

# AN IMPROVED LONG LIFE DURATION CERAMIC MATRIX COMPOSITE MATERIAL FOR JET AIRCRAFT ENGINE APPLICATIONS

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#### Abstract

A new concept of Ceramic Matrix Composite (CMC) material, mainly based on the use of a self-sealing technology for matrix and the use of a multilaver woven reinforcement, has been developed by Snecma for achieving high performance levels targeted by future jet engines. The driving force for this development has been to increase both lifetime and temperature capability of previous C/SiC and using SiC/SiC composites materials а monolithic SiC CVI matrix and finishing treatment against oxidation.

# **1 INTRODUCTION**

In order to meet high performance levels targeted by future jet engines components [1], Snecma Propulsion Solid (SPS), has developed a new concept of self-sealing Ceramic Matrix Composites (CMC) materials named CERASEP<sup>®</sup>A410 and SEPCARBINOX<sup>®</sup>A500 combining improvements of fiber reinforcement, interphase and matrix [2]. These materials, manufactured by SPS are designed for thermo-mechanical applications such as long term parts for gas turbine engine. The development of these CMCs has been milestoned by steps, which as specified in different papers [3].

The aim of this paper is to present thermomechanical characteristics and analysis validated by sub-element testing of these CMCs. After reviewing the material development story, the CMC's development phases, we focus on the measured thermomechanical properties and life time evaluation by fatigue testing. This characterization program provides a basic database used for the stress analysis of CMC composites through mechanical behavior models using Continuum Damage Mechanics. A thermomechanical analysis is presented with the comparative behavior of two materials. A sub element testing validate the orthotropic nonlinear law.

#### 2 Materials for gas turbine engine

The application of advanced materials has played a vital role in the development of gas turbine engines over the last fifty years. An estimated 50% of the increase in efficiency and performance has been directly attributed to material development. Material developments come in two forms:

- the easiest being the study development of a known material system through iterations of material and processing : In this way, the temperature capability of nickel base superalloys has increased by over 350°C since the 1940's,
- the other way is the introduction of new types of advanced material, tailored to the specific requirement of gas turbine engines.

This approach is more difficult and requires considerable continuous investment over many years. The introduction a new materials takes typically a minimum of fifteen years. The necessary levels of safety and reliability dictate that all risk associated with the introduction of a new technology are adequately assessed and addressed. In gas turbine engines the components are subjected to many potentially detrimental factors including high temperature and loads, vibrations and load cycling thermal shock, oxidation, corrosion, abrasion, erosion, handling and occasional impacts. In this context very new materials, however promising, cannot instantly be put into engines

The silicon bases ceramics, primarily Silicon Nitride and silicon Carbide have been the focus of much interest and development during the last twenty years. These materials retain strength at over 1200°C where there has been no other material available (Fig. 1).

**Fig. 1** Usable strength for high temperature materials for gas turbine engines.



However monolithic ceramic technology was considered unsuitable for structural components : in an environment when catastrophic failure is to be avoided at all cost. The very low defect tolerance and low failure strain of this material was considered to impact too high of a risk and consequently has not been specified in production engines. Composite material combining the advantages of ceramics with a material capable of offering an improved tolerance to strain have captured all the recent efforts to use ceramics in larger aero-engines. The incorporation of continuous ceramic fibers offer the ultimate in the ability to control the properties of the material.

# **3 BACKGROUND**

SiC/SiC (Silicon Carbide fiber/ silicon carbide matrix) composite material series A300 developed by SPS (originally SEP) before 1992 were made of a 2D reinforcement (2D : 2 directions of reinforcement), using Nicalon<sup>TM</sup>

C.G. from Nippon Carbon, with pyrocarbon interphase, and a SiC matrix deposited by chemical vapor infiltration (CVI).

CERASEP<sup>®</sup>A373 (A300 serie) material was characterized by a high specific strength, at room temperature, of about 300MPa and a nonbrittle behavior, with an enhanced failure strain. of about 0.5%. This material, protected by an appropriate finishing treatment against oxidation was also characterized by a relatively good life time capability, in flexural creep, under air, at temperature between 873 to 1473K. Nevertheless, testing in more severe conditions, especially for tensile/tensile fatigue tests performed at a stress level of 120MPa, failure of the material occurred rapidly, due to a partial oxidation of the interphase. This was illustrated by a life time duration of about 10 to 20 hours at 873K and less than 1 hour at 1123K. In conclusion, for these series of SiC/SiC composites life time duration in air, at moderate and high temperature was considerably reduced beyond the elastic yield point (about 80MPa). A first evaluation of the SiC/SiC material series A300 behavior was established in real conditions by the design, manufacturing and bench testing of Rafale engine M88-2 inner nozzles flaps in 1992. This highlighted early cracking of some components which resulted from local stresses. These stresses were generated by the combination of non-uniform thermal gradient and higher temperatures than anticipated by thermal analysis. It was found that these stress levels were twice than that of the SiC/SiC series A300 design allowable (80 MPa) and induced micro-cracking of the material leading to an oxidation rate incompatible with long life span.

