

Effect of Double-Step Solution Treatment on Rejuvenation Heat Treated Microstructure of IN-738 Superalloy

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Abstract. This work investigates the effect of rejuvenation heat treatment, with double-step solution treatment at the temperature from 1150 °C to 1200 °C, on the recovered microstructure of IN-738 cast superalloy. The superalloy has been long-term exposed as a turbine blade in a gas turbine prior to this study. After double solution treatment and aging at 845 °C for 12 h and 24 h, the recovered microstructures were examined by using a scanning electron microscope. Coarse γ' particles, that have presented in damaged microstructures, could not be observed in the samples after the rejuvenation heat treatment. In addition, the image analysis illustrates that the reprecipitated γ' particles in the samples with double-step solution treatments increase significantly in sizes during aging than that in the samples with the single-step solution treatment. Furthermore, the measurement of the samples hardness presents that the as-receive sample hardness is improved after rejuvenation heat treatment studied in this work.

Introduction

The Inconel-738 (IN-738) is a Ni-based superalloy with excellent creep strength and corrosion resistance at high temperature. It is usually used as many engine components in gas turbine industries, such as blades, vanes and integral wheels [1]. The strength of the alloy [2], is due to the solid solution of chromium, tungsten and cobalt, the strengthening of carbide at grain boundaries, and the precipitation hardening of gamma prime (γ') particles that is the major strengthening phase of Ni-based superalloys [3, 4].

However, the operation of Ni-based superalloy components at high mechanical and thermal stress conditions gradually degrades the microstructure of Ni-based superalloys, finally resulting in the depletion of creep strength, fatigue, and corrosion resistance. The damaged microstructures of the alloys, e.g. IN-738, have been observed coarse γ' particles surrounding by small in size of γ' particles and also continuous $M_{23}C_6$ carbides along grain boundaries of γ matrix [5–7]. The coarse γ' particles are expected to be dissolved during solution treatment in rejuvenated heat treatment, and then reprecipitate in new sizes and morphologies throughout aging time. A previous study in the effect of temperature dropping during solution treatment on the recovered microstructure [7], has presented that the solutionising at temperature of 1155 °C/3 h and 1185 °C/3 h without temperature droppings shown fine γ' precipitates homogeneously distributed throughout the matrix. A multi-step solution treatment [8], from 1221 °C/2 h to 1260 °C/2 h, was applied to as-cast CM 247LC Ni-based superalloy. This contributes to an increase in volume fraction of fine γ' precipitates, yield strength at 300 K, creep rate and rupture life of the alloy. However, the effect of multi-step solution treatment in rejuvenated heat treatment on recovered microstructure of Ni-based superalloys is still rarely to be discussed.

This work aims to investigate the microstructure and hardness of nickel based superalloy grade IN-738 after rejuvenated heat treatment with double-step solution treatment, 1150 °C and then heating up to 1200 °C, in comparison to the results of the samples with single-step solution treatment at 1200 °C.

Materials and Experimental Procedure

A piece of IN-738 turbine blade after a long time exposure was studied here. The chemical composition of the alloy is shown in Table 1.

Table 1 Chemical composition of IN-738 in wt.%.

Ni	Cr	Co	W	C	Al	Ti	Mo	Ta	Nb	Fe	Zr	B
62.32	15.17	8.34	4.77	0.09	2.13	3.37	1.60	0.88	0.62	0.66	0.04	0.009

The rejuvenation heat treatment programs consist of double-solution treatment operated from 1150 °C to 1200 °C for different times, 1 h and 2 h, and the single-step solution treatment at the temperature of 1200 °C for 1 h and 2 h followed by air quenching, and then aging at 845 °C for 12 h and 24 h, as described in Fig. 1. and Table 2. This created 6 conditions of rejuvenation heat treatment for this study. After that, all specimens were examined microstructures by using a scanning electron microscope (FEG-SEM) and ImageJ analysis software [9]. Finally, the hardness values of the samples were measured by operating Vickers microhardness tester HVS-1000Z with the load of 500 gf.

