

OM Study of Effect of HIP and Heat Treatments on Microstructural Restoration in Cast Nickel Based Superalloy, IN-738

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Received Aug. 2, 2007

Accepted Sept. 19, 2007

Abstract

The present work aims at studying and searching for the most suitable and practicable repairing condition for microstructural restoration, which could provide the desired microstructural characteristics by rejuvenation method of hot isostatic pressing (HIP), followed by 12 heat treatment conditions for long-term serviced gas turbine blades made of cast nickel base superalloy grade IN-738 operated by Electricity Generating Authority of Thailand (EGAT). During heat treatment process, coarse carbides and over-exposed coarse gamma prime precipitates, which had formed previously during service, would dissolve into the matrix during solution treatment. Then specimens were processed through a series of precipitation aging, which re-precipitated the strengthening phase to form the proper morphology in size and shape as well as distribution that is almost similar to the new one. Metallography examination had been performed by using light optical microscopy after hot isostatic pressing and heat treatments to evaluate the rejuvenated microstructures.

Key words : Hot Isostatic Pressing (HIP), Rejuvenation, Microstructural Repair, and Nickel-Based Superalloy

Introduction

Nickel-based superalloys have been developed to be utilized at high temperature applications. The microstructures and mechanical properties (at low to high temperatures) can be related to their manufacturing processes. Many mechanical properties are strongly related to the microstructures. In the new, heat treated alloy, the gamma prime (γ') particles are arranged properly in a structure, which results in an optimum balance of tensile, fatigue, and creep properties.⁽¹⁾ Several previous research works⁽²⁻¹²⁾ had been carried out to investigate these relationships of microstructure-mechanical properties. One of these processes is heat treatment which solutioning is generally followed by a single or a double aging sequence to precipitate homogeneous distributions of either

cuboidal or spherical gamma prime within the grains interior as well as discrete grain boundary carbides.⁽¹⁾ The size, volume fraction and distribution of gamma prime phase are vital to control the creep strength at intermediate to high stresses. The proper heat-treated microstructure can provide their phase stability, and adequately high strength and good ductility even after long-term thermal exposure. The heat treatment processes for nickel-based superalloys continue to change in order to optimise numerous mechanical and physical properties.⁽¹³⁻²¹⁾ This allows for the selection of heat treatment parameters to become more advantageous.

The aim of this research work is to determine the most suitable and practicable repair-condition which could provide the proper

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microstructural characteristics by rejuvenation method of hot isostatic pressing (HIP) followed by various methods of heat treatment for gas turbine blades made of casting nickel based superalloy grade IN-738 after 50,000-hour service operated by Electricity Generating Authority of Thailand (EGAT).

Material and Experimental Procedure

The cast nickel-based superalloy in this study was IN-738. The chemical composition of the alloy is shown in Table 1. Rectangular plates, having a dimension of 1 cm², were cut from the most severe degradation zone of turbine blades. HIP condition is as follows: specimens were HIPed at pressure of 100 MPa for 86.4 ks at 1473 K, and then the HIPed specimens were heat treated according to heat treatment conditions including solution treatment, primary and secondary precipitate aging treatments in vacuum furnace (see experimental heat treatment details in Table 2). Heat treated plates were cross-sectioned in order to observe the microstructure compared to those of parallel grinded and polished surface of turbine blades. All sectioned samples were polished using standard metallographic techniques and were subsequently etched in marble etchant, which has the following chemical composition: 10 g CuSO₄, 50 ml HCl, and 50 ml H₂O. The microstructures of heat treatment samples were studied by scanning electron microscope with secondary electron mode and image analyser.

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

Ni	Cr	Co	Ti	Al	W	Mo	Ta	Nb	C	Fe	B	Zr
Bal.	15.84	8.5	3.47	3.46	2.48	1.88	1.69	0.92	0.11	0.07	0.12	0.04

Table 2. Heat treatment conditions applied to long term exposed IN-738

No.	Solution annealing	Primary precipitate aging	Secondary precipitate aging
1	-----	-----	1118 K / 86.4 ks (AC)
2	-----	1198 K / 3.6 ks (AC)	1118 K / 86.4 ks (AC)
3	-----	1328 K / 3.6 ks (AC)	1118 K / 86.4 ks (AC)
4*	1398 K / 7.2 ks (AC)	-----	1118 K / 86.4 ks (AC)
5	1398 K / 7.2 ks (AC)	1198 K / 3.6 ks (AC)	1118 K / 86.4 ks (AC)
6	1398 K / 7.2 ks (AC)	1328 K / 3.6 ks (AC)	1118 K / 86.4 ks (AC)
7	1448 K / 7.2 ks (AC)	-----	1118 K / 86.4 ks (AC)
8	1448 K / 7.2 ks (AC)	1198 K / 3.6 ks (AC)	1118 K / 86.4 ks (AC)
9	1448 K / 7.2 ks (AC)	1328 K / 3.6 ks (AC)	1118 K / 86.4 ks (AC)
10	1478 K / 7.2 ks (AC)	-----	1118 K / 86.4 ks (AC)
11	1478 K / 7.2 ks (AC)	1198 K / 3.6 ks (AC)	1118 K / 86.4 ks (AC)
12	1478 K / 7.2 ks (AC)	1328 K / 3.6 ks (AC)	1118 K / 86.4 ks (AC)

