DEVELOPMENT OF DAMAGE TOLERANT INCO 718

FOR HIGH TEMPERATURE USAGE

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A specific heat treatment has allowed to develop a Damage Tolerant grade of INCO 718, which is called INCO 718DT. When applied on standard INCO 718 (TR), this treatment enables to get a very large improvement on crack propagation resistance under creep-fatigue cycles at 700 and 750°C. However, the static properties, both yield strength and creep resistance, are lowered at intermediate temperatures, while remaining acceptable for most applications. All those mechanical properties have been correlated with the microstructure evolutions, and specifically, the γ' precipitates.
A similar way of improvement on DA 718 revealed to be unsuccessful, due to an unacceptable decrease of creep resistance. For the DA grade, another specific treatment has to be determined to get high temperature damage tolerance capability.

Superalloys 718, 625, 706 and Various Derivatives
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Background

INCO 718 alloy is widely used for aeronautical turboengines applications, both on forged parts (disks, blades, shafts, supports...) and cast frame sections. This Fe-Ni rich superalloy has been developed more than 30 years ago and exhibits attractive mechanical properties up to 650°C. The standard heat treatment of the alloy consists in a solutioning treatment between 950 and 1000°C, followed by a double aging at 720°C (furnace cooling) and 620°C.

At elevated temperature, the γ'' phase (Ni_3Nb) is less stable than γ' (Ni_3Al,Ti) (1,2) and a dramatic deterioration of crack propagation resistance occurs above 650°C (3, 4).
However, in some areas of critical turboengine parts, such as slots and rims in turbine disks, temperatures higher than 650°C are more and more frequently encountered and classically processed INCO 718 is unsuitable for such applications.
Several attempts have been carried out to improve the damage tolerance capability of INCO 718 above 650°C.

- grain morphology modification (coarsening, necklace structure, serrated grain boundaries...(5,6)), but minor improvements of crack growth resistance are obtained, in comparison with important decrease of tensile and LCF (7) properties,
- composition variations and heat treatment modifications, to change the γ'/γ'' precipitates ratios and sizes (8, 9),
- changes of precipitates morphologies without modification of chemical composition, through specific aging treatments (8,10).

The third method has been chosen by SNECMA and an adequate heat treatment has been searched for on standard INCO 718 (ie solutioned + double aged) hereafter denoted as <u>ST 718</u>, and DA INCO 718 (Direct Aged) in order to try to improve the damage tolerance capability at elevated temperature (650 - 750°C).

Experimental Procedure

Two rough forgings have been used in the present study:

- a stamped LP turbine disk of ST 718, heat treated as follows :
    - solutioning 955°C - 1 h - air cooling
    - aging 720°C - 8 h -> 620°C - 8 h - air cooling

The basic microstructure consists in homogeneous equiaxed grains, (ASTM 6-8) and intergranular δ phase precipitates (fig. 1a).

- a hot die forged pancake (ϕ 580 mm) of DA 718 (720°C - 8 h -> 620°C - 8 h - air cooling).

A very fine grained microstructure is obtained (ASTM 13-14) and numerous δ phase platelets are observed in the grain boundaries (fig. 1 b).
On these two forgings, lab tests specimens have been machined for tensile, creep, low cycle fatigue (smooth cylindrical specimens) and crack propagation tests (CT specimens).

