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SILICON NITRIDE CERAMIC DEVELOPMENT IN THALES ALENIA SPACE : QUALIFICATION ACHIEMENT AND FURTHER DEVELOPMENTS FOR FUTURE APPLICATIONS

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I. INTRODUCTION

Dealing with ceramic materials for more than two decades, Thales Alenia Space – France has identified Silicon Nitride Si3N4 as a high potential material for the manufacturing of stiff, stable and lightweight truss structure for future large telescopes. Indeed, for earth observation or astronomic observation, space mission requires more and more telescopes with high spatial resolution, which leads to the use of large primary mirrors, and a long distance between primary and secondary mirrors. Therefore current and future large space telescopes require a huge truss structure to hold and locate precisely the mirrors. Such large structure requires very strong materials with high specific stiffness and a low coefficient of thermal expansion (CTE). Based on the silicon nitride performances and on the know how of FCT Ingenieurkeramik to manufacture complex parts, Thales Alenia Space (TAS) has engaged, in cooperation with FCT, activities to develop and qualify silicon nitride parts for other applications for space projects.

II. QUALIFICATION OF AN INDUSTRIAL GRADE OF SILICON NITRIDE FOR TRUSS STRUCTURES

FCT Ingenieurkeramik develops and produces different ceramic materials inside silicon nitride family : various sintering additives at different rates lead to different formulations or grades. During the manufacturing of the material, the chosen processes, in term of preform preparation (slip casting, cold isostatic pressing, uniaxial pressing), sintering modes (gas pressure sintering, hot isostatic pressing) and final machining (laser cutting, grinding, polishing), have also a great influence on the materials final properties.

In order to select the most appropriate grade and the associated processes, the final application of ceramic parts must be defined precisely. At the beginning of their cooperation, FCT and TAS have thus worked together to determine the most suited material for the manufacturing of a telescope truss. Then this material has been tested and qualified through a methodology which has been built in the frame of a study on a global structural strength reliability approach for structures.

This article focuses on the methodology followed to qualify the mechanical behavior of the tubes in silicon nitride (other tests are performed to determine properties like coefficient of thermal expansion, or glued or bolted junctions, but they are not developed here; some details are available in [1]).

The different steps of the testing methodology for the determination of tube strength are detailed hereafter.

A. Evaluation phase

The objective of this phase is to check that the material properties are in agreement with the specifications, and also to confirm the choice of the material for the future application. It is also verified that the material behavior is consistent with what is already known on the material (comparisons with database of similar materials for example).

In the frame of that phase, a large part, representative of future truss beam geometry, has been manufactured and mechanically tested (Fig. 1). The objectives were to check that the selected processes were adapted to the manufacturing of such large components, and also that the obtained strength was in line with the expectations. Some elementary samples have been also manufactured and tested : mechanical strength and thermal expansion have been tested to check that the properties were in agreement with current silicon nitride properties. These preliminary tests have allowed to definitely select the material for the truss application.

Fig. 1. Test of the first beam in silicon nitride (evaluation phase)

B. Characterization phase

The objectives of the characterization phase are numerous. During this phase, the manufacturing parameters have to be consolidated, and in particular their influence on the strength of the material has to be evaluated. The behavior of the material has also to be better understood, in particular with respect to its final geometry and application. The industrialization phase is thus prepared thanks to these first results.

During this phase, a first evaluation of the allowables of the future parts must also be done. This evaluation relies on the Weibull theory applied to tests results. At each test campaign, the apparent Weibull parameters (σ_θ, m) are directly derived from the Weibull plot. Depending on the fracture mode, the calculation of the effective volume or surface allows to determine the material characteristics (σ_0, m), which are material specific values and not relative to a particular set-up [2]. The allowables are then calculated with the help of these material characteristics. Determining these parameters require numerous tests and samples due to the statistic behavior of ceramic material. That is why it is important, as early as possible in the study of a new material, to try to choose the most judicious tests.