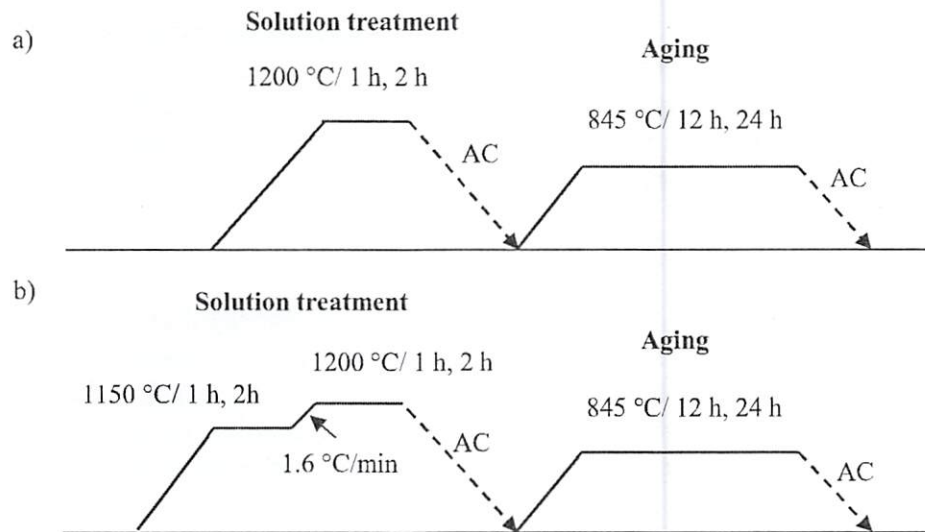


Fig. 1 The rejuvenation heat treatment conditions in this work with (a) single-stage solution treatment, and (b) double-step solution treatment. AC represents air cooling.

Table 2 Heat treatment programs applied to a long term serviced IN-738.

Condition	Solution Treatment	Cooling	Aging at 845 °C
A1	1200 C/2h	Air cooling	12h
A2	1200 C/2h	Air cooling	24h
B1	1150 C/1h+1200/1h	Air cooling	12h
B2	1150 C/1h+1200/1h	Air cooling	24h
C1	1150 C/2h+1200/2h	Air cooling	12h
C2	1150 C/2h+1200/2h	Air cooling	24h

Results and Discussion

As-receive microstructure. The observation of as-received IN-738 turbine blade after a long time service presents inhomogeneous microstructure that includes of coarse γ' particles dispersed among the fine γ' particles in size and large carbide particles, as shown in Fig. 2. When the sample hardness was tested, the average value is 419.47 HV.

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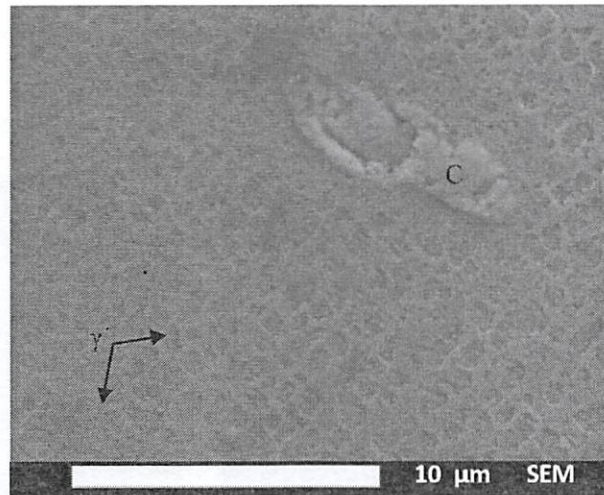


Fig. 2 The degraded microstructure of IN-738 turbine blade after a long term exposure including of γ' particles in various sizes and coarse carbides indicated as C.

