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## *Si<sub>3</sub>N<sub>4</sub> ceramic application for large telescope development results*

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# Si<sub>3</sub>N<sub>4</sub> ceramic application for large telescope development results

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**Abstract**— Thales-Alenia-Space has identified the ceramic Si<sub>3</sub>N<sub>4</sub> as an interesting material for the manufacturing of stiff, stable and lightweight truss structure for future large telescopes. Si<sub>3</sub>N<sub>4</sub> ceramic made by FCT has been selected for its own intrinsic properties ( high specific Young modulus, low CTE, very high intrinsic strength for a ceramics ) and its cost effective beams manufacturing capabilities.

In order to qualify beam and beams end fittings for future large and thermo-elastic stable truss structure for space telescope, full development and tests activities have been performed. Manufacturing process has been optimised in order to obtain a very high reliable strength.

Full scale beams with thin wall have been manufactured and tested in bending and in tension. Full scale beam assembly with integrated junctions have been manufactured and tested up to ultimate loads and have been space qualified.

Beams end fittings made also in Si<sub>3</sub>N<sub>4</sub> and its direct bolting capabilities have been also space qualified by tests.

Beside this qualification for current space telescope, developments are continuing thank to CNES R&T to develop high loaded brazed junction between Si<sub>3</sub>N<sub>4</sub> parts, enhanced thermal conductivity and mechanical strength through Si<sub>3</sub>N<sub>4</sub> formulation and manufacturing process tuning.

*Keywords*- Structure, truss, ceramic, telescope, space

## I. INTRODUCTION AND OBJECTIVES

### A. Context

More and more space mission requires for earth observation or astronomic observation, telescope offering very high spatial resolution leading to telescope with large M1 mirror and with a M2 far from M1. Therefore actual and future large space

telescopes require a huge truss structure to hold and locate precisely M2 mirror.

Such large structure requires very strong material with high specific stiffness and low CTE.

Based on the Si<sub>3</sub>N<sub>4</sub> performances associated to FCT experience to manufacture rather complex parts, Thales Alenia Space has engaged since some years with FCT development activities to develop and qualify Si<sub>3</sub>N<sub>4</sub> parts for space projects.

Thanks to the high strength, its low CTE and manufacturing capability it has been decided to develop future large truss structure fully composed of Si<sub>3</sub>N<sub>4</sub> elements to be as much as possible stable, stiff and lightweight. Demanding high stability large space telescope structure could be made fully in Si<sub>3</sub>N<sub>4</sub> components to link with very high stability M2 mirror to M1 supporting baseplate.

### B. Definition of Si<sub>3</sub>N<sub>4</sub> telescope truss structure

Following deep material trade-off, TAS has selected the Si<sub>3</sub>N<sub>4</sub> to perform the large M1-M2 truss structure for next high resolution earth observation telescope for French MOD. The large truss structure is composed of 6 long Si<sub>3</sub>N<sub>4</sub> beams, with Si<sub>3</sub>N<sub>4</sub> end fittings connecting the beam to telescope optical bench. On the top of the beams Si<sub>3</sub>N<sub>4</sub> endfittings link the beams to a M2 hexagonal frame.

End-fittings made in Si<sub>3</sub>N<sub>4</sub>, allow to obtain very lightweight, links between beams, such part carrying heavy loads from the truss structure to the bottom part of the platform.

Therefore foreseen telescope truss structure is made of Si<sub>3</sub>N<sub>4</sub> beams of d=80mm with a thickness of less than 3mm, truss structure length being dependant to the project application typically between 1 to more than 2 meters.

Truss beams have been sized vs requested space loads and stiffness to fulfil satellite requirements and to be compatible of

launch and space environment. Diameter and thickness of the beams has been determined to limit dynamic amplification and avoid deep secondary notching at M2 level.

Elementary manufactured 1,3 meter beams are joined together by glued junction as today FCT furnace capability limit the length to around 1,5 meter, but a new furnace now in operation allow to built beam of 1,8 meter.

Such structure has been now fully qualified and all flightparts have been produced and flight proof tested..

Taken benefit of this qualification, TAS proposes also in the frame of ESA Cosmic vision to perform the truss structure of Euclid telescope in Si3N4.

Euclid instrument is foreseen to observe dark energy and dark matter and scheduled to be launched in 2017. The telescope is a Korsch telescope with pupil diameter of 1,23 m and a distance between M1M2 of 1,8 meter.