Indeed, at the very beginning of the characterization phase, it is essential to define the geometry of the future parts, their surface condition, and also the stress state in operation. This definition will condition the kind of tests to be performed in order to cover the whole operating conditions.

In the case of silicon nitride industrial grade, its main application is tubular parts with polished surface condition. The stress state in operation is both volume and surface loads, on both internal and external surface (close to tensile load).

After having specified the application, a list of points to be addressed during the characterization phase has been established. These points are the followings :

- The influence of granulation batches and sintering runs on mechanical strength and other properties;
- The influence of the loading direction on the mechanical behavior of the material;
- The homogeneity of the final part with respect to mechanical strength
- The link between elementary tests and the behavior of the final part (in order to predict the behavior of the final part from elementary tests)
- The definition of the witness samples for future manufacturing campaigns and the preliminary definition of the associated criteria.

In order to answer these questions, the following tests have been settled :

- Comparative tests : in order to assess the influence of manufacturing parameters. In that case the samples do not need to be fully representative of the final part but need to be sensitive to the same properties variations;
- Understanding tests : the samples need to be representative of the final part, with respect to geometry and manufacturing conditions.

Comparative tests.

The influence of manufacturing batches on the strength of silicon nitride parts have been assessed through 4-point bending tests on ground samples. Even if the manufacturing processes and the surface condition of the samples are not the same as the final parts, the behavior of material from different batches can be compared, and the influence of manufacturing batches evaluated. After these tests, fractographic analyses have been performed in order to identify the defect population responsible for failure. Bending test is a very interesting test, as it is easy to settle and to exploit. A specific test jig was developed to evaluate the strength on larger surfaces than those usually tested in other labs, with adjustable distances between supports and load application points. Details are available in [3].

A lot of granulation batches have been characterized with the help of this kind of tests. The treatment of all this data aims to check if all the different batches are compatible. The k-sample Anderson Darling Test (ADK) is used to check if the data coming from different batches can be pooled to be analyzed together (the definition of this test is given in [4]). If the data coming from different batches are poolable, the Weibull parameters are computed for all data groups and thus are valid for the whole manufacturing batches. If not, some procedures have to be defined in order to take into account the influence of the manufacturing batch on the mechanical behavior of the parts (cautions for the test plan in order to separate and determine the influence parameters, but also for the determination of allowables). Fortunately, all the batches tested in the frame of the characterization phase were compatible : some Weibull parameters have thus been defined for the whole data coming from this kind of samples. This showed also that the material was robust and reproducible : this was a good result for the industrialization of this material.

The information coming from comparative tests described previously is only valid for 4-point bending ground samples: they are not directly usable for the determination of allowables of the final part. Indeed, the behavior of the final part may not be predictable from these preliminary tests, because of the different surface condition, but also because of the manufacturing process : the manufacturing process of a tube is completely different from that of plates (e.g., compaction process, environmental conditions during debinding and sintering, …)

So after these results obtained thanks to several tests campaigns and numerous samples, complementary tests have been used : these are understanding tests, and they are depending on each geometry and application.

Understanding tests.

In the case of massive parts, 4-point bending tests on plates with the same surface condition can be useful. But in the case of tubes in silicon nitride, different kinds of understanding tests were necessary.

In particular O-ring and C-rings tests have been performed on samples machined through the slicing of a representative tube (Fig. 2). These tests allow to evaluate the strength homogeneity along the tube, but also between internal and external surface of the tube.

During O-ring test, a compression load is applied on the external surface of the ring in contact with compression machine fixtures (Fig. 2). The tensile stress is thus maximized in the internal surface of the ring, near the fixtures. Complementary C-ring tests have been also performed : during these tests, the maximum tensile stress is then located in the external surface of the ring. The combination of these two tests allows to determine if internal and external surface can afford the same loading, or if one surface is more sensitive than the other.

Tensile tests on long slices of tubes and on scale 1 tubes have been also performed. This kind of test is more difficult to settle than elementary tests like 4-point bending tests or O-ring / C-ring tests : specific tooling is necessary, to interface the sample and the test machine. A good alignment between the axis of the tube and the axis of the applied load is necessary. The symmetry of the loading must be checked by the use of strain gauges bonded on the samples (Fig. 3).