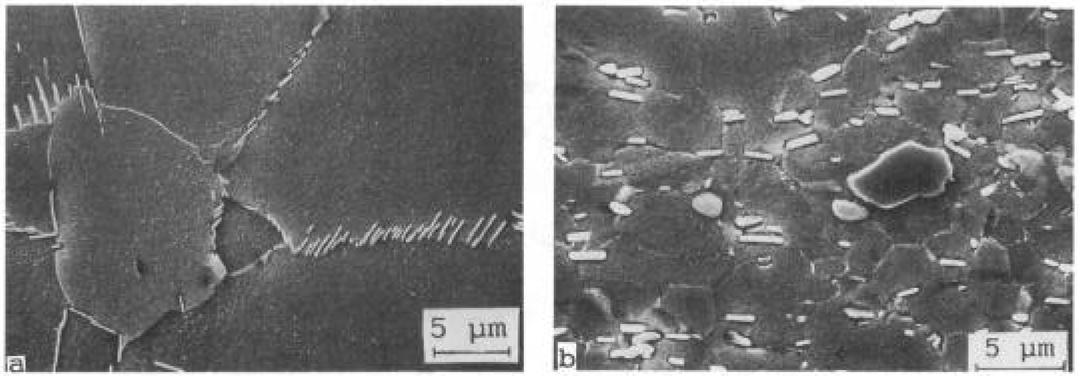
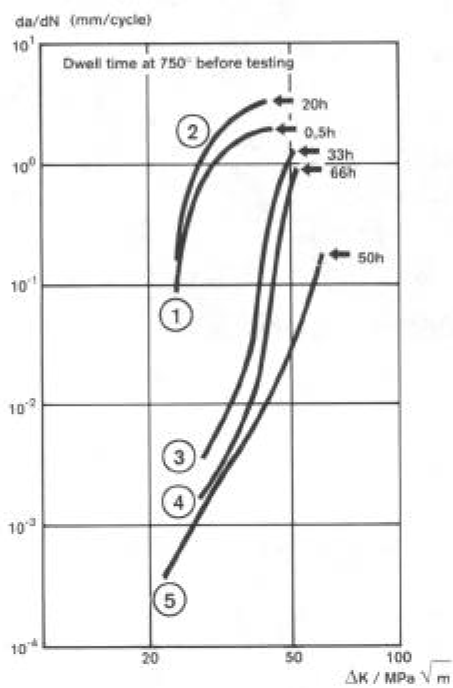


Figure 1 - Basic microstructures in ST 718(a) and DA718 (b)

<u>Determination of adequate post-heat treatment on ST 718</u>

Preliminary crack propagation tests have been performed on basic ST 718, at 750°C with trapezoïdal wave 10-90-10 (R=0,05) in order to assess the alloy reference crack resistance at high temperature.
At low ΔK values (20 to 35 MPa$\sqrt{m}$), two sets of curves da/dN (ΔK) are recorded as shown in fig. 2 : in some cases, the crack propagates very rapidly (curves 1 and 2) and the total duration of the tests is short (less than 20 h). In other cases (curves 3 and 4), the crack is blocked for several hours before propagating at a much lower crack growth rate than the first specimens (100 times slower). It is then assumed that a



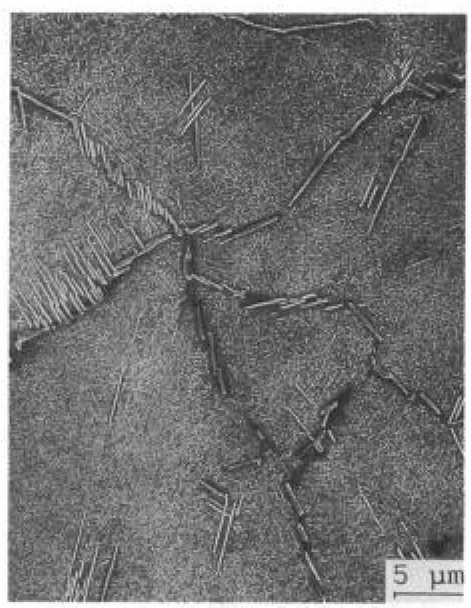


Figure 2 - Crack propagation curves for various post-aging treatments (90s dwell time)

Figure 3 - DT 718 microstructure

microstructural change has occurred in the alloy before that the crack effectively progresses, during the "imposed" dwell time at 750°C (longer than 33 h). From those results, a 750°C - 50 h post-aging treatment has been applied on ST 718 to check the above assumptions.
The 750°C crack growth curve (n° 5 on fig. 2) shows that the chosen heat treatment greatly improves the damage tolerance capability at high temperature of ST 718, which will be referred subsequently as INCO 718 DT or DT 718 (11).

Characterization of DT 718

Microstructural investigations

As expected, the overaging heat treatment applied to ST 718 leads to microstructural alterations (9, 10, 12, 13, 14).
The grain sizes and δ phase amount are unchanged as compared to reference ST 718 (fig. 3). The grain aspect is however more granulary, which indicates early stages of precipitation (14). TEM examinations evidence coalescence of γ' and γ'' phases : fig. 4a shows the microstructure of reference ST 718 with hardly detectable circular γ' ($\phi \sim 10$ nm) and lenticular shaped γ'' (L $\sim$ 10 to 15 nm). On DT 718, 50 nm dia. γ' spheres are observed (fig. 4b) as γ'' platelets the length of which is comprised between 100 and 300 nm.
These observations are in good agreement with the precipitation TTT diagram (13) in which the γ' precipitation is observed for 20 h dwell time at 750°C.

