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NEW MATERIALS FOR HIGH TEMPERATURE TURBINES;  
ONERA'S DS COMPOSITES CONFRONTED WITH BLADE PROBLEMS

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16. Abstract The problem of advanced aircraft turbines operating at higher temperature immediately raises the question: are there new required materials available for such a realization? ONERA's refractory DS composites answer the question. These materials are not a laboratory invention looking for an eventual application: on the contrary, they represent the outcome of research work directed precisely to new materials for better and more reliable turbine blades in advanced aircraft engines. The requirements for a blade material in aircraft turbines operating at higher temperatures are compared with the actual performance as found in COTAC DS composite testing. The paper specifies the structure and the properties of the more fully developed 74 and 741 types. In particular, high temperature structural stability, impact of thermal and mechanical fatigue, oxidation resistance and coating capability are more thoroughly evaluated. The great gain in operational temperature of these materials can be immediately exploited in the field of uncooled solid blades. The problem of cooling passages in DS eutectic blades is also outlined.			
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NEW MATERIALS FOR HIGH TEMPERATURE TURBINES;  
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Introduction

In analyzing the development of turbine engines, an improve- /20-1\*  
ment in performance customarily is provided by an increase in the  
temperature of the heat source. Actually, during the last twenty  
years, the inlet temperature of the turbine has gone from about  
1100°K to more than 1500°K in civilian engines and nearly 400°K  
more in military engines, and the short-term (1980) targets pro-  
vide for an increase to 1600 and 2100°K, respectively. Meanwhile,  
the tendency to increase the constraints is no less significant.  
To take only the last five years, the strength required of turbine  
blading has increased by nearly 30%, and the rate of this progress  
does not appear to have to slow down in the coming decade.

Such a scaling up would not have been considered without par-  
allel progress in the area of materials: metallurgical research  
leading to structurally more stable and better hardened alloys,  
offering an increase in operating temperature and strength; uni-  
directional solidification, permitting more dependable cast parts  
to be obtained, free of the risk of premature breaking by loss of  
intergranular cohesion; varied and better adapted protective tech-  
niques; technological and industrial progress in the design and  
making of parts. Besides, investigation of the laws of heat flow  
and transfer has permitted optimization of the cooling circuits;  
the difference between the turbine inlet temperature and the max-  
imum blade temperature can reach and exceed 500°K.

The turbine engines of the future will require still higher /20-2  
performance materials. Cooling systems have reached such a degree  
of sophistication, that the technology of circuit layout will have  
difficulty in following this pathway, without prohibitive cost and  
efficiency penalties, unless revolutionary designs can provide a  
still unforeseeable gain. The heat experts can only guess at it.  
But, as far as materials science is concerned, the risks can hardly  
be taken of stating that an additional jump of 50°K or of 30% in  
strength cannot be considered, by the improvement of present Co  
or Ni superalloys.

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\*Numbers in the margin indicate pagination in the foreign text.

On the other hand, refractory composites, prepared by unidirectional solidification, represent a class of metallic materials which can provide relief. The method of preparation, characteristics and possibilities of application of such composites have been fully analyzed in specialized conferences [1,2] or AGARD meetings [3,4], to mention only the most complete reviews. The refractory variations intended for production of turbine engine blades are fiber or lamellar composites, i.e., essentially biphasic materials, although prepared from multiconstituent systems (number of constituents greater than the number of phases). The complex Co or Ni base matrix is reinforced by high resistance refractory carbon fibers [5], or by alternating lamellae of intermetallic phases [6]. In very high temperature creep, these composites give a spectacular increase in strength and operating temperature, as stage which there can be no hope of exceeding by improvement of present alloys.

As great as it may be, the high temperature creep advantage is insufficient to promote a new material to the production of hot parts of the future turbine engines. To be accepted, a new material first has to satisfy the set of characteristics that the engine builders are already accustomed to handling in calculating their forecasts, but also, whether some less common characteristics of composite materials can lead the engine builder to go along with a change in design, analysis of which requires, in turn, experimental testing of the new properties. The question to be examined here is how the composite materials developed by ONERA, to improve the performance and reliability of future turbines, will answer the questions of the engine builders.

## Current Properties of COTAC 74

### Structure

The ONERA refractory composites, presently in the development stage, belong to the family designated COTAC 74 [7].<sup>1</sup> The matrix is a Ni, Co or Cr base oriented crystal superalloy, hardened in solid solution together with a high W content and by coherent precipitation of a phase of complex composition, of the  $\gamma'$  Ni<sub>3</sub>Al type, produced by addition of aluminum; the reinforcing fibers are refractory NbC monocarbide, of the same CFC crystallographic structure as the matrix. Solidified under a high thermal gradient ( $150-200^\circ\text{K}\cdot\text{cm}^{-1}$ ) at a relatively slow rate ( $\leq 2.4 \text{ cm}\cdot\text{hr}^{-1}$ ), the system becomes organized at the very first, under conditions close to thermodynamic equilibrium, as an oriented fiber composite, of which Fig. 1 shows the metallographic structure observed in a transverse section by scanning electron microscope.

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<sup>1</sup>Rated composition; Ni, 20 Co, 10 Cr, 10 W, 4 Al, 4.9 Nb, 0.6 C.