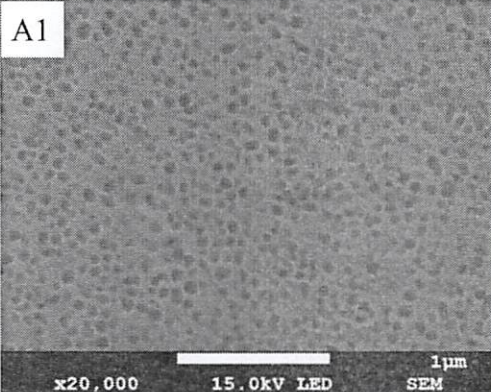
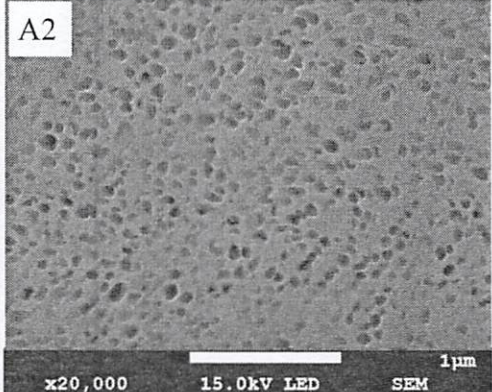
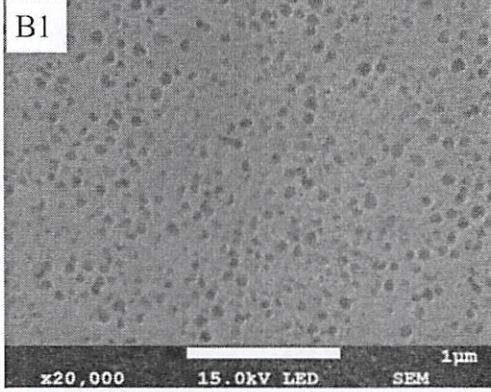
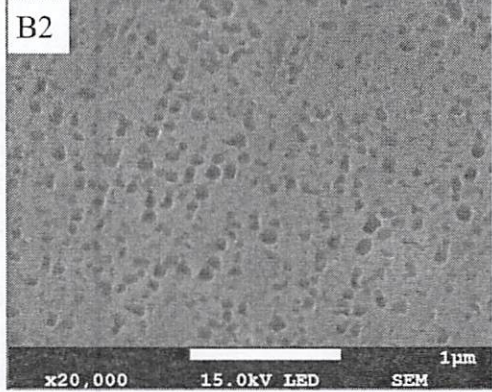
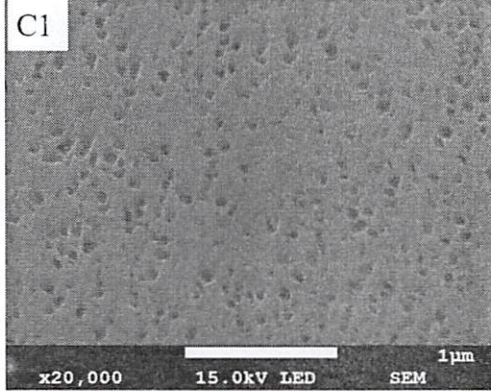
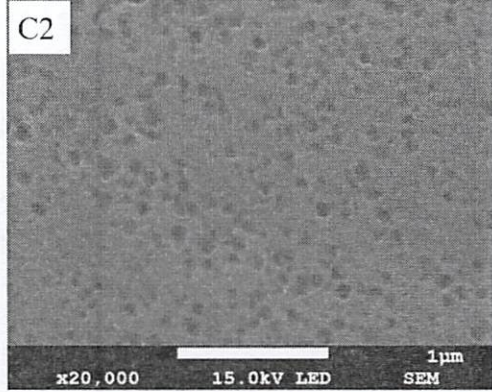
Solution treated and aged microstructure. Later, the deteriorated samples were heat treatment with various conditions, as shown in Table 2, resulting in the recovered microstructure with fine γ' precipitates depicted in Table 3. The γ' particles in large size as shown in the long time exposed sample cannot be observed in all after aged samples. This presents that the solution treatment temperatures and times applied herein are enough to dissolve the previous degraded γ' particles. The morphology of reprecipitated γ' phase after aged at 845 °C for 12 h (A1, B1, C1) and 24 h (A2, B2, C2) are in between spheroidal and cuboidal shapes, even though the samples are solution treated in different programs.

The rejuvenated microstructures are then analysed by using ImageJ analysis software. The results of number density of γ' particles per area in μm^2 are plotted in Fig. 3. It can be seen that all specimens present similar values, but the specimens with heat treatment program A (solution treatment at 1200 °C/ 2 h) are likely to show the highest value and the samples in program B (solution treatment at 1150 °C/ 1 h + 1200 °C/ 1 h) are likely to have more number of γ' particles per area than that of the samples heat treated with program C (solution treatment at 1150 °C/ 2 h + 1200 °C/ 2 h). In the same solution treatment condition, the samples in program A and B show similar number of γ' particles per area after aging at 845 °C for 12 h and 24 h. However, a slight reduction in the number of γ' precipitates per area with the increase in aging time can be seen. Only the sample in program C1 presents a significant decline in has the number γ' precipitates per area in comparison with that of the sample C2. The reason for this will be explained later.