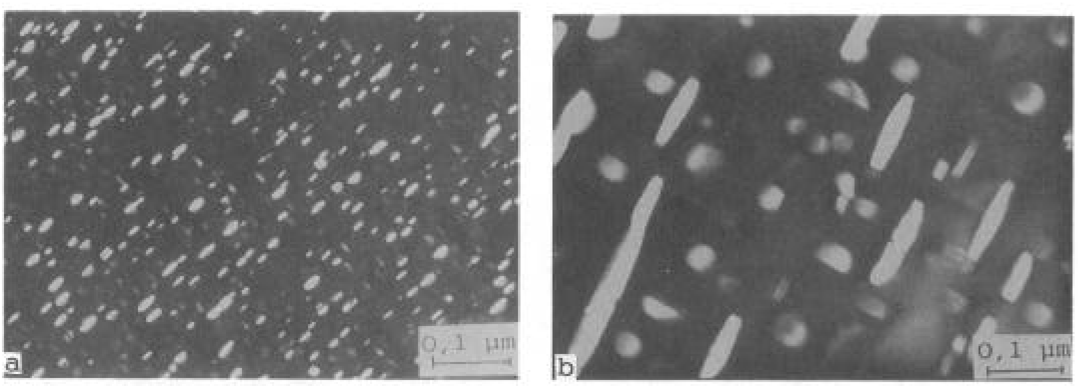


Figure 4 - γ'-γ'' precipitates in ST 718 (a) and DT 718 (b)

Mechanical properties

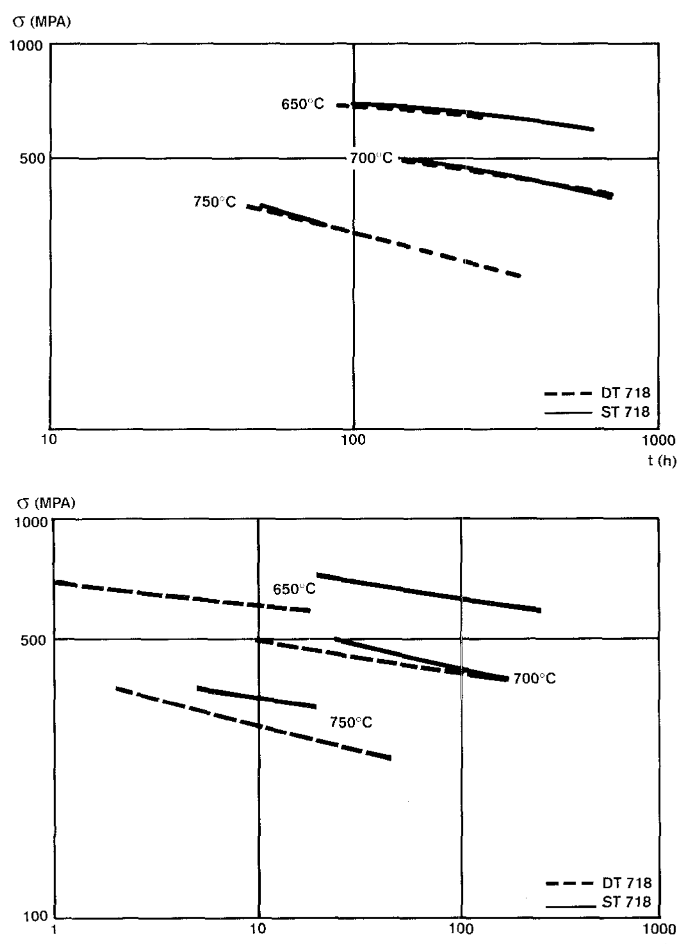
Tensile resistance : the tensile properties (ultimate and yield strength) of DT 718 have been evaluated between 550 and 750°C and compared to ST 718 values (table I).
It must be noticed that the yield stress of DT 718 is significantly lower than that of ST 718 (about 20 % at 550°C) while the ultimate tensile stress is nearly unchanged at that temperature. At 650°C and above, the tensile resistance of DT 718 is roughly inferior (about 10 %) to that of ST 718. Conversely, the ductility is dramatically increased.

