# Advanced Composite Materials and Technologies for Large Solid Rocket Motors

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#### Abstract

The solid propulsion is confirmed since several decades as one of the more reliable, effectiveness and cost-sustainable technologies for the space launchers. The architecture of Ariane 5 includes strap-on solid boosters for takeoff, similarly to most of the heavy worldwide launchers currently in service and the future Ariane evolution will keep the solid technology as base configuration as well.

Therefore, the improvement of the solid rocket motor in the perspective to increase performance, reduce costs and enhance reliability is within the priority themes for the future developments.

The introduction of the structural composite materials for the motor cases replacing the steel, is one of the main steps ahead performed in the past years. The new launcher Vega is conceived with three SRMs with composite cases, having great advantages in the reduction of inert mass.

The extensive use of composite cases and the fact that the carbon-fibers epoxy prepreg for space motors are quite strategic materials, require to include the composite materials in the research and development activities, as additional area, in order to carry on the improvement on this core technology.

In particular, since several years Avio has been developing in-house carbon-epoxy prepreg, through extensive research on pure epoxy resin and impregnation process by the pilot plant of prepreg tow and tape.

The in-house prepreg named HXE-23 has specific characteristics tailored for the filament winding technology on large motor case and a rheology to optimize the mass curing, on the lessons learned of the Vega qualification.

Several tests have been carried out either in the configuration of specimens as well in the configuration of small and intermediate scale of composite case.

The HXE-23 prepreg will be tested at full size level through the Zefiro40 SRM, the new second stage rocket motor under development in Avio, with extremely high strength fibers.

# 1 Introduction

Since the development of the Vega solid rocket motors, the structural composite materials have been considered in Avio as strategic line to invest on for research and development, as for the other space materials belonging to the core business: solid propellants, thermal insulations, bonding interfaces.

The choice done at the beginning to work with carbon fibers reinforced pre-impregnated with epoxy resin in place of wet winding or electron beam technology, was justified for the mitigation of the risk, having the successful technical heritage of the Zefiro 16 composite case.

The experience acquired on the large motor case manufacturing with unidirectional carbon-epoxy prepreg tow and tape, by means winding cycles, curing cycles, root cause analysis and problem solving of the several troubles faced until the successful qualification of all the cases for the three stages, allowed to determine a solid background to use for approaching the design of a new specific prepreg material.

The core subject is the resin, that must be compliant to several constraints enabling to have a robust manufacturing cycle, giving at the end the established homogenous case performance. The commercial materials are technically satisfactorily at border line and have the not negligible drawback of the transportation that requires deep incoming test, adding costs on the already high materials costs.

The long research activity in Avio leads at present time to a patent under application on resin and prepreg tow and tape, designed for specific large solid rocket motor cases, fully characterized and tested until the intermediate scale. The configuration has been optimized with fibers for second stage (high strength, space grade) and for first or boost stages (commercial grade) and will be tested at full size with the Zefiro 40 italian program.



# Figure 1.1 P80 SRM filament winding manufacturing

# 2 The Z40 Program

After the success of the VEGA launcher (two flawless missions, maiden flight performed on 13<sup>th</sup> February 2012), Avio is currently working on future developments of the actual configuration.

The current four staged launcher (three solid rocket motors – P80, Z23, and Z9 - and one liquid propellant stage) is moving to a three stages configuration with enhanced performances that will allow to double the payload mass from the actual 1500 kg up to 3000 Kg into LEO orbit.

In a first evolution step named VEGA-C, the first stage motor P80 will be substituted by a new enhanced first stage SRM. The second step, VEGA-E, will see the introduction of the Z40 solid rocket motor and a new cryogenic upper stage in place of the Z23, Z9 and AVUM motors.



Figure 2.1 VEGA launcher

Being composite material for motor case the key for high performances achievement, developments for new solid rocket motors are based again on high strength carbon epoxy material, with Avio resin system.

The Z40 project is an Avio self funded program. This second stage motor case is designed for extreme structural performances, with a diameter of 2.4m and a boss-to-boss length of about 6m. The filament wound body vessel and the skirts will be fully manufactured with HXE-23, the patented Avio prepreg system with innovative resin, constituting the first full scale demonstrator for the new material.



Figure 2.1 Z40 solid rocket motor cross section view

Avio approach for material mechanical characterization, consolidated in the frame of the VEGA development, is based on tensile tests at different scale level, from basic rings (ASTM D 2290), standard 6'' small vessels, up to 1:5 prototypes of the vessel. Interlaminar shear strength and compressive properties are characterized as well in this first testing phase.

