Microstructure-Crack Tip Interactions in P/M Superalloy Material at Elevated Temperatures

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ABSTRACT. *The influence of microstructure, in particular secondary gamma prime precipitates, γ'_S, on intergranular cracking of the P/M IN100 is investigated. A series of heat treatments using coupons made of as-received material is carried out in order to identify conditions leading to minimum and maximum variation in the size and volume fraction of secondary gamma prime, γ'_S. These specific heat treatments are applied to compact tension specimens to regenerate the corresponding γ'_S statistics. In addition to these microstructures, specimens subjected to extended aging time as well as long thermal exposure at 650°C have been generated and characterized. Fatigue crack growth experiments are performed on compact tension specimens having as received as well as modified microstructures at both 650°C and 700°C in air. The loading cycle includes a dwell time ranging from 100 seconds to 7200 seconds and superimposed at the maximum load level. Results of the as received material show that the length of the dwell time does not influence the crack growth rate. Results of the heat treated microstructures at 650˚C showed that a reduced volume fraction of γ'_S enhances the growth rate. The influence of the volume fraction of γ'_S on altering the intergranular crack growth rate is explained in terms of the crack tip stress relaxation.*

INTRODUCTION

Precipitate strengthened nickel based superalloys have an excellent resistance to creep and stress rupture up to 650°C. They are widely used in aircraft engines, nuclear structures, oil fields, gas production and power generation industries. The typical applications of these alloys include long hold times at elevated temperature, thus promoting intergranular fracture. It is generally assumed that this cracking growth mode is governed by the grain boundary cohesion strength, as well as matrix microstructure precipitates which influence the viscous flow and stress relaxation across the grain boundary crack path. Numerous investigations were carried out to determine these influences [1-7]; however, no general agreement has been achieved yet. For example, Jackson [1] concluded that in nickel based superalloys neither the size nor the volume fraction of the γ' precipitates influence intergranular cracking unless they are in the direct vicinity of the carbides along the grain boundaries. Zhao et al [2] found that the size, morphology, distribution, lattice mismatch and segregation of particles along the grain boundaries control the strength of the grain boundaries, thus, the intergranular cracking. Schirra et al [3] observed that in the P/M materials SR3 and KM4, intergranular crack growth rates decrease with increasing tertiary γ' size. On the other hand, Telesman et al [4] showed that increasing the size of the secondary not tertiary γ' lead to a decrease in the intergranular crack growth rate in Alloy 10 and ME3 alloy. Byrne et al. [5] attributed the observed reduced dwell crack growth rates at elevated temperature to alterations in the precipitates along grain boundaries during long term thermal exposure. The influence of changes in the precipitates in RR1000 on the intergranular crack growth rate has also been reported by Knowles and Hunt [6]. They observed the dissolution of the tertiary γ' due to high applied loading which resulted in crack tip stress relaxation and in turn reduced formation and growth of voids. Ma et al [7] in their work on IN783 did not observe a reduced intergranular crack growth rate due to the dissolution of particles but due to the increase of the γ'' size.

The objective of this paper is to examine the influence of variations of γ'_S statistics on intergranular crack growth behaviour in the powder metallurgy IN100 alloy. For this purpose, a series of heat treatment experiments are carried out to determine conditions providing minimum and maximum variation in the size and volume fraction of γ'_S. These specific heat treatments have been applied to compact tension specimens to regenerate the corresponding γ'_S profile. Dwell-time, fatigue crack growth experiments are carried out on compact tension specimens having as received, as well as modified microstructures, at 650°C and 700°C in air environment.

MICROSTRUCTURE CONTROL

The material used in this study is the powder metallurgy IN100 superalloy, the composition of which is 4.85Al, 4.24Ti, 18.23Co, 12.13Cr, 3.22Mo, 0.71V, 0.071 Zr, 0.02 B, 0.072 C Balance Ni. A typical microstructure detail of the material is shown in Fig. 1.