TABLE I. Tensile properties of ST and DT 718

Temp. °C / Grade	550			650			700			750		
	Y.S. MPa	U.T.S. MPa	E %	Y.S. MPa	U.T.S. MPa	E %	Y.S. MPa	U.T.S. MPa	E %	Y.S. MPa	U.T.S. MPa	E %
ST 718	1040	1200	18	960	1100	18	880	950	17	750	790	18
DT 718	870	1170	20	850	1000	29	810	850	27	690	700	29
$\frac{DT\ 718}{ST\ 718}$ %	- 17	- 3	+ 11	- 11	- 9	+ 61	- 8	- 10	+ 59	- 8	- 11	+ 61

On a qualitative point of view, these results are consistent with those obtained elsewhere on overaged INCO 718 up to 760°C (10, 15).

Creep : rupture creep tests have been performed at 650, 700 and 750°C. Times to 0,2 % elongation have been recorded. The diagrams σ/t (fig. 5a and b) reveal similar lives to creep rupture for both ST 718 and DT 718, but also a quite significant decrease of time for 0,2 % creep elongation in DT 718 at 650°C : factor 30 (in time). At 700°C and 750°C, this effect is much more moderate, with a time reduction factor less than 3.



a) rupture lives

b) 0,2 % elongation times

Figure 5 - Creep curves

Low cycle fatigue : stress and strain controlled LCF tests have been achieved at 650°C on DT 718, at low frequency (0,5 Hz) and under R = 0 load ratio.
The LCF resistance of DT 718, for strain controlled loading, is slightly higher than that of reference ST 718 (fig. 6).The cyclic stress-strain curves (fig. 7) have been drawn for both 718 grades, and compared to monotonic tensile curves. It appears that cyclic softening is noticeably less important on DT 718 than on ST 718. Moreover, while ST 718 displays an extensive cyclic softening, the DT 718 cyclic behavior remains rather close to the monotonic one.
As stress controlled tests are concerned, DT 718 reveals a lower resistance than ST 718 (fig. 8), the gap in stress amplitude being about 5 %. This result can be, in first consideration, related to the tensile strength values, which are about 10 % lower for DT 718 as compared to ST 718.

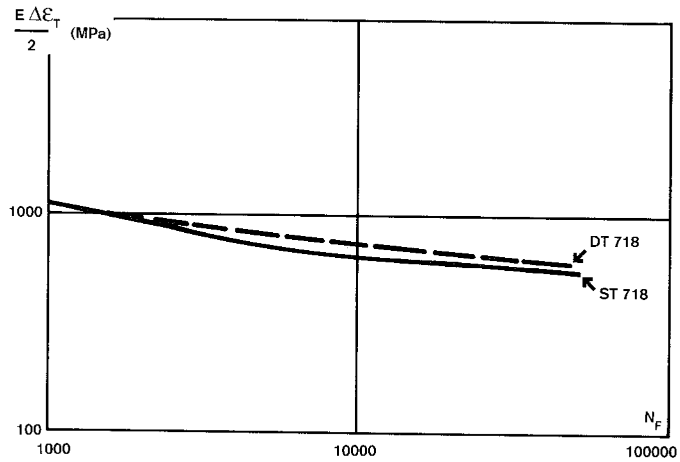


Figure 6 - Low cycle fatigue at 650°C ($R\epsilon$ = 0 - ν = 0,5 Hz)

