N 18, A NEW DAMAGE TOLERANT PM SUPERALLOY FOR HIGH TEMPERATURE

TURBINE DISCS

C. DUCROCQ , A. LASALMONIE, Y. HONNORAT

SNECMA – MATERIALS AND PROCESSES DPT – BP 81 – 91003 EVRY CEDEX

ABSTRACT

This paper describes the microstructure and the properties of N 18. This new P.M. alloy was designed for application in high temperature turbine discs. N 18 is strengthened by a high volume fraction of γ' (55 %). We describe how the N 18 composition was selected to fit the SNECMA requirements ; we show that it is possible in this alloy to control, the respective volume fraction of inter and intragranular γ', as well as the grain size by thermomechanical treatments ; as compared to other P.M. superalloys such as Astroloy, IN 100 and René 95, N 18 offers a unique combination of tensile strength, creep resistance and damage tolerance.

INTRODUCTION

The development by SNECMA of jet engines with improved efficiency and specific power requires, for the turbine discs, up to 750°C, the use of a limited density forgeable Ni based superalloy which has, a good compromise of mechanicals properties :

- high yield strength [R 0,2 $>$ 1000 MPa for T $\leqslant$ 750°C] so that the disc can tolerate overspeed without burst,

- high creep resistance,
- good damage tolerance : under cyclic deformation, the crack growth rate should be as low as possible even when environmental effects and hold time under stress are taken into account.

Several P.M. superalloys fullfil the strength specification such as René 95, IN 100, MERL 76 and Astroloy. Unfortunately the crack growth rate is an increasing function of yield strength (fig. 1) so that none of these alloys can satisfy the SNECMA goal, although the closest one is Astroloy.

As a consequence a research program was undertaken involving SNECMA, two laboratories : ONERA and EMP, and an alloy producer : IMPHY S.A., to define and develop, a new P.M. alloy, at the industrial scale.

EXPERIMENTAL TECHNIQUES

The conception and development of the new alloy progressed in three phases :

PHASE A : laboratory scale : a systematic study of the microstructural properties relationships based on physical considerations was made. Small batches of powders (10-30 kg) were atomized by REP and compacted by HIP. 47 alloys derived from Astroloy and René 95 were prepared to optimize the following parameters in three steps :
1 - the carbon content
2 - the nature and amount of γ and γ' forming elements
3 - the minor elements.

PHASE B : pilot scale : from the results of phase A, five compositions were selected for a more extensive characterization of mechanical properties and structural stability. The following fabrication route was used :

- atomization by rotating electrode process (50 kg)
- screening at 65 mesh (224 μm)
- compaction by HIP and isothermal forging.

The heat treatments were : a solutioning treatment (4h at 15°C below the γ' solvus) followed by a cooling at 100°C/min + 700°C/24 h air cooled + 800°C/24 h air cooled.

The comparison was made with the help of the following tests :

- tensile deformation (T $\leqslant$ 750°C)
- creep on smooth and notched specimens (650°C $\leqslant$ T $\leqslant$ 750°C) ; fatigue crack growth rate measurements at 650°C with hold time 300 s.

PHASE C : industrial scale : the best composition (N 18) was argon atomized in the IMPHY S.A. plant. Several atomization batches of 100 kg were mixed to form large blends.

A blend of 300 kg of screened powders (diameter $<$ 106 μm) was used for the N 18 characterization.

The composition range of the N 18 family [1] is given in table 1. The choice of the elements obeyed the following objectives :

- the peak strength of the γ' phase must be shifted toward high temperatures,

- the intrinsic strength of the γ matrix must be high.

ALLOY	Cr	Co	Mo	Al	Ti	Nb	Hf	Other	C ppm	B ppm	Zr ppm
N 18 Patent	11-13	8-17	6-8	4-5	4-5	<1,5	≤1		≤500	≤500	≤500
N 18 this study	11,5	15,7	6,5	4,35	4,35		0,5		150	150	300
IN 100	12,4	18,5	3,2	5,0	4,4			V:0,8	700	200	600
MERL 76	12,4	18,5	3,2	5,0	4,3	1,65	0,75		250	200	450
RENE 95	13	8	3,6	3,5	2,5	3,5		W:3,5	250	75	500
ASTROLOY	14,6	16,6	5,0	4	3,5				280	280	600

