CHARACTERISATION OF A COMPOSITE MATERIAL: NEEDS AND METHODOLOGIES

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ABSTRACT

Despite the difficulties encountered in the recent past and despite their cost, composite materials are already widely used, and this will increase in near future. Launch vehicles are not an exception to this tendency and composites are usually employed especially on the upper part. Indeed, they are involved in the structures interfacing the last stage and the satellites, as for ARIANE. But composites are also more and more used for solid propellant stages, as for VEGA.

For a long time, composite structures have been designed with rules and methodologies employed for metallic ones. The heterogeneity of this type of materials is not fully exploited and their potential to sustain damage is not totally mastered. Thus, because composites are more than never an answer to mass saving, we need to revise rules and methods related to their use. In this context, this article intends to focus on the basis of a composite structure development, the material characterisation.

In a first part, the European habits are recalled. The main drawbacks will be underlined through examples of failures or lacks of robustness. Then, an complementary plan of characterisation will be built, using as much as possible the latest gains from research advances. After discussing the compromise between a full test plan and the industrial possibilities, the third part of this paper will outline recommendations on the characterisation of composite materials for future developments proposed for introduction in the evolution of the ARIANE design rules. The conclusion will resume the challenges indispensable to overcome, in order to foster the extensive utilisation of composites for designing launch vehicle structures. The intention is to focus on the work to be done to demonstrate that composites are a promising solution for mass and cost savings, ensuring the competitiveness of European launch vehicles.

1. INTRODUCTION

The development of Ariane 5 took its basis on different general specifications. For the structures, the specification is dedicated to “design, dimensioning and tests” [1] and is currently under update to take benefits from recent developments. The specification gathers requirements and recommendations to conduct a development up to the qualification. Thus, one full chapter is dedicated to requirements for materials and especially for their characterisation.

On Ariane 5, structures on launchers can be metallic – it is the case for the main tanks (for first and upper stages), the solid booster envelope and structures as engine thrust frames – or composite. Composites are mainly chosen for structures where specific stiffness is searched. This gain can be directly turn into performance (payload mass) for structures of the last stage. Then, Ariane 5 possesses equipment bays, payload adaptors, interstage skirt and payload made of CFRP (carbon fibre reinforced plastics). Figure 1 illustrates this fact.

Ariane 4 already used composites in the upper parts above the last propulsive stage : the SPELDA (carrying structure for payloads in a double launch configuration), the equipment bay and the fairing were already constituted of sandwich panels, and specific equipment like highly-pressurized Helium tanks were already composite wrapped on metallic liners.
Figure 1: Composite structures on Ariane 5.

Figure 2: Ratio of composites for the main planes compared to the Ariane 5-ECA configuration.
In term of contribution of composite material to the whole system, the launcher situation can be directly compared to airplanes, as it is schemed on figure 2. If the launcher Ariane 5 ECA shows only 5% in weight of composite, the amount of composite in the upper part is equivalent to the one of the Airbus A380.

The concern here is to make a status of the existing habits for the characterisation of composite materials for launcher structures. These habits will be confronted to experiences gained from the development of the different Ariane 5 configurations. Current reflections are on-going in the frame of the rewriting of the general specifications. The rewriting has the objective to take into account the recent past-experience and gained knowledge, as well as the progressing improvements achieved through the research and technology programs. Gathering these data in this paper should allow to give recommendations for the future developments and their preparations.

2. CHARACTERISATION OF COMPOSITE MATERIAL

2.1 Current status

2.1.1 Specific use of composite on launcher structures

The composites usually selected are carbon/epoxy combinations, either sandwich with an aluminium nida-type core and thin skins, or thicker composites called “monolithic”. The couple fibre/matrix is chosen to answer to the main requirements of a launcher structure: stiffness and/or strength and mass. To answer to these requirements, carbon fibres and their high specific characteristics are a natural answer. The selection of the most appropriate carbon fibre can be performed among categories as intermediate modulus (IM7, T800), high modulus (M40J, M55J) or high strength (T300, T700). Resins are always epoxies.

The selection of the couple is also the result of the industrial experience. It means that the choice of the material is often strongly linked to the existing manufacturing tools and to the knowledge of already used fibre/matrix couples for other developments. Besides, industry involved in the space field has often an activity also linked to the aeronautical field. Thus, when appropriate, the composite materials for space structures are common to the aeronautical developments, in order to take benefit of the already existing mastering and of the larger batches necessarily supplied for the more consuming aeronautical activity. If this logic provides obvious advantages, the question of the selection of the most suitable fibre/matrix couple can be raised, since there can be a bias linked to already existing “in-house” solutions.