Thanks to the very low CTE both of the zerodur mirrors and the M1-M2 Si3N4 truss structure we reach a very thermo-elastic stable telescope, element essential for the Euclid mission.

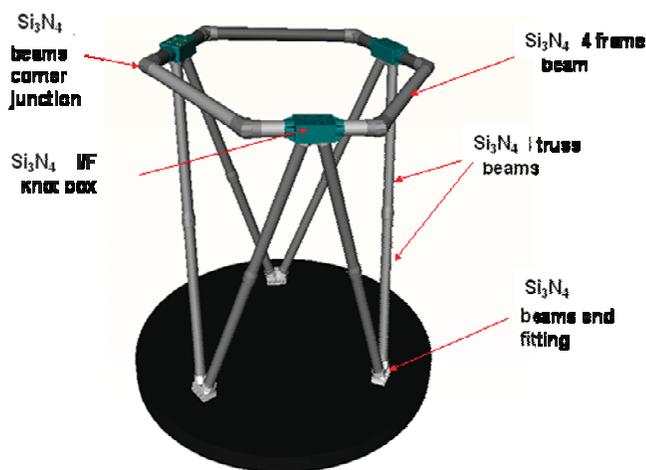


Figure I-1 Example of a 2 meter telescope Si3N4 truss structure designed for Euclid telescope in the frame of ESA cosmic vision project

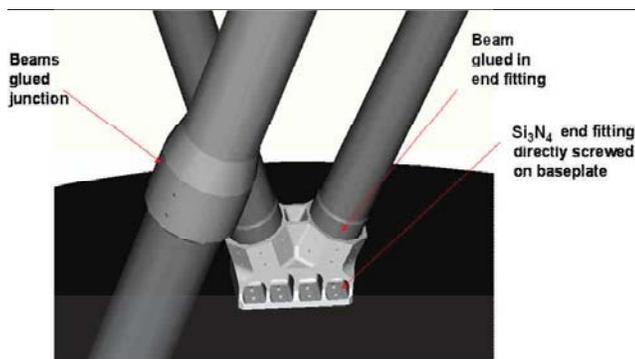


Figure I-2 Full Si3N4 junction allowing a direct connection of the truss to the instrument baseplate

## II. Si3N4 PROPERTIES AND MANUFACTURING PROCESS

### A. Si3N4 Material properties

The Si3N4 standard grade for gaz pressure sintering GPSN is a composition of 90% Si3N4, 6% Al2O3, 4% Y2O3 which is sintered at 1 MPa of nitrogen gas pressure. It can be brought to improved mechanical properties using HIP process up to 1,850°C and pressure up to 200 MPa.

Material		GPSN	HIPSN
Density	g/cm <sup>3</sup>	3.25	3.22
Youngs modulus	GPa	320	330
Thermal cond.	W/mK	25	30
Bending strength	MPa	700	1,000
CTE RT	10 <sup>-6</sup> /K	1,4	1,4

Table II-1 Properties of silicon nitride ceramics for lightweight structures

Si3N4 offer also a very high weibull modulus up to 20, offering a high strength reliability allowing to size structure with very low probability of failure even for large area highly loaded.

One of the most sensitive parameters for large optical components is the CTE. At room temperature, CTE of Si3N4 has been precisely measured and offer a very low CTE of 1,4 10<sup>-6</sup> m/mK and is therefore one of the lowest available value in combination with low density, high stiffness, strength and long term stability. CTE reproducibility has been also measured using samples issued from different batches and thank to the mastering of manufacturing precise parameters, such value is fully reproducible. In the range of 150 K the CTE is near 0 and is 0,9 10<sup>-6</sup>m/mK at 235 K (Euclid operational temperature). The CTE of corresponding materials for optical use is shown for the temperature range between -100°C and +100°C.

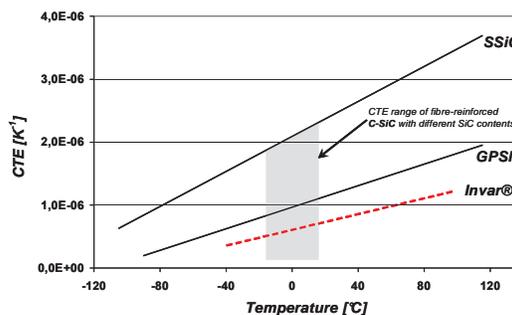


Figure II-1 Coefficient of thermal expansion CTE for structural “optical” materials

B.  $Si_3N_4$  manufacturing capabilities

FCT Ingenieurkeramik has developed different ceramic material like  $Si_3N_4$  and  $SiC$  as well as some composite Materials like  $C_f/CSiC$  and the corresponding process for the commercial fabrication of a broad range of components with mainly large and complex sizes and shapes and e.g. very narrow tolerances.

