High temperature creep-fatigue crack initiation in 718-DA Ni based superalloy

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ABSTRACT: *Low cycle fatigue tests were performed on IN 718-DA alloy at 600°C, both in air and under vacuum environment. The effect of an hold time (90s) at maximum applied total strain was investigated. All the specimens were taken from forged discs. These test results were compared to those obtained on conventional 718 (annealed and aged) tested under vacuum. SEM observations of the fracture surfaces and of the gauge length of the specimens showed various types of initiation sites. Both in air and under vacuum, the fatigue cracks initiated from surface connected particles (carbides and nitrides) or from subsurface particles. The deleterious effect of oxidizing environment on the fatigue life is explained by a preferential oxidation of carbide particles. A mechanism accounting for cleavage of nitride particles due to preferential carbide oxidation is proposed.*

INTRODUCTION

For its high strength, good oxidation, fatigue and creep resistance at high temperature Inconel 718 is the most widely used superalloy for aerospace applications [1]. This material can be used under annealed and aged conditions (TR). More recently, significant developments have been made to reduce the grain size of this material. This was mainly achieved by using another thermomechanical heat treatment, referred to as Direct Aging (DA).
The low cycle fatigue (LCF) proprieties of this material have been extensively investigated [2-4]. Recently, it was shown that in this material the reduction in grain size may lead to preferential initiation of LCF cracks at second phase particles, such as niobium carbides, or titanium nitrides, which produces a significant decrease in LCF life [5].
The aims of the present study are to investigate the effect of environment on preferential initiation of fatigue cracks from second phase particles.

Moreover a limited number of tests were performed to compare TR and DA materials.

MATERIAL AND EXPERIMENTAL PROCEDURES

The material is a fine grained austenitic γ matrix which is strengthened by the precipitation of γ' (Ni_3(Ti-Al)), and γ'' (Ni_3(Nb-Ti)) particles. A third type of precipitate is also present in the alloy which is the Ni_3Nb δ phase. The δ phase particles are found both at grain boundaries and inside the grains. The last family of second phase particles present in the alloy are nitrides (TiN) and carbides (NbC) with a size from 5 to 20 µm. These particles are preferentially located at grain boundaries. The nominal composition of the alloy used in this study was : 19%Cr, 18 % Fe, 5 % Nb, 3 % Mo, 1 %Ti, 0.5 % Al, 0.05 % Ta, 0.05 % C (weight %).
Most tests were performed on specimens extracted from a forged disc which was directly aged after forging. This material, referred to as DA_2, had a small grain size of 5-10 µm. The specimens were cut along the tangential direction of the disc. Another DA material, named DA_1 was also investigated. A limited number of tests under vacuum were performed on a conventional (TR) material with a grain size of about 20 µm.
LCF tests were carried out in air at 600°C ± 2.5°C with a quartz lamp radiation furnace using a MTS 458-20 10T servo-hydraulic machine. The specimen temperature was controlled by two K-type thermocouples welded on each head of the specimens (out of the gauge length). The LCF specimens had a diameter of 8mm and a gauge length of 12.5mm. They were polished using a 1µm diamond finish. The specimens were tested under 10-10 or 1Hz triangular cycles and trapezoidal 10-90-10 cycles under strain control condition with $R\varepsilon=\varepsilon_{min}/\varepsilon_{max}=0$ and a hold time of 90s applied at maximum applied total strain. The vacuum tests were also performed at 600°C using a secondary vacuum chamber (1×10^{-4} Pa).
The tests were interrupted when the maximum load had decreased by 20%. SEM observations of the gauge length were performed on the unbroken specimens. Then the specimens were fatigued at room temperature using 10 Hz cycle under load control. SEM examinations of the fracture surfaces were also made to identify the fatigue crack initiation sites.

RESULTS AND DISCUSSION

Mechanical tests

The conditions used to test the materials are given in Table 1. Most experiments were carried out at a total strain amplitude $\Delta\varepsilon_t$=1.1%. In table 1 the values of the test parameters determined at mid-life, including the plastic strain amplitude, $\Delta\varepsilon_p$, the maximum and minimum stress, σ_{max} and σ_{min}, are also given. The number of cycles to failure, N_f, is also reported.