Table 1 : composition (Wt%) of N18 and other high strength P.M. superalloys

γ' forming elements :

The strenghtening capability of γ' can be improved by the addition of Ta or Nb (René 95). Because of its high density Ta was eliminated ; as for Nb, it has the drawback of increasing the notch sensitivity and the high temperature fatigue crack – growth rate [see René 95 on fig. 1] , so that the maximum content of Nb was limited to 1,5 weight %. As a consequence Al and Ti are the main γ' forming elements ; the required strength necessitates a volume fraction of γ' higher than 0,5. The sum Al + Ti was taken as 0,1 with a near unity ratio Al/Ti.

γ forming elements :

Mo and W are very efficient as high temperature solid solution strengheners of γ . A detailed study (2) evinced that Mo should be preferred to W :

- the partition ratio of Mo between γ and γ' was twice that of W

- for a given W + Mo content, the notch sensitivity during creep at 650°C increased drastically with the amount of W, whereas the rupture time for smooth specimen was only slightly improved (fig. 2) ; moreover the need of keeping a low density was in favour of Mo.

Other elements :

The choice of the chromium content (11-13%) gives to the N 18 family a good oxydation resistance, without excessive precipitation of intragranular carbides, detrimental for the ductility. For the same reason a low carbon content is prefered. Some Hf (≤1%) is added to promote the formation of MC carbides without scavenging Mo, Ti and Cr ; thus Hf has an indirect effect on the strengthening ; some Hf remains in solid solution within γ and γ' and contributes directly to the strenghtening.

The composition range of the N 18 family is given on table 1 ; on the same table is given the composition which was extensively tested : on table 2 its properties are compared with those of the reference commercial alloys.

Fig.1 : Yield strength and fatigue crack
       growth rate at 650°C of PM alloys
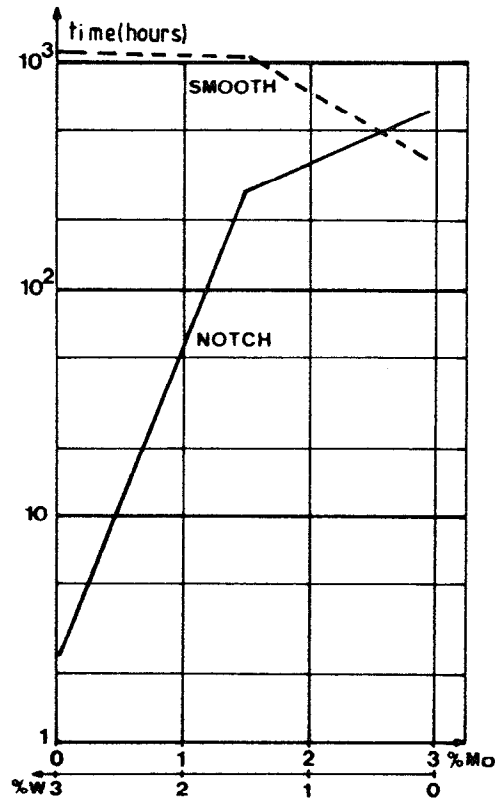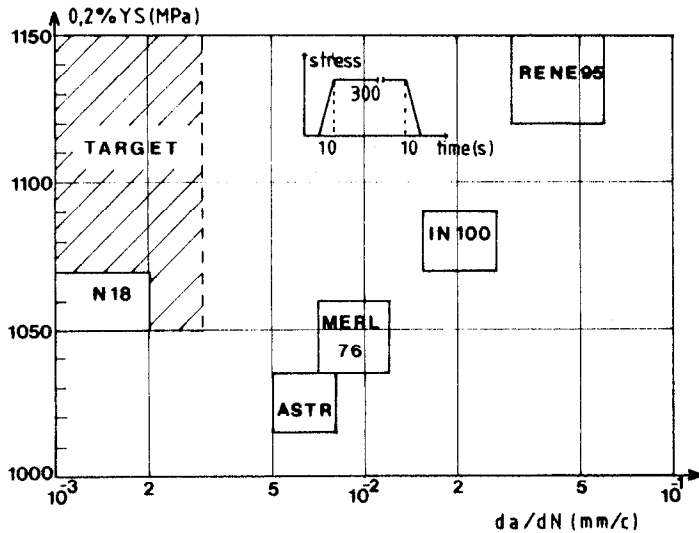   SNECMA results, except René 95[9]