$Si_3N_4$  material has been identified as key interest for space instrument structural parts using already mastered manufacturing processes :

- slip casting for complex, thin walled components or
- cold isostatic or uniaxial pressing of preforms
- green machining up to thin and precise shape,
- sintering, gas pressure sintering, HIPing
- final machining by laser cutting, grinding, honing, drilling, lapping and polishing.

One of the key manufacturing properties is to be able after sintering to drill without difficulty holes allowing very precise assembly without loss of strength .

Rather large components with one dimension up to 1.8 m in length and/or 0.7 m in diameter could be now manufactured . FCT produce prototypes according to customers design requirements but also small and intermediate series up to 10,000 pieces per year or fabrication lot, whenever prototypes were successfully tested.

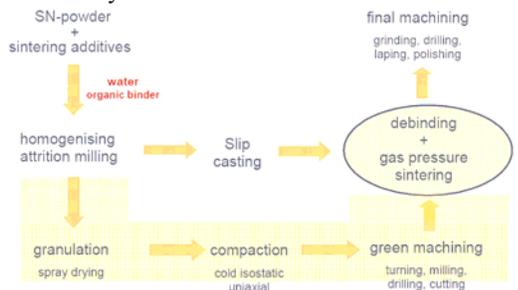


Table II-2 production process for silicon nitride lightweight component

III.  $Si_3N_4$  TRUSS STRUCTURE DEVELOPMENT AND QUALIFICATION TESTS

Based on the truss design for space telescopes, beams and end-fittings have been manufactured by FCT and space qualified by TAS-F.

Material has been firstly fully characterised in order to determine its basic properties and more over strength probability and reliability through a large tests campaign on different manufacturing batches and furnace runs.

The chosen mechanical tests were 4-point bending tests, which is among the possible tests on ceramics, the easiest to perform and the easiest to exploit. This test is frequently done in TAS on different ceramic materials, which allow the statistical behaviour of the new material to be compared directly with an

important material database. A 4-point bending test jig was developed to evaluate the strength on larger surfaces (800 mm<sup>2</sup>) than those usually tested in other labs allowing to have more reliable data on sufficient loaded surface area. The samples dimensions are thus chosen in order to be representative of the stress area encountered in the real flight structure. With this test jig it is also possible to measure the Young's modulus of the sample.

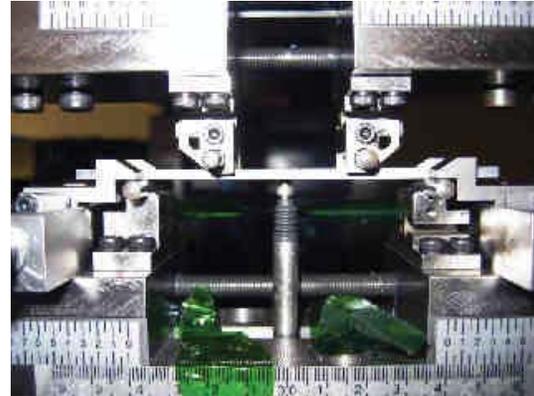


Figure III-1 4 pt bending test set up on large samples

Strength probability and Weibull modulus are therefore determined and batches compared between each other showing a good results reproducibility between batches. During the flight part manufacturing a lot of witness samples are manufactured following all the flight part process and using the same run. For the beam , an extra length of the beam is sintered with the beam and this extra length is cut in 14 bending samples .

To validate the full truss strength a statistical approach based on stochastic method is implemented. Qualification and sizing has not be only performed though classical tests campaign on 4pt bending samples but also on numerous beams and end fittings through tensile tests up to rupture to validate the statistical correlation between samples strength repartition and the beams and end fittings strengths level and statistical repartition.

Such crossed tests has allowed to determine the real truss safety margin vs launch loads with a good reliability, statistically spoken.

A lot of Beams of 1.3 meter length, d = 80mm with a thickness of 2.5 mm to 3 mm have been manufactured by FCT for the development and qualification campaign .