TABLE 1: Fatigue test conditions and results

Material	Spec	Environment	Cycle	$\Delta\varepsilon_t$ %	$\Delta\varepsilon_p$ %	σ_{max} MPa	σ_{min} MPa	N_f cycles
TR	TR1	Vacuum	Trap	1.1	0.25	680	-760	10735
TR	TR2	Vacuum*	Trap	1.1	0.31	630	-660	8682
DA_1	DA3	Vacuum	Trap	1.1	0.24	687	-830	11101
DA_1	DA4	Air	Trap	1.1	0.26	727	-756	6551
DA_1	DA5	Air	Trap	1.1	0.24	777	-774	9395
DA_2	DA23	Air	Tri 1Hz	0.6	0.00	872	-220	1052800
DA_2	DA11	Air	Trap	1.1	0.14	915	-655	3691
DA_2	DA12	Air	Trap	1.1	0.23	770	-820	5126
DA_2	DA21	Air	Trap	1.1	0.23	800	-805	3529
DA_2	DA32	Vacuum	Trap	1.1	0.23	740	-790	9816
DA_2	DA22	Air	Tri 10-10	1.1	0.18	990	-680	2440
DA_2	DA42	Air	Tri 10-10	1.1	0.26	982	-760	1470
DA_2	DA43	Air	Trap	1.5	0.55	900	-980	690
DA_2	DA34	Air	Trap	1.5	0.59	870	-950	890

* TR2 preoxidised for 15h at 550°C in air and vacuum tested

These results show that DA_1 material exhibits slightly better fatigue properties compared to DA_2 alloy. It is worth noting that, for similar test conditions, DA_2 alloy exhidits a slightly higher strength than DA_1 material (compare test DA21 with DA4). This might explain the difference in fatigue life in these materials. It is also observed that, in DA_2 material, the LCF life with a hold time is larger than that determined under triangular wave cycle. This might also be related to the difference in maximum stress corresponding to these two types of cycling. A similar behaviour in crack propagation rate was reported by Lynch & al [6]. In a disc material, it has been shown that the effect of hold time on the fatigue crack growth rate was strongly dependent on a number of metallurgical factors, in particular the

specimen orientation. This was related to the orientation of the fatigue crack with respect to the alignment of δ phase particles [7].
In both DA materials, it is observed that the fatigue lives determined under vacuum are significantly larger than those measured under air environment, by a factor of about 2. Further tests performed on TR material show that a preoxidation of the specimen before testing under vacuum tends to reduce the fatigue life (compare TR1 and TR2 tests). This clearly underlines the environmental effect on fatigue life and in particular, on fatigue crack initiation.
The results obtained on DA_2 material tested under trapezoidal hold time cycle are reported in Figure 1 where it is observed that, the slope in the Manson-Coffin plot is equal to about –0.65. The large scatter observed in test results was attributed to the position of the specimens in the disc [8].

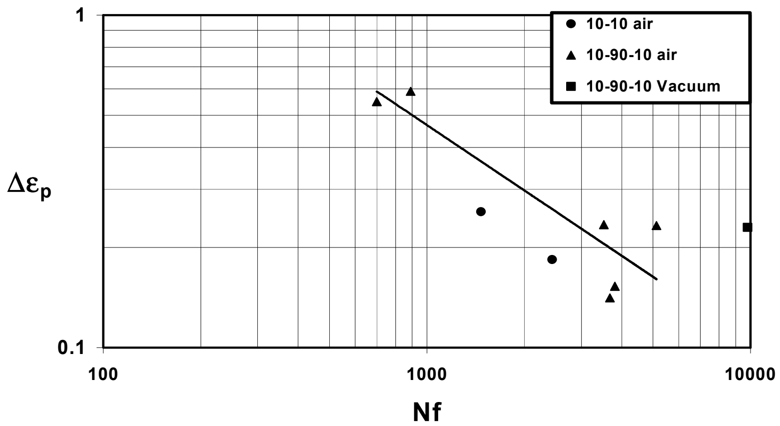

Figure 1: Fatigue life *vs.* Plastic strain range for DA_2 specimens.

Initiation sites

Fractographic observations showed different types of initiation sites. In all cases, these sites were related to the presence of a second phase particle with a size of 10-20µm. Figure 2a shows that, in air environment, the fatigue crack was initiated from a TiN particle. In DA23 specimen tested at lower strain, an internal initiation from a TiN particle was evidenced, as shown in figure 2b.In DA23 specimen tested under vacuum, surface initiation from a NbC particle was observed (figure 2c & d).
SEM observation on the gauge length of the specimen showed the presence of secondary fatigue cracks which were also systematically related to

second phase particles (Figure 3). In many cases these particles were niobium carbides which were strongly oxidised and transformed into Nb_2O_5 type oxide (see Figure 3a) when the material was tested in air environment. Under vacuum, both carbides and nitrides were observed as initiation sites (Figure 3b).