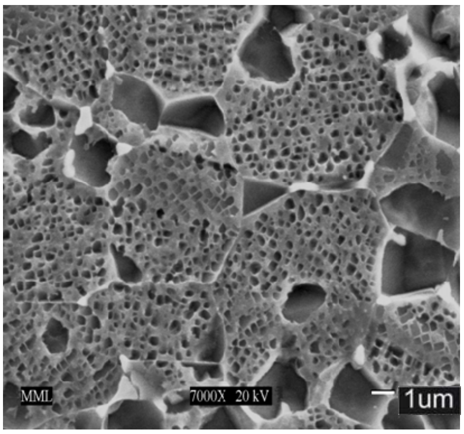

Figure 1. Typical microstructure of the as received material

The as received microstructure has seen a three stage heat treatment cycle. In order to maximize or minimize the volume fraction (*Vf*) or size (*d*) of the γ'_S, additional heat treatment cycles were then performed on this material. Details of these heat treatments and corresponding microstructure details are listed in Refs. [9,10]. In order to quantify the γ'_S structure, scanning electron microscopy (SEM) was performed at two different magnifications and images were analyzed using image processing software to determine the size and volume fractions of γ'_S. Since all heat treatments are carried out in sub-solvus solutioning, no changes were detected in the grain size measured as ~ 5μm. Similarly, the volume fraction of the primary gamma prime remained constant for all heat treated conditions. Significant changes in sizes and volume fractions of the γ'_S are observed. However, they do not occur in the same specimen i.e. specimens with the smallest and largest size γ'_S do not exhibit maximum or minimum variations in volume fraction. The five conditions examined here (*Vf*, *d*) are (as received: 28%, 207 nm), (19%, 130 nm), (26%, 258 nm), (15%, 208 nm) and (46%, 97 nm).

MICROSTRUCTURE- CRACK INTERACTIONS

Two sets of Dwell-fatigue crack growth experiments are carried out. The first set is performed on CT specimens all of which are made of the as received condition. The second set was carried out on specimens subjected to the heat treatment procedures described above. The loading cycle consists of 1s loading, 1s unloading and a dwell time of 0s, 100s, and 7200s superimposed at the maximum load level. All tests, detailed in Ref. [9], were performed at a stress ratio of 0.1 at 650°C in air environment. Results of the first set are plotted in term of da/dN versus ΔK and are shown in Fig. 2a. These results show that the crack growth rate increases with temperature and hold time duration. In order to unify these effects, the crack growth curves are plotted in terms of da/dt versus K_{max} as shown in Fig. 2b. This figure shows that the crack speed is temperature dependent, but independent of the length of the hold time duration. Attention is then placed on crack growth results of the second set of specimens having modified sizes and volume fractions of γ'_S. These set of crack growth experiments has been carried out at 650°C and results are shown as da/dN vs ΔK in Fig. 4a, and in terms of da/dt vs K_{max} in Fig. 4b. Fig. 4(a) shows effects of hold time and γ'_S statistics on the crack growth rate. Fig. 4(b), exhibit only effects of γ'_S statistics, since da/dt is independent of hold time durations. Typical grain boundary crack path generated by the hold time tests is shown in Fig. 5a showing secondary cracking which has been used by other authors as a quantitative measure of the crack tip driving force [8]. Fig. 5b illustrates the dominant nature of intergranular fracture observed at all dwell time tests.