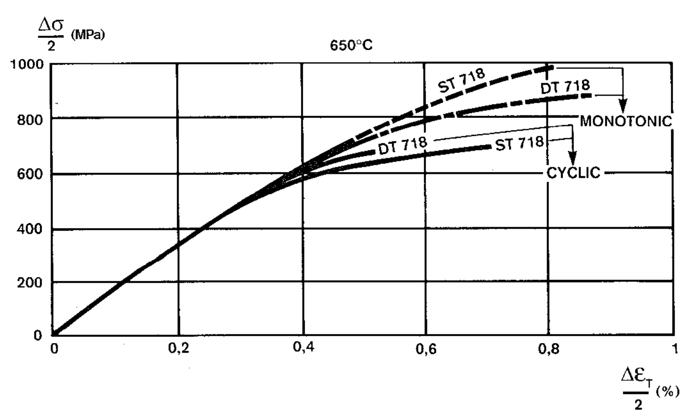


Figure 7 - Cyclic and monotonic stress-strain curves at 650°C

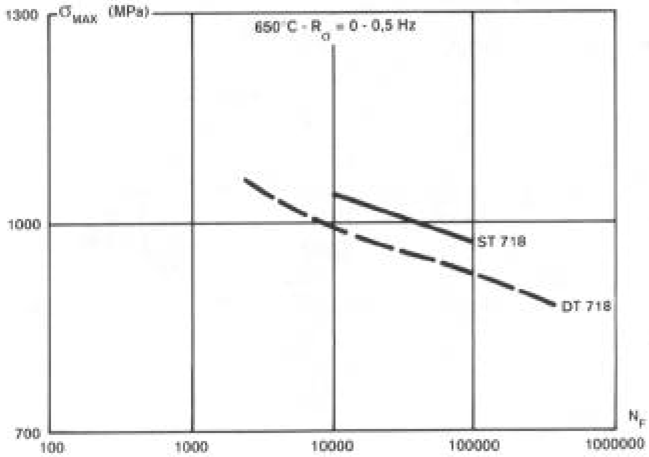


Figure 8 - Low cycle fatigue at 650°C (Rσ = 0 -ν = 0,5 Hz)

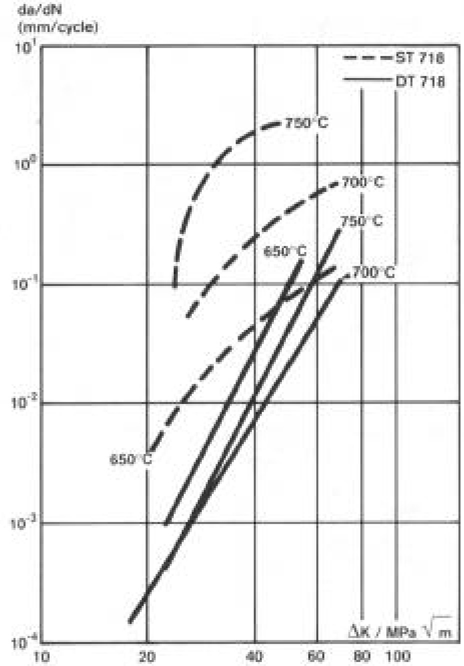


Figure 9 - Crack propagation curves (cycle 10-90-10 -R=0,05)

<u>Fatigue crack propagation</u> : CT specimens have been tested at 650°C and 700°C in the same mechanical loading conditions as at 750°C (trapezoïdal wave 10-90-10s - ratio R = 0,05).
At 650°C, the crack growth rate is twice lower for DT 718 than for ST 718 up to ΔK = 40 MPa√m. This difference between the two INCO 718 grades is highly emphasized at 700°C : the crack growth rate is fifty times lower for DT 718. A synthetic chart (fig. 9) summarizes the crack propagation properties of DT 718 compared to ST 718. The lowest crack growth rates are recorded at 700°C, and in all cases, the benefit of DT 718 over ST 718 increases with the temperature : at ΔK = 30 MPa√m, factor 4 (in rate) at 650°C, 50 at 700°C and 400 at 750°C.
Such improvement have already been noticed at 650°C through other specific treatments (5, 6, 7) but the dramatic decrease of crack growth rates at higher temperature have not been yet observed.

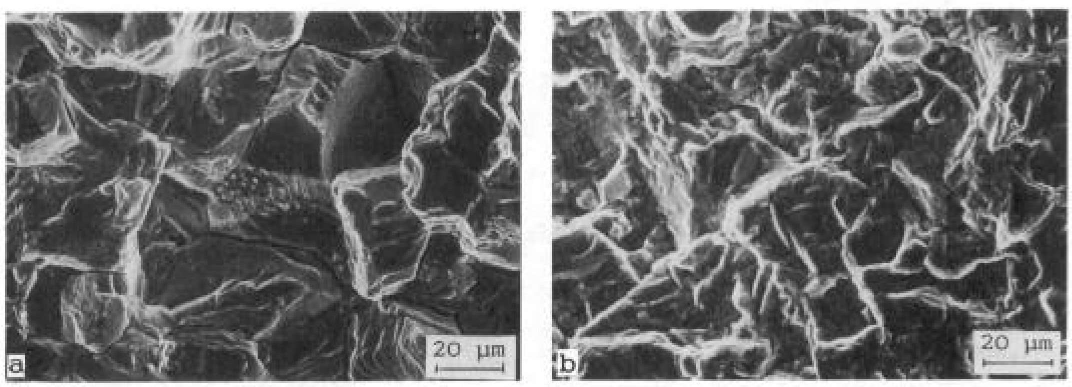


Figure 10 - Rupture surfaces on specimens tested at 700°C
a) ST 718 b) DT 718

Fractographic investigations on failed specimens have revealed a pure intergranular propagation mechanism on ST 718 (fig. 10a) while a more ductile aspect, with predominating transgranular mode is evidenced on DT 718 (fig. 10b).