In the same period, Snecma developed a C/SiC (SEPCARBINOX<sup>®</sup>, Carbon fiber/Silicon carbide matrix) material for engine nozzles operating at intermediate temperature and life span objective over one thousand hours. A typical example of application is the M88-2 engine outer flaps.

SEPCARBINOX<sup>®</sup>A262 is made of a multilayer reinforcement with carbon fiber, a pyrocarbon interphase, a SiC CVI matrix and an

enhanced finishing treatment to improve its behavior atmosphere. in oxidative SEPCARBINOX<sup>®</sup>A262 is characterized by a non-brittle behavior with failure strength of about 250 MPa at room temperature, and life time duration, for temperatures below 973 K, the application requirements. matching Qualification of the SEPCARBINOX<sup>®</sup>A262 technology was pronounced in 1996 for the M88-2 engine outer flaps, after completion of endurance testing based on accelerated mission cycles. The lifetime objective of the component has now been demonstrated on the M88-2 engine and serial production is underway.

Moreover, destructive evaluation, after representative engine life duration (1000 hours engine test, resulting from 375 hours ground test and 625 hours flight test), performed by tensile properties measurements, at room temperature, has confirmed the excellent capabilities of such material :

- $-\sigma/\sigma_{\text{initial}} > 0.90,$
- $-\epsilon/\epsilon_{\text{initial}} > 0.90,$
- E/ E<sub>initial</sub> > 0.90

# **4 NOVEL SELF-SEALING MATERIALS**

# 4.1 General approach

SPS decided in 1992 to engage the development of a new CMC material concept, matching long life span at high temperature with higher stress allowance.

The initial objective was to develop a SiC/SiC material useable beyond its mechanical vield point for temperature range up to 1373K, and capable of operating life exceeding 1000 hours in oxidative atmosphere integrating 100 hours in severe conditions (stress level up to 120MPa). The material concept was mainly based on the use of a novel self-sealing technology for the matrix and the use of a multilayer woven reinforcement to reduce delamination sensitivity during the The manufacturing process. development approach for the composite has already been described [2].

More recently, considering the high potential of the self-sealing matrix, and the

know-how achieved on C/SiC A262, the combination of a carbon reinforcement with a self-sealing matrix has been studied. This approach provides better economical outlook considering the gap between the cost of carbon and carbide fibers.

# 4.2 A410 and A500 description

multi-layer reinforcement Plane named GUIPEX<sup>®</sup> has been developed, in order to prevent the natural delamination sensitivity of 2D materials. GUIPEX<sup>®</sup> preforms are made of layers, linked together and the technology is applicable to carbon and ceramic fibers. Number of layers is adjustable, in order to obtain composite thickness range between 2 and 7 mm, or variable thickness composite. These reinforcements have been optimized to obtain orthotropics composites, with in-plane characteristics close to a 2D material.

A hardening step is necessary to obtain the desired shape, with the desired fiber volume fraction, before the matrix infiltration. Specific CVI route, combining both interphase and hardening process has been selected.

A self-sealing route has been selected for the matrix in order to eliminate finishing treatment. The principles of the self-sealing approach are to consume part of the incoming oxygen and to prevent access of the residual oxygen to carbon interphase through the microcracks. A novel matrix technology, combining carbides deposited by CVI process with specific phases, has been developed.

The functioning of the self-sealing matrix is illustrated in Figure 2, where the consumption of a carbide (black area) sequence by oxidation and formation of a sealing glass in a microcrack is well shown after tensile fatigue test, in air, at high temperature.