Fig.2 : Creep rupture time at 650°C
        1000 MPa – Influence of W
        substitution (W+Mo=3%)

	Fraction γ'	solvus γ'	Density g/cm3	Nv	Md
IN 100	61 %	1185°C	7,9	2,471	0,928
MERL 76	64 %	1190°C	7,95	2,591	0,941
RENE 95	48 %	1155°C	8,27	2,217	0,915
ASTROLOY	45 %	1145°C	8,0	2,357	0,926
N 18	55 %	1190°C	8,0	2,361	0,932

Table 2 : Comparison between N 18 and commercial alloys

INDUSTRIAL PROCESSING

Powder production :

In view of mechanical characterization IMPHY S.A. produced a 300 kg blend
of N 18 powder ; it was argon atomized, and then containerized in the clean
rooms of IMPHY S.A. ; the transfer of powder in smaller extrusion containers
was done in the clean rooms of SNECMA.

Powder control :

The present résults were obtained from powders smaller than 106 μm (150 mesh)
50 % of which was below 50 μm. The cleanliness was checked by the water elu-
triation procedure described in the SNECMA patent [3] .

The cleanliness was equivalent to that of industrially produced Astroloy as
shown in table 3. The gas content of the powder was 86 ppm oxygen and 34 ppm
nitrogen (compared to 65 ppm O and 30 ppm N for Astroloy powder).

Size of inclusions	N 18*	ASTROLOY*	Specif. SNECMA
80 < size < 106 μm	6	3	< 20
63 < size < 80 μm	8	6	

* N 18   300 kg Ø < 106 μm (150 mesh)
* ASTROLOY   2700 kg Ø < 106 μm (150 mesh)

Table 3 : Water elutriation results on N 18 and Astroloy powders

Powder consolidation

Two routes were tested :
- compaction by HIP and conventional forging of 50 kg billets giving rise to a necklace structure,
- extrusion of 30 kg billets on the 4000 T. SNECMA press and isothermal forging of 5 kg billets on the 600 T. SNECMA press.

The second route proved to be better, because of an easier industrial practice and because of a better ultrasonic inspectability [4]. Small discs(Ø 170mm) were forged from bars (Ø:90 mm). The heat treatments were similar to those given above for phase B ; several solutioning temperatures were tried, results are presented here for two cases :

4 h at 1110°C giving a small grain size (5 μm) (fig. 3a) or solutioning
4 h at 1165°C giving a medium grain size (12 μm) (fig. 3b).

In both cases the cooling rate after solutioning was 100°C/min and the precipitation treatments were 700°C/24 h A.C. + 800°C/4 h A.C.. For the medium grained alloy discs were forged at a reduced scale (Ø 180 mm) and bench tested (specially for overspeed resistance) at SNECMA (fig. 4).
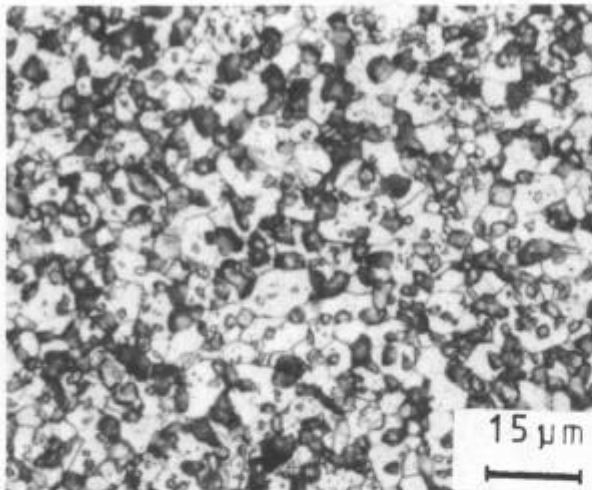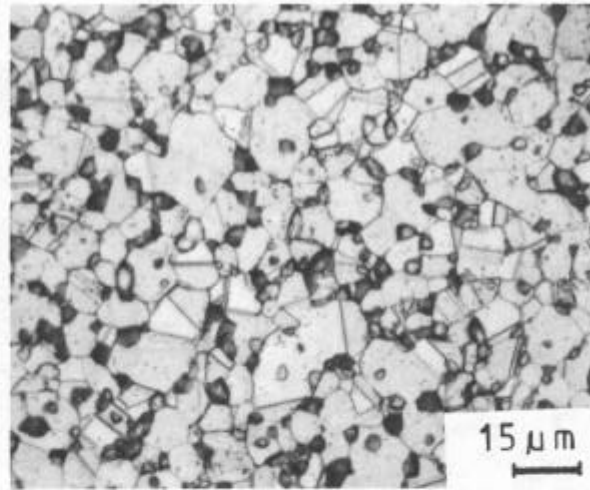