In a second step, moisture absorption characteristics, material fracture toughness, interface with other materials (metals and rubber) are performed. The last step is composed by tests on skirt and body prototypes for bolted joint and impact damage characterization

The preliminary characterization phase led to an estimation of the material strength at full scale level that is in line with the expected performance at the beginning of the development (estimated case mass passed from 900 Kg at the beginning of material development to about 850 Kg after material characterization).

## **3** Resin research and development

Since the strategic role of the prepreg is mainly linked to the possession of the resin system know how, large efforts were devoted in the field of the research and development of several resin compositions having as final target the following performance:

- Low environmental impact;
- Medium-high Glass Transition Temperature;
- High stability at room temperature;
- Rheology consistent with the impregnation process and suitable for Filament winding application of the prepreg;
- Elongation at rupture as higher as possible.

The next paragraphs summarize the fields investigated, the results achieved and the choices done for the prepreg application and manufacturing.

#### 3.1 Environmental impact

The base processes for the production of epoxy resin systems addressed to prepreg are mainly two:

- Solvent process;
- Hot melt.

In the first case the resin viscosity desired for the carbon fibers impregnation is obtained by solvent addition to the resin system composition while for the hot melt the viscosity is lowered acting on the impregnation temperature.

Considering the impact of the solvent removal after the impregnation Avio selected a process without solvent even if this choice obliged us to deeply investigate a large number of raw materials and optimize the viscosity vs the temperature without affecting the resin stability.

#### 3.2 Glass Transition Temperature (Tg)

This performance is directly linked to the epoxy resin functionality and the relevant hardener. However, the drawback of a base resin with high Tg is a poor processability during the impregnation process.

Then, the experience achieved in this research, outlined the need to define a compromise between the max Tg achievable and an acceptable level of stability and processability. This compromise was further investigated during the selection of the most suitable curing agent. The different resin compositions were added both with several hardners at different percentage and verifying either the Tg as well as the reactivity, by means calorimetry (Differential Scanning Calorimeter DSC) and rheology measurements.

Typical calorimetric diagrams, obtained with two cycles on the same resin sample, are given in figure 3.2.1:



Figure 3.2.1: 1<sup>st</sup> and 2<sup>nd</sup> run at DSC

At the present time several set of resin composition are available showing a Tg in the range of 130 to 350°C associated to different out-life (resin stability at room temperature).

#### 3.3 Resin and prepreg stability

The prepreg stability is another property very important for large size composite motor case manufacturing, because the winding cycles could be quite long or it can be necessary to absorb

without defects on the product, any possible delay due to accidental troubles during the manufacturing.

Transferring this need to the resin material, the issue is to have formulation with high Tg and simultaneously a process temperature to enable suitable viscosity at the impregnation level able without any effect of destabilization.

In this perspective specific epoxy resins were investigated (with very low viscosity) and reactive diluents able to reduce the viscosity of the system composition without lowering the cured resin Tg. Experimental results, based on comparison of rheology and thermal tests conducted on fresh and aged resins show stability up to 6 months at ambient temperature.

## 3.4 Viscosity and Rheology

The viscosity profile of the resin in function of the temperature plays not only a fundamental role for the carbon fibers impregnation but also during the prepreg application by filament winding technology and motor case curing. It is the core issue for the product processability and the absence of defects, enabling the flow through the multiple layers of prepreg, during the curing under compaction.

The reference base constraints were:

- Viscosity at impregnation temperature;
- Viscosity at room temperature after impregnation;
- Viscosity at the bstage;
- Onset of the curing kinetic
- Curing kinetic and thermodynamic .

For fully achieving above performance the focus was necessary mainly for the bstage profile. Several components were investigated and simultaneously the preservation of the overall resin/prepreg performance was verified.

The figures 3.4.1 and 3.4.2 show the viscosity profiles at resin and prepreg level in function of specific temperature profile.

The test performed was by the TA Instruments Ares Dynamical mechanical Rheometer with the multistep thermal cycle.

#### 3.5 Elongation at rupture of the net resin

The need of a medium high level of elongation at rupture of the resin is mainly linked to the impact of this performance on the transfer of the loads, among the different layers of prepreg, even in presence of defects such as voids or delaminations normally occurring during the filament winding and curing.

The best performance were achieved acting both on the epoxy resins selection and modifying the resin system by the adding of elastomeric components at different percentage able to improve the elongation at rupture from 3,0% up to 15%. Since the percentage of elastomer in the composition reduces the Tg, also in this case it was necessary to reach a compromise between the elongation at rupture and the glass transition temperature.



Figure 3.4.1 : HXE-23 Resin curing



Figure 3.4.2: Tow preg curing

## 4 Prepreg manufacturing and characterization

#### The manufacture of composite materials motor cases includes mainly two prepregs:

- Tow preg (for filament winding application)
- Unidirectional (UD) Prepreg tape (for reinforcing specific motor case zones)





Figure 4.1 Tow preg HXE-23 and tape

While for achieving the technology to produce UD tape it was quite easy to identify in the market Suppliers of the relevant machines not the same was for the tow preg having this technology a very limited application (the most part of Companies using the filament winding prefer the wet winding and not prepreg).