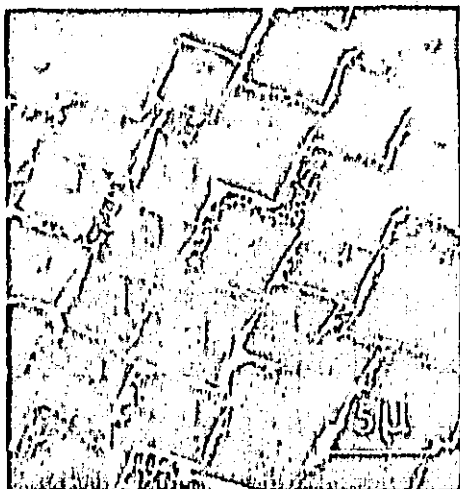


Fig. 1

With its precipitates, the matrix here recalls the microstructure of the best Ni base superalloys; in fact, like the highest performance modern types prepared by directional solidification, the composite is free of transverse grain joints, which improves high temperature reliability, for the principal applications directed along the axis of crystallization. Further, the unique orientation of the grains of the matrix, which follows a crystallographic direction with a low modulus of elasticity, permits the thermal constraints to be minimized. Reinforcement is provided by NbC refractory fibers, very long perfect monocrystalline filaments (whiskers), about 0.8  $\mu\text{m}$  in diameter, the extra-

ordinary mechanical strength of which approaches theoretical values.

### Properties

Although reinforced by practically inseparable fibers, the composite does not retain less complete ductility under tension (Fig. 2), like under impact (Table 1), in the entire temperature range. We note in passing the relative insensitivity to cutting: the impact strength of notched samples is greater than that of the commercial IN-100 alloy.<sup>2</sup> Despite the anisotropic behavior, the transverse properties of the composite appear to be satisfactory: at the temperature of the base of the blade, for example, the transverse impact strength is greater than 8  $\text{J}\cdot\text{cm}^{-2}$ , while, under transverse tension, deformation is 2.5%.

With respect to creep, for a lifetime of 1000 hours, COTAC /20-3 74 surpasses IN-100 below 900°C (Fig. 3).

It is seen that, despite the density handicap, the specific stress advantage is more than 30% at 950°C, 40% at 980°C and 60% at 1000°C; at equal specific stresses, the temperature advantage is from 42 to 80°K. For longer duration, a 30 to 40% increase in stress corresponds to a temperature advantage of 100°K and the improvement in lifetime reaches a factor on the order of 20. Meanwhile, it must be noted that the composite is of less interest with respect to creep at medium temperature and is also more sensitive to variations in stress than the equiaxial alloy: at 900°C, to exceed an average lifetime of 150 to 1000 hours, the creep stress of IN-100 must be reduced by more than 25%, while

<sup>2</sup>Rated composition: Ni, 15 Co, 10 Cr, 5.5 Al, 4.7 Ti, 3 Mo, 1 Ti, 0.18 C, Zr.

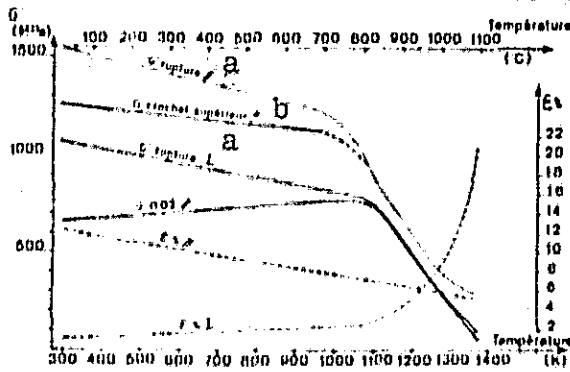


Fig. 2. COTAC 74 -- longitudinal ( $\Rightarrow$ ) and transverse ( $\perp$ ) tension vs. temperature.

Key: a. Breaking  
b. Upper yield point

a slight reduction in stress, less than 10%, is enough to reach the same failure time in COTAC 74.

For a turbine blade, the association of the creep characteristics and the distribution of stresses  $\sigma$  and temperature  $t$  along the blade permits estimation of the possible lifetimes of the material, by localization of the critical point, where the  $(\sigma, T)$  curves along the blade meet the corresponding lifetime curve of the material. In conventional superalloys, a family of curves, of 300, 1000 or 2000 hours lifetime at permissible temperature as a function of stress, to

TABLE 1. WORK OF RUPTURE COMPARED WITH IMPACT BENDING ( $J \cdot cm^{-2}$ )

Température (°C)	b		c		d
	Eprouvilles lisses COTAC 74	IN 100	Eprouv. entaillées COTAC 74 IN 100		Eprouv. travers COTAC 74 entaillé*
20	77 - 110	57 - 120	25 - 31	14 - 28	6 - 7
700	37,6 - 40	43	21 - 23	12	0,3 - 0,7
1000	48 - 50	26	25 - 26	111	13 - 14

\*Groove parallel to fibers.

Key: a. Smooth test pieces  
b. Grooved test pieces  
c. Transverse test pieces  
d. Grooved

cause 1% deformation, generally is plotted; meanwhile, as to the COTAC 74 composite, creep failure occurs with relatively low deformation, on the order of 1.2-1.5%; therefore, not so much the limiting deformation, as the lifetime of the material affected has to be taken into account here with a correction factor. For the IN-100 alloy, for example, experimental results show that the 1% deformation time corresponds practically to the creep failure half-life. For the COTAC 74 calculation, the left-limited lifetime divided by 2 usually is used, which leaves a good margin of safety, since 1% deformation is close to the total lifetime. Fig. 4 for example, compares the possibilities of the materials, for the production of a noncooled, low pressure blade, i.e., which is subjected to relatively low centrifugal stress. The