* *Standard Heat Treatment condition*

Results and Discussion

The Microstructure of As-received Alloy

An optical micrograph, obtained from the transverse sections at about mid blade height of the airfoil, is shown in Figure 1. The microstructure of as-cast alloy generally consists of extensive precipitation of ordered L1₂ γ' intermetallic phase within dendrite core and in the interdendritic region. The agglomerated gamma prime particles can also be seen. Coalescence of the gamma prime particles, as a result of long-term service, seems to occur continually, causing larger and more rounded particles. In this study, the coarse gamma prime particle size was approximately 1.2 μm. The airfoil microstructure shows significant degradation in service. The gamma prime particles had spheroidised and coarsened in the airfoil samples. This type of microstructure is theoretically expected to provide lower creep resistance during loading at high temperatures.

Heat Treated Microstructure after HIP Process Investigated by OM

All of the reheat treatments after HIP process in long-term serviced turbine blade IN-738 provided the various microstructural restoration characteristics with more homogeneous structure compared to the long-term serviced one, as shown in Figures 2-13. It was found that the heat treated microstructures, according to programs No. 1-3,

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consist of uniform distribution of very fine gamma prime particles precipitating in the matrix in near cubic shape, Figures 2-4. It should be noted that each microstructure contains only a single size of precipitated gamma prime particles. These have sizes in the narrow range of between 0.15-0.3 μm . In these structures, it could be counted that the heating during HIP process practically worked as solutioning at 1473 K for 18 ks and then followed by aging at 1118 K for 86.4 ks. The microstructure after HIP process was already prepared for the next step precipitation aging where the previously existed coarse gamma prime particles were almost dissolved already into the matrix. When precipitation (secondary) aging was applied, fine gamma prime particles re-precipitated in the matrix at a high volume fraction.

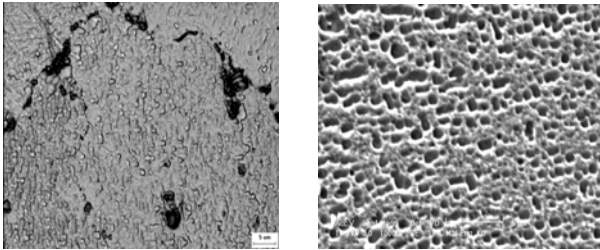


Figure 1. As-received microstructure after long-term service showing the coalescence of γ' particles, areas of γ - γ' eutectic and grain boundary carbides

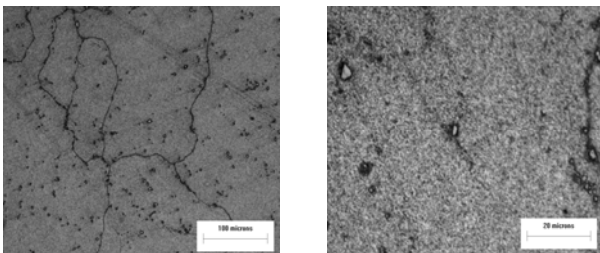


Figure 2. After heat-treatment at 1118 K for 86.4 ks (AC); Condition No. 1

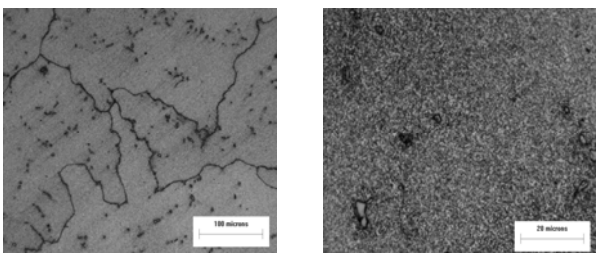


Figure 3. After heat-treatment at 1198 K for 3.6 ks (AC), and 1118 K for 86.4 ks (AC) ; Condition No. 2

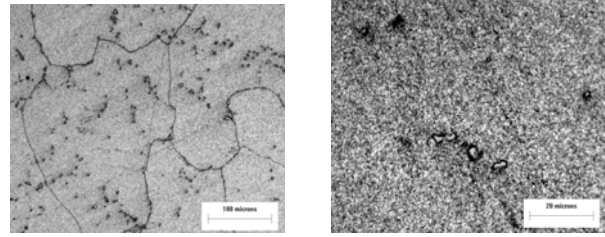


Figure 4. After heat-treatment at 1328 K for 3.6 ks (AC), and 1118 K for 86.4 ks (AC) ; Condition No. 3

The solution treatment at 1398 K for 7.2 ks had more effect on microstructure characteristics. The solutioning at this temperature provided morphology with coarser γ' phase precipitation, as shown in Figures. 5-7. The average diameter size of γ' particles was about 0.35 μm for coarse particles. The solutioning at 1398 K for 7.2 ks caused more dissolution into the matrix of previous or residual coarse γ' particles. After final aging at 1118 K for 86.4 ks with or without primary aging, coarse γ' particles could uniformly reprecipitate into the matrix with coarser size and higher volume fraction of total γ' precipitated phase compared to the final microstructures according to conditions No. 1-3. The addition of primary aging (at both 1198 K and 1328 K for 3.6 ks) had only a slight effect on microstructural characteristic investigated by OM.