15 μm

Fig. 3 a

15 μm

Fig. 3 b

Fig. 3 : the two tested microstructures a) solutioning at 1110°C
b) solutioning at 1165°C

Superplasticity

N 18 is superplastic at high temperature, for deformation rates smaller than 5.10^{-3} s^{-1}. The strain rate sensitivity exponent is about 0,7 as in Astroloy (fig. 5) ; for a 5 μm grain size, the flow stress is smaller than that of Astroloy and IN 100 ; this low flow stress allows the extrusion of larger bars for a given press.
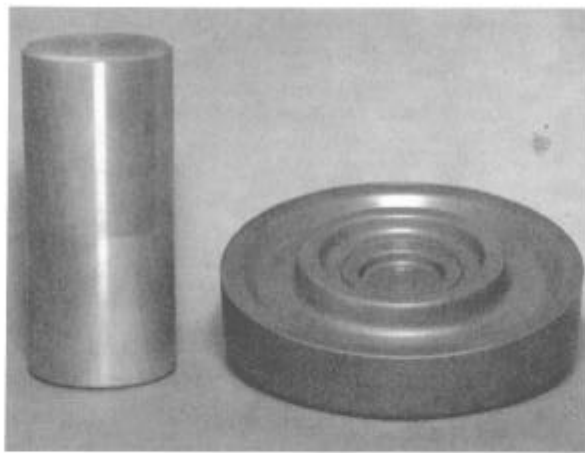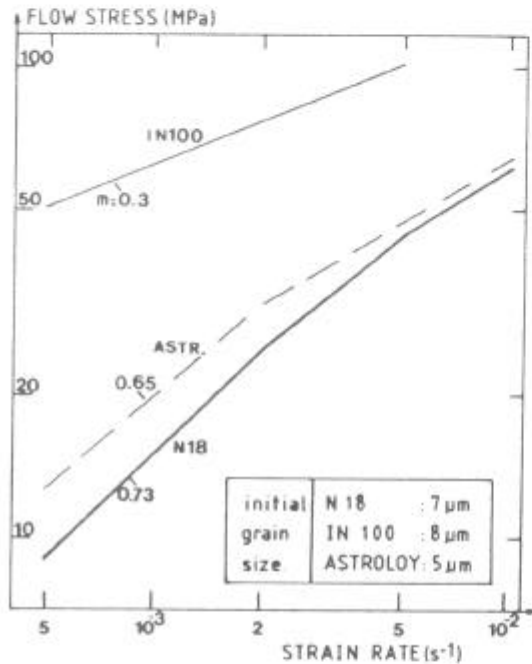
Fig. 4

Isothermally forged disc preform

Fig.5 : flow stress of superplastic N18, IN 100 [5] and Astroloy at 1100°C
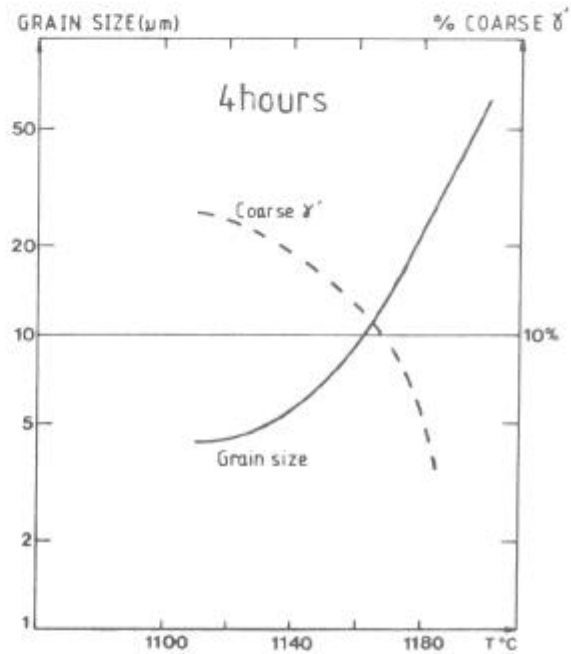