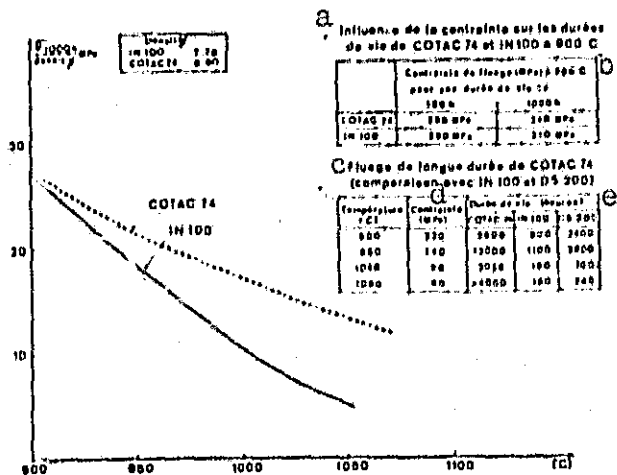


Fig. 3.

- Key: a. Effect of stress on lifetime of COTAC 74 and IN-100 at 900°C.  
 b. Creep stress (MPa) at 900°C for a lifetime of  
 c. Long term creep of COTAC 74 (comparison with IN-100 and DS 200)  
 d. Stress (MPa)  
 e. Lifetime (hours)

maximum permissible temperature which causes 1% creep deformation in IN-100 and DS 200 superalloys in 2000 hours is compared here, to the 5000 hour lifetime of the COTAC 74 composite.

Such a representation has the advantage of showing the possibility of use of the composite for long periods, under moderate stress. In the double flow turbine engines of the next generation, of which the low pressure stages will operate at temperatures of more than 1000°C, COTAC 74 appears to be of special interest, for the production of solid blades, thus avoiding the troublesome technical complications of supplementary cooling circuits.

Meanwhile, with respect to the creep properties determined, i.e., of stabilized operation, thermal problems and

vibrational stresses remain, which, with oxidation and dry corrosion, constitute the principal factors in blade lifetime and reliability. We shall now review each of these points.

### Thermal Problems

With density equivalent to that of most of the Ni base superalloys (DS 200, X-40, Rene 120), and lighter than the Co base alloys (MAR-M 509, L-605, WI-52), COTAC 74 is penalized meanwhile, by the exceptional lightness of IN-100 alloy, to which we compare it here (Table 2), notably with respect to thermal diffusivity. In return, the incipient melting temperature advantage is considerable. This certainly is a serious advantage, for the guide blades in particular, an advantage paid for, meanwhile, by technological difficulties: the more the melting temperature of the material is raised, the more production difficulties build up. Added to the necessity for large thermal gradients and low oriented crystallization rates of the composite, this is a handicap, which must be taken into account in cost estimation.

Comparison of the thermal characteristics of the COTAC 74 /20-4



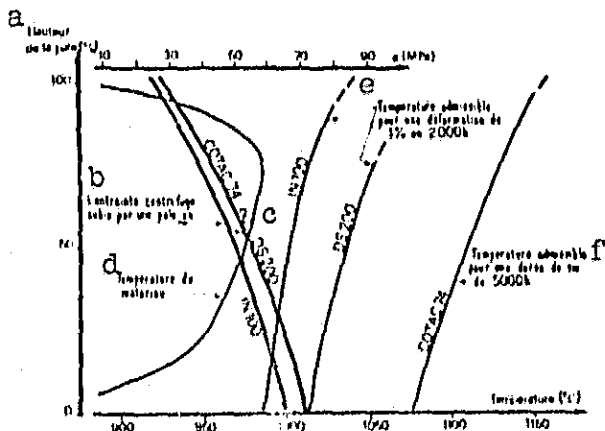


Fig. 4.

Key: a. Blade height (%)  
 b. Centrifugal stress experienced by a blade of  
 c. Or  
 d. Temperature of material  
 e. Permissible temperature for 1% deformation in 2000 hours  
 f. permissible temperature for lifetime of 5000 hours

specific heat,  $c$ , finally results in a small effect on the thermal diffusivity  $\lambda/cp$ .

Therefore, it can be said that a change in material, which consists of replacing the IN-100 alloy in a cooled blade by the COTAC 74 composite does not appreciably modify the temperature field [8]:

At constant load, the increase of  $\lambda$  involves a decrease in the temperature gradients, which has a favorable effect on blade behavior; sometimes, the average temperature of the blade is practically unchanged;

In transient operation, (a) at low temperature (taxiing  $\rightarrow$  full throttle), the increase in  $\lambda$  and the relative decrease of  $\lambda/cp$  involves an increase in the temperature gradients; meanwhile, since the temperatures of the metal are low, this effect is not dangerous; (b) at high temperature (full throttle  $\rightarrow$  idling), the simultaneous increase in  $\lambda$  and diffusivity involves much reduced thermal gradients; the temperature field should be little affected.

oriented composite and the IN-100 equiaxial superalloy calls for the following remarks:

1. The thermal expansion,  $\alpha$ , of the two materials is of the same order of magnitude; the differences in longitudinal and transverse behavior of the composite are small.

2. The thermal conductivity,  $\lambda$ , of COTAC 74 is greater than that of the commercial alloy, but this difference is reduced at elevated temperatures; within the accuracy of measurement,  $\lambda$  of COTAC 74 is identical longitudinally and transversely.