Consequently Avio defined the concept and the specification requirements of the tow preg machine which was developed and manufactured in cooperation with a European Company.

# 4.1 UD tape

The process to produce the prepreg tape include the following machines:

- Resin mixer;
- Resin filmer (this machine allows to film the desired amount of resin on a suitable substrate);
- Fiber Fibers Creel : delivery system to carry a number of single carbon tow to the prepreg machine;
- Prepreg machine.

The next figures show the overall cycle of the UD tape and some detailed views of the machines installed in Avio.



Figure 4.1.1 Tape prepregging cycle

All the process parameters (temperatures, line speed, gaps, pressures while impregnating) were fully investigated by producing samples and testing it by chemical, physical, thermal and rheological determinations and were optimized for a wide selection of carbon fibers (commercial and aerospace grade, with different numbers of filaments and produced by different Suppliers).

The same samples, having fixed a reference curing cycle, were cured and tested to evaluate mechanical properties (stress, strain and modulus on tensile specimens; stress on shear specimens).

# 4.2 TOW preg

The impregnation of single tow carbon fibers is performed by a machine able to carry the dry carbon fibers in a hot zone where meets the resin on the application roll. The resin is pumped from a resin melter to the application roll with a pump delivery consistent with the line speed to reach the desired resin content on the prepreg. After the application roll, the prepreg crosses a number of kneading rolls to let the resin fully wet the carbon fiber. At the end the tow preg enters the cold area of the machine for achieving an increasing of viscosity before the tow preg rewinding to obtain the final prepreg tow bobbins.

As for UD tape also for this machine it was necessary to fully investigate the process parameters which were optimized for different carbon fibers. These last were selected in a wide range both from different Suppliers and with different technical performance. Basically we investigated the

performance achievable by the use of both commercial carbon fibers as well as aerospace grade having different numbers of filaments (from 12k to 24k) and different fibers sizing.



Figure 4.2.1: Rewinding step of the tow-preg process and some bobbins of HXE-23 for second stage motor case

#### 5 Tests and technological demonstrators

Several tests have been carried out either in the configuration of specimens as well in the configuration of small and intermediate scale of composite case.

Z40 TOW	Requirements		HXE23	
FIBERS # 1	NOL RING (MPa) >=2550	SBS (MPa) >=58	NOL RING (MPa) 2700-3100	SBS (Mpa) 65
FIBERS # 2			NOL RING (Mpa) 3000-3500	SBS (Mpa) 66
Z40 TAPE	Requirements		HXE23	
FIBERS #3	NOL RING (Mpa) >=2120	SBS (Mpa) ≻=58	NOL RING (Mpa) 2200	SBS (Mpa) 68
FIBERS # 4			NOL RING (Mpa) 2200-2300	SBS (Mpa) 73
P120 TOW	Requirements		HXE23	
FIBERS # 3	NOL RING (Mpa) >=2119	SBS (Mpa) >=58	NOL RING (Mpa) 2400	SBS (Mpa) 66
FIBERS # 5			NOL RING (Mpa) 2700-3100	SBS (Mpa) 64
FIBERS # 6			NOL RING (Mpa) 2800-3300	SBS (Mpa) 67
FIBERS # 4			NOL RING (Mpa) 2100	SBS (Mpa) 69

Figure 5.1 Main results on specimens of HXE-23 in configuration with different fibers.

Other than tests at specimens level, also the small and intermediate scale vessel have been performed in order to evaluate the transfer coefficient between the resin and the fibers. The acquired experience on Vega SMRs composite case and the statistics, allowed to establish a reliable scale factor in order to extend the coefficient to the full scale case. The reliability of the manufacturing cycle already qualified with the P80 allows to calculate the HXE-23 composite performance at the Z40 and P120 level.

The test with different fibers was aimed to a first technical screening on the performance, in order to have element for the final selection to be done also considering the costs.



Fig. 5.2 Small vessel burst test of HXE-23



Figure 5.3 Intermediate scale case test with HXE-23-Z40

#### 6 Conclusions

The technology readiness level achieved for the HXE-23 carbon-epoxy prepreg have been presented, together with some highlights of the resin development in Avio. This material has been specifically designed and formulated for the large composite cases of space solid rocket motors, having the consolidated experience of the Vega SRMs cases, successfully qualified at the ground level and in flight.

Other than the technical drivers for the resin formulation and impregnation, also the costs have been taken into account, testing several kind of fibers to enable suitable tradeoff for all the future motor cases.