ANALYSIS AND DISCUSSION

The influence of γ'_S statistics, in terms of volume fraction, *Vf*, and particle size, *d*, on intergranular crack growth rate has been examined at 650°C. In addition to as received microstructure, four conditions of heat treatments were carried out resulting in variations of *Vf* ranging from 15% to 46% and variations in the mean size *d* ranging from 97 nm to 258 nm. These are compared to those of the as-received condition which are measured as *Vf* = 26% and *d* = 207 nm. The rapid cooling rate (quench cooling) from subsolvus has generated the largest *Vf* (46%) and smallest *d* (97nm). While a heat treatment in which stabilization and aging cycles are reversed has generated the lowest *Vf* (15%). A correlation between volume fraction and size of γ'$_S$ is shown in Figure 6 indicating an increase in the size as the volume fraction decreases. Fig. 7a, shows that a decrease in *Vf* has a beneficial effect on the crack growth rate; a decrease from 46% to the 15% results in a decrease of da/dt by approximately an order of magnitude. Effects of the mean particle size on da/dt, see Fig. 7b, did not show a consistent pattern. *W*ithin the range examined (97nm to 258nm), da/dt decreases as *d* increases reaching a plateau at about 160 nm above which the crack growth rate increases as the size increases. It could be speculated that this plateau is a limit defining the shearing/looping mechanisms occurring during dislocation /particle interactions. In an attempt to explain these observations, the relationship between *d* and hardness, being a measure of the matrix yield, was examined. Results show that hardness decreases as particle size increases; a pattern similar to that obtained by Jackson and Reed [11]. It was, however, observed that that the crack growth rate is insensitive to the matrix hardness.

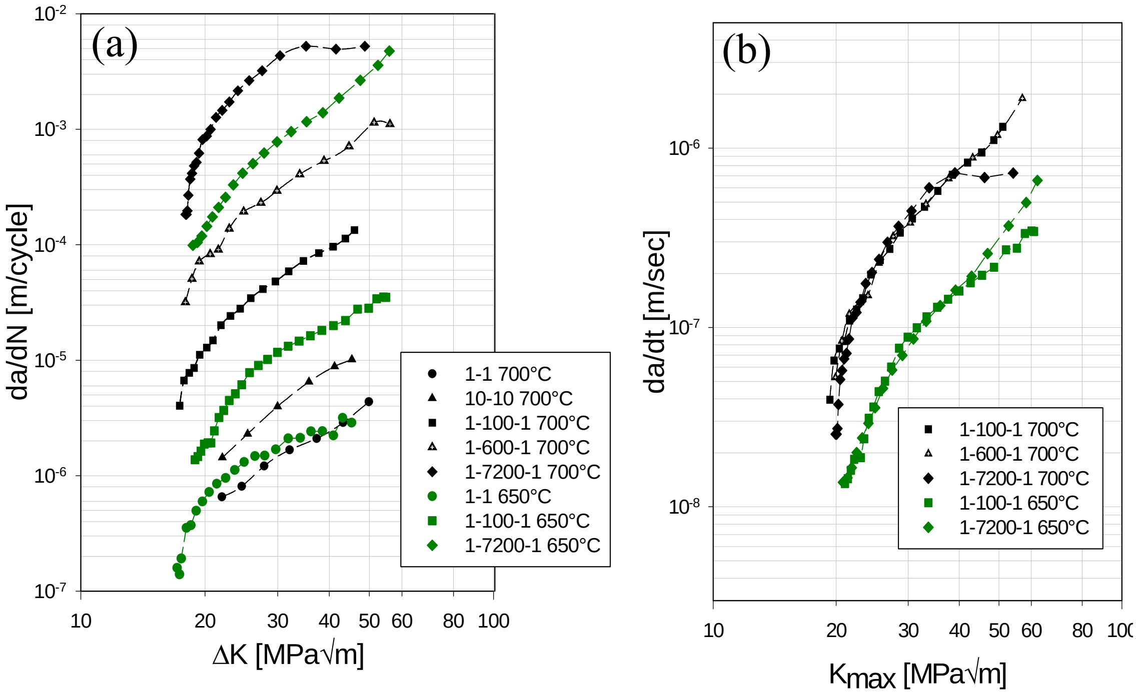

Figure 3: Crack growth rate as a function of temperature and hold time duration for as received material in terms of a) da/dN vs. versus ΔK and b) da/dt versus K_{max}.