Fig. 8 full scale  $Si_3N_4$  beam (L=1.3 m, D=80 mm,  $W_{th}$ =2.5 mm)

TAS-F has then performed on first beam a full scale 4 pt bending test up to rupture. The test was followed by an ultra rapid camera (22,000 images per second) allowing to follow the cracks initiation and its propagation. Cracks start as

predicted at the most tensile loaded area, at a level corresponding to the probable rupture domain, domain determined by Weibull law considering the loaded surface.

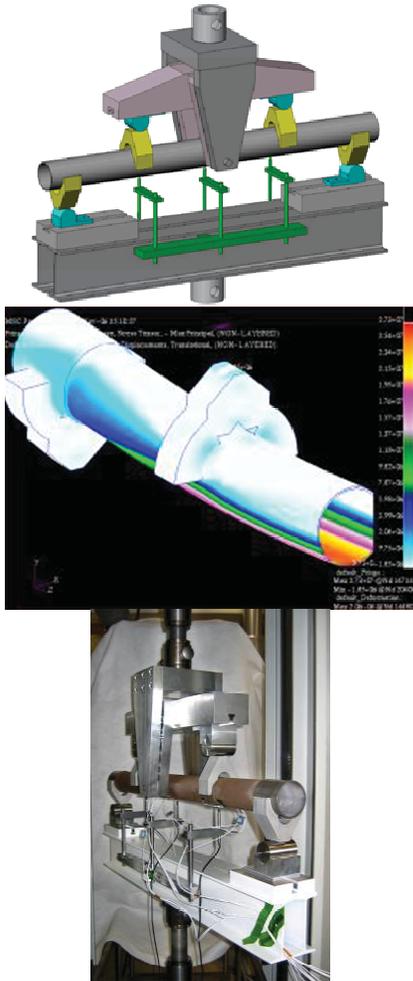
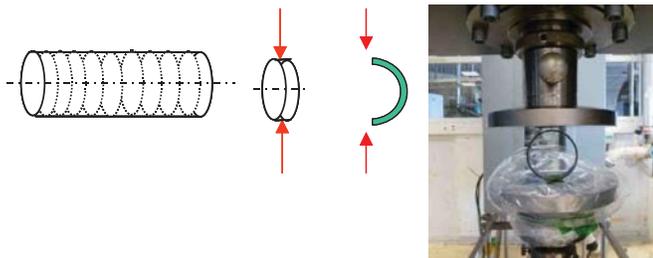


Figure III-2 bending tests up rupture including FEM comparison and rupture mode

Manufacturing process improvement have been also conducted prior the qualification campaign to optimise the strength statistic; Beams and endfitting are submitted after sintering to a polishing step which allow to obtain a surface with very low roughness ( $Rz < 1\mu m$ ) internally and externally allowing to avoid any surface voids susceptible to generate early cracks propagation .

To validate the improvement of the process, full scale beams have been manufactured then cut in slices leading to rings. Rings have been tested in compression leading to a tensile stress on the internal surface. After rupture the 2 remaining parts C ring are then tested in compression leading to tensile stress on the external surface.

Remarkable high and homogeneous strength have been then measured in the current section of the beams .



strength of the beam measured on slices internal t

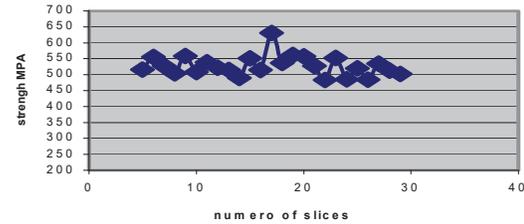


Figure III-3 Tensile strength repartition along beam length

Full tensile tests on representative beams up to rupture have been also performed, results being compared to test prediction and confirming the stochastic approach..

Beams are equipped with strain gauges to follow up stress development in the current area and in the main loaded areas.

The results have allowed to confirm the stiffness of such assembly and to validate the strength level. Such early tests have allowed to better understand the failure mechanism and determine residual safety margin of flight representative beams vs the flight loads.



Figure III-4 qualification beams with polished surface and glued tests I/F

Gluing techniques have been developed and space qualified through a complete tests campaign including mechanical cycling and thermal ageing. To realize beam of more than 2 meter length 2 beams are assembled together by gluing using a female and a male beam .