3. The similar evolution of the difference in

TABLE 2. PHYSICAL PROPERTIES

a Propriétés	b Matériaux	c Cotac 74 orienté		f IN 100 équiaxe
		d en long	e en travers	
g Densité, $\rho$ ( $10^3$ kg·m <sup>-3</sup> )		8,6		7,8
h Température de fusion commençaute (K)		1605		1536
i Dilatation thermique linéaire moyenne, $\alpha$ ( $10^{-6}$ K <sup>-1</sup> ) entre la température ambiante et	1000 K	13,7	14,6	14,9
	1100 K	14,3	15,3	15,5
	1200 K	15,2	16,3	16,3
	1300 K	16,2	17,0	17,4
j Chaleur massique, $c_p$ (J·kg <sup>-1</sup> ·K <sup>-1</sup> ) à	1000 K	625		625
	1100 K	675		660
	1200 K	725		760
	1300 K	800		815
k Conductivité thermique, $\lambda$ (W·m <sup>-1</sup> ·K <sup>-1</sup> ) à	1000 K	26		18
	1100 K	27,5		20
	1200 K	29,5		23,5
	1300 K	31,5		27

- Key: a. Property  
 b. Material  
 c. Oriented COTAC 74  
 d. Longitudinal  
 e. Transverse  
 f. Equiaxial  
 g. Density  
 h. Incipient melting temperature  
 i. Average linear thermal expansion,  $\alpha$   
 ( $10^{-6} \text{K}^{-1}$ ) between ambient temperature  
 and  
 j. Specific heat,  $c_p$  (J·kg<sup>-1</sup>·K<sup>-1</sup>) at  
 k. Thermal conductivity,  $\lambda$  (W·m<sup>-1</sup>·K<sup>-1</sup>) at

TABLE 3. COMPARATIVE THERMAL DIFFUSIVITY

T <sup>0</sup> (K)	$\lambda_{\text{Cot}}$	$\lambda_{\text{IN}}$	$\frac{\lambda_{\text{Cot}} - \lambda_{\text{IN}}}{\lambda_{\text{IN}}}$	$c_{p\text{Cot}}$	$c_{p\text{IN}}$	$\frac{c_{p\text{Cot}} - c_{p\text{IN}}}{c_{p\text{IN}}}$	$\frac{\lambda/c_{p\text{Cot}} - \lambda/c_{p\text{IN}}}{\lambda/c_{p\text{IN}}}$
875	24,3	15,5	0,57	585	390	0,50	-0,05
1225	30,5	24,2	0,26	745	785	-0,05	0,20

## Thermal Fatigue

Superimposed on the creep (centrifugal stress) and fatigue (high frequency vibratory pulsations) stresses, the phenomenon of thermal fatigue, i.e., the repeated application of combined mechanical and thermal stresses at a localized point on the blade, is one of those which can most unfavorably degrade the lifetime of a blade. Meanwhile, study of the laws of creep-fatigue superposition has only started [9,10]. The materials have been characterized by laboratory tests, generally in a unidirectional application. But, the mechanism of failure of a blade depends on a combination of complex stresses, among which, priority must be given to consideration of loads connected with transient operation. Therefore, the number of flight cycles of which the blade is capable must be determined, and this determination requires knowledge of the stress and temperature ( $\sigma$ , T) spectrum during one cycle. Of course, the type of flight cycle, like the number of cycles, is different, from which it follows that the question is of a commercial, medium or long-range aircraft or of a fighter, for example.

It is simplest to calculate the mechanical deformation cycle /20-5 experienced by each blade element during the transient period, and to compare the results to repeated cycles in the laboratory. If, to simplify, it is assumed that the blade sections are plane and parallel to each other, because of the intensity of the centrifugal field, the level of local thermal stress in the elastic domain will be of the type

$$\sigma = E(T_{\text{mean}} - T_{\text{local}}).$$

Superimposed on the centrifugal stresses, the level of stresses thus calculated easily surpasses the elastic region. Meanwhile, by means of the abovementioned simplifying hypothesis, if the laws of behavior of the material are known, calculation becomes possible for the stop  $\rightarrow$  full throttle phase, as well as for the instantaneous reverse transition (NB. in the case full throttle  $\rightarrow$  stop, each cycle differs from the preceding one for, in the plastic region of the metal, each blade element already has its own mechanical deformation. Meanwhile, experience shows that the deformation cycles rapidly converge towards a stabilized cycle, which permits the conduct of tests on simple test specimens in the laboratory, which are representative of the cycle experienced by each blade element).

Experimentally, two types of tests were carried out on the COTAC 74 composite: on the one hand, thermal cycling tests intended to define the structural stability of the material during repeated heating-cooling cycles and their effect on mechanical properties; on the other hand, so-called thermal fatigue tests, on wedge test pieces, under conditions simulating the cracking of the trailing edges of the turbine blades.

## Thermal Cycling

The difference in the coefficients of thermal expansion of the fibers and of the matrix can introduce strong internal stresses into the composite, during repeated heating and cooling. If the stresses exceed the elastic region of the matrix, the thermodynamic equilibrium between the phases is destroyed, and the accumulation of repeated plastic deformations can cause degradation of the reinforcement phase, which involves lowering the characteristics of the material. Therefore, it was decided to search for COTAC 74 composites, with strongly hardened matrices and an elevated elastic limit, so as to provide the composite a cycled creep lifetime equivalent or as close as possible to the creep isotherm [11]. This was the meaning of the choice of Ni base matrices which are advantageously hardenable in solid solution and by coherent precipitation from the ordered  $\gamma'$  phase.