Discussion

In order to precise the advantage of DT 718 as damage tolerance is considered for part designing, the crack propagation curve at 700°C has been plotted in comparison with PM alloys Astroloy and N18, whose microstructures are reinforced by several populations of γ'precipitates, and which are known to offer a good crack propagation resistance at high temperature (16, 17) : For $\Delta K \approx 30$ MPa$\sqrt{m}$, DT 718 crack growth rates are quite comparable to N18 ones (fig. 11) and twice lower than that of Astroloy. In the same loading conditions, a factor of about 50 for da/dN increase is noticed on ST 718.

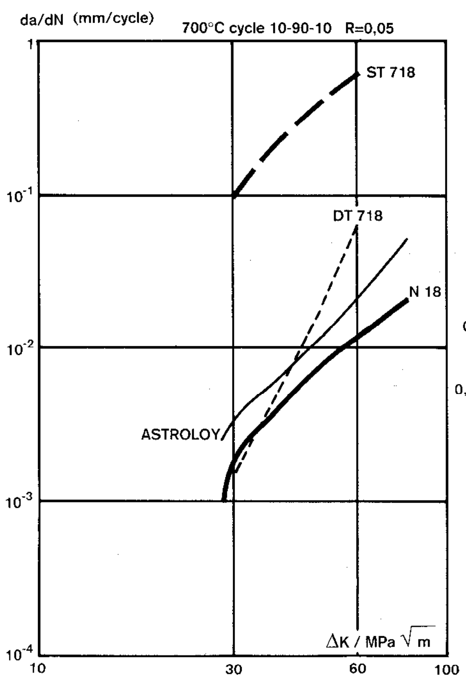


Figure 11 - Crack propagation curves on discs superalloys

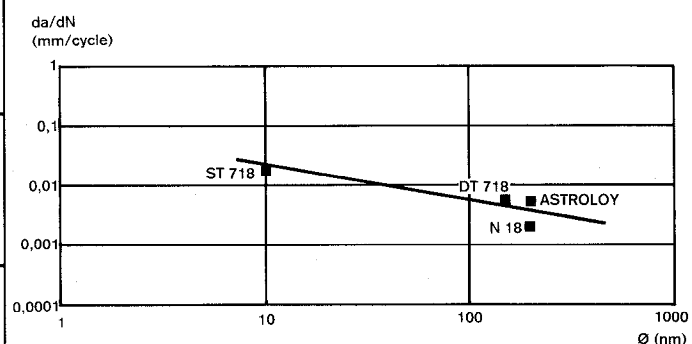


Figure 12 - Crack propagation rate versus precipitates sizes at 650°C (cycle 10-90-10 - ΔK = 30 MPa$\sqrt{m}$)

A microstructural parameter has been sought to correlate those observations. A relationship can be established, in a first approach between the crack propagation rate da/dN and the strengthening γ'-γ''precipitates size (fig. 12) : the crack growth resistance is increased with the precipitate size, but other parameters (grain boundaries morphology, intergranular carbides, δ phase...) may play a role too in cracking processes. However, it has been shown that in nickel base superalloys, a strong coupling exists between deformation mechanisms and precipitates sizes (18,19) : small precipitates are mostly sheared, which is associated with heterogeneous planar deformation, while large ones are by-passed, which means an homogeneous deformation. In fact, planar reversible slip would be favourable to low crack growth rate, and that is quite opposite to the best resistance of DT 718 (large precipitates -> planar slip). However, another mechanism is worth to be considered at grain boundaries level (20). In the case of homogeneous glide process (DT 718), the slip lines are coincident across the grain boundaries and the resulting homogeneous form of deformation reduces the localized stress concentration. Therefore, intergranular cracking is expected to be unlikely and that is in line with observations made on DT 718 failed specimens.

Moreover, on INCO 718, a huge environmental effect has been observed (2, 10, 21) and may contribute to explain the better crack growth resistance of DT 718 at high temperature as compared to ST 718 . On one side, the intergranular brittle fracture (<-> high crack growth rate) results from a combination of an heterogeneous deformation and an oxidation process, involving Ni and Fe oxides (ST 718). On the other side, an homogeneous slip deformation reflects an increase of the grain boundary activation energy for diffusion (DT 718), thus limiting oxygen penetration along the affected grain boundary (22). So, very rapidly, a selective oxidation process of chromium occurs (23) and leads to a protective film. Consequently, the oxygen attack at the crack tip which contributes to high cracking rates, is prevented.

