Investigation of heat treatment parameters effect on the microstructure and on the mechanical properties of a powder metallurgy nickel-base superalloy

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INVESTIGATION OF HEAT TREATMENT PARAMETERS EFFECT ON THE MICROSTRUCTURE AND ON THE MECHANICAL PROPERTIES OF A POWDER METALLURGY NICKEL-BASE SUPERALLOY

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Abstract

The relationships between heat treatment parameters and microstructure, and between microstructure and mechanical properties were investigated in the SMO43 disc P/M superalloy. Various heat treatments were applied to this superalloy to determine the effect of the solution temperature on the grain size, and of the cooling path and aging temperature on the γ' precipitates distribution. Microstructural features were characterised through the careful study of γ' precipitation. Fatigue crack growth tests with dwell time were performed at high temperature for comparison of the mechanical properties of some selected microstructures.

Keywords:

Superalloy, heat treatment, microstructure, mechanical properties, fatigue crack growth.

1. INTRODUCTION

High-performance nickel-base superalloys, which can sustain high temperatures and high loadings, are required for turbine disc applications in aeronautical gas turbine engines. The nickel-base superalloy, SMO43, processed by powder metallurgy, was designed for this purpose [1,2]. This alloy contains a lower γ' volume fraction and has a lower γ' solvus temperature than other superalloys such as N18. Therefore, this alloy can be either subsolvus or supersolvus heat-treated, with less risk of reaching the incipient melting temperature [3].

In order to increase its potential for turbine disc application, the effect of changes of microstructural features of this alloy on its mechanical properties is investigated. Actually, numerous studies – see for instance references [4] to [7] – have been dedicated to examine the effect of grain size, γ' size and γ' distribution on the mechanical properties at elevated temperatures of powder metallurgy superalloys.

The present work deals with the relationship between heat treatment parameters and microstructural characteristics, and with the effect of changes of these microstructural characteristics on crack growth rate evaluated for fatigue crack growth tests with dwell time at high temperature.

The optimisation of the alloy will ultimately be reached by the identification of the set of heat treatment parameters that generates the microstructure resulting in the best compromise of mechanical properties, crack growth rate being an important one.

2. MATERIAL AND PROCEDURE

Alloy powder was produced by Aubert & Duval using VIM, VAR and argon atomisation. The chemical composition of the P/M SMO43 under study is given in Table 1 in weight percent. After compaction by HIP and extrusion, SMO43 pancakes were isothermal forged at SNECMA.

Table 1 Chemical composition of SMO43.

Ni	Cr	Co	Mo	W	Al	Ti	Nb	Hf	B	C	Zr
Bal.	13.3	12.2	4.6	3.0	2.9	3.6	1.5	0.25	0.01	0.015	0.05

2.1 Heat treatments investigation

Cylinders (12 mm diameter and 20 mm long) were machined from parts of the pancake forgings. Heat treatments were performed on these specimens using a halogen lamps furnace having a low thermal inertia and an integrated forced-air cooling system. This furnace was specially developed at the Centre des Matériaux, Mines Paristech, to allow control of the cooling path from the solution temperature with a wide range of cooling rates, from 10°C/min to 400°C/min. Cooling paths with two or more cooling rates can be achieved using this furnace with control of the transition temperature between the two cooling rates. The temperature was controlled using thermocouples embedded in the specimen and in its clamping rods in the furnace.

Each specimen was heated either below (subsolvus solution treatment) or above (supersolvus solution treatment) the solution temperature of primary γ' precipitates. Then, the specimens were cooled from the solution temperature with either a fast (100°C/min) or a slow cooling rate (25°C/min). Some specimens were cooled using a cooling path with two cooling rates (two-step cooling path). Finally, specimens were aged for 8 hours at four different temperatures. Table 2 summarizes the parameters of those of the heat treatments that were thoroughly investigated (temperatures are not mentioned to preserve data confidentiality).

Table 2 Heat treatment parameters.