The sizes of particles presented here are determined by the diameter of the circle that have the area equivalent to the particle analysed by using ImageJ analysis software. Considering the average size of particles in the rejuvenated samples after aging for 12 h, indicated as A1, B1, and C1 in Fig. 4, the values are likely to reduce from heat treatment condition A1 to C1. When the aging time is extended to 24 h, the γ' precipitates in A2, B2 and C2 increase in size. In A solution treatment condition, the sizes of γ' precipitates during ageing from 12 h to 24 h expand slightly. In contrast, the sample with solution treatment in condition B and C, the γ' precipitates grow significantly in size. The reason for this can be the dissolution during solution treatment. The more γ' particles taken into solution during solution treatment, the smaller in size and volume fraction of γ' precipitates would presents [2]. That also provides more driving force for the growth and coarsening process in aging time.

The percentage area of γ' precipitates in all aged samples were also determined, as plotted in Fig. 5. It is worth noting that the percentage area of γ' particles of C1 and C2 samples are nearly constant. Taking account of a considerable reduce in the number of γ' particles per area (Fig. 3) and a rise in the particle sizes (Fig. 4) of C1 and C2 samples with increasing aging time from 12 h to 24 h, the γ' particles observed in C1 and C2 samples are in coarsening which is the final step of precipitation process [10]. However, the hardness of all samples after aging are higher than that of the as-received specimen, as presented in Fig. 6.

Table 3 The microstructure of specimens after rejuvenation heat treatment with condition A1, A2, B1, B2, C1, and C2.

Solution treatment	Aging at 845 °C/12 h	Aging at 845 °C/24 h
1200 °C/2 h		
1150 °C/1 h + 1200 °C/1 h		
1150 °C/2 h + 1200 °C/2 h		

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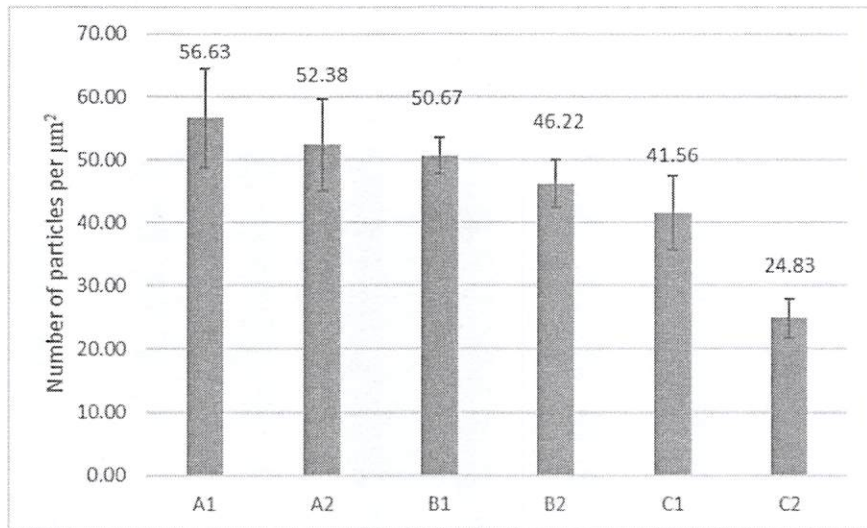


Fig. 3 The comparison between the number of γ' precipitates per μm^2 in each sample after rejuvenation heat treatment with different conditions.

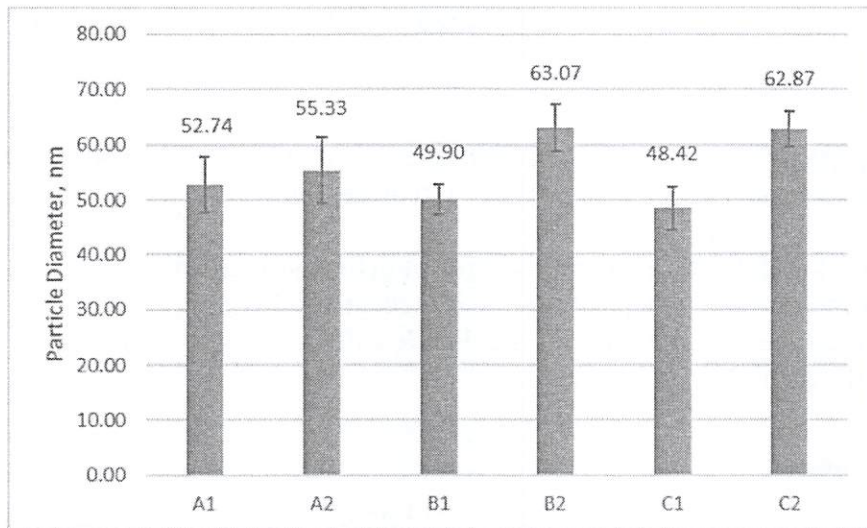


Fig. 4 The average diameter of γ' precipitates in each sample after rejuvenation heat treatment with different conditions.