Glue is introduced by injection through small holes located on the female beam at junction area. I/F junction have been carefully machined after sintering to offer very high accuracy positioning and constant glue thickness. Thanks to appropriate surface preparation only cohesive rupture has been observed with sufficient margin.



Figure III-5 Beams assembled by gluing and glued in Ta6V end fitting for tensile tests

Glued junctions between two beams has been mechanically tested up to rupture and tests have been performed on numerous assembly beam parts to check the reproducibility of the strength.

One assembly beam has been submitted first to 5 thermal cycling (-20°C, +50°C) then to 30 cycles at 60000 N and then 30 cycles at 72000 N loads cycles covering maximal stress level including margin see during qualification launch load of such a truss and then truss has been tested up to rupture, demonstrating the safety factor.

The cycles have not impacted the strength of the beams and of the junction. Rupture occur all the time in the thinnest part of the beam and not in the junction area.



Figure III-6 Glued joined assembled beams for tensile test under tensile tests machine

To qualify the full beam assembly two beams at the real flight length have been glued together leading to a beam of more 2 meters . This full scale beam assembly has been submitted to:

- mechanical cycles with residual elongation measurement → no elongation have been measured even in the glued area
- CTE measurement which have confirmed the CTE measured on samples
- Up to rupture in tensile and in this test more than 120000mm2 was loaded → rupture was nevertheless largely above the success criteria showing the large mechanical margin offered by such assembly.



Figure III-7 2meter joined beams tested in tensile

To join together the beams and join the truss to main platform structure, 2 beams are connected together to a Si<sub>3</sub>N<sub>4</sub> end fitting , this end fitting being bolted to the bottom structure. End fitting design has been optimised and sized by FEM model to allow full loads supporting including high safety margin.



Figure III-8 view of the Si<sub>3</sub>N<sub>4</sub> end-fitting part

Bolting tests with large screws up to M10 diameter have been performed on Si<sub>3</sub>N<sub>4</sub> assembly. Thanks to high Si<sub>3</sub>N<sub>4</sub> fracture toughness, for all the tests, rupture of titanium screws has been reached before any damage of the Si<sub>3</sub>N<sub>4</sub> parts.

To tests all the assembly, a Si<sub>3</sub>N<sub>4</sub> beam has been glued in the Si<sub>3</sub>N<sub>4</sub> end-fitting and tested up to rupture.

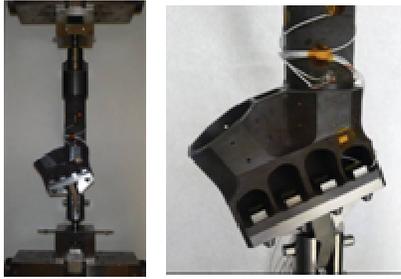


Figure III-9 beam end fitting junction tensile test and view of the screwed end fitting on the I/F plate



Figure III-10 FEM prediction of the test showing the stress distribution in the assembly (local highest stress peak is in end fitting when major large stressed area is in the beam)

During tensile test the end fitting was screwed flight representatively to I/F plate demonstrating the screwed junction capability to such heavy loaded I/F. Rupture occurs in the main section of the tube as predicted by Weibull probabilistic law, even if the maximum peak stress is locally obtained in a small stress concentration area of the end fitting. This confirms that, as predicted by Weibull law, failure occurrence is linked not only to the maximum stress but also to the loaded surface area. To qualify the end fitting themselves, tests of the end fitting alone have been performed. Test of the strength of the Si3N4 end fitting in the bolted attachment area has been subjected to special attention: The strength of the bolted Si3N4 bracket has been validated by tests, even in the worst cases of attachment. This verification of Si3N4 end fittings strength have been done in the worst interface configurations for the bolted attachment with the Titanium insert:

- Traction load applied with a degraded flatness of the interface: 30µm for the tool Insert instead of 10µm for the flight one,
- Traction load applied with a misalignment of the bracket w.r.t the tool insert: bolts decentred of 0.4mm in the bracket hole.

Detailed FEM Simulation of the unflatness of the interface has been performed showing the slight stress increase due to the I/F unflatness.