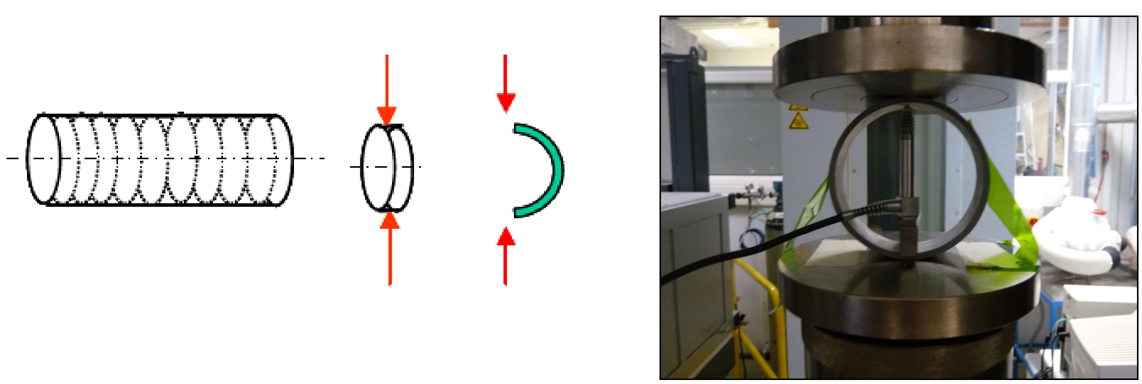

Fig. 2. O-ring and C-ring test principle and jig for ceramic tube slices

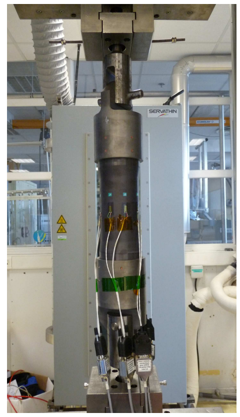

Fig. 3. Tensile test on a long slice of tube

The combination of the results obtained from these different tests allowed :

- To check that the material has the same answer depending whatever the load direction is (axial for the tensile tests, tangential for the O-ring and C-ring tests);
- To determine which flaw population is responsible of the failure for each kind of test : volume defects, surface defects (in the case of surface defects, the tests allow to determine which population is the most dangerous for the part, on internal surface or external surface);
- To evaluate the homogeneity of a tube. The results of O-ring and C-ring tests have shown that some inhomogeneities were present on the long tubes. This phenomenon has been further studied during the characterization phase : it appears that it is reproducible and it has been taken into account for sizing. This point has been identified for further improvements, and is currently studied (cf. §III.A);
- To establish a link between the different tests with the effective volume or surface correction. Indeed, as the material has the same behavior for the two loading directions, the results of the O-ring tests can be used to predict the fracture strength of tubes under tensile load, by the calculation of the material specific characteristics (σ_0, m) through effective volume or surface analysis.

C. Qualification phase

The rule for the qualification phase is that the manufacturing processes must be the same as those that will be used for the manufacturing of flight parts.

The manufacturing processes have thus been frozen at the end of the characterization phase.

All the tests performed during characterization phase have also allowed to define the most discerning tests in order to qualify the material for its future application.

The performed tests were the followings :

- Still 4-point bending tests at each new manufacturing batch (granulation batch or sintering run) : on ground samples machined from plates. The Weibull modulus and characteristic stress are compared with those of the database, determined on previous tests performed during characterization phase ;
- O-ring tests from samples machined from representative tubes
- Tensile tests on representative tubes.

The link between O-ring tests and tensile test results is checked and compared with database results.

At the end of characterization phase, the strategy of witness samples (number, kind of samples, location, ..) has been settled and the associated criteria have been defined. This strategy was validated during the qualification phase.