The stress tests used are generally 30 minute cycles, with maintenance at maximum temperature (800-1100°C) for 28 minutes, followed by rapid cooling to about 250°C. Under these conditions, the effect of thermal cycling on fiber stability and COTAC 74 composite performance are zero at moderate temperatures (for example, nonfailure in 5000 hours of creep cycling, more than 10000 cycles, between 800 and 250°C, under a stress of 400 MPa); at maximum temperatures of 1100°C, thermal fatigue causes slight degradation of the characteristics: to obtain a lifetime equivalent to that of a creep isotherm in creep cycling, the stress has to be reduced about 10% [7].

## Cracking in Thermal Fatigue

These tests, the object of which is the study of the formation and growth of cracks, under conditions representative of actual cracking of the trailing edges of turbine blades, were carried out with thin edged wedge test pieces ( $r=0.25$  mm), by heating to 1100°C in 60 seconds with a propane burner and cooling to about 100°C in 20 seconds in a forced air jet (Fig. 5).

For the purpose of comparison, the tests were carried out under identical conditions, on COTAC 74 composite and on two superalloys, Ni base IN-100 and Co base MAR-M 509.<sup>3</sup> The three materials were given a preliminary surface protection. Since the heating and cooling take place by direct attack on the thin edge, this results in establishment of isotherm which are practically parallel to the edge, to the reinforcing fibers and to the grain joints of the composite. Under these conditions, the cracks appear on the thin edge of the test pieces. Fig. 6 compares the

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<sup>3</sup>MAR-M 509, rated composition: Co, 23 Cr, 10 Ni, 7 W, 3.5 Ta, 0.6 C, Zr, Ni.

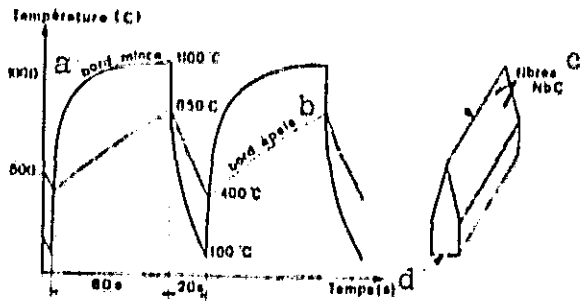


Fig. 5. Thermal fatigue of COTAC 74; diagram of test piece and of cycle.

Key: a. Thin edge  
 b. Thick edge  
 c. NbC fibers  
 d. Time, sec.

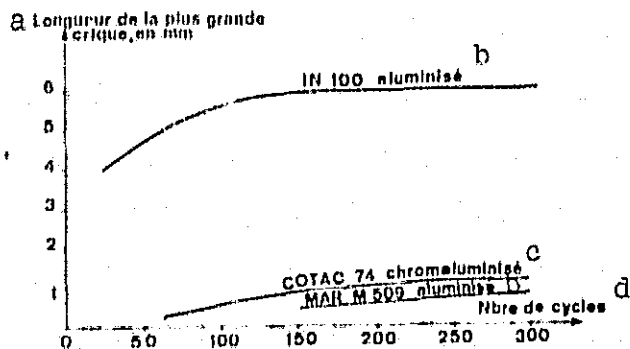


Fig. 6. Thermal fatigue: evolution of longest crack vs. temperature.

Key: a. Length of largest crack, in mm  
 b. aluminized  
 c. chrome-aluminum plated  
 d. Number of cycles.

propagation of the longest crack of each material, as a function of time (number of cycles).

It is established that the behavior of COTAC 74 composite, clearly better than that of the MAR-M 509 Co base alloy, known for its good thermomechanical cracking behavior [12]. A finer analysis of the test permits the differences in the cracking mechanisms in these two materials /20-6 to be brought out. In MAR-M 509 alloy, formation of small cracks, on the order of 0.5 mm, which slowly, but continuously propagate, until reaching 5-10 mm in length. On the other hand, in COTAC 74, the small cracks stabilize at a length of from 0.5 to 1.5 mm, which corresponds to the distance between the thin edge and the first grain joint. Meanwhile, if the cracks do not progress towards the inside, the number of them increases with number of cycles, and it can reach a density of 20 cracks per cm. Although the thermal fatigue cracks do not develop further in COTAC 74 after a few hundred cycles, some tests were conducted well beyond that. After 4500 cycles, the surface protective coating can be seen to swell and come off, which involves increased local oxidation at the level of the composite-protection interface (Cr Al standard protection; new protective coatings, on which there will be a question further on [13], have not been tested in these tests).

With respect to IN-100 equiaxial alloy, the slower progress of the cracks is explained here, on the one hand, by the lower modulus of elasticity of the composite, which permits the formation

of a plastic zone in the front of the crack. (In this highly anisotropic material, the transverse modulus is about 8% higher; meanwhile, it must be noted that the specific modulus  $E/\rho$  remains lower and that this leads, in pieces of the same geometry, to about 20% lower frequencies.) On the other hand, the absence of grain joint perpendicular to the trailing edge apparently also is favorable to good thermal fatigue behavior.

### Less Conventional Tests

For isotropic materials, there are a number of methods of calculating and testing behavior, based on the theory of deformation of a homogenous solid. The appearance of synthetic composites in aircraft structures has, on its part, spurred the development of analytical tools which are better adapted to anisotropic materials. Meanwhile, the region concerned, for the time being, does not exceed the range of moderate temperatures, on the order of 500°K. It would be desirable to see today techniques for natural refractory composites, which have to operate in the region of elevated temperatures. Experimentally, we present here a certain number of the results of less conventional tests, such as bending creep, notch creep or, further, shearing creep, to characterize the behavior of the base of the blade [14].