Heat Treatment	Solution temperature	Cooling path	Aging temperature
A	Supersolvus	100°C/min	High temperature
B	Supersolvus	100°C/min	Medium temperature
C	Supersolvus	100°C/min	Low temperature
D	Supersolvus	25°C/min to a temperature below the solvus temperature followed by 100°C/min	Medium temperature
E	Subsolvus	100°C/min	Medium temperature

Characterisation of primary and secondary γ' precipitates was conducted by scanning electron microscopy (SEM), after etching in Glyceregia. Characterisation of tertiary γ' precipitates was completed using transmission electron microscopy, TEM. Grain sizes were measured by the intercept length method performed on crystallographic orientation maps obtained by Electron BackScatter Diffraction (EBDS).

Sizes of γ' precipitates were measured using image analysis, the mean size corresponding to the average Feret's diameter.

2.2 Fatigue crack growth study

Cylindrical mechanical testing specimen blanks (13 mm diameter by 60 mm long), were machined from the pancake forgings.

SENT (single edge notched tension) specimens were electrical discharge machined (EDM) from the specimens blanks after heat treatment according to the five sequences given in table 2. Fatigue crack growth tests at 650°C in air were performed on these SENT specimens using a servo-hydraulic machine fitted with a radiation furnace. The loading cycle consisted in a 10 seconds loading at constant loading rate, followed by a dwell time of 300 seconds at constant loading and a 10 seconds unloading to the minimum load at constant unloading rate. The load ratio was equal to 0.1. Crack growth was measured by potential drop monitoring, calibration of this technique being obtained by optical microscopy measurements.

3. RESULTS

3.1 Microstructural investigation

Solution temperature

After subsolvus heat treatment, primary γ' precipitates, with a size from 1 µm to 4 µm, are observed at grain boundaries, as shown in Figure 1. The mean grain size of this microstructure is about 5 µm in diameter. After supersolvus heat treatment, primary γ' precipitates are totally dissolved and the mean grain size is about 20 µm. A higher solution temperature results in the development of coarser grains.

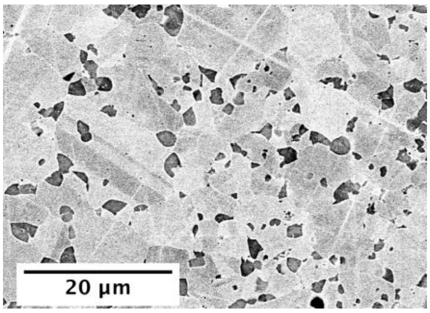


Fig. 1 SEM micrograph (back-scattered electrons) of primary γ' precipitates from specimen subsolvus heat–treated.

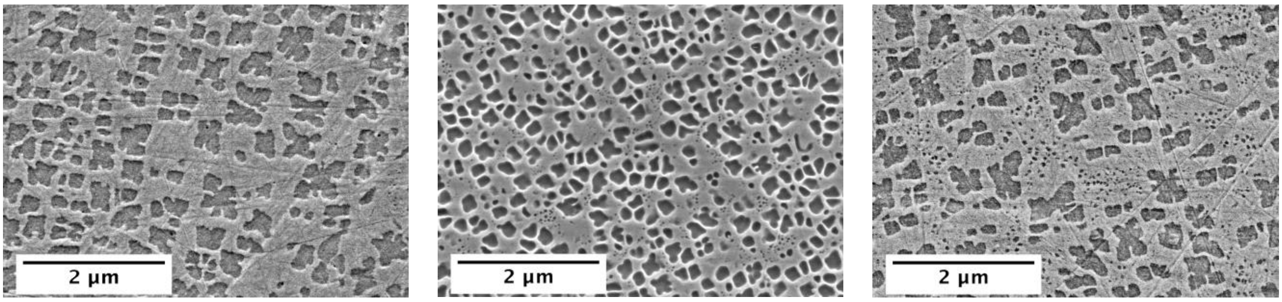
Cooling path

The cooling rate has a strong effect on the secondary γ' size, shape and distribution.

Slow cooling rate leads to the precipitation of large secondary γ' with an octo-cube shape and a size ranging from 250 nm to 500 nm (Fig. 2.a).

Increasing the cooling rate leads to the precipitation of smaller secondary γ' with a cube shape having an average size of around 190 nm (Fig. 2.b).