D. Tests associated with flight parts

The strategy defined for the manufacturing of flight parts is the following:

- 4-point bending tests at each new granulation batch (granulation batch or sintering run)
- Tests of the witness samples, coming from the same compaction preform and sintered with the flight part;
- Proof-test of the flight part after non destructive controls.

This methodology has allowed to fully qualify an industrial grade of silicon nitride and to manufacture several flight parts in numerous campaigns. All these flight parts have been proof tested successfully without any failure. Processes and design are thus qualified for the selected space applications.

Thanks to this great success and gained know how on the use of silicon nitride for space applications, other perspectives are under reflection. Some developments are thus in progress in order to identify and qualify a new optimized grade and its associated processes.

III. FURTHER DEVELOPMENTS

These developments are performed in the frame of studies funded by CNES and in cooperation with FCT Ingenieurkeramik. Improvements of some properties are possible in order to enhance the mechanical strength or the thermal conductivity. Others developments in the frame of high mechanical capability brazed junctions between silicon nitride parts are also ongoing, through a collaboration with PMB, specialist in metallic – ceramic brazed junctions.

These developments are described hereafter.

A. Enhancement of mechanical strength

Large parts homogeneity improvements

Heritage on large parts has shown that it was existing, at one extremity of the tube, an area with a higher density of defects, which causes a decrease of strength of the whole part. A study has been initiated in order to understand the origin of these heterogeneities, and try to improve manufacturing processes in order to limit this phenomenon. The test plan, written in collaboration between FCT and Thales Alenia Space, foresaw to manufacture some reference parts and also some specific parts, manufactured with some modifications in the process.

The heterogeneity problem has been reproduced on the reference parts, as shown in Fig. 4 (on this figure the O-ring tests results obtained on slices machined from different locations of the tube are plotted versus the location of the slice inside the tube). On the specific parts, it appears that the heterogeneity problem has been strongly limited : Fig. 4 shows that for this tube, the differences between the different areas of the tube, and especially the extremity and the current area, were less noticeable. This problem can thus be avoided by a simple modification of the handling of the parts during the manufacturing process. This modification of process will be applied for each new manufactured part, when it is possible.

Due to some specific design of some parts, this modification of process is not always feasible : that is why the interest of an additional post-treatment after sintering has been also studied in order to further improve the strength.

Complementary post-treatments.

On the industrial grade, a final treatment of polishing is performed on the tubes after sintering. It has been proven by numerous tests during the characterization phase of the reference silicon nitride grade that thanks to the very smooth surface condition obtained with this treatment, the strength of the parts was strongly enhanced.

Nevertheless, FCT has experimented another post-treatment on flexure samples, on which some surface defects were created after manufacturing, by grinding (different grit size of the grinding wheel, grinding direction in rectangular direction) or artificial cracks by Vickers indentation [5] [6]. This crack healing treatment have brought very interesting results : the strength decrease caused with the artificial defects was strongly reduced.

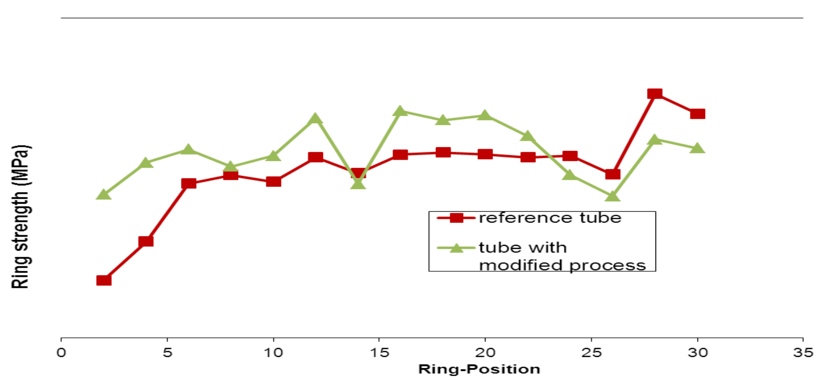

Fig. 4. Strength of O-ring samples machined from reference tube and tube obtained with modified process