If oxidation assisted phenomena, more than purely mechanical modes transitions, can explain the excellent resistance of DT 718 as crack propagation is concerned, the good LCF strength is assumed to be due to the mechanical effects associated with coarsening of precipitates. Large γ'-γ'' precipitates have been observed to be responsible of cyclic hardening (24), as observed on DT 718 : the coarse precipitates retain more coherence through interaction with dislocations and cannot be sheared easily by cyclic straining. As a result, cyclic softening is not as noticeable in DT 718 as in ST 718 : cyclic softening is considered to be deleterious because the load bearing capacity of the material is reduced with cyclic strain accumulation.

As <u>static properties</u> (creep, tension) are concerned, it has been evidenced (3, 12, 18) that an increase of γ'-γ'' precipitates sizes is cause of decrease of creep and tensile resistance : due to precipitates coarsening and spacing between particles increase, the mechanism of dislocation motion is altered from a mode involving shearing of small precipitates particles to that of by-passing large precipitates by the Orowan process. Consequently, a decrease of tensile strength and an increase of primary creep are observed. An investigation of particle size on steady state creep rate ($\dot{\varepsilon}_s$) has shown (25) that for γ'' size exceeding 30 nm, $\dot{\varepsilon}_s$ dramatically increases.

The above considerations corroborate the observations of weaker resistance of DT 718 under tensile (up to 750°C) and creep loadings (at 650°C) as compared to ST 718. At higher temperature, the effect is quite less important for creep at 700 and 750°C, due to assumed environmental effect and microstructural alterations of ST 718 (coarsening of precipitates).

Attempt of DA 718 overaging

For critical parts applications in turboengines, various specific thermomechanical treatments have been developed in order to get high tensile and LCF resistance up to 600°C (26) : highly stressed at moderate temperature DA 718 is presently used for disks manufacturing in jet engines. In order to keep high yield strength while improving damage tolerance capability at elevated temperatures, several post-aging heat treatments have been applied on DA 718, as on ST 718.

Influence of post-aging treatment on resistance of DA 718

The same overaging heat treatment as for DT 718, i.e 750°C - 50 h, applied on DA 718, leads to a very important lowering of static properties at 650°C : - 20 % in yield and ultimate tensile stress, life to creep rupture divided by about 10 (table II).

TABLE II. Effects of post-aging treatments on tensile and creep resistance of DA 718

	Tensile resistance (650°C)			Creep 650°C - 650 MPa		
	Y.S. MPA	U.T.S. MPa	E %	$t_{0.2}$ % h	t_R h	E %
DA 718	1120	1190	35	37	176	23
DA 718 + 700°C - 100 h	1040	1090	29	20	123	21
DA 718 + 750°C - 5 h	980	1100	35	27	115	21
DA 718 + 750°C - 15 h	950	1010	37	14	96	21
DA 718 + 750°C- 50 h	870	950	35	< 1	16	33

Modifications of the post-aging conditions have been applied : the chosen heat treatment (750°C - 5 h) allows limited decrease of tensile and creep properties as compare to reference DA 718.

Characterization of modified DA 718

Microstructural investigations

The grain sizes and δ precipitates are quite unaltered by the overaging treatment. TEM investigations revealed lenticular γ'' (length about 10 to 20 nm) and circular γ' precipitates (diameter $\sim$ 10 nm) in DA 718 (fig. 13a). After aging 5 h - 700°C, a slight coalescence is observed : 30 nm γ'' - 10 to 20 nm dia γ' (fig. 13b), much less important than in DT 718.