Fig. 6 : Influence of N18 solutioning temperature on fp and d

MICROSTRUCTURE

The main microstructural features which determine the mechanical properties are :
- the intergranular $\gamma'p$ precipitates formed at high temperature ; their volume fraction and size are fp and dp
- the intragranular $\gamma's$ precipitates formed during the heat treatments having a volume fraction fs and a size ds
- the grain size - d -.

The ability to control these parameters is critical when searching a balance between a high strength at low temperature and low creep rates above 600°C. For instance d must be small enough to induce a high Hall-Petch strengthening; on the contrary small d promote rapid grain boundary sliding during creep ; moreover it is well known that the crack growth rate at 650°C is a decreasing function of grain size [6] .

Fig. 3 shows extruded - isoforged structure with the two different solutioning temperatures (1110°C and 1165°C). In both cases the grain size is homogeneous. In high strength P.M. superalloys, the grain growth kinetics is very low below 1100°C and drastically rapid near or above the γ'solvus ; as a consequence

grain size control by heat treatment is difficult in Astroloy ; in N 18 it is easier to obtain a grain size in the range 15 μm - 30 μm due to the high γ' solvus (fig. 6). When the solutioning temperature increases from 1115°C to 1165°C, the fraction fp decreases continuously from 0,25 to about 0,15 ; since fs + fp $\simeq$ 0,55, the fraction fs of intragranular γ' is an increasing function of the temperature.

The γ' size resulting from the standard heat treatments is ds $\simeq$ 0,13 μm (fig. 7), whereas dp $\simeq$ 4 μm ; ds depends strongly on the cooling rate from solutioning but no systematic investigation of this parameter was made.
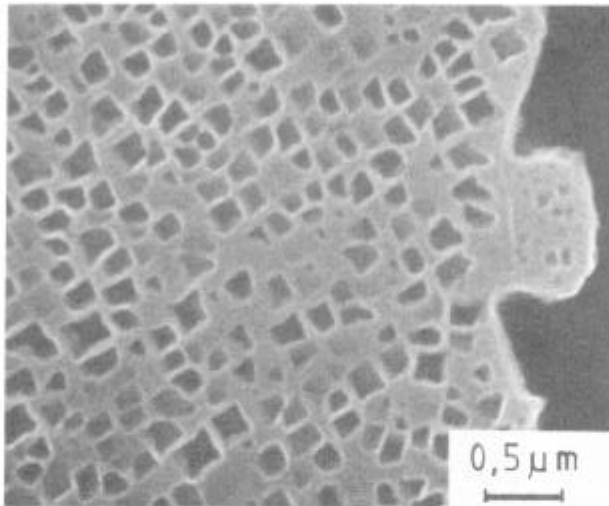

Fig. 7 : intragranular γ' after the
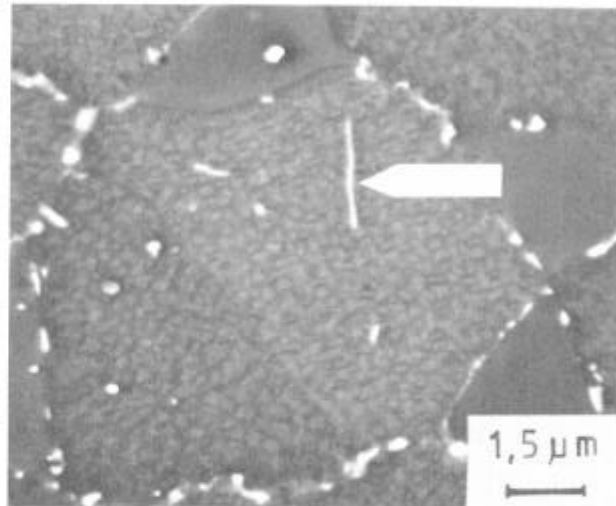standard precipitation
treatment

Fig.8 : intergranular carbides
and platelet precipitation
(arrow)

