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EVALUATION OF ULTRASTABLE CARBON/CARBON SANDWICH STRUCTURES JOINED WITH CERAMIC CEMENT

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The purpose of this paper is to present the evaluation of inorganic joining technology to bond Carbon/Carbon (C/C) sandwich (C/C skins to C/C honeycomb) with the aim to make ultra stable structures that could offer high level of stability requested by space science high performances instruments. This bonding solution has been identified as a solution to overcome the stability issues faced with the ultra-stable structure supporting the GOCE gradiometers for which an organic solution has been used. To fulfil the tight stability requirements of GOCE, it has been necessary to take into account the influence of hygrometry on stability during the overall project duration.
Compared to organic joining technology, inorganic materials are at their early stage for space applications and present promising characteristics: they are insensitive to moisture (no moisture-induced distortion) and have a very low thermo-elastic sensitivity (Coefficient of Thermal Expansion (CTE) < 7.10^{-6} °C^{-1}); in addition, they offer a competitive stiffness-to-mass ratio and can withstand high temperatures of more than 350°C without damages. Inorganic bonding process are also suitable for large structures applications (>1 square meter). Beside the Hyper Stable systems (interferometric or aperture synthesis), other potential applications have been identified for this technology such as high temperatures systems (high power radiators, large high power antennas, solar arrays…)
The ceramic bonding technology which has been developed can fulfil the requirements for the future ultrastable structures as, by nature the coefficient of moisture expansion (CME) of the ceramic adhesive is null and its CTE is close to that of the bonded materials. A ceramic powder with a negative CTE has been added to a commercial graphite base adhesive to better tune the CTE of the bonding line to that of the materials to be bonded. The addition of a ceramic powder has shown no detrimental effect on the mechanical strength of the graphite base adhesive: the results in flatwise tensile tests are comparable to those obtained with an organic bonding. The scalability of this process has been assessed on a quite large sandwich panel: with an optimized cement application process. From the tests performed, there is no scale effect on the mechanical strength of the assembly, which was not the case for the previous inorganic bonding developments.
This work has been performed with Politecnico di Torino for cement development, a laboratory of the University of Torino with a great competence in the preparation and characterization of glasses and ceramic matrix composites for several applications, and thanks to ESA support.

I. CARBON/CARBON TECHNOLOGY FOR SPACE APPLICATIONS

For high performance space instruments, there is a growing demand for structural dimensional stability that is required to guarantee accurate relative positioning of components.
Among various advanced materials, Carbon / Carbon (C/C) can enable to achieve ultra stability as it is not sensitive to moisture absorption.
This composite can be specifically designed and manufactured in order to offer a remarkable quasi null coefficient of thermal expansion (CTE). This property, combined with good mechanical characteristics, allows its use for large stable and heavily loaded structures.

GOCE gradiometer (Fig. 1)

Carbon/Carbon technology has been selected for the construction of the sandwich panels, which support the accelerometers and their electronics, as well as the thermal control unit for the Electrostatic Gravity Gradiometer (EGG), the main payload of the Gravity field and steady state Ocean Circulation Explorer (GOCE) satellite.

GOCE has been launched 17th March 2009 and, since September 2009, it delivers data to build the best Earth gravity map ever. The tiny differences between the measurements of the six gradiometers that compose the core of the scientific payload can be related to variations in the Earth gravity. This is made possible by to the high stability of their supporting structures that ensure a constant relative positioning of the gradiometers.

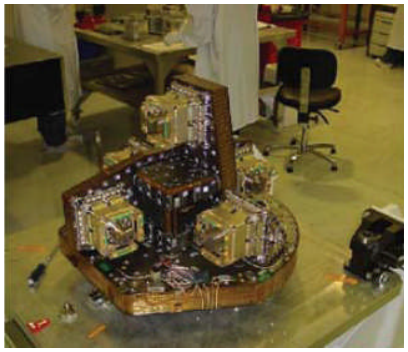

Fig. 1 : GOCE gradiometer

PLEIADES telescope (Fig. 2)

The monolithic cylinder between the primary and secondary mirror is made of Carbon/Carbon. Besides the stability and mechanical performances, a high level of cleanliness has been achieved for the structure so as to avoid optics contamination.

Fig 2 : PLEIADES telescope

2. SANDWICH MANUFACTURING

Carbon / Carbon composites are made of carbon fibers, the “textile preform”, linked together by carbon material, the “matrix”. Several types of carbon fibers are able to be arranged in 1D, 2D or 3D architecture to make the textile preform. A high temperature heat treatment can be performed to improve the thermal characteristics of the C/C composite. The carbon matrix is introduced into the fibrous textile preform using a Chemical Vapor Infiltration (CVI) process.

