen by considering approximately half of the standardized material out time limit, 11 days [13], as a sufficient period for the detection of properties variation. The linear sampling allows to get homogeneous data over the time that may be taken as a start base for future adjustments accordingly to the future needs. The six days intervals lead to a total aging time of 30 days which, added to the five days that came with the material when received, result in 35 days of out-time for the last sample to be tested.

According with the testing standards and the available resources, the sampling of the material was made and six samples, named "kits", were stored individually in perforated plastic bags. All the material initiated its aging process simultaneously and then, with a periodicity of six days, the required quantity for one kit was stored in the freezer, stopping its aging.

Regarding the specimens for testing, the uncured specimens were directly cut from the material sam-

ples while for the cured ones, lay-up, cure and cutting processes were needed. These processes had to be harmonized within the factory environment so to cause a minimum impact possible in the production rates.

The lay-up was made by hand, hand lay-up, using a steel plate as mold. During this process several parameters were controlled in order to minimize the differences between laminates and consequent influences in the tests results. Between others, orientation, stacking, foreign objects intrusion, compaction, etc..

The laminates were autoclave cured according to the cure cycle stated by the NCAMP material qualification standard. [14] After cure, the parts were demolded, inspected through ultrasonic imaging and, without any compromising defect, sent for trimming.

To ensure the necessary dimensional tolerance and faces smooth finishing, it was decided to cut all the specimens using a CNC water jet machine. This decision implied the learning of CNC programming skills but due to the number of specimens needed it was the most sensible option.

3 Testing

The testing program was conducted by international standards and supported by the Embraer industrial know-how. The standardization of the results, besides mandatory in any certification procedure, becomes a focal point to ensure results reliability and repeatability.

Known that the matrix is the most affected component by external aggressions such as the temperature during out-time condition Miracle et al. [15], the methodology of work was oriented to conduct tests where the results are more dependent from the matrix than from the fiber properties.

In order to completely characterize the material, besides mechanical and physical-chemical tests, pro-

cessability tests were performed.

3.1 Processability

The processability tests were useful to ascertain the ability of the material to be used during lay-up processes. They consisted in a tack and drape test. Tack testing evaluates the adhesion capacity of the prepregs. Without tack the layers will not adhere to each other and/or to the mold precluding the lay-up. The drape test evaluates the capacity of the prepreg to be molded without filament damage. Tack was tested attaching two specimens, one over the other, to a vertical steel plate and evaluate its adhesion and separator release properties during 30 minutes. For the drape, a similar procedure was performed but with only one piece of material and over a 90° mandrel and during 15min. Both tests are qualitative and the material evaluation criteria is provided in the NCAMP [13] standard.

3.2 Physical-chemical

The physical-chemical tests were useful to verify the material properties mainly in terms of fiber/resin content and resin condition.

With uncured material was performed a flow and solubility test. Both tests evaluate the influence of the cure degree increase and hence resin viscosity increase, due to the out-time exposure. The solubility was done dissolving a piece of material with an appropriate solvent, M.E.K., following the ASTM D3529 [8] standard. The amount of resin that will be dissolved should be proportional to the material degree of cure since only uncured resin is soluble. F. Lucas and B.G.S.E.M. [12] By weighing the specimens before and after the solvent application it is possible to find the solubility percentage. The flow test evaluates the capacity of the prepreg resin to flow during the cure. The percent flow is covered by the ASTM D3531 [9] standard and obtained through the differ-

ence between the cured and uncured weight of a simple laminate.

Regarding the tests with cured material, the thickness variation and the void content were evaluated. The thickness measurements were gathered using a micrometer and ultrasonic inspection. They presented coherent results and both showed that the laminates thickness variation was within 5% of the theoretical values. The void content is one important criteria of the composite material quality. It is estimated by the difference between the material physical and theoretical densities and represents point defects that ruin the material homogeneity and could result in its failure. The test procedure is covered by the ASTM D2734 [5] standard.

3.3 Mechanical

The performed mechanical tests were useful to characterize the aged material under certain loading cases. The chosen tests were suitable to evaluate the resin properties which are the most affected due to the aging. Specimens were evaluated with interlaminar shear strength (ILSS), tensile +45°/-45°, impact and compression after impact (CAI) tests. Moreover a dynamic mechanical analysis (DMA) was also used to characterize the material properties in function of temperature.