#### Bending Creep

It concerns determination of how the relatively low deformability of the composite in high temperature tension creep is emphasized or attenuated, when the stress is not uniform in the entire cross section of the test piece. By way of comparison, these tests were carried out in parallel on COTAC 74 and IN-100, at temperatures of 900 and 1050°C.

The test pieces are mounted in a three point bending assembly, distance between supports 20 mm, at 900 and at 1050°C. The creep deformation corresponds to measured deflections of the recordings 0.25 hour before failure.

TABLE 4. COMPARATIVE MODULI OF ELASTICITY

		Cotac 74		IN 100
		a	1	
Modulo d'elasticité dynamique (GPa) a	300 K	158	171	218
	1000 K	122	137	175
	1200 K	108	118	158

Key: a. Dynamic modulus of elasticity (GPa) at

TABLE 5. BENDING CREEP

Température (°C)	Charge (kg) <sup>a</sup>	IN 100		Cotac 74	
		t <sub>rupt.</sub> (h) <sup>b</sup>	flèche (mm) <sup>c</sup>	t <sub>rupt.</sub> (h) <sup>b</sup>	flèche (mm) <sup>c</sup>
900	90			1273	11
	105	150	6	110,4	11
	126	40,5	7,0	76	11
1050	27	332	0,0	600	1,0
	35	124	4,5	163	2,3
	40	35	5,0		
	45	21	5,2	28,5	2,7

Key: a. Load  
 b. t<sub>fail</sub>, hours  
 c. Deflection (mm)

It is noted (Table 5) that the creep lifetimes increase rapidly as the load decreases, while the deformation of the breaks remains practically constant. At 900°C, the composite does not demonstrate particular fragility. The plasticity is satisfactory, superior to that measured on the commercial superalloy for comparable lifetimes. At 1050°C, relatively low capacity for plastic deformation is noted in the composite, although, for an identical load, the failure time of COTAC 74 remains longer than the lifetime of IN-100.

Creep of Notched Test Pieces (K<sub>t</sub>=3.4)

Cotac 74 test pieces were selected parallel to the reinforcing fiber axis. Fig. 7 permits comparison of the creep lifetimes of smooth test pieces and notched test pieces (K<sub>t</sub>=3.4) of COTAC 74, under different stresses, at 800, 1000 and 1050°C.

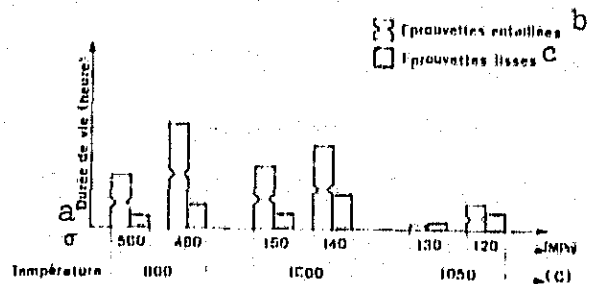


Fig. 7. Comparative smooth creep and notch creep lifetime of COTAC 74 (K<sub>t</sub>=3.4).

Key: a. Lifetime (hours)  
 b. Notched test pieces  
 c. Smooth test pieces

It can be noted that the presence of a K<sub>t</sub>=3.4 notch does not have a detrimental effect on the creep behavior of the composite. The lifetimes of the notched test pieces are equivalent to those determined on smooth test pieces at 1050°C; at 1000°C, they increase by a factor of 2.5 to 4.1 and, at a temperature of 800°C (base of blade), the presence of the notch improves the failure time by a factor of 4 to 4.3.

Shearing Creep (Base of Wedge)

The aim of these tests is to subject the composite to shearing stresses parallel to the fibers, the type of application which is found in the blade attachment prongs to the turbine disc. Comparative tests were carried out on COTAC 74 composite and on IN-100 alloy, at a temperature of 700°C, under a stress of 400 MPa (load directed to the theoretical shearing surface, in the meaning of the force applied). By analogy with the behavior of synthetic composites, it can be feared that a shearing force parallel to the fibers will cause premature failure under a low stress, along the matrix-fiber interfaces. This is the reason for the choice of the particularly severe conditions, with relatively increased shearing stresses, with account taken of the actual applications, to which the base of the blade is subjected.

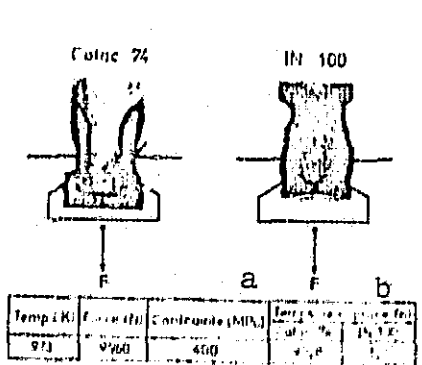


Fig. 8. Shearing creep.

Key: a. Stress (MPa)  
b. Failure time, hours

It is noted (Fig. 8) that, under these conditions (973°K, 400 MPa), failure of the composite occurs by shearing creep entirely parallel to the fibers, while, under the same conditions, IN-100 equiaxial alloy breaks rapidly by separation under tension. In consideration of the great difference in recorded lifetimes in this test, it appears that the behavior of the base of the wedge of COTAC 74 blades is no particular cause for concern.

Oligocyclic Fatigue (Base of Blade)

In order to complete the characterization of the behavior of the COTAC 74 blade attachment, oligocyclic fatigue tests were carried out at a temperature of 923°K. As a result of the combination of thermal and mechanical stresses, a part is subject to low frequency vibration, which attain the highest levels in transient operation during an acceleration or rapid deceleration of the engine.