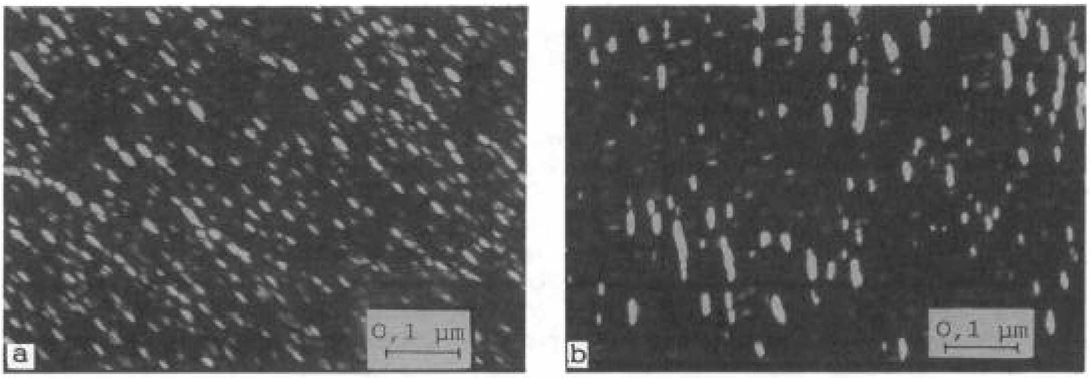


Figure 13 - $\gamma' - \gamma''$ precipitates in DA 718 (a) and DA 718 + overaging (b)

Mechanical properties

Tensile resistance : Tests between room temperature and 750°C show that the tensile properties of DA 718 are noticeably lowered by post-aging treatment. In particular, the yield strength of modified ST 718 is close to that of ST 718 and the benefit of D.A. treatment has totally disappeared (fig. 14).

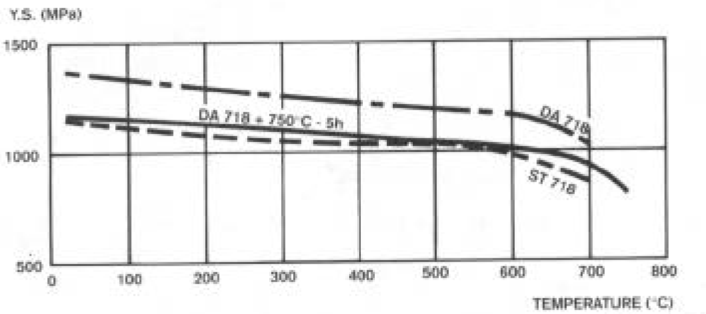


Figure 14 - Yield stress versus temperature for DA 718 (basic and overaged) and ST 718

Creep properties : Decreases of creep rupture stress (- 5 %) and especially of time to 0,2 % elongation which is divided by a factor 5 at 700°C (fig. 15) are recorded. ST 718 and basic DA 718 exhibit quite higher rupture strength, mainly at 700°C where the creep properties of modified DA 718 are too low to allow any relevant application of this grade for highly loaded parts at elevated temperatures.

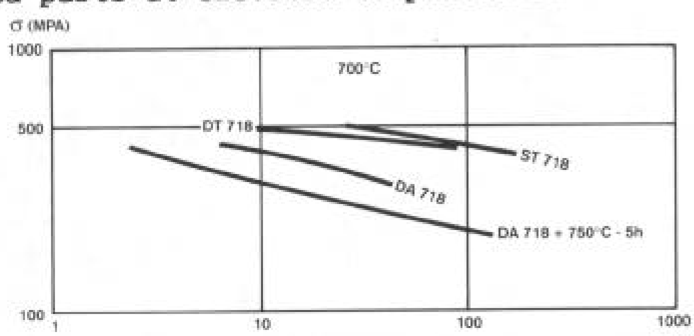


Figure 15 - 0,2 % elongation creep curves on ST, DT and DA 718 (basic and overaged)

Conclusion

An extension of INCO 718 alloy application for high temperature, up to 750°C, is quite possible through a slight modification of heat treatment. Overaging for 50 h at 750°C standard INCO 718 leads to an excellent crack propagation resistance in dwell fatigue over 650°C : for that reason, the modified standard INCO 718 was named DT 718. The tensile properties at lower temperatures (550 - 650°C) as the creep resistance at 650°C are decreased of about 20 %, which may remain acceptable for specific applications on turboengine parts. LCF properties are slightly altered by overaging in the 550 - 650°C temperature range. INCO 718 could so offer quite relevant applications in turboengine, for moderately loaded parts encountering high temperatures, up to 750°C : turbine and rear casings, various static parts for which creep is the main damaging mode (nozzles, cowls...) could attractively be made of DT 718, which would offer a dramatically better crack propagation resistance.
A similar approach has been carried out to develop a damage tolerant material from high strength DA 718, but it revealed unsuccessful, due to an unacceptable decrease of creep resistance. Opposite to DT 718 in which a double precipitation/coalescence of $\gamma' - \gamma''$ phases accounts for good resistance at high temperatures, the metallurgical modifications in post-aged DA 718 which could be responsible of alterations of mechanical properties are not totally explicited.

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