2.1.2 Characterisation of composite materials

The material properties are the input data for the dimensioning. The current methodologies used for dimensioning are deterministic. Within a deterministic approach for strength design, the structure has to sustain ultimate loads without failure using lower limit material resistance (R1) (Figure 3). Ultimate loads (UL) are obtained multiplying limit loads (S1) by the ultimate load factor of safety (SF) and limit loads are considered as the maximum loads likely to be encountered during the service life of the structure. Getting the example of ARIANE 5 Launcher’s structures, SF of 1.25 is generally required against rupture [2], S1 is defined as the load having a 99% probability of not being exceeded in service with a confidence level of 90%, and R1 is defined as the value which has a 99% probability of being exceeded with a confidence level of 90%. Considering the typical dispersion of loads and material resistance,
the correct use of such values gives a probability of failure (Pf) typically less than $10^{-5}$, which is compatible of global vehicle reliability.

Figure 3: Statistical distributions of resistance and load.

R1 is also called the allowable value. For static characteristics, the allowables for Moduli (Young and shear) are mean values and the allowables for strength are values at 99% of probability with 90% of confidence.

The determination of the allowable mechanical properties of a material shall be based on a minimum number of valid tests (on samples) to obtain sufficient statistical confidence in the value of the analysed parameter. According to [3], B-value (90% of probability with 95% of confidence) requests more than 30 repetitions whereas A-values (99% of probability with 95% of confidence) requests more than 75 repetitions. This amount of tests is not compliant with the industrial possibilities, all the more at the beginning of a development. Thus, [1] requests for 10 valid results. It is then assumed that the dispersion is a normal law. The allowable is

$$X_{\text{allowable}} = \bar{X} - k(N,P,Q)\sigma,$$

where $\bar{X}$ is the mean value, $\sigma$ is the standard deviation and $k(N,P,Q)$ being a function which depends on a number of tests $N$, on the confidence level $Q$ and on the probability $P$ with which the allowable value is obtained.

Up to recently, the characterisation of a composite material copies the characterisation of a metallic, i.e. simply modulus and strength. This characterisation is performed on the elementary ply (unidirectional, or UD, tests), then on the laminate with the stacking sequence representative of the structure (multidirectional, or MD, tests). Tests are performed in the plane of the laminate, in the main directions (longitudinal and transverse), in tension, compression and shear. Properties shall of course be obtained in accordance with the flight structure environment: when necessary, temperature and moisture sensitivities are assessed, as well as compatibility with oxidizers and space environment.

Following this first step of elementary tests, composites shall be characterized in their structural configuration. Thus, sandwich should be tested on its own in order to take into account the composite as two thin skins separated by and bonded to a rather thick core. Flatwise and edgewise tests are the usual tests performed in order to obtain properties as strengths and moduli in the in-plane direction and normal to the sandwich plane. The composite is characterised in its structural configuration, i.e. taking into account the reality of the structure: manufacturing processes as laying-up (by hand or by fibre placement) and curing phase in autoclave mainly (including the bonding of the skins on the core).

Since a few years, taking into account the damage in composite is one of the most challenging step towards an optimised utilisation of composite. Tools dedicated to damage modelling may need specific properties to be identified. In CNES, a tool called DAMSTRAT is used to assess the damage in the ply (see also paper reference 535 “Validation of the DAMSTRAT software
for progressive failure analysis in composite launcher structures” in ECCM13). The identification of the damage parameters requires simple tests of loading and unloading cycles on dedicated stacking sequences selected to activate specific damage modes.

Once the characterisation of the sound material achieved, dedicated characterisation is performed to feed the damage tolerance analysis. It consists in identifying the main manufacturing defects (delamination, skin wrinkle, skin/core debonding, hole to cover any fibre ruptures) and to implement them artificially in the composites (with the help of Teflon patches for delamination and debonding for instance). After cycling the samples with the load spectrum representative of the life, residual strength is assessed by a tension or compression test up to failure. In addition to the manufacturing-type defects, the impact due to tool drop shall be considered and its effect on the allowable shall be addressed. That being so, these low velocity impacts are not always characterised with the same rigor (see also the considerations related to limitations in the next subsection).