The tests were carried out by repeated tension, at a frequency of 1 cycle per minute. Each cycle includes rapid placing under stress (10 sec), followed by a stage of maintenance at  $\sigma_{max}$  for 20 seconds; unloading (10 sec) was followed, in turn, by a stage of maintenance at  $\sigma_0$  for 20 seconds (Fig. 9).

On flat test pieces grooved on one side ( $K_t=2.4$ ), comparison of  $10^4$  cycle endurance results shows a clear superiority of COTAC 74, nearly 40%, over the commercial alloy.



## High Frequency Fatigue

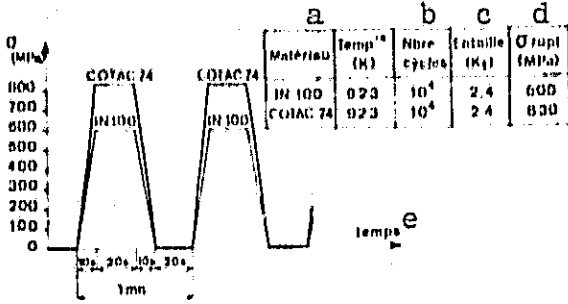


Fig. 9. Comparative oligocyclic fatigue at 923°K on notched test pieces ( $K_t=2.4$ ).

Key: a. Material  
 b. Number of cycles  
 c. Notch  
 d.  $\sigma_{fail}$   
 e. Time

In a general way, in high frequency fatigue, the endurance is greater than that of conventional superalloys [15]. For COTAC 74, the results are shown in Table 6. The transverse fatigue behavior is at 75% of the longitudinal result and, with regard to the effect of the surface protection, the endurance limits determined by torsion flexure indicate a minimum reduction on the order of 2%. Fractographic examination of the surfaces shows that about 70% of the cross section is of the fatigue failure type.

TABLE 6. HIGH FREQUENCY FATIGUE OF COTAC 74

Température (K)	Eprouvette a	Solicitation b		d $\sigma_{non\ rupt}$ , en 10 <sup>7</sup> cycles (MPa)
		Type	Fréquence (Hz) c	
293	Torçure, // aux fibres e	Flexion rot. f	49	400
293	Torçure, ⊥ aux fibres g	Flexion rot. f	49	510
973	Torçure, // aux fibres e	h Traction répétée	87	20 680
1073	Torçure, // aux fibres e	h Traction répétée	87	20 520

Key: a. Test piece  
 b. Load  
 c. Frequency (Hz)  
 d.  $\sigma_{nonfail}$  in 10<sup>7</sup> cycles (MPa)  
 e. Cylindrical parallel to fibers  
 f. Torsion flexure  
 g. Cylindrical perpendicular to fibers  
 h. Repeated tension

These results must not make it be forgotten that failures frequency reveal more complex mechanisms, for example, superposition of stationary applications on high frequency vibrational phenomena. Likewise, the risk of resonance with the natural frequency of the blade, reported above, must be watched for.

Ballistic Impact

In these technological type tests, the development of damage at high temperature, caused by projectiles fired at different velocities is compared. A 3 g truncated tip projectile strikes small 30 x 70 mm target plates, 2.5 mm thick, fitted into a recess at one end. The targets are raised to temperatures of 800, 950 or 1100°C, by means of an oxyacetylene torch, and the temperatures are monitored on the opposite face, at the impact level, by two thermocouples. The tests of COTAC 74 composite and IN-100 and MAR-M 509 alloys allow it to be stated that, at the firing temperatures and conditions, the composite has the best impact behavior [14].

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At 800°C and comparable damage, the muzzle velocity gain of COTAC 74 is 20 m·sec<sup>-1</sup> over IN-100 and 30 m·sec<sup>-1</sup> over MAR-M 509.

At 950°C, the advantage over IN-100 is 40 to 50 m·sec<sup>-1</sup> and, over MAR-M 509 targets, 25 to 30 m·sec<sup>-1</sup>.

At 1100°C, COTAC 74 was only compared with MAR-M 509 alloy. The apparent deformations are smaller than at 800 and 950°C, cracks develop parallel and perpendicular to the fibers, and the impact behavior of the composite remains superior to that of MAR-M 509 alloy. Meanwhile, poor adherence of the surface protection deposit (test pieces protected by standard Cr Al) is noted; it cracks (Fig. 10).

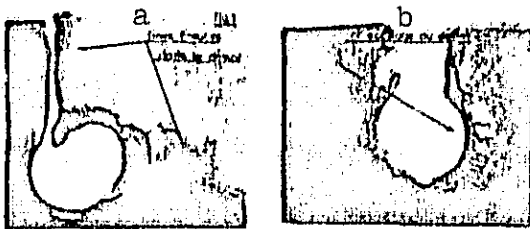


Fig. 10. COTAC 74 protected by standard Cr Al: ballistic impact tests.

- Key: a. Fine closed cracks of deposit
- b. Separation of deposit.

Dry Corrosion, Oxidation and Protection of COTAC 74

Near accidental damage caused by injection of foreign bodies or by engine trouble, the hot parts undergo progressive deterioration of their properties in operation, with respect to the original characteristics, and that is as much the cause of structural change of the material, as the result of surface aggression by the ambient medium. Concerning structural stability (problems of accelerated thermal diffusion, appearance of undesirable phases, coalescence, etc.), the results of long-term tests of COTAC 74, reported above, demonstrate amply that this point is the object of the alert attention of metal physicists. The problem of the effect of prolonged exposure of the composite to a highly oxidizing atmosphere, aggravated at more moderate temperatures by sulfurizing attack in a saline environment, remains. We deal with it below.