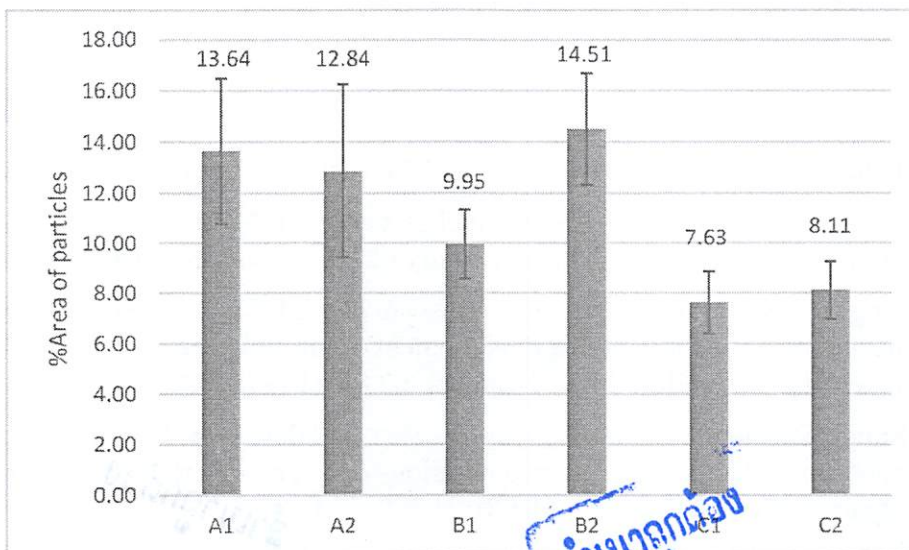


Fig. 5 The percentage of γ' particle area in each sample after rejuvenation heat treatment with different conditions.

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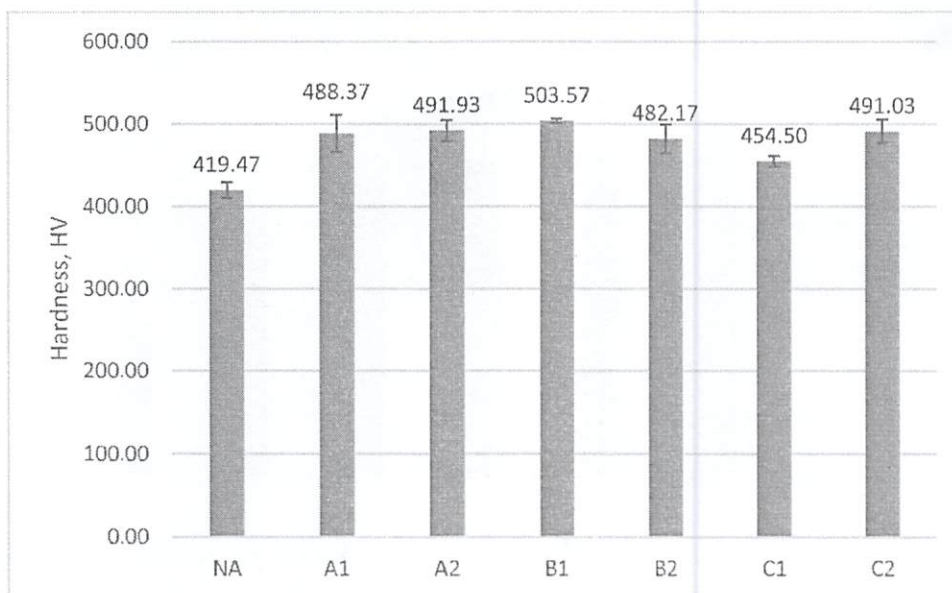


Fig. 6 The hardness of the samples after rejuvenation heat treatment with different conditions in comparison to the as-received sample. NA indicates the as-received sample.

Summary

1. The coarse γ' particles in degraded microstructure cannot be observed after double-step solution treatment conditions, 1150 °C/1 h + 1200 °C/1 h and 1150 °C/2 h + 1200 °C/2 h, and aging at 845 °C for 12 h and 24 h.
2. Double-step solution treatment conditions result in the more growth in γ' precipitate sizes during aging relative to the single-step solution treatment at 1200 °C/2 h.
3. The rejuvenation heat treatment with the double solution treatment conditions can improve the hardness of the degraded IN-738.

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