In addition to these elementary characterisations, tests are more and more required to master the understanding of singular areas, seats of stress concentrations. These tests are mandatory to obtain the correct rupture criterion for areas like holes, door corners, lay-up drop out, sandwich core thickness change, variation of ply as a result of drape over corners, …).

Therefore, the composite characterisation became more and more specific. In addition to classical UD and laminate tests, specific tests related to the structure configuration have to be performed. The following paragraph gives two examples of structural component tests performed during Ariane 5 structure developments.

2.1.3 Examples for characterisation of singular areas

The characterisation logic based on successive UD, MD and sandwich (when necessary) tests is the usual one for European launcher structures. For a long time, this has been sufficient to ensure a correct dimensioning with positive margins. Nevertheless, taking back the development or the qualification of structures with the same design but with increased load specifications showed limitations. They are mainly due to lacks of knowledge of some structural behaviour which were not tackled from that moment on margins of safety remained positive. With increasing loads, margins became sometimes negative and requested to think again about the suitability of input data to represent correctly the real behaviour of the materials. This is particularly the case for structural singularities.

Therefore, it is now established that component tests shall be performed as early as possible in the development. They aim at characterizing the complex behaviour of a structural situation as connections between sandwich and interface metallic rings or integrated interface composite parts. These structural parts often show failure modes which are not direct composite failure modes, but a combination failure mode where composite but also rings, connection elements and the specific geometry can play a role for the initiation and the propagation of the failure. This characterisation is generally used to extract allowables (after statistical treatment of the test results as described in the previous paragraph) directly from the rupture load obtained during the test. These allowables are directly used for the dimensioning and for the assessment of the margin of safety.

On this topic, two examples from Ariane 5 development are detailed below.

The first one is related to the connection between the EPS (Etage à Propergols Stockables, or storable propellant stage) structure. This structure, manufactured by EADS – CASA Espacio, consists in a metallic sandwich spherical platform, an external CFRP sandwich cone, stiffened by two panels organised as a cross and linked to this cone or the platform through metallic “Pi-fittings” (see Figure 4). This fitting allows the connection of a sandwich panel with an
aluminium part through rivets making the link between one fork of the fitting on one side and on an added doubler on the other side. An adhesive is inserted between sandwich and aluminium pieces for shimming and tightness.

During re-qualification of the structure due to a new configuration of the launcher, margins appeared to be negative in some connections. If the former methodology was considered to be conservative, no quantitative demonstration of this conservatism was available. Thus, it has been decided to perform dedicated tests on the real configuration to extract an allowable more representative of the structure behaviour in flight. Detailed finite element models have been used to determine the test set-up and the most appropriate location of strain gauges.

The failure mode observed on samples is always the same whatever the loading is (tension or compression, combined compression + shear): failure initiates in the skin in the connection itself at the level of the upper raw of rivets and propagates out of the connection in the skin at more or less 45° of the longitudinal axis. This failure process is characteristic of a metallic ring/sandwich connection.

Figure 4: EPS configuration with Pi-fitting localisation (surrounded by dotted lines) and scheme of the connection.

The second example deals with the cone 3936 which is placed at the interface between the upper stage and the payload adaptor (see figure 5).

Figure 5: Cone 3936 and scheme for the cobonded integrated CFRP interface for the cone 3936.

This structure manufactured by EADS - CASA Espacio is constituted by a monolithic thick CFRP laminate layered by fibre placement. The upper and lower interfaces with the adjacent
structures are performed through composite integrated rings. They are manufactured by the cobonded technique, which allow to place net-shape RTM carbon parts between the two layered skins on the circumference and to cure the resin of the fibre/matrix couple simultaneously with the bonding between RTM parts and skins. Because the failure modes can not be apprehended through the elementary failure modes of the different elements (RTM parts and skins), neither by elementary tests nor by complex 3D FE modelling, component tests have been performed on samples totally representative of the real configuration aiming at understanding the behaviour up to rupture and at extracting the allowables for the dimensioning. Tests in tension and compression have been performed. The constant observed failure mode is rupture in the RTM part initiated at drilled holes for bolts. This is demonstrative of a failure mode typical of the connection.