**Fig. 2** *Micrograph of self-sealing matrix after tensile fatigue at* 600°*C* 



#### **5 MATERIAL CHARACTERIZATION**

#### 5.1 Experimental procedure

CERASEP®A410 and SEPCARBINOX®A500 test coupons are machined from standard  $200x200 \text{ mm}^2$  plates. The first manufacturing step of these plates is the interphase deposition and the CVI hardening. The second step consists in performing, after demolding, two CVI cycles for carbides deposition. Test specimens for high temperature mechanical testing in oxidative atmosphere, are machined after the first CVI cycle, in order to efficiently protect all the specimens edges. Most tests are performed according to current European Standard, as already mentioned [3]. In-plane tension, creep and tensile fatigue coupons are dogbone specimens with a width of 16 mm in the gage section in order to limit scattering. Low cycle fatigue tests (LCF) are carried out using efficient strength and not apparent strength. In fact, it is assumed that the seal-coat does not play a structural role. Hence, the thickness of the coupons after the first CVI cycle is taken into account to calculate the load. Database generation is focused, first on basic mechanical and thermal properties versus temperature. In parallel, assessment of the durability, in air, based mainly on tensile fatigue at 0.25Hz (trapezoidal cycle), typical engine cycle fatigue and tensile creep are carried out, taking into account environmental effects, in order to determine design allowable.

| Table I. | CMCs | physical | properties |
|----------|------|----------|------------|
|          |      |          |            |

| Materials | Fiber Vol.<br>Fraction (%) | Density<br>(g/cm <sup>3</sup> ) | Porosity<br>(%) |
|-----------|----------------------------|---------------------------------|-----------------|
| - A410    | 35                         | 2.20 - 2.30                     | 12 - 14         |
| - A500    | 40                         | 1.90 - 2.10                     | 12 - 14         |

Physical properties of these two CMCs are described in Table I.

#### 5.2 Mechanical and thermal database

A410 material is characterized by mechanical properties similar to those obtained on previous SiC/SiC materials (Table II). At room temperature, 40 coupons corresponding to 8 manufacturing batches have been tested. Standard deviations obtained are 20 MPa for the ultimate strength and 25 GPa for the elastic modulus. These monotonic tensile properties do not change significantly up to 1473 K. Typical stress-strain curve with unload/reload loop is shown in Fig.3, for the A410.

Fig. 3 Tensile stress-strain curve, at RT and dir.1 for A410



As already mentioned by other researchers [4], three domains can be observed: a linear elastic portion, a non-linear elastic domain due to microcracking starting at a macroporosity level followed by a quasi linear domain due to microcracking at the tow level. Residual strain after loading and hysteresis loops is negligible which is typical of a damageable linear elastic behavior.

A500 material shows tensile properties similar to those obtained on

SEPCARBINOX<sup>®</sup>A262 (Table II). It is characterized by a low elastic modulus in comparison with the A410. In addition, the asproduced A500 is microcracked due to the thermal expansion mismatch between carbon fiber and carbide matrix. This explains the absence of a linear elastic domain on the stressstrain curve (Fig.4). At high temperature, more particularly at 1473 Κ. the A500 is characterized by a slight increasing of the elastic modulus in agreement with microcracks partial re-closing

| Table II. Results from tensile test in dir. 1 |                           |       |      |       |  |  |
|-----------------------------------------------|---------------------------|-------|------|-------|--|--|
| Temp.                                         | Number                    | S     | е    | Е     |  |  |
| (K)                                           | coupons                   | (MPa) | (%)  | (GPa) |  |  |
|                                               | CERASEP <sup>®</sup> A410 |       |      |       |  |  |
| RT                                            | 40                        | 315   | 0.50 | 220   |  |  |
| 873                                           | 5                         | 320   | 0.60 | 210   |  |  |
| 1473                                          | 9                         | 325   | 0.60 | 205   |  |  |
|                                               | SEPCARBINOX® A500         |       |      |       |  |  |
| RT                                            | 15                        | 230   | 0.80 | 65    |  |  |
| 873                                           | 3                         | 230   | 0.80 | 70    |  |  |
| 1473                                          | 3                         | 230   | 0.70 | 90    |  |  |

Fig. 4 Tensile stress-strain curve, at RT and dir.1 for A500



The two CMCs exhibit a significant difference of in-plane thermal expansion coefficient . In fact, the respective values are about 5  $10^{-6}$ K<sup>-1</sup> for A410 and about 2.5  $10^{-6}$ K<sup>-1</sup> for the A500, at 1273 K (Fig.5).

On the other hand, the thermal conductivity of the A500 material is slightly higher than the thermal conductivity of the A410 material at high temperature for directions 1 and 2 (Fig.6).

# 5.3 Determination of the life time duration in oxidative atmosphere

Characterization of the tensile fatigue behavior versus temperature, using a trapezoidal cycle at 0.25Hz has been performed on two CMCs.