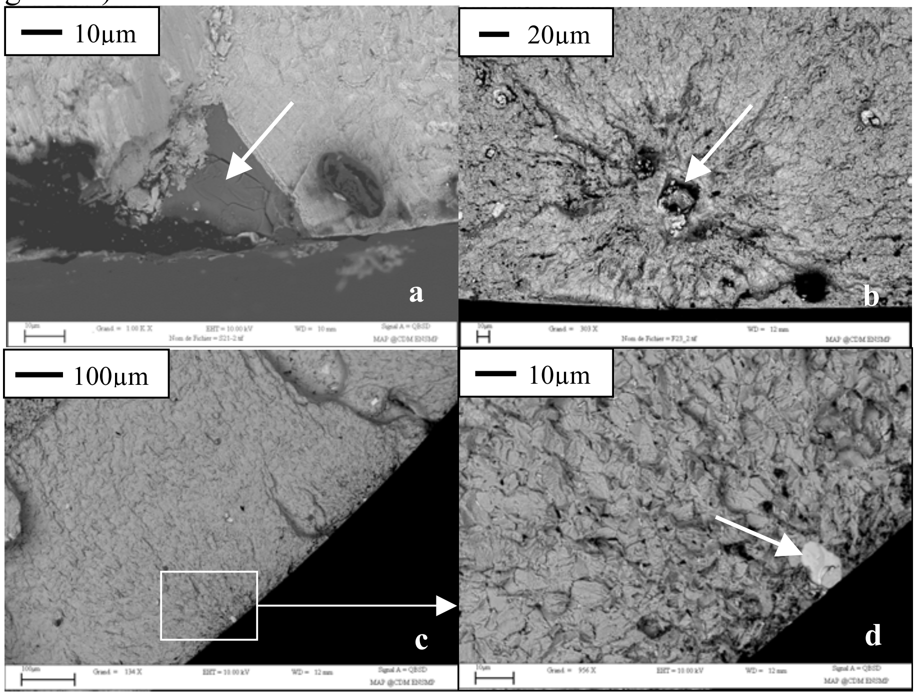

Figure 2: Fatigue crack initiation sites a) surface TiN, DA21 ; b) internal TiN, DA23 ; c) and d)surface NbC, DA32

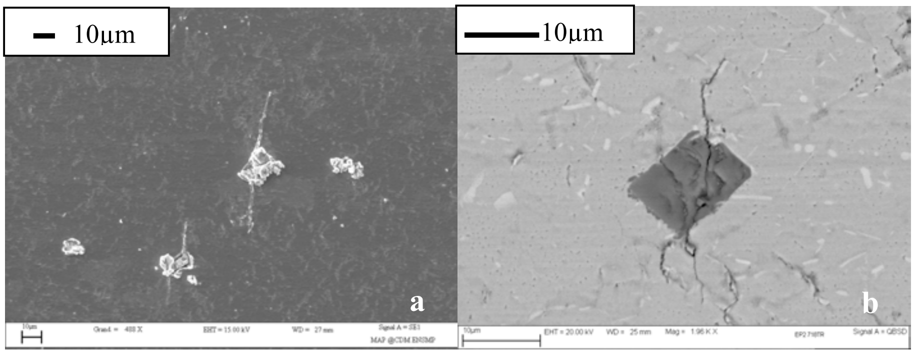

Figure 3: Secondary fatigue crack initiation sites observed on the specimen gauge length a) oxidised Nb carbides , DA34 ; b) TiN particle, TR1

Oxidation

The test results have shown a strong effect of environment on the fatigue life. This is the reason why, in addition to the tests performed under vacuum, preoxidation experiments were carried out. Polished surfaces were exposed to oxidation in air at 500, 600, 650°C for various times, between 1 and 120 hours. Figure 4 illustrates two types of oxidation mechanisms. Preferential surface oxidation of NbC particles is shown in figure 4,a, c & d. Figure 4a. corresponds to the formation of Nb_2O_5 type oxide occuring with a volume expansion ratio larger than 2. This explains the particular shape of oxidised Nb carbide observed in this figure. In many cases, Nb carbides are located around the Ti nitride particles. It was often observed that preferential oxidation of Nb carbides around Ti nitrides lead to the formation of cleavage like microcracks in TiN particles (see figure 4d.). The second oxidation mechanism is illustrated in figure 4b. This mechanism corresponds to internal oxidation of NbC particles located just beneath the free surface of the specimens. The volume expansion due to carbide oxidation leads to the formation of a protrusion at the free surface. Similar observations have been reported by others [2,3].