2.2 Limitations

Even if the habits are evolving in the good way, i.e. to consider the composite as a specific material deserving its own methodology of characterisation, some limitations appear in the current procedures. Without trying to be exhaustive one can quote and explicit a few ones: the inter-lot characterisation, the structural component characterisation, the damage tolerance characterisation and the bonding in structural assemblies.

The inter-batch characterisation is requested in the general specification in order to check the stability of the characteristics within different batches of supplied components for composite structure manufacturing. This characterisation is seldom performed during the early stages of the development because manufacturing does begin after the design is frozen. Moreover, several lots of material can only be available once the production has really started. At last, the needed characterisation not only consists in tests to check for the resin or prepreg stability but shall also be performed on samples which can be considered as witness samples, like for instance tests on the laminate representative of the structure configuration. Thus, this inter-batch characterisation can not be available during the development whereas one would need to rely on it to consolidate the allowables.

Both examples described in § 2.1.3 reveal the need to test structural details such as structure singular zones. These tests demonstrated also the need to implement representative loadings, which can implies rather costly and complex test set-up. That being, preliminary study with the help of detailed FE models allows to select the correct configuration of the test set-up as well as the loading sequence. Anyhow, in a context of constant research for optimisation, this type of tests is now considered as necessary, and it has to be foreseen in the early steps of every development. To take full benefit of this characterisation, instrumentation shall also be optimised in order to get data at several levels (especially global and local) to enrich the understanding of the behaviour. For instance, Lévêque [4] showed that multi-instrumentation as strain gauges coupled with image correlation, integrated optical fibre Bragg gratings, infrared thermography, X-ray radioscopy or acoustic emission, gives complementary measurements (local/global, on surface/in the material) and consequently an extensive database for deeper exploitation.

As described previously, the damage tolerance demonstration relies on dedicated tests with virtually implemented defects. Even if mentioned in the actual rules, low velocity impacts such as tool drops on composite structures are not always considered as typical initial defects to study. When taken into account, one can observe a lack of coherency from an industrial to another in the test to assess the knockdown allowable due to the presence of the defects. Moreover, one can also notice the lack of validity of the procedures, especially with respect to the representativeness of the impact event.
Finally, because of the obvious gain in mass and potential gain in the manufacturing costs, structural bonding is of great interest for the highly loaded structures of a launcher. Connections between parts (rings and sandwich panels for instance) are mainly riveted or bolted but structural bonding is also used and already shown its feasibility. Nevertheless, in addition to a dedicated design, specific characterisation has to be performed, for the adhesive itself but also for the bonded assembly. This specific topic underlines the obligation to link the development and the process aspects in order to build an appropriate characterisation plan.

This last point is one of the major lesson for any improvement in methodologies: to emphasize the dialog between development teams and production/manufacturing ones.

3. PROPOSALS FOR IMPROVEMENT

The feedback experience gained from the Ariane 5 structure developments and the consequent limitations listed above give cause for improving the current methodology for composite characterisation. Besides, numerous research works are running through Europe, particularly motivated by the aeronautical field. They give a lot of tracks and means to improve the understanding and the use of composite for lightweight and highly loaded structures. The AMERICO program is one of the major one run in France between 2002 and 2007 which provide a quantity of ways of improvements, especially for the characterisation of composites. One can list a few of them which are considered sufficiently mature to be implemented in the industry and especially for future launcher structure developments.

3.1 Database consolidation

Leroy [5] and Rollet [6] worked on the improvement of the exploitation for existing databases with the objective to improve the confidence in the extracted allowables and to decrease the number of tests without reducing the final margin of safety. Working on the modelling of the experimental dispersions and taking into account the correlation existing between the different elementary tests, they show that one can limit the minimum required conservatism in the test results which consequently allow to expect higher performances in the structural dimensioning or, from another point of view, to decrease the number of tests in qualification with the same level of performance. This reduction can be more than 15% of the current characterisation.

In parallel to this result, these approaches, when used in parallel to the characterisation, allow to adjust the test campaign by re-sizing it in progress. It can also help for the re-assessment of A or B-values and to improve the confidence for small sampling of results, which is the case for launcher structures. Furthermore, coupling with the techniques of virtual testing which give information on the existing correlation between results, one can think about assessing other stacking sequences from tested ones.