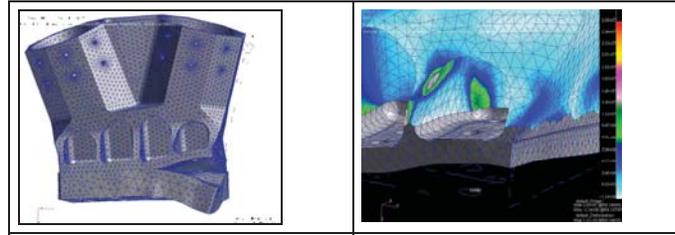


Figure III-11 A unflatness of 30µm at I/F generate 50 Mpa stress increase

For these both degraded configurations, no loss of strength have been measured with regards to the nominal configuration (30µm of flatness and bracket centred): rupture over 200 000 N for a level of stress around 500 MPa.

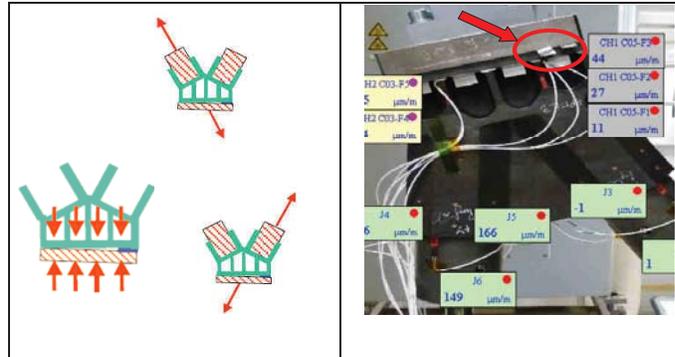


Figure III-12 Tensile test on end fitting with 30 µm unflatness at the I/F

All these tests have allowed to qualify the end fitting and endfitting junction with large margins and thank to Si3N4 strength and high fracture toughness I/F bolting is done without precaution and any risk.

For the connection to the M2 frame today a solution with 2 parts has been used the Si3N4 endfitting being connected to a Titanium M2 frame bracket. To save mass and reduce the number of pieces FCT has developed a more complex end fitting allowing a direct connection of the beams of the truss with the beam of the M2 frame through an integrated truss connecting Si3N4 node.

Hereafter the Si3N4 demonstrator for the monolithic "truss bracket - M2 frame insert": This monolithic part could replace if necessary for thermo elastic local behaviour, the nominal assembly: Si3N4 bracket and titanium insert



Figure III-13: Si3N4 demonstrator for the monolithic "truss bracket - M2 frame insert"

#### IV. PROOF TESTING OF FLIGHT BEAMS AND END FITTING

For the acceptance of the flight parts, NDT control are first performed ( dye penetrant test ) and witness samples tested.

Nevertheless, to prevent any risk, each flight part is then proof tested before use. Therefore all the beams and all end fitting are individually tested under tensile loads which correspond to 1,3 time the qualification loads .

For this test each beam has an extra length on which dedicated test tool end fitting will be glued. After the proof test flight beams will be cut to the appropriate length and joined together. The beams are proof tested up to 60 000 N while during qualification test campaign beams have been conducted up to rupture occurring above 100 000 N .



Figure IV-1 Tensile proof test on flight beam

For the end fitting, a special gluing procedure has been developed, therefore loads are introduced in the both end of the endfitting through invar blades tools. After the proof test at 1,3 the qualification loads the invar blades are unglued by heating .



Figure IV-2 Proof test on the flight end fitting

Following the qualification process, 24 flight beams and 12 flight end fittings ( corresponding to two flight models ) have been manufactured and proof tested with success without any rejection .

#### V. IMPROVEMENT IN PROPERTIES

As Si<sub>3</sub>N<sub>4</sub> is multiphase ceramic material with large content of additive it is possible to enhance its properties by adapting the type of the additive which were selected at the origin for high temperature application. In the frame of a past study in the frame of an CNES R&T contract (R&T 2008), the interest of working on the properties of Silicon Nitride in order to obtain a material with higher mechanical properties, lower CTE and higher thermal conductivity has been highlighted for future applications. A study has thus started in partnership with FCT in Germany, it consists in defining specific Silicon Nitride formulations in order to increase the thermal conductivity, without decreasing the mechanical strength and not increasing the coefficient of thermal expansion (CTE). The targeted value of thermal conductivity was 60 W/mK. A bibliography study has allowed to identify the different ways of thermal conductivity improvement, taking into account the properties of the reference material, the thermal conductivity aimed and the intended application to an industrial material for beam and truss structures :

- The modification of sintering additives;
- The replacement of standard powders with higher grade Si<sub>3</sub>N<sub>4</sub>-nanopowders for higher thermal conductivity and lower sintering temperature;
- The addition of high conductivity particles, like SiC or AlN or Carbon NanoTubes (cubic BN or diamond powder).