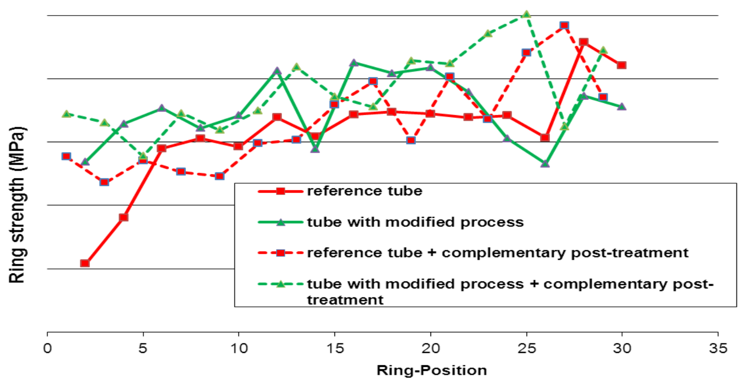

Fig. 5. Strength of O-ring samples machined from reference tube and tube obtained with modified process, with and without complementary post-treatment

It was thus interesting to study the influence of this post-treatment on the mechanical strength of tubes for space applications. The same samples as those previously studied to increase the homogeneity of tubes have been tested. The results are given on Fig. 5.

Fig.5 shows that the complementary post-treatment has an influence on the extremity of the reference tube (ring position between 1 and 5). The complementary post-treatment has not any influence on the current section of the refence tube (ring position between 6 and 30) nor on the whole length on the tube with the modified manufacturing process in order to improve the homogeneity.

As a conclusion, the post-treatment seems to be useful to reduce the impact on defects on strength. But in areas where the defects are already small, this treatment has no additional improvement.

B. Increasing the thermal conductivity

The interest of working on the properties of Silicon Nitride in order to obtain a material with higher thermal conductivity without decreasing mechanical properties and without increasing CTE has been highlighted in 2008 and studied in the frame of R&T contracts. The first study consisted in defining specific Silicon Nitride formulations in order to increase the thermal conductivity. Indeed, as silicon nitride is a multiphasic ceramic material with a large content of sintering additives, it is possible to enhance its properties by adapting the type of additives. After an exhaustive bibliographic study and several tests in order to select the most adapted solutions, it has been assessed that the work on sintering additives was necessary and sufficient before any other modification (replacement of standard powders or addition of high-conductivity particles). The first part of the study was to manufacture and test small samples. Very good results have been obtained in term of thermal properties and mechanical properties. Nevertheless, the sintering cycle parameters applied for the manufacturing of these small samples were quite specific.

Next step of the study has consisted in manufacturing some bigger parts with this new selected formulation, in order to demonstrate the manufacturability of scale 1 parts. This industrialization phase requires that it is possible to sinter the new material with other parts from compatible grades in the same furnace runs, and thus with the same sintering cycle parameters. It has thus been decided to make a compromise on the properties of the new material, in order to insure the industrialization of the new grade and to secure its reproducibility and future maturity.

The thermal conductivity is lower than the one obtained on first small samples, (around 50 W/mK) but it allows the manufacturing of large parts with a good mechanical behavior. Nevertheless it can be possible, by tuning sintering parameters while keeping a sintering cycle compatible with the sintering of other parts, to improve slightly the thermal conductivity value (this will be done in a future study).

Some plates have been manufactured with different manufacturing batches et some strength tests have been performed. A good reproducibility between the different batches has been obtained. Four tubes have been also manufactured with this grade. The length of the tubes is 500mm; apart this length, other parameters are representative of scale 1 tubes. One of this tube has been sliced and the slices have been tested in O-ring tests. The obtained average strength is slightly lower than the one of the reference grade, but with a remarkable good reproducibility, really improved compared to reference grade.