The air-fuel ratio has a great excess of air, and the combustion gases always attack the blades at high temperature with an oxidizing flame. On the other hand, in aircraft engines which, nevertheless, use refined kerosene, the combustion gases do not contain less of such impurities as sulfur and salt, which come from the fuel, as well as from the air.

Since there are no metal alloys which can, at the same time, satisfy the multiple mechanical conditions required at high temperature and provide perfect inertness toward the aggression of the ambient medium, the material is selected, as a function of the first criterion (high mechanical and thermal strength), on condition, of course, that it at least have the capability of surface protection by an appropriate coating. In fact, whenever metallic material is chosen, the turbine blades receive a protective cladding.

The protective qualities of the cladding (chemical attack, erosion, stability) have to be accompanied by good adherence to the substrate, relative stability with respect to it so as to minimize exchange by diffusion, and a plasticity capable of compensating the differences in thermal expansion between the part and its coating. For safety reasons, it also is necessary for the oxidation and dry corrosion resistance of the alloy itself to be sufficient to eliminate the risk of catastrophic damage, leading to rapid destruction of the part as a result of accidental damage to the protective coating.

There are no particular problems on this last point. The dry corrosion or oxidation behavior of unprotected COTAC 74 composite at high temperature is generally on the same order as, if not better than that of most of the alloys actually used, IN-100 in particular. This is attested to by numerous oxidation and direct corrosion tests of bare COTAC 74, as well as by long-term creep and fatigue tests in the air, on test pieces with and without protection. On the other hand, the problem of protection of the composite turns out to be more critical.

First, there is the fact that, if the composite is to provide an operating temperature advantage, the protective coating itself also has to be able to resist at least 42 to 60°K higher temperatures, than is required of actual commercial alloy blades. In addition, it is true that biphasic, anisotropic composites did not have the same coating capability as homogenous alloys.

The development of ONERA methods of protection has permitted an efficient solution to be found, which consists of putting an intermediate diffusion layer between the composite material and the surface cladding, which is made up of a Ni-Cr alloy with stabilizers. This point will be developed later [reference 13]. It is sufficient to state here that the coatings developed, called DE 77,

are relatively ductile and provide effective protection of the composite against high temperature oxidation, as well as against saline corrosion, in cycling tests on an experimental wheel. The regular thickness of the cladding, predetermined with precision, practically does not change during prolonged holding at high temperature.

The effect of the DE 77 cladding on the mechanical properties of COTAC 74 have been examined in detail. Up to 900°C, creep tests give identical results on protected test pieces and on bare test pieces; at higher temperature (1000 and 1070°C), some loss in stress could be estimated at about 10%. More precise determinations are under way.

### Conclusions

We have set up a balance sheet of the characteristics of the native COTAC 74 composite, a material intended for the production of the blades of future turbine engines. More resistant, capable of an increase in temperature and stress, with respect to the performance of present alloys, the composite is no less than an unusual, highly anisotropic material, produced by special techniques. It is not evident, a priori, that its exceptional properties, measured parallel to the fibers, are sufficient to cause spontaneous adherence to it by the engine builder, even if performance in other areas seems reassuring to us. COTAC 74 is a new material, and its possible use depends less on the appreciation of those who produce it, than on the interest of those who have to apply it. Also, rather than make a judgement on quality, we have limited ourselves to comparing the characteristics, conventional and less conventional, with the properties of the best metallic materials of today, in order to see the measure in which COTAC 74 offers the possibility of satisfying the requirements of the engine technology of tomorrow. The question remains open, and the elements of the response have to be sought in a continued exchanged with future users. /20-9

For ourselves, the satisfying results already obtained on COTAC 74 do not hide from us the importance of the work still to be done. We must deepen study of the fundamental problems of solidification and progressively decrease the number of simplifying hypotheses, in order to place at the disposal of industry an elegant and simple method of production, which permits composite parts to be obtained with the required properties and at reduced cost. We must improve foresight by thermodynamic calculation of the diagrams of more complex systems, avoiding trial and error.

This is the path we are following. While we have limited ourselves here to the presentation of COTAC 74, which has been most completely characterized in laboratory tests and on the engine test stand, refinements in composition has already permitted us to enlarge the COTAC 74 family with types 741 and 742, which are

stronger, more refractory and more reliable. New methods of preparation, which have actually been tested, permit the direct preparation of blades in finished form, and the program of calculation in the study will permit its extension to the simultaneous industrial solidification of clusters of oriented blades. But, progress should be achieved in the field of ceramics, for the production of cores with good cohesion, which are easy to remove.

The drilling of transverse cooling channels in COTAC 74 blades can be done by electrochemical machining, with electrolytes and relatively simple electrodes adapted to the materials; high drilling rates have been achieved by electrical erosion. Encouraging results by diffusion welding or diffusion brazing indicate the possibility of producing hollow blades by this technique, a procedure which could compete with the technique of ceramic cores, which are too difficult to remove.

The results of transverse and shearing creep tests of COTAC 74 are reassuring, and they do not appear to have to involve modifications of the design of the base of the blade. But, a closer analysis, for example, a complete three-dimensional calculation by the finite element method of the state of stress of the blade attachment, will be better if, with the elastic stress of COTAC 74 taken into account, the form of the present base can be preserved.

In the present stage of development, the required progress in the use of natural composites in turbine engines, can only be accomplished by close collaboration between those looking for materials, the heat specialists and the engine builders. This is the manner in which we are engaged and we can only congratulate the organizers of the Conference, for having facilitated such a confrontation here.

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