After several manufacturing campaigns in order to evaluate these different solutions, it has been assessed that the work on sintering additives was necessary and sufficient before any other modification. Three formulations with different sintering additives have been selected and tested. After a first industrialization manufacturing campaign, one grade which seems more promising concerning the scalability to industrial parts, has been selected.

Some mechanical and thermal tests have been performed on samples manufactured in the industrial facilities of FCT (granulation and sintering facilities).

Then 4 pt bending on large samples have been performed to be compared to the values qualified on the standard grade.

The results have shown that the Young modulus is quite the same than the one of the reference grade and shown an expected slight decrease of the strength : the strength of the new grade is 25% lower than the industrial grade..

The fractographic and SEM observations of the samples have shown that some residual porosities were still visible : these defects can be at the origin of the failure. Some fine tuning of the sintering process are still possible in order to improve the sintering cycle and thus reduce the presence of these porosities.

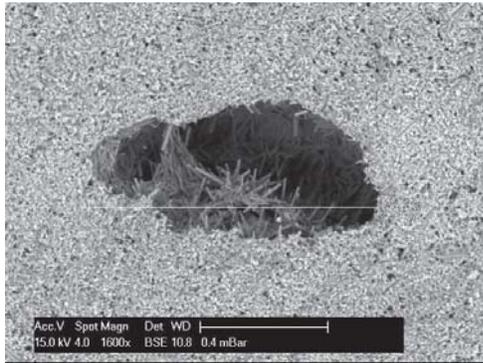


Figure V-1 : residual porosity found on the tested samples

The CTE measurement is performed with a Michelson laser interferometer : 2 different samples can be tested in parallel. The measurements have been always performed with the same reference sample : a sample from the industrial silicon nitride grade. The results have shown no significant impact on the CTE compare to the industrial .

The thermal conductivity test jig used in Thales Alenia Space Cannes are based on a permanent measurement under secondary vacuum . The sample is put between a cold spring and a warm spring inside the test facility and a constant thermal flow is crossing the sample between the warm spring and the cold spring. The temperatures are measured near the cold and warm springs and also on the sample during the temperature stabilization phase and after the thermal flow injection.

The thermal conductivity, measured on 4 samples on the new grade, is very close to 80 W/mK, to be compared with a conductivity of 15W/mK for the reference industrial grade.

As a conclusion, the thermal conductivity of the new grade is well improved and compliant with the objective of the study. The recommendation for the next step is to understand and optimize the associated manufacturing processes and to manufacture large parts with this grade and test it under the same condition as the qualification tests performed on the standard grade.

## VI. BRAZING DEVELOPMENT

In order to benefit from the large strength of Si<sub>3</sub>N<sub>4</sub> on extended size or for more complex shape ( closed back design ) metal brazing development activities has been launched under CNES funding. After an important trade off , active metallic brazing has been selected as it offers the most important strength capacity.

Active metal brazing is a well established technique for joining ceramics to themselves and/or to metallic compounds. The active element in the brazing filler such as Ti or Zr, is characterized as having sufficient thermodynamic driving force to destabilize the ionic or covalent bonding of the ceramic by reacting with one or more of the ceramic elements in order to form a reaction layer. Ag-Cu-Ti especially shows good wettability to Si<sub>3</sub>N<sub>4</sub> ceramic.

The brazing has been developed and performed by PMB Incusil ABA paste was then used to braze Si<sub>3</sub>N<sub>4</sub> substrates to each other.

Two brazed configurations were achieved: overlapping brazing and endwise brazing. In the case of overlapping brazing the joint was sheared through compression test while endwise brazing was dedicated to 4 points bending test.

The aim of the latter configuration is to test the strength of the joint and to compare the failure stress obtained to the one performed on the same sample that was not brazed ( 120mm length Si<sub>3</sub>N<sub>4</sub>)

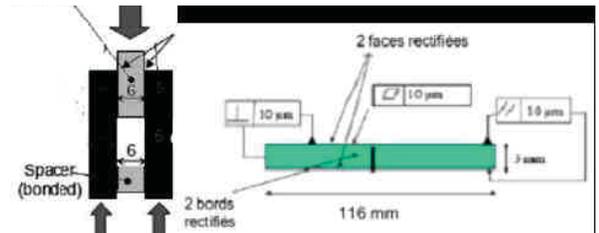


Figure VI-1 shear tests 4 pt bending tests samples

The brazing paste was placed between two samples of silicon nitride and the joint was calibrated to 100μm for both configurations. Such large thickness has been selected to be representative in worst case of tolerance on large part, as more thick is the brazing less is the strength.