3.2 Multi instrumentation

Recent techniques as full-field measurements are now well-mastered and sufficiently mature to fit to industrial constraints. In the frame of the AMERICO program, this technique was applied to various composite laminates and at two different scales: the laminate and ply-scale [4,7]. Some out-of-the-plane elastic properties were thus obtained and the technique was found to be of great interest for the detection of damage zones and to quantify their increase,
including in high gradients zones (near hole). This is important to note that this technique can be considered to be multi-scale since it allows to have information at the material level (for instance, determination of the density of delamination or of cracks appearing in the material by monitoring the edges of the sample) but also at structure level (e.g. assessment of the damage on structural elements such as plates with holes, tubes or connections).

Another promising technique is the use of FBG (optical Fibre Bragg Grating) as proposed by Collombet [8]. After demonstrating that the fibre can be considered as non intrusive, Collombet showed that implementing this type of sensor during the laminate elaboration allows to monitor the full life cycle of the sample or of the composite structure itself. Apart from checking the existence of damage, it can give precious information on the level of residual stresses after curing (especially the through thickness ones, causes of potential delamination). At last, FBG are used for the identification of the thermal and mechanical properties of the composite.

3.3 Technological evaluator

One of the very promising contribution of the AMERICO program is the use of a so-called “technological evaluator”. Two main objectives are pursued: to understand till the early steps of a development the behavior of complex structural areas (as connections or high-gradient zones) and to take into account the intrinsic variability of the material.

This structure is specially designed very early in the program to improve numerical simulations of singularities and to promote innovative designs on the structural scale. It can be used throughout the development (up to the large component level) in order to validate the different choices of conception performed. Thus, the technological evaluator is a multi-instrumented sample representative of the industrial structure (in term of manufacturing process and major characteristics of the design).

The evaluator is not a sub-scale of the final structure but simpler samples introducing the main difficulties of the design. Its definition is adjusted with the help of sensitive analyses performed through numerical calculations. The fact that these samples shall remain less complex than a test representative of the structure (even in sub-scale) gives the possibility of manufacturing a significant amount of evaluators. This is a very important aspect because it can allow parametric and/or statistical study of variability and its influence (mainly the material intrinsic variability or the variability brought by the process).

Collombet’s team, helped by DDL Consultant, showed on an industrial example that it can bring drastic reduction of manufacturing costs and tests: an overall reduction of 30% is waited for costs. It has to be noted that, if the global number of tests during the development can be reduced, the amount of calculation is increased.

3.4 Improvement of the dialogue test / calculation

The previous example fits to the general trend for composite analysis which is the need of improving the dialogue between tests and calculations. In the same way, the use of techniques as full-displacement measurements (see §3.2) will give its entire interest if it is accompanied by numerical calculations to exploit the monitoring in an optimal manner.

As for both examples above, Huchette [9] shows evidences of the interest of coupling test and calculations. In his work, Huchette developed a model thanks to numerical and experimental tests. In fact, the experimental tests allowed to extract the different kind of damage
mechanisms acting in the material as well as quantitative information on the damage kinetics. This information is used in the numerical tests to model the effects of the damage on the elastic behaviour of the damaged plies, and then, the identification of the effective tensor is fully realized numerically. Nevertheless, the identification of the evolution law of each mechanism needs experimental tests. These experimental tests allow to identify the damage threshold and the parameter coupling the transverse cracking and delamination induced by transverse cracks. This shows the complementarity of both components (experiment and calculation) for the achievement of the analysis.

Besides, the capitalization of experiments will permit to improve the models for using them as means of identification of properties, all the more that these experiments will be well instrumented. This is the goal and the challenge of the virtual testing.

For information, one can also refer to [10] in which dialog between tests and calculations is realized using FBG for the assessment of the thermal expansion of a laminate.

4. CONCLUSIONS

The description of the usual characterization of composite materials for launcher structures gives an overview of the practices and of their limitations. Among them, one can quote the need for tests for any singular area in order to consolidate the dimensioning of a structure for which optimization in term of strength, stiffness, mass and cost is a constant research. To reach these requirements, improvements are needed. These improvements shall correspond to techniques mature enough to be brought in the industrial context.

Some recent activities seems to answer to the demand, and, among them, the French research advances gained in the frame of the program AMERICO. It seems now clear that the database consolidation, the multi instrumentation of characterization tests, the technological evaluator concept and the improvement of the dialogue between test and numerical calculations are considered as steady foundations for improving the mastering of the composite materials, maintaining them as pertinent solutions for space structures.

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