For Carbon/Carbon sandwich panels, the bonding between facesheets and honeycomb core can be ensured either through codensification of facesheets and honeycomb core (leading to a pyrolitic bonding) or through an organic adhesive bonding like for standard aluminum or Carbon Fiber Reinforced Plastic (CFRP) panels.

The preferred method is a pyrolitic bonding because it yields better stability characteristics: low CTE, no sensitivity to moisture or ageing. Unfortunately, this kind of bonding which works for sandwich with thin facesheets, is not efficient for sandwich panels combining high thickness (1.5 to 3 mm) and large size (around 1 sq m) like for GOCE gradiometer: an organic bonding has been qualified for these panels [1].

Further to GOCE, a development has been undertaken in order to obtain an ultrastable secondary bonding of the facesheets onto the honeycomb core.

The main requirements are summarized in Table 1.

Mechanical characteristics	Required performances (room temperature)
Shear strength	Ideal : σ_U = 12 MPa Acceptable : σ_U = 6 MPa
Young modulus	As low as possible Ideal 20-30 GPa Acceptable 40-60 GPa
Coefficient of Thermal Expansion -50 / +50°C	Ideal: $\leq 5.10^{-6}$ °C^{-1} Acceptable: $\leq 7.10^{-6}$ °C^{-1}
Coefficient of Moisture Expansion	0

Table 1: Main requirements for the bonding

But there are also manufacturing needs such as:

- Curing temperature lower than 300°C if possible, in order to realize the bonding with standard curing facilities
- Duration of the curing cycle as short as possible
- Bond thickness : as thin as possible (if possible in the range of that of an organic adhesive : approximately 200 µm)
  Process compatible with differences in height across the honeycomb core(honeycomb manufacturing tolerance : ± 0.1 mm)
- Process compatible with different substrates (different providers for C/C, different skins and honeycombs thicknesses).
- Scalability to large structures (1m^2) :
  - Solidification delay long enough to bond large structures (at least 2 hours);
  - Restraint not too important (difficult to quantify at this point);
  - Pot-life : at least 2 months.

3. CEMENT DEVELOPMENT

The joining materials have been selected by Politecnico di Torino, taking into account the requirements listed above. Metal brazing alloys as well as polymeric precursors of ceramic adhesives, with and without ceramic powder, have been studied and characterized through the following tests:

- preliminary wetting tests on C/C skins;
- measurement of the apparent shear strength of the joints during compression tests (single lap tests); observation of the fracture surface morphology
- optical microscopy on polished cross-sections;
- measurement of the Young modulus of the joint with a nanoindenter.

A trade-off has been performed in Thales Alenia Space to select the most interesting solution, with respect to numerous criteria :

- characteristics of the bonding;
- applicability
- manufacturing parameters
- supplying.

According to this trade-off, the chosen baseline was a commercial graphite base adhesive, with ceramic fillers with a negative coefficient of thermal expansion (CTE).

Complementary tests like X-ray diffraction and Thermal-Gravimetric Analysis (TGA), as well as mechanical tests like single lap shear have been performed by the Politecnico in order to optimize the choice of the negative CTE fillers, the filler content and the joining process on C/C plates (curing parameters and external pressure) [2].

The application of the bonding process to sandwich geometry has needed a lot of complementary tests, in order to evaluate the enduction process on honeycomb. These tests have been performed in Thales Alenia Space, at first on small samples, then on larger samples. These tests included the following: examinations on polished cross-sections, evaluation of shear strength on C/C flat samples, evaluation of flatwise tensile strength on sandwiches. All these tests have allowed defining a process which is compatible with an industrial manufacturing, and applicable to large dimensions sandwich structures.

4. EVALUATION OF SANDWICH PERFORMANCES

Coefficient of Thermal Expansion (CTE)

This measurement has been performed in a test facility which allows measuring CTE from –175°C to 150°C (performed at PMIC, United States of America).
The CTE of the sample has been determined with a Michelson laser interferometer measurement system (following ASTM E 289-04), under vacuum.
Length change measurements are taken as the specimen temperature is cycled between 150°C and –175°C for 3 full temperature cycles. Specimen length change and temperature have been continuously recorded during the tests (Figure 3).