The ILSS test estimates the apparent interlaminar shear strength, which represents the maximum shear stress at half thickness of the specimen, at the moment of failure. The specimen is tested with a three-point bending where the span is small compared with the specimens thickness. This is a geometrical necessity since it is responsible for the generation of interlaminar shear forces that will promote the material delamination. The ILSS specimens lay-up was [0]₁₁.

The tensile test with +45°/45° lay-up specimens combines the usual tensile test characteristics with others from the IPS test. The aim of the test is to ob-

serve the induced shear stress between the laminate layers that for small strains is governed by the resin properties. Regarding the test procedure, identical to the one for the uniaxial tensile test, the test is governed by the ASTM standard D3039 [6]. Moreover, since the lay-up is the one used for IPS tests, some information was gathered from the ASTM D3518 [7]. The analysis of the obtained data allowed the determination of the material modulus and ultimate shear strength. The specimens lay-up for this test was $[+45/-45]_{4s}$.

The impact and CAI tests were useful to characterize the material in terms of brittleness or ductility. The test is divided into two parts: the first when the specimens are subjected to a specific impact energy and the second when the specimens are subjected to a compression test after a impact. From the first part of the test it was possible to study the barely visible impact damage (BVID), i.e., the indentation level provoked by a set of impact energies. From the second part results the material compressive strength after a BVID energy impact. The standard which defines this test is the EN6068 [17]. For the impact and compression test the lay-up used was $[[0,+45,90,-45]_3]_s$.

Addressing the DMA, it is a testing technique that can evaluate the behavior of a material when subjected to cyclic conditions of stress and temperature. The analysis is made by applying a small deformation to a sample and repeating that condition sinusoidally at a certain frequency. This continuous load case together with the presence of a temperature profile gives a relation between the stiffness and damping, respectively reported as storage modulus and tangent delta, with the temperature. When using relatively high temperatures for a certain material, the DMA test can also give information about the material transition phases such as the glass transition temperature. Dawkins [11], F. Lucas and B.G.S.E.M. [12]

The standard used to conduct this test was the

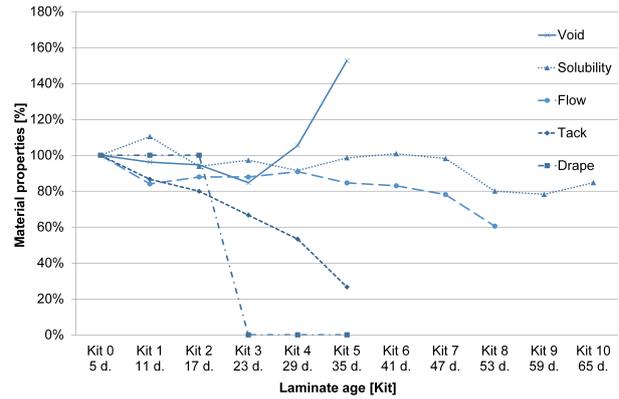


Figure 4.1: Overall results comparison - physical-chemical and processability properties.

ASTM 7028 [10] and the lay-up used was identical to the one of the ILSS, $[0]_{11}$.

4 Results and discussion

4.1 Overall discussion regarding the material properties results

Observing the various test trends, figure 4.1 and 4.2, one can easily identify common behaviors that suggest relations between the properties being evaluated in different tests. The tack decrease, together with the loss of material flexibility due to residual matrix polymerization and cross-link, contributes to the failure of the draper test. Also at this point the decrease rate of the apparent shear strength in the ILSS test is accentuated conducting to a total drop of about 11%. Apparently, the drop in the tack and draper hampers the adherence between the laminate layers which is reflected in a lower ILSS resistance. Moreover, when the tack level drops below 50%, between kit 4 and 5, the ILSS failure mode changes, ceasing of be internal and undetectable through visual inspection and becoming external with visible delaminations.