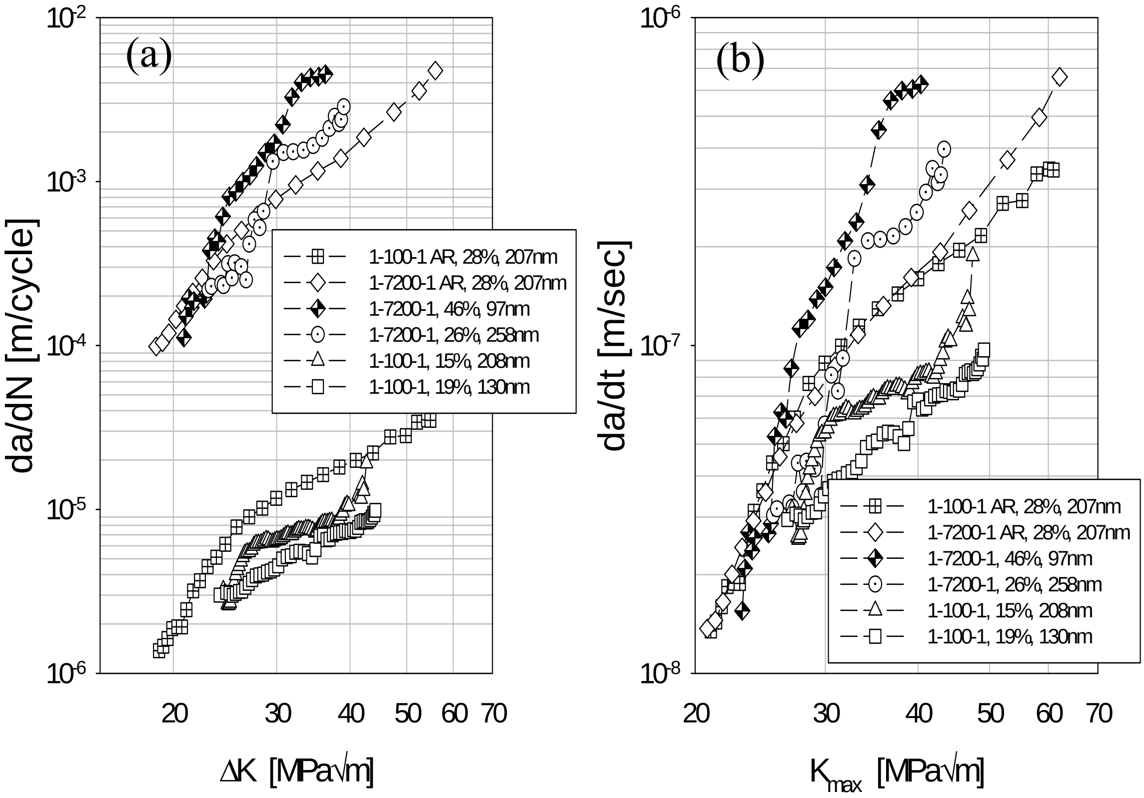

Figure 4: a) Crack growth rate as a function hold time for tests conducted at 650°C, and b) Effects of γ'_S statistics on da/dt for tests conducted at 650°C

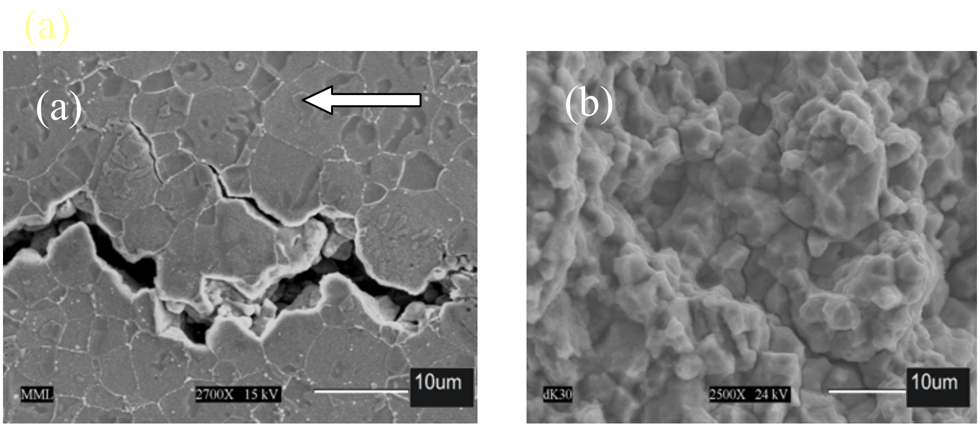

Figure 5: a) Typical grain boundary crack path showing secondary cracking (as received, 1s-7200s-1s, 650ºC), and b) typical intergranular fracture surface corresponding to dwell time tests (as received, 1s-7200s-1s, 650ºC)