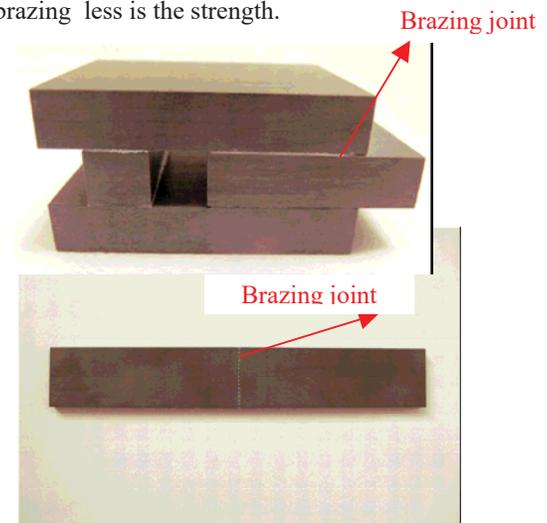


Figure VI-2 View of the shear and 4 pt bending samples

. Then the assembly was brazed around 700°C using specially designed graphite tools. During brazing process a pressure was exerted on the samples in order to ensure each part contact closely in spite of CTE mismatch between graphite and silicon nitride.

During brazing, Ti diffused toward the Si<sub>3</sub>N<sub>4</sub> substrate in order to form a continuous layer of TiN. The free energy of formation of TiN and Si<sub>3</sub>N<sub>4</sub> are both negative at brazing temperature. The strength of the brazing joint relies on the good formation of this layer at the interface with silicon nitride substrates.

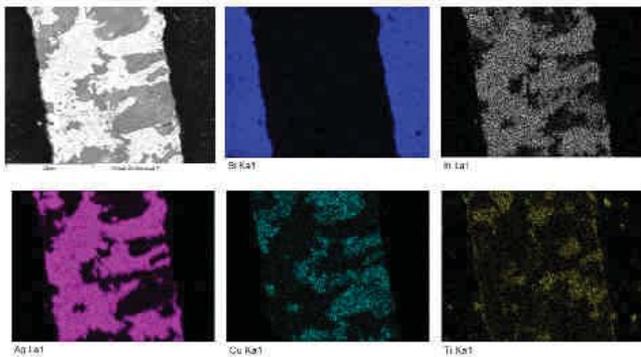


Figure VI-3 composition repartition of the different agent inside the bonding

After brazing the parallelism and perpendicularity of the shearing samples and 4 points bending tests samples sides was below the  $25\mu\text{m}$  requirements. This condition was fulfilled by PMB as well as flatness between the two sides of 4 points bending test samples of  $30\mu\text{m}$ . 6 samples for each type was then tested up to rupture. Each value is a mean value issued from these 6 tests. The standard deviation was below 10%.

During the compression shear test, loads was so high 120 000 N ie 800Mpa in compressive in  $\text{Si}_3\text{N}_4$  that the failure occurred simultaneously in the ceramic substrate and in the joint which is an indicator of the strength of the bonds between the joint layer and the silicon nitride substrate.

In the case of 4 points bending test the failure occurred in tensile in the joint at 262 MPa which is relatively high compared to other ceramic brazing..

Today final optimisation of brazing composition and brazing condition are still running before to test the brazing on real large parts ( beam direct junction for example).

## VII. CONCLUSION

$\text{Si}_3\text{N}_4$  structural ceramic is now fully space qualified thank to intensive manufacturing campaign performed by FCT and intensive and exhaustive tests campaign performed by TAS. This new technology is now offered for the realisation of very lightweight stiff strong and stable structure for space use.

Qualification results and proof tests results obtained on new French MDO space large telescope could benefit to the realisation of the  $\text{Si}_3\text{N}_4$  truss structure of Euclid telescope which is similar in terms of design and performances .

Current development are still running to improve from the present standard material its properties as well parts assembly through promising results in thermal conductivity enhancement and through first results obtained in brazing of  $\text{Si}_3\text{N}_4$ .

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