A second common trend that can be identified is the drop relation between the flow and solubility tests. Both rely on the resin viscosity that was increased by the material cure during the aging. In the flow test,

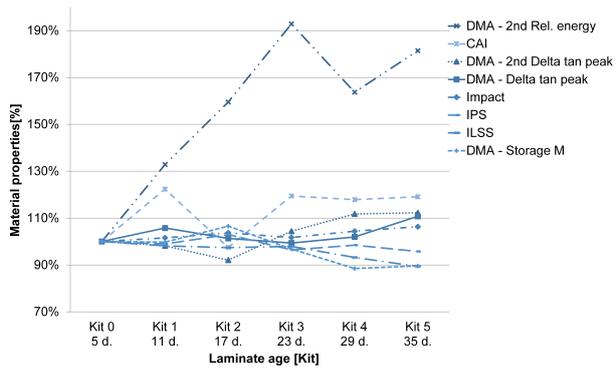


Figure 4.2: Overall results comparison - mechanical properties.

a decrease is visible beyond kit 5 and it becomes sharper after kit 7. Likewise, in the solubility test and by the kit 7, this viscosity increase, i.e., the advancement in the resin polymerization and cross-link, turns into a lower wet resin content, about 20% decrease, by preventing the resin dissolution.

Regarding the material strength tested by mechanical methods, it was observed that the storage modulus and the shear stress graph were almost identical. The trend similarity could be explained by the dependency relation of the shear modulus from the storage modulus. However, in terms of global variation, in the DMA the drop was about 10,4% while in the tensile it was about 4,3%. Thus, apparently the tests support each other trends. Besides this, the ILSS results were also coherent with those from DMA and tensile, presenting a drop of around 11% in the interlaminar shear.

Another interesting fact that combines two completely different tests is the relation between the DMA test, i.e., secondary relaxation delta tangent peak, and the impact test. This relation has already been observed in thermoplastics and states that large low-temperature relaxations occur for all polymers with a high impact strength. [11] In the case of this project and although we were dealing with thermosets, this relation was *a priori* unknown but apparently was verified. Thus, since the secondary delta tangent peak

increases with the material out-time, it was expected that the impact resistance of the material increases. The tests results proved so (see figure 4.2).

4.2 Overall discussion regarding the testing methods

Through the developed analysis of each test, some trends emerged, directing advice for the most suitable test for a certain material condition and available resources. With this in mind, a summary of the performed tests in terms of material and apparatus requirements, temporal demands and results obtained was made, figure 4.3.

Starting by the material and apparatus needs, the main constraints found were the material quantity and the processes that should be applied to it before and after testing. In this regard, the most favorable tests were the flow, the solubility and the tack tests. The void test was not included in this list since its associated experimental error could easily return unreliable results. The solubility and flow used a low material quantity and needed a more complete laboratory apparatus when compared with tack and drape. Addressing the temporal resources demand, although the tensile and ILSS tests used a larger material quantity, they are faster to execute and for that reason were positioned near the flow and solubility. In this regard, three main time intervals were distinguished: aging time, testing intervals, testing set-up time and testing time. According to the test sensitivity, the aging interval can be reduced or increased in order to get a better resolution of the material properties variation. About the set-up time and testing time, it was verified that for mechanical tests the set-up time was in general the higher of the two. For the physical-chemical tests the opposite behavior was verified. Summing the set-up and testing time contributions, the mechanical tests were in general, exception for the I+CAI and DMA tests, quicker than the physical-

chemical.

Besides resources constrains, the quantity and quality of the results are a major concern, surpassing the others in many cases. For the drape and especially the tack properties, the human influence was clear once the results are qualitative and hence, dependency from a subjective judgment is inevitable. On the other hand, the void and solubility were more influenced by the experimental uncertainty. The mechanical tests, although they were in general less affected by the experimental uncertainty and human interference, they were sensible to specimen manufacturing errors such as deviations in the layers orientation, lay-up flaws or inadequate dimensional tolerances.

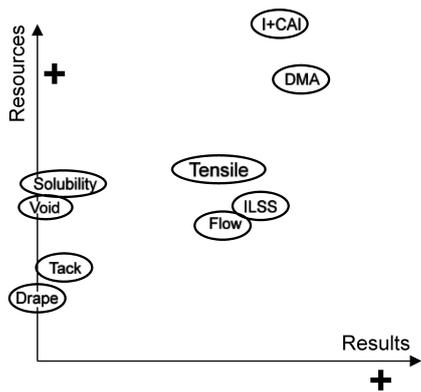


Figure 4.3: Tests comparison regarding results versus resources.