THERMAL STABILITY

The Nv [7] and Md [8] parameters are given in table 2 for N 18 and other P.M. superalloys. The stability of N 18 as respect to T.C.P. phases (i.e. σ phase) is equivalent to that of Astroloy. This was confirmed by microscopic observations on specimens annealed between 600°C and 800°C. At 650°C a treatment of 3000 hours induced a precipitation of fine intergranular carbides but no σ phase was found, at 700°C some rare intragranular platelets were observed after 600 h ; these platelets disappear above 850°C ; their length was about 1 μm and thickness 0,1 μm (fig. 8), they were enriched in Mo and Cr and were found to contain carbon ; there is however some ambiguity about their nature : from the diffraction patterns they can be M_7C_3 or σ . In N 18 the precipitation of the platelets can be retarded by increasing the solutioning temperature. Up to 750°C the volume fraction of the platelets remains always small and does not seem to be detrimental for ductility.

No platelet but a much more abundant precipitation of carbides was observed in Astroloy annealed in the same conditions.

MECHANICAL PROPERTIES

Tensile properties

As explained before, the most extensive characterization was made on extruded iso forged structures, with grain sizes 5 μm and 12 μm.

The tensile properties are ploted on fig. 9 and compared to Astroloy and IN 100 (grain size $\simeq$ 5 μm). N 18 has the highest rupture stress ; its yield stress is intermediate between Astroloy and IN 100 up to 650°C but is less thermoactivated above 650°C.

The tensile properties below 650°C of N 18 are almost insensitive to the grain size for the range 5 μm - 15 μm. Indeed, when the grain size goes from 5 μm to 15 μm, the softening due to the Hall Petch mechanism is $\Delta \sigma_1 = k$ $(d_2^{-\frac{1}{2}} - d_1^{-\frac{1}{2}}) = - 140$ MPa for a Hall-Petch constant $k = 0.7$ MPa $\sqrt{m}$ [10]. It is balanced by an increase of the γ's volume fraction (fs goes from 0,3 to 0,4) giving a strengthening $\Delta \sigma_2 / \sigma_2 = n \Delta f/f$ [for a constant ds] . $n = \frac{1}{2}$ if γ' is sheared and $n = \frac{1}{2}$ in the case of OROWAN bending so that 100 MPa $<$ $\Delta \sigma_2 <$ 150 MPa. The total variation - 40 MPa $< \Delta \sigma_1 + \Delta \sigma_2 <$ 10 MPa, is inside experimental dispersion.

- The properties are given for a cooling rate 100°C/min from the solution treatment. The yield stress can be modified by changing the cooling rate as shown in other alloys such as Astroloy (fig. 10).
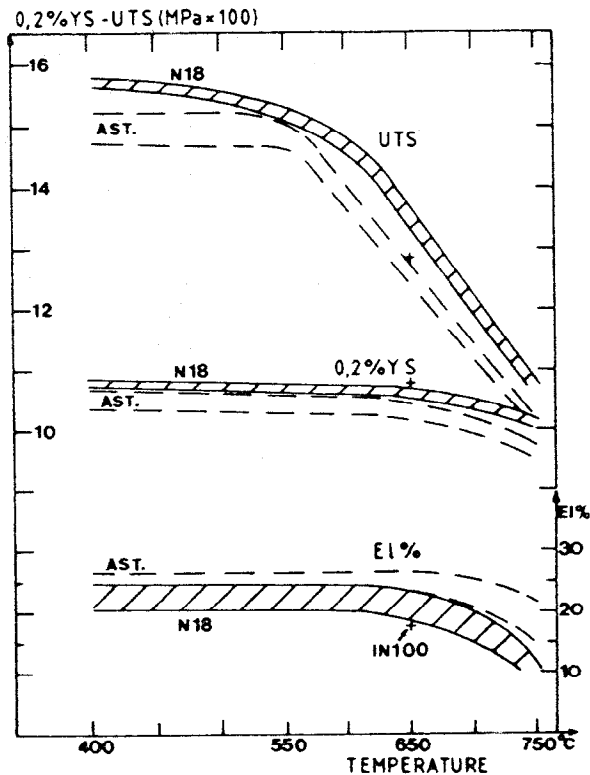- The ductility of N 18 is always higher than 12 .