The two-step cooling path conducts to a bi-modal precipitation of secondary γ' (Fig. 3.b): one octo-cube shaped population with a size ranging from 200 to 500 nm that may precipitates during the slow cooling step; a second population is constituted by small spherical shaped γ' precipitates with a size ranging from 10 nm to 40 nm that may precipitate during the fast cooling step.

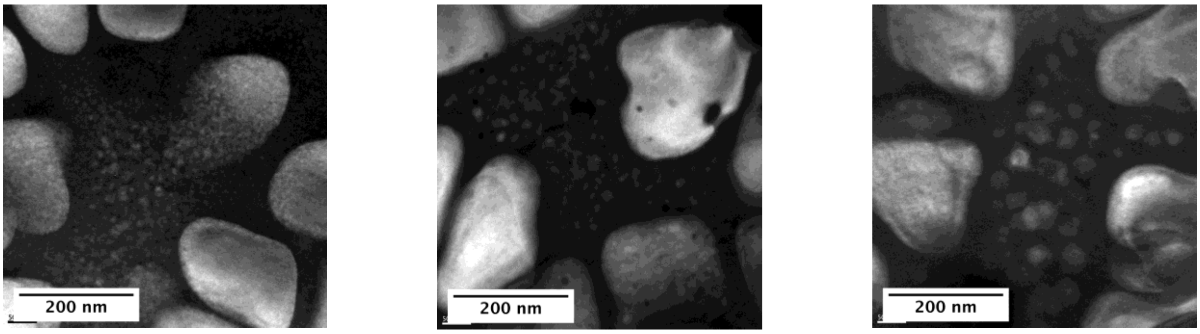


a) Slow air cooling *b) Fast air cooling* *c) Two-step cooling path*

Fig. 2 SEM micrographs (secondary electrons) of microstructure after the different cooling paths from the supersolvus solution temperature.

Aging temperature

Coarsening of tertiary γ' precipitates, that nucleate when cooling has reached temperature where diffusion becomes difficult, occurs during the aging treatment. The effect of the aging temperature on the tertiary γ' size was qualitatively investigated from TEM dark field micrographs (Fig. 3).



a) Low aging temperature *b) Medium aging temperature* *c) High aging temperature*

Fig. 3 TEM of microstructure after supersolvus heat treatment followed by aging at a) low, b) medium and c) high temperature.

As the aging temperature increases, the maximum size of tertiary γ' precipitates increases from less than 10 nm to 30 nm. This precipitate growth is mainly produced by Ostwald ripening and, then, results in the decrease of the number of tertiary γ'.

3.2 Mechanical characterization

Fatigue crack growth with dwell time

The results of the crack growth tests with dwell time conducted at 650°C are given in Figure 4, which presents the crack growth rate versus ΔK for each of the microstructures generated by the heat treatments given in Table 2.

Crack growth rate relative to the subsolvus microstructure appears to be more than ten times higher than the supersolvus one. Among the supersolvus heat-treated specimens, the three ones cooled at 100°C/min display the same crack growth behaviour, whatever the aging temperature. The fourth supersolvus heat-treated specimen, with a two-step cooling, shows a slightly lower growth rate than the other supersolvus heat-treated specimens.

First observations of crack path tended to reveal that cracking was mostly intergranular, which is consistent with other studies that showed that crack propagation became intergranular for dwell crack growth tests [8,9].