Regarding the accuracy and nature of the results, four main criteria were evaluated: results type (qualitative or quantitative), results quantity, correlation with the material out-time and tolerance to the material aging. Quantitative tests with mathematical relations between the results and material aging and that are also sensitive to the aging factor, were the best suitable for the issue at hands. In this evaluation category, the more advantageous tests were the DMA and CAI+I for the mechanical and the flow for the physical-chemical.

Overall, the DMA test proved to be the most complete one, allowing obtaining conclusive sets of data

that can be related with various material properties at once. The only evident drawback of the method, besides the testing time that it is function of the material and test settings, is being dependent from a complex testing machine. By contrast, if resources availability are a constraining factor, the ILSS test revealed to be an effective method. Regarding the more tolerant to the issue at stake and with a higher susceptibility to experimental uncertainty physical-chemical tests, the most profitable proved to be the flow.

4.3 A methodology proposal for out-time characterization

During this work, it becomes clear that the material properties sensibility to out-time was not uniform. Instead, they appeared to change at different rates and at different times, suggesting that a more adequate testing schedule should account different time steps according to each test and evaluated property. This leads to consider an un-evenly distribution in time that should be selected based on the variation of some specific material properties. Some properties require larger aging times to be affected so the aging time should also be long enough for clear and meaningful results to be obtained.

Due to uncertainty in properties of previously untested materials and even variation in known materials, a flexible methodology that acts as a general guideline was proposed, figure 4.4.

Six evaluation moments are proposed, i.e., main evaluations, where specific material properties should be tested. In the first evaluation moment, T0, when material revalidation is not allowed anymore, a complete battery of tests is performed in order to establish a baseline. In the remaining main evaluation moments the more resource demanding tests are performed, e.g. DMA, tensile and I+CAI. Doing this, their use was moderated and restricted to the moment where they would be more profitable. Between

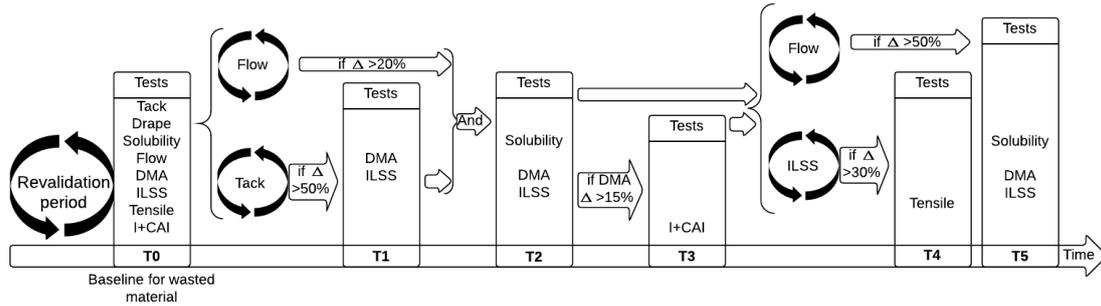


Figure 4.4: Methodology proposal for prepreg out-time aging study.

each main evaluation moment, a set of less demanding tests, e.g., tack, flow and ILSS, should be made to ascertain the main evaluations most appropriate time. These "cheaper" tests can be done with a higher periodicity, until the degradation targets are achieved, triggering then the main evaluations. With this procedure, it is possible to manage resources in an optimized way without disregard any material property and obtaining clearer and, consequently, more reliable results.

The last evaluation moment should take place when the flow test presents a minimum drop of 50%. With less than 50% of flow, the material will hardly be consolidated and large drops in its properties are expected. Therefore, for materials of this nature, even if a lay-up could be performed in this condition, it would result in laminates with poor interlaminar adherence and its use would be no more profitable. Thus, at this stage it is proposed the end of the evaluation process. However, to establish a last baseline and to verify this condition, a last main evaluation is performed.

Taking the material previously studied, a particularization of the methodology proposed is presented in figure 4.5.

The new approach should be able to follow the material aging from around 22 days of out-time until around 70 days spending less than 50% of the material and, consequently, also saving time resources. Although these resources optimization, the results

gathered should be much more reliable since a much clear signaling of variation in properties is expected.