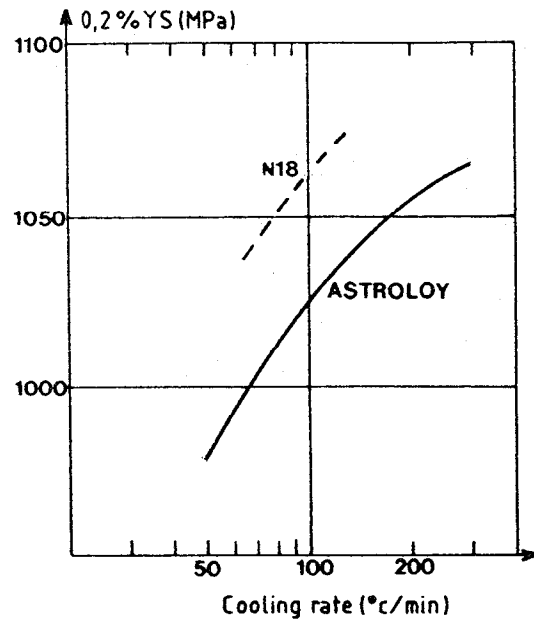Fig. 9 : tensile properties of N 18 Astroloy and IN 10

Fig. 10 : influence of cooling from the solutioning treatment on the yield stress at 650°C

Creep properties (600°C - 750°C)

In the usual Larson Miller plot, N 18 (d = 12 μm) is better than Astroloy and IN 100 (fig. 11). For a 5 μm grain size the creep properties are about the same as for Astroloy with a 5 μm grain size ; the creep ductility is about 7 % in the all range of temperatures.

Low cycle fatigue and crack growth rate

Fatigue life was measured both is stress and strain controlled tests at 550°C - 600°C and 650°C.
High stress (or deformation) levels were used so that the rupture initiated always on surface inclusions (mainly alumina) ; the fatigue life was found to be the same as in Astroloy tested in identical conditions : for instance at 600°C [f = 0,5 Hz, $\Delta \varepsilon = $ 1% (R ε = 0)] the number of cycles ot failure is 210^3 for both alloys. The crack growth rate at 650°C with a dwell time of 300 s is given on fig. 12 ; da/dN follows the usual relatioship da/dN = C. $\Delta K^{2.5}$ [6] and is about half the rate observed in Astroloy which is one fo the most damage tolerant alloys. As shown on fig. 13 the rupture is in-
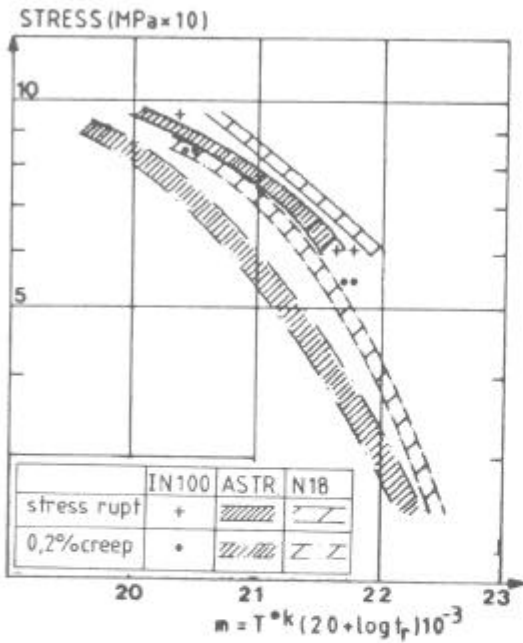
STRESS (MPa×10)

	IN100	ASTR	N18
stress rupt	+		
0,2% creep	•		

$m = T^{\circ k}(20+\log t_r)10^{-3}$

Fig. 11 : Larson Miller curve for
creep deformation and
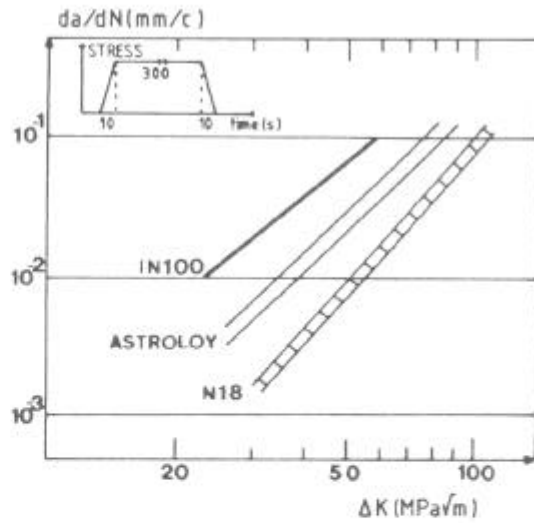creep rupture

da/dN (mm/c)