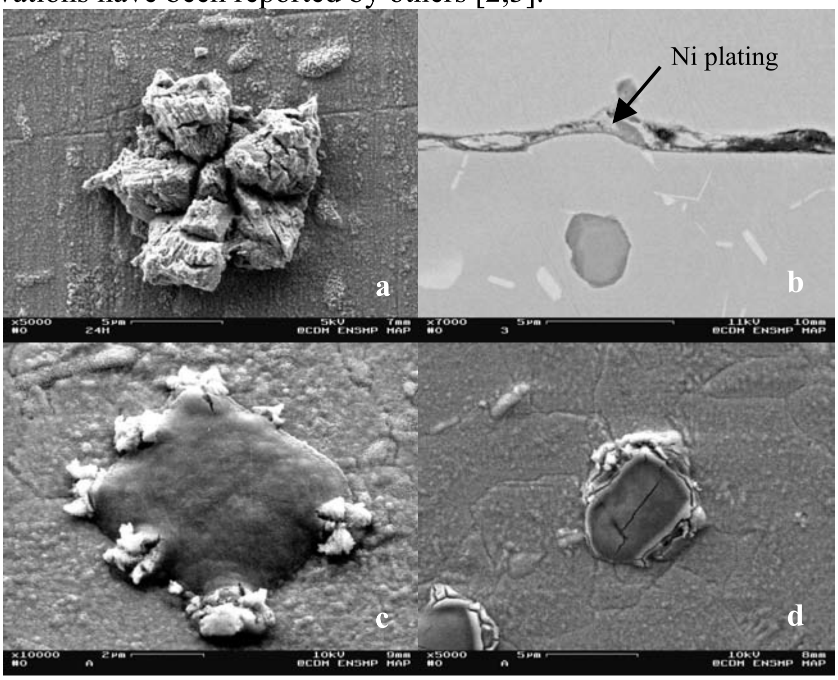

Figure 4: SEM examination of thermal exposure effects on particles a) on surface carbide ; b) on sub-surface carbide c)and d) on surface nitride

A mechanism for cleavage fracture of TiN particles due to preferential oxidation of Nb carbides is proposed in figure 5 where it is suggested that the volume change associated with NbC oxidation may induce tensile stresses of the outer free surface of the TiN particles. These stresses can initiate the formation of cleavage microcracks in these particles. Those microcracks are potent sources for the initiation of fatigue cracks and may contribute to the reduction in fatigue life observed in preoxidised specimens.

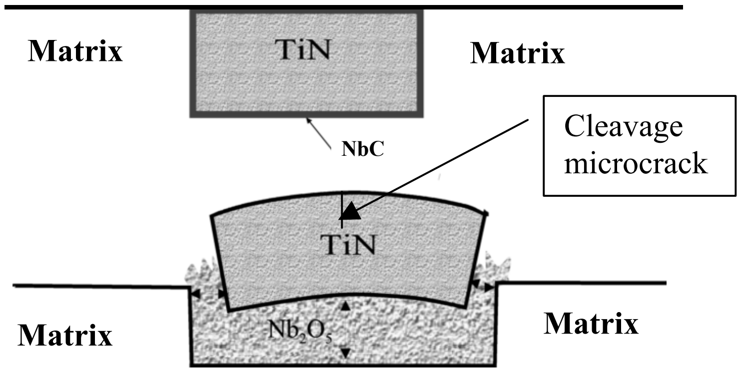

Figure 5: Schematic representation of thermal exposure effects on nitrides. Preferential carbide oxidation expansion may lead to bending stresses in nitride particles.

CONCLUSIONS

1- The application of a hold time at maximum strain in LCF tests produces a small increase in fatigue life when In 718 DA is tested at 600°C.
2- The fatigue life measured in air environment is lower than that determined under vacuum.
3- Preoxidation in air of a specimen subsequently tested under vacuum leads to a decrease in fatigue life.
4- These environmental effects are related to preferential oxidation of NbC particles which can occur externally either at the free surface of the specimen or internally just beneath the free surface.
5- Cleavage microcracks are nucleated within TiN particles located at the free surface when these particles are surrounded by Nb carbides. This phenomenom is related to the volume change accompanying NbC oxidation. These cleavage microcracks are potent sources for the initiation of fatigue cracks.

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