5 Concluding Remarks

First of all, is important to note that there are inherent results variations originated by the raw material, manufacturing process and testing uncertainties that cannot be neglected. In the presented study, some of the variation in the results was small enough to be affected by those factors. Therefore, to achieve more reliable results, more tests should be done or a higher level of degradation achieved in order to promote a higher properties variation. Regarding the the degradation found in mechanical and physical-chemical properties of the material employed in this study, it was moderated due to the available exposure time. In spite of this, the use of the aged material is also conditioned by its processability properties, i.e., tack and drape. These were sharply affected by the out-time.

Concerning the evaluation of the testing methods, globally the DMA test proved to be the more complete, giving clear results and relating various material properties at once. If the DMA test apparatus is unavailable, the ILSS test is a suitable alternative to characterize the material. For the physical-chemical tests, the flow test was the most practical and with clearer results.

Based on the developed work and regarding

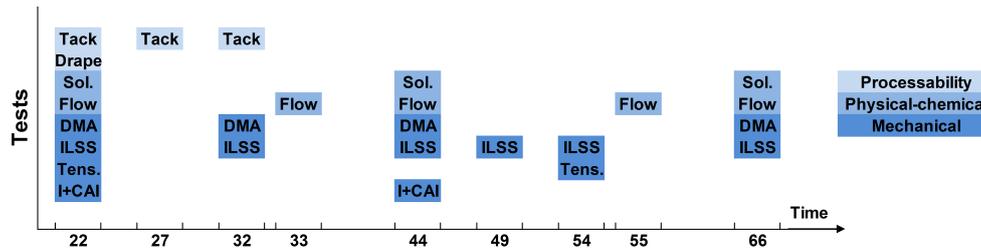


Figure 4.5: Application of the methodology proposal to the material studied in this thesis.

prepreg material of similar nature, a new methodology to study the out-time influence in the material properties was proposed. The implementation of this methodology for the studied case was simulated resulting in more than 50% resources savings, an extended evaluation period and more reliable results.

The material proved to be able to maintain reasonable properties that could certainly satisfy non critical applications. However, it must be considered that the material aging highly degrades its adherence and flexibility abilities preventing their utilization with conventional lay-up methods. Thus, the results suggest that since only the processability properties are significantly reduced, after the end of the lay-up process, the parts could have a limited and controlled time tolerance beyond the stated out-time until they enter the autoclave. If confirmed, this would allow higher flexibility and optimization of autoclave scheduling, with clear advantages to the company. However, a more thorough work to implement these practices is needed and should mainly consider larger material samples so to gather a statistically sufficient amount of data to support them.

In conclusion, regarding the properties of the material addressed and within the project timescale, it was shown that the out-time aging did not critically affect the material properties required in terms of part project and design. However, the re-use of this material is constrained by industry and international standards and by its processability properties. Overcoming these two drawbacks by, for instance, supply the

material for external companies or internally use it in less controlled and non-critical parts, with simpler geometries and applying tack and drape enhancers or non-conventional lay-up techniques, could lead to a favorable balance by the cost benefit standpoint and result in a more sustainable and cleaner industry.

5.1 Recommendations for future work

A different aging schedule could be made, possibly considering the proposed methodology or other unstudied techniques. The influence of the exposition method in the material aging could be evaluated beside a a method to control the moisture absorption effect. The relation between the material properties and its storage-time could also be evaluated.

Some tack and drape enhancers could be tested, both for hand or automated lay-up, to verify their effectiveness when dealing with out of validity material and evaluate their influence in the material properties.

About the cure processes, it would be interesting to minimize the specimen's differences with a unique cure for all the involved specimens and evaluate the relation between the room environment curing during aging versus a corresponding autoclave cure time. Moreover, a detailed explanation of the reasonable material properties variations, gathered through the DMA test, would be a valuable help to better understand this test and its usefulness in this kind of study.

Following the results suggestion that the physical-chemical and mechanical properties are only slightly affected by the aging, further tests to ascertain the

consequences of exceeding the material out-time after lamination, i.e., during the stand-by to cure time, would be helpful for optimize the autoclave usage.

Concerning the testing and analysis methodologies, different tests with different lay-ups could be made, e.g., 90 ° tensile, compressive, flexural, uncured DMA or DSC, +45°/-45° DMA etc..

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