Illustration of LCF tensile behavior of CERASEP<sup>®</sup> A410, using trapezoidal cycle at 0.25 Hz is presented in figure 7. At 873 K, duration of 1000 hrs without failure is reached

for a stress level of 170 MPa, and a duration higher than 100 hrs at 200 MPa. At 1473 K, a duration of about 100 hrs is still obtained at 160 MPa. The potential of the CERASEP<sup>®</sup>A410, decreases at 1673 K, in accordance with the fiber thermal capacity. Furthermore, duration of about 100 hrs are still observed for stress levels lower than 100 MPa.

Fig. 7 A410 T/T fatigue, in air, 0.25 Hz



Fig. 8 A500 T/T fatigue and tensile creep, in air



A comparison of the SEPCARBINOX<sup>®</sup> A500 behavior tested in tensile fatigue at 0.25 Hz and in tensile creep is provided in figure 8. The life time duration, in tensile fatigue, is around 100 hrs for a stress level closed to 160

MPa and in the temperature range of 873 - 1473 K. In tensile creep, life time durations are debited, but still remain significant.

#### **6 THERMOMECHANICAL ANALYSIS**

The life design of CMC components is based upon both thermal and thermomechanical MARC<sup>©</sup> analyses using Finite Element modeling. Thermal analyses are transient in order to determine the change with time of the temperatures during representative jet engine cycles. Thermal analysis also yields important results for thermomechanical calculations. High temperature gradients are responsible of internal loads due to differential thermal expansion (Fig.9). Thermomechanical analysis also takes into account pressure loads and interface conditions between parts.



In order to size CMC components, an knowledge of the accurate non-linear orthotropic behavior throughout the entire range of operating temperature is needed. That is experimental characterization mean within tension. compression and shear for all reinforcement directions of the composite. While stress-strain relationship of in-plane testing is relatively easy to measure, it is often more difficult in the case of shear and Poisson effects. Because of the damaging character of CMC behavior. tensile testing must be incremental performed using load-unload cycles. In compressive testing, stress-strain curves are generally linear. In addition to the set of curves defining the stiffness matrix, it is necessary to add the 3 thermal expansion curves.

#### 6.1 Comparative thermomechanical behavior

For components submitted to heterogeneous thermal flow, like nozzle flaps, the combination of thermal properties and elastic modulus of A500 material, is very favorable to decreasing thermal gradients and as a result to lowering thermal stresses [5]. An example of thermomechanical analysis, performed on a component representative of a nozzle flap, and taking into account the specific characteristics of each material is given in Fig.10 and Fig.11.

Fig. 10 thermomechanical stress field on a nozzle flap



Fig. 11 Longitudinal stress - A410 and A500 cases



The lower sensitivity to thermal gradients of SEPCARBINOX<sup>®</sup> A500 compared with A410 is a way to increase its design margins as outlined by the following example : referring to the same design case, Fig.11 shows max. longitudinal stresses respectively of 60 MPa and 145 MPa for A500 and A410 materials. The relevant stress allowable for a

life time objective of 100 hours, issued from the fatigue and creep curves plotted in Fig. 7 & 8, are respectively 160 MPa and 190 MPa for A500 and A410 materials. These data end up in a sizing coefficient of 2.7 for A500 material which is twice than that of the A410 sizing coefficient.

#### 6.2 Coupling damage and oxidation

Snecma Propulsion Solide has developed an uncoupled orthotropic elastic non-linear behavior law, which take into account a network of strain stress curves [6]. More recently, coupled mechanisms of damage and inelastic strains have been taken into account using a damage model, named ODM, and developed by ONERA [7]. This approach is essential to improve modeling of CMC behavior. Fig. 12 shows the tensile-compressive stress-strain curve with unload-reload loop, obtained with damageable model, for A500.



**Fig. 12** Tensile-compressive stress-strain ODM curve, at RT and dir.1 for A500

The quality of CMC component sizing, based on thermomechanical analysis depends on the understanding of both fatigue and creep yields and failure limits. Once again, fatigue and failure limits are easily obtained for in-plane, uniaxial and isothermal conditions, while anisothermal, multiaxial and non-proportional loading need non standard characterization.

In contrast with metallic materials, there is no general validated failure criterion. The analysis could be based on known criteria taken from the literature, as Hill, Hoffman or Tsai-Wu. The current criterion is based on maximal stress or strain, which is directly compared to stress-duration curves issued from fatigue and creep testing. In most cases, stress distribution is preferentially uniaxial (Fig.10). But, criterion currently used is sometimes insufficient, because knowledge of the failure mechanisms and associated characterization are still in progress.

To improve lifetime justification with selfmatrix, introduction of coupling sealing between macroscopic damage mechanism and oxidation is needed. This work had been initiated with a macroscopic model, which takes into account the relationship between oxidation kinetics and fissuring mechanisms at microscopic scale [8]. This new method gives first good results concerning fatigue-creep behavior for A410 database. Improvements of the method will be extended and validated for A500 material and complex thermodynamic conditions.