Fig. 12 : Crack growth rates at
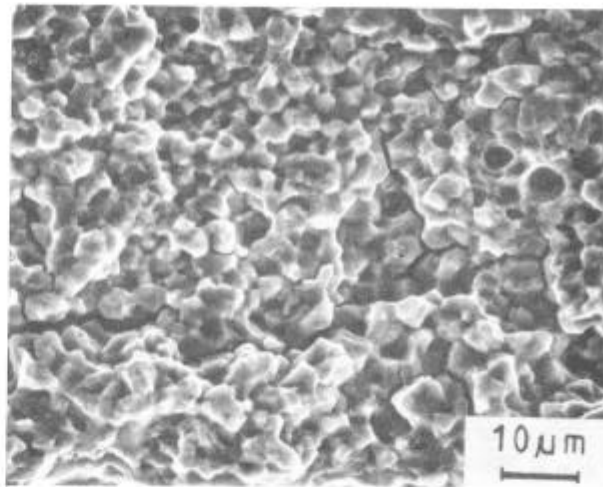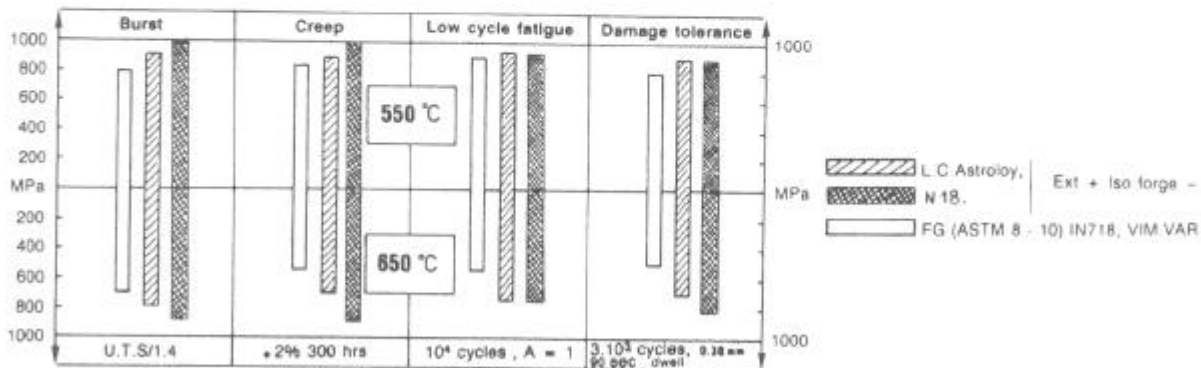650°C

Fig. 13 : Intergranular rupture
at 650°C in 12 μm N 18

10 μm

Fig. 14 : Minima design data at elevated temperature

L C Astroloy, | Ext + Iso forge –
N 18.
FG (ASTM 8 - 10) IN718, VIM VAR

U.T.S/1.4   + 2% 300 hrs   10⁴ cycles , A = 1   3.10³ cycles, 0.38 mm

tergranular so that da/dN is unlikely to be strongly affected by the strengthening of the matrix ; the main advantage of N 18 is obviously in the possibility of getting a medium grain size (10-20 μm) while keeping, at the same time, a high volume fraction of intergranular γ'.

CONCLUSION

Figure 14 summarizes the mechanical properties which are critical for the turbine disc designer. The figure shows clearly the superiority of N 18 over Astroloy and fine grained IN 718 which are presently used by SNECMA for compressor discs fabrication.

N 18 is characterized by better creep properties and damage tolerance at 650°C combined with a high rupture strength ; the mechanical properties of this new P.M. alloy fall within the initial target (fig. 1) ; these properties are the result of easy grain size control and γ' morphology combined with optimum strengthening of the grain and of the grain boundaries. Several tons have now been extruded for bench testing of actual turbine discs.

ACKNOWLEDGEMENTS

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