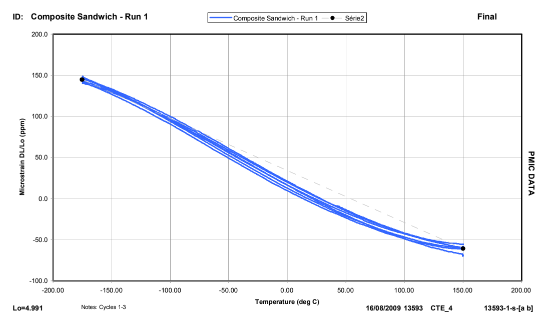

Figure 3: Measurement of the Coefficient of Thermal Expansion

The obtained value is – 0.6 10^{-6}/°C which is more negative than the value measured for the faceskin (-0,3 . 10^{-6}/°C around room temperature), but in a wider thermal range and with a different testing device.
Further measurements of CTE are being implemented.

Thermal conductivity

Even if the thermal conductivity of the adhesive is expected to be good (graphite base), the transverse thermal conductivity of the whole sandwich is linked to the transverse conductivity of the honeycomb : calculations can show that the adhesive layer is not a strong contributor in the value of the conductivity.

The value measured at room temperature for the sandwich is 1.1 W/mK.

Flatwise tensile

The flatwise tensile strength has been evaluated at different steps of the development because it gives good indication about the quality of the bonding. The result is correlated with the quantity of inorganic adhesive. With the final process, the tensile strength varies between 3 and 4 MPa, which is a very high value for this kind of test.

Compressive

9 specimens coming from 2 large sandwiches have been tested at different temperatures. Some facing

blocks are bonded to the samples in order to apply the compression loads (for the samples tested at room temperature, these blocks are in resin; for the other samples, the resin is loaded with short carbon fibers). The obtained results are reported in Table 2.

T°C	Average modulus (GPa)	Standard deviation (GPa)	Average strength (MPa)	Standard deviation (GPa)
20	70.7	0.5	167	6
150	74.1	1.5	151	11
-175	83.8	8	145	3

Table 2: Sandwich compressive properties for different temperatures

The failure mode is purely compressive for all the samples. The average compressive properties are of the same range order than those obtained on GOCE geometry. The effect of temperature remains low with regards to the tested temperature: -10% for 150°C strength with respect to 20°C results; -13% for a testing temperature of –175°C. This confirms the stability of the inorganic adhesive within a large range of temperature.

Scalability to large structures

Since the beginning of this program, as lesson learned from past activities, the process scalability has been one of the most important concern. Indeed, joining skins to honeycomb core through co-densification during a CVI process (Chemical Vapour Infiltration) which enables to consolidate the C/C structure, cannot provide an efficient bonding for large structures: there is a gradient of the carbon deposition from the edges to the centre of the panel leading to a poor consistency of characteristics.

So, even the first manufacturing campaigns with the developed cement have been performed with operations suitable for an industrial process and for large dimensions samples. The scalability of the process has been demonstrated on a panel of 420x480 mm2. The flatwise tensile strength of this panel has been evaluated on several samples taken at different locations: the obtained results are the same than those obtained on smaller panels. There is no scale effect on the mechanical strength, which was not the case for the previous inorganic bonding developments.

5. CONCLUSION

A new inorganic joining technology for C/C sandwich panels has been successfully developed which can very likely fulfill the main requirements for the future ultrastable structures needed by space industry:

- The CME of the ceramic adhesive is null hence hygrometry control is not necessary
- The CTE of the bonding material can be tuned by adding filler material – It has been lowered by adding negative CTE ceramic particles
- The mechanical strength of the bond is high, comparable to that of organic bonded assemblies and not adversely impacted when adding a filler having a negative CTE;

This bonding technology can address a lot of applications, according to the good thermal and mechanical performances obtained during this study, at room temperature but also for higher temperatures.

To assess the potential of this bonding solution further and expand the range of potential applications, the cement will be tested on other substrates (like ceramic substrates) and on larger temperature ranges. In parallel, the cement will be used on different sources of C/C.

The graphite nature of the adhesive is favourable for thermal conductivity, which evidences the technology to future high temperature antennas or mirrors.

[1] Development of Carbon-Carbon sandwich panels – 9th International Symposium on "Material in Space Environment" – Noordwijk, the Netherlands 2003

[2] Study of joining of Carbon/Carbon composites for ultra stable structures - Journal of the European Ceramic Society, ISSN 0955-2219; 2010, vol.30, n°7, pp 1751-1759