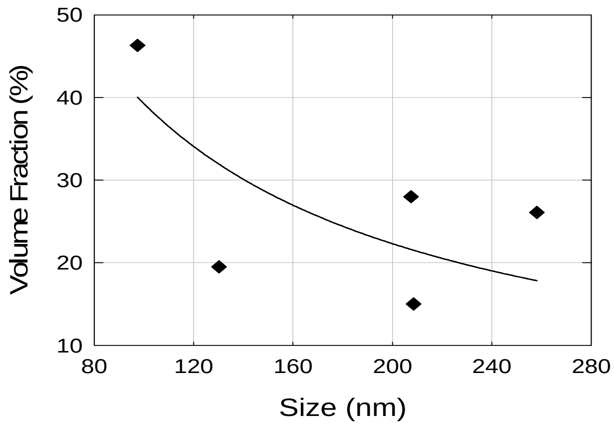

Figure 6: A correlation between the volume fraction and the size of γ'$_S$

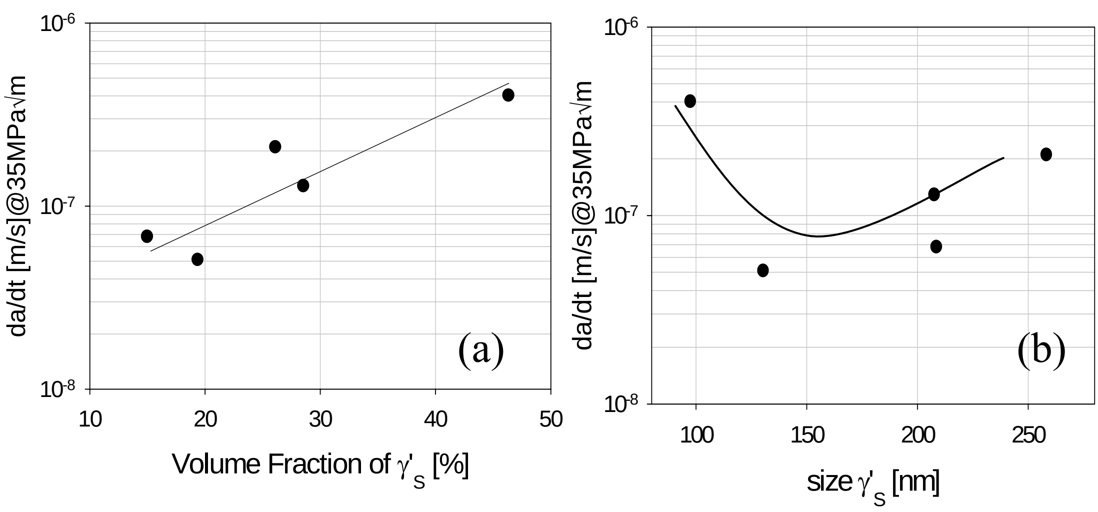

Figure 7: a) da/dt versus volume fraction of γ'$_S$, and b) da/dt versus size of γ'$_S$