#### 7. SUB-ELEMENT TESTING

Required changes in the method, i.e. multiaxial fatigue-failure criteria and life time predictive laws, must be validated by subelement testing. Sub-element testing means instrumented CMC component with a geometry as near as possible of the engine component one and taking into account both boundary conditions and representative loading of jet engine application.

A first example of sub-element testing at room temperature is shown on figures 13 & 14.

Fig. 13 flexure-torsion sub-element testing and its instrumentation



Fig. 14 deformed shape for flexure-torsion sub-element testing



The A500 plate was instrumented with 10 strain gauges. This testing was dedicated to validate both orthotropic non-linear elastic and ODM laws using Finite Element analysis. The plate was loaded by an uniform pressure on the top and was simply supported on the other side. One of the supports had a longitudinal deviation equal to 10% or 20% of the transversal size. These boundary conditions consisted of a combination of flexure and torsion loading. Several cycles with incremental pressure have been carried out

Mechanical analysis has also been carried out, as shown on fig. 15 for displacement and fig. 16 for transversal stress and translaminar shear.

**Fig. 15** deformed shape (x10) and normal deviation field for flexure-torsion analysis



**Fig. 16** transversal stress and translaminar shear field for flexure-torsion analysis



The comparison of calculated strains and measured strains by a 10 gauge set shows good agreement. Figure 17 gives the strain response of a  $[0^{\circ}, 45^{\circ}, 90^{\circ}]$  strain gauge, implemented near the center of the plate (Fig. 13). Similarly the Orthotropic Non-Linear Elastic Law (ONLEL) load responses are correctly simulated. However, because of elastic assumption, unloading and residual strains are not reached.

**Fig. 17** Calculated and measured strain, at RT and dir.  $0^{\circ}$ ,  $90^{\circ}$  &45° for flexure-torsion testing (ONLEL)



When introducing damage effects, it is possible to obtain a more realistic CMC behavior, taking into account damage history and permanent strains, as shown on fig. 18, for an unidirectional gauge at the center of the plate. In this case and concerning A500, subelement analysis validates the identification of both coupling parameters and damage kinetics of ODM behavior law.

**Fig. 18** Calculated and measured strain, at RT and dir. 90° for flexure-torsion testing (ODM)



In a second time, the same sub-element had been tested under torsion loading at 873 K in air, as shown on Fig. 19. The plate was simply supported on two adjacent sides and a deviation equal to 10% of the transversal size was applied on the opposite corner. Testing was performed

during 550 hours, with unloading every 18 seconds (110 000 cycles). No apparent failure was visible after this high duration. Before and after torsion fatigue, the plate has also been tested at room temperature with a 10 gauge set (Fig. 19) in order to validate behavior law of both virgin and older CMC. A final flexure-torsion testing at maximum pressure and deviation was applied without any failure of the sub-element.

**Fig. 19** Torsion sub-element testing at ambient temperature (static) and 873 K (fatigue).



Translaminar shear field is shown on fig. 20. Throughout the entire plate surface, shear is in the range from 70% to 90% of mean Iosipescu ultimate shear measured on A500 specimen. Once again, orthotropic non-linear behavior law led to model only the first loading. As shown on fig. 21, strain responses from ODM analysis are in a good agreement with experimental results. While damage and residual strain after loading is reached, hysteresis loops are not negligible anymore.





**Fig. 21** Calculated and measured strain, at RT and dir.  $45^{\circ}$  for torsion testing



The complete results of sub-element testing demonstrates substantial improvement:

- Ø A500 component shows strong potential for providing high life duration considering multiaxial loading at 873 K in air;
- Ø The validation of orthotropic non-linear elastic law, which is the current method used in the most CMC jet-engine development, is confirmed;
- Ø The ODM damageable behavior law is an excellent candidate in order to perform future detailed sizing analysis and to improve both fatigue-failure criterion and life time predictive law with oxidation coupling.

#### 8. Conclusion and Perspectives

A new concept of Ceramic Matrix Composite (CMC) material based on Self Sealing Matrix is expected to become an emerging technology which opens "new frontiers and horizons" in the 21st century. Field service evaluations of Seals are planned in the near future. It will be a new challenge for these materials to be tested in "flight". Consistent efforts are underway to improve the technology readiness level (TRL) as well as towards an improvement of the material life capability above the performances to date. To this end, modeling tools addressing material aging and life prediction are good help for long term projections.

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