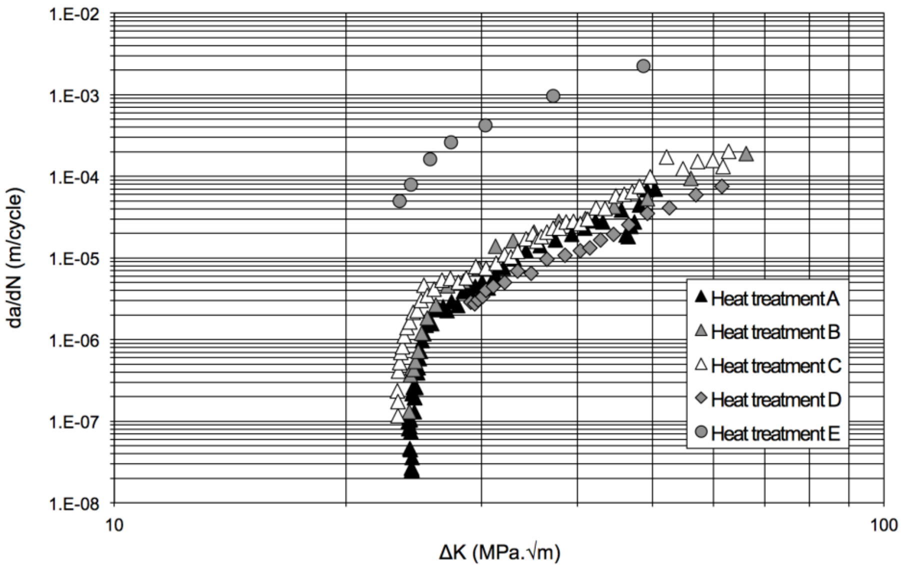


Fig.4 Results of crack growth tests with dwell time at 650°C for the heat treatments investigated on SMO43.

4. DISCUSSION

The crack growth test results clearly show that the solution temperature has a strong effect on the crack growth rate. Actually, it is the effect of the grain size and of the grain boundary structure and chemistry on the crack growth rate, which is revealed here: depending whether the solution temperature is higher or lower than the solvus temperature of the primary γ' precipitates, grain size increases or not, and coarse grain microstructures, that were supersolvus heat-treated, showed lower crack growth rate than the microstructure with fine grain size, which was subsolvus heat-treated. It is worth emphasizing that previous studies on older P/M alloys like Astroloy [9] and wrought nickel-base superalloys [10] have shown that this effect was mainly an effect of intergranular oxidation kinetics. These results are in agreement with those obtained by Chang *et al.* [7]. They were able to show that the grain size, the grain boundary morphology and chemistry were controlling the time dependent part of creep-fatigue crack growth rate. In particular, it was shown in model alloys based on René95 with and without chromium. Gayda *et al.* [11] and Telesman *et al.* [12] measured higher crack growth rate under creep-fatigue conditions at 704°C for the fine grain subsolvus microstructure than for the coarse grain microstructure. Gayda *et al.* suggested that the coarse grain size was the dominant parameter for high creep-fatigue crack growth resistance when comparing subsolvus and supersolvus heat-treated batches [11].

Among the supersolvus heat-treated specimens, the lack of difference between the crack growth rate of the three specimens cooled at the same rate of 100°C/min but aged at three different temperatures, tends to suggest that the tertiary γ' precipitate distribution, which is the only microstructural difference between these specimens, does not have any significant effect on the crack growth. However, the fourth supersolvus heat-treated specimen with a two-step cooling displays a slightly slower crack growth rate than the other supersolvus heat-treated specimens. This lets suppose that the secondary and the tertiary γ' distribution and/or the grain boundary morphology and chemistry may have an effect on the crack growth during fatigue crack growth test with dwell, and that the grain size is not the only influential parameter. Then, we suggest that the lack of effect of the aging temperature on the fatigue crack growth rate, that is to say the indifference of the crack growth rate to the tertiary γ' distribution, may result from the small size of the secondary γ' precipitates and, more precisely, from the most common narrowness of the γ matrix channels between them (Fig. 2b). Such narrow channels of γ matrix does not allow the development of a proper tertiary γ' distribution which may have an effect on the crack growth rate, even if cracking is mostly intergranular.

5. CONCLUSION

A study was conducted to investigate the relationships between heat treatment parameters and microstructural features and between microstructure and dwell crack growth rate in the SMO43 superalloy.

The cooling rate is a key parameter controlling the secondary γ' size and distribution. A cooling path with a slow cooling rate above a certain transition temperature followed by a fast cooling rate conducts to the precipitation of two types of secondary γ'.

The grain size was found to have a strong influence of the fatigue growth rate. Tertiary γ' precipitate distribution was not found to have an effect on the fatigue crack growth rate. However, it is suggested that this may result from the narrowness of γ matrix channel between the secondary γ' precipitates.

Effect of cooling path is to be studied in order to assess whether the secondary γ' size has an effect on the crack growth rate with dwell time.

ACKNOWLEDGEMENTS

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