As a conclusion, this study has allowed to identify a grade with an improved thermal conductivity and which can be easily manufactured. First results obtained on tube prototypes are very encouraging : in particular a very good reproducibility is obtained along the length of the tubes, and also between the different tested batches. Next step is now to manufacture scale 1 tubes with this new grade, and to test them (measurement of mechanical strength with tensile tests, O-ring tests; measurement of CTE and thermal conductivity on samples machined directly from the tubes).

C. Brazed junctions

In order to benefit from the good properties of silicon nitride on extended size or for more complex shapes without jeopardizing stability or integration time, brazing developments activities have been launched under CNES funding, in collaboration with FCT Ingenieurkeramik for the manufacturing of ceramic parts, and with PMB for the brazing junctions (PMB is a specialist of metallic – ceramic brazed junctions). Brazing technology allows to assemble structurally some parts, and thus to avoid adhesive bonds.

At the beginning of the study, an extensive bibliographic study and tests on several solutions have allowed to select active metal brazing as the most interesting solution, in term of mechanical strength of the brazed joint.

Then further tests have been performed on brazed samples, in order to freeze the brazing parameters : composition, pressure to be applied, form of the braze alloy, thickness of the joint, brazing cycle. Mechanical tests and complementary analysis like optical microscopy and Scanning Electronic Microscopy (SEM) have allowed to select the most adapted parameters. In the case of 4-point bending test, the failure occurred under tensile load in the brazed joint at 262 MPa, which is a very good result (Fig. 6).

The strength of the joint after thermal cycling and thermal ageing has been also studied. No decrease has been noticed after this kind of treatment. The aspect of the joint is the same before and after such treatment.

After this first phase of the project, some tests on real geometry have been launched, especially for two applications : the brazing of tubes, and the brazing of plane sections for the manufacturing of mirror supports.

The study of the brazing of tubes is in progress. The first phase was to determine the best process to apply the pressure during the brazing cycle. The design of the parts to be brazed has been specifically modified in order to be compatible with the brazing tooling : this work has been performed through an intensive collaboration between FCT, PMB and TAS. Some rings have been manufactured and brazed using the developed tooling (Fig. 7). Some optical micrography and SEM observations have shown that the brazed joint has the same characteristics as obtained on previous samples. Some samples have been machined from the brazed rings and have been tested in order to put the brazed joint in flexure : approximately the same strength as the one measured on elementary samples has been obtained. The same brazing parameters and tooling principles will be thus applied for the next step : the brazing of tubes with a length of 300mm. This assembly will be tested under a tensile load.

Fig. 6. Silicon nitride sample with a brazed junction at the middle of the sample, for 4-point bending tests

Fig. 7. Ceramic rings after brazing

The other application is the brazing of plane sections for the manufacturing of mirror supports. Once again, the same brazing alloy and brazing parameters as those selected during the first phase of the study are used. The point is to develop a special tooling in order to maintain the different parts together during the whole brazing cycle, and to apply the pressure. A first prototype has been manufactured and brazed. Non-destructive testing has been performed in order to check the presence of brazing alloy. A mechanical global test will be performed (the preparation of the test is in progress).

IV. CONCLUSION

An industrial grade of silicon nitride is qualified for the manufacturing of beams and beams end fittings, through full development and test activities. This technology is now offered for the realisation of very lightweight, strong and stable structures for space use.

In parallel to the complete qualification of this industrial grade, improvements of some properties are under development, in the framework of studies funded by CNES and in cooperation with FCT Ingenieurkeramik. The objectives of these studies are numerous : to improve the homogeneity of large parts, to enhance mechanical strength and to increase thermal conductivity. Many encouraging results have been obtained recently : the new grades and associated processes are about to be qualified for large parts.

Other developments are on-going, in particular to develop strong brazed junctions between silicon nitride parts (in cooperation with PMB) : after very good results obtained on samples, the study is now transposed to geometries which are representative of large parts.

All these technical and industrial achievements have been made possible through intensive and open-minded cooperation between end-user and producers under the management of CNES, and will open the path for new and even more ambitious applications, such as wider and more complex parts, aiming to the building of a complete instrument structure in silicon nitride.

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