As such, one could conclude that the secondary gamma particle size is possibly not the prime factor in determining the intergarnular crack growth resistance which may be more sensitive to localized effects brought up by variations in the particle volume fraction. As mentioned earlier, many studies have discussed the role of volume fraction on intergranular crack growth rate leading to contradictory conclusions. In a recent dwell crack growth experiments conducted by Telesman et al [8] on LSHR disk P/M superalloy, a conclusion was made that the γ'$_S$ statistics do not significantly influence the dwell crack growth at 704°C yet they observed a large influence of the tertiary gamma prime, γ'$_t$. In order to examine if the differences in their conclusions regarding the influence of the γ'$_S$ and γ'$_t$ may have resulted from difference in test temperatures between the current work (650°C) and that of reference [8], a da/dt vs K_{max} comparison is made between as-received condition and a microstructure that has been previously treated to produce a microstructure with Vf = 16.3% and d = 155.4 nm. This comparison shows that at 700°C, the role of microstructure is diminished indicating that the crack growth is possibly environment controlled. Attention is then focused on an explanation for the role of Vf of γ'_S on da/dt as has been observed here. One explanation is that a decrease in Vf of γ'_S triggers a modification in the internal stresses in the vicinity of the grain boundary fracture path that affect the crack tip driving force. A suggested mechanism by which this modification could be examined is by considering the effect of the reduction of Vf of γ'_S on the volume fraction of the tertiary gamma prime since both particles share the same elements, Al and Ti, in their formation. The work of Gabb [12] examined the volume fractions of the two particles in same specimen of heat treated superalloys. His results providing a correlation between the volume fractions of γ'_t and γ'_S when applied to the current study, a prediction of Vf of γ'_t versus γ'_S in the modified microstructures is plotted in Fig. 8. It is apparent from this figure that the decrease in Vf of γ'_S which increases the available Ti and Al in the solid solution, leads to an increase in the Vf of γ'_t. While speculations can be made regarding the effects of the heat treatment conditions on the dissolving of γ'_S and the subsequent formation of γ'_t, this topic is not discussed in this paper. One could however, assume that an increase in the volume fraction of the tertiary gamma prime results in higher dislocation/particle interactions. As a result of this increased interaction, the back stress component that constitutes the viscous flow in the matrix and particularly in regions adjacent to the crack path would be magnified. Using continuum formulations to describe the evolution of hardening characteristics in superalloy microstructures, the amplification of the back stress results in a decrease of the viscous strain which have a direct impact on the affected grain boundary decohesion strain resulting in a decrease of da/dt.

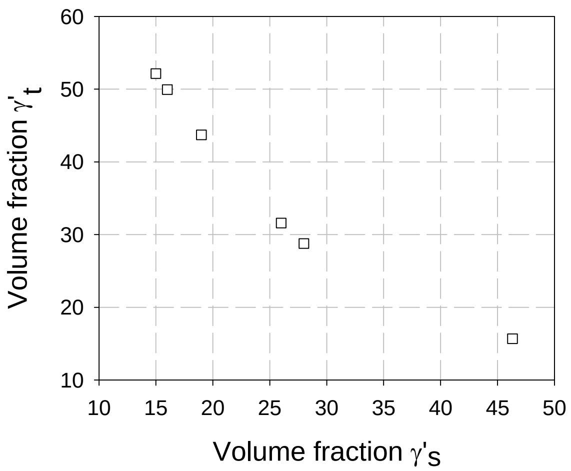

Figure 8: A correlation of volume fractions of γ'_t and γ'_S in the heat treated microstructures

CONCLUSION

A series of heat treatments is carried out on P/M IN100 alloy in order to identify conditions leading to variation in the size and volume fraction of γ'_S. These specific heat treatments are applied to compact tension specimens to regenerate the corresponding γ'_S statistics. Dwell-time fatigue crack growth experiments are performed on these specimens at both 650°C and 700°C in air. Results of the as-received material show that the crack growth rate at 700°C is faster than that at 650°C. At the latter temperature, da/dt is shown to be independent of the hold time duration thus indicating a minimum role for creep damage. Results of the heat treated microstructures show that at 700°C the intergranular crack growth is independent of the γ'_S statistics thus pointing out to the dominant role of environment. At 650°C the intergranular crack growth decreases with the decrease of γ'_S volume fraction. This influence is discussed in terms of the inverse correlation between the *Vf* of γ'_S and γ'_t ; a decrease in Vf of γ'_S leads to an increase in *Vf* of γ'_t which could in turn alter the internal viscous stress field governing the strain field across the grain boundaries in the crack tip zone. An increase in the viscous stress results in crack tip stress relaxation and consequently a decrease in the crack growth rate.

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