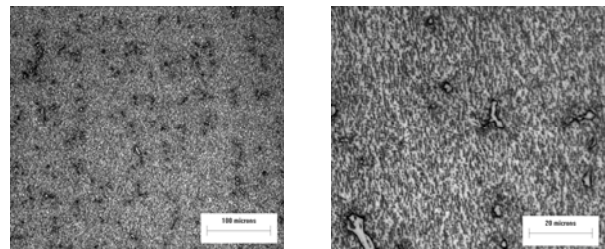


Figure 5. After heat-treatment at 1398 K for 7.2 ks (AC) and 1118 K for 86.4 ks (AC) ; Condition No. 4

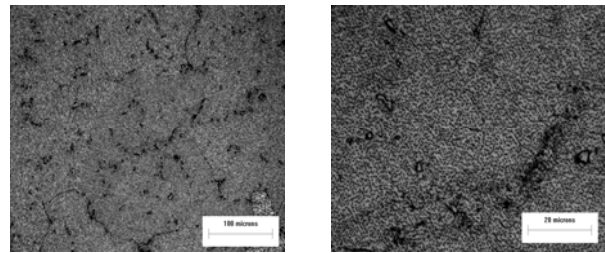


Figure 6. After heat-treatment at 1398 K for 7.2 ks (AC), 1198 K for 3.6 ks (AC), and 1118 K for 86.4 ks (AC); Condition No. 5

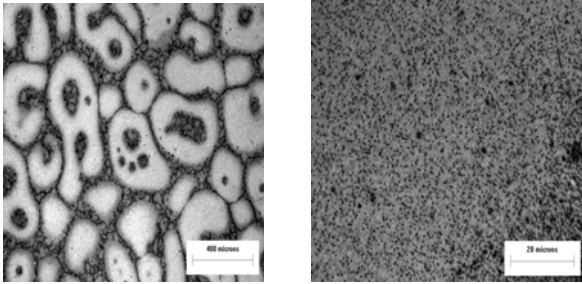


Figure 7. After heat-treatment at 1398 K for 7.2 ks (AC), 1328 K for 3.6 ks (AC), and 1118 K for 86.4 ks (AC); Condition No. 6

Figures 8-13. show the effect of solution treatment at high temperature of 1448 K and 1478 K for 7.2 ks on final microstructures. Compared to microstructures with lower solutioning temperature (at 1398 K) of conditions No. 1-3, it was found that these higher temperatures of solutioning provided the coarser average size of precipitated γ' particles. However, the volume fractions of finer γ' particles in these final microstructures were less than those of final microstructures according to conditions No. 1-6. The dispersed finer γ' particles in the matrix should be the result of γ' precipitation during long term aging after solutioning. The primary aging at 1198 K for 3.6 ks (see Figures 9 and 12) provided a slight effect on the microstructure, but only resulted in lower volume fraction of γ' particles compared to microstructures without primary aging. However, in contrast, when primary aging at the higher temperature of 1328 K was applied for 3.6 ks, the γ' precipitation could take place rapidly, resulting in slightly higher volume fraction and coarser size of very fine γ' particles after final aging.

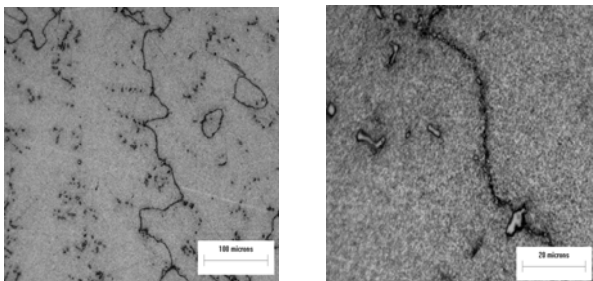


Figure 8. After heat-treatment at 1448 K for 7.2 ks (AC) and 1118 K for 86.4 ks (AC) ; Condition No. 7

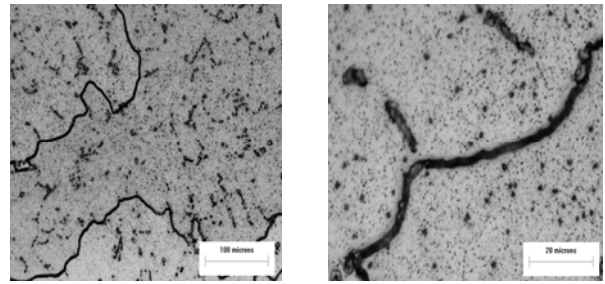


Figure 9. After heat-treatment at 1448 K for 7.2 ks (AC), 1198 K for 3.6 ks (AC), and 1118 K for 86.4 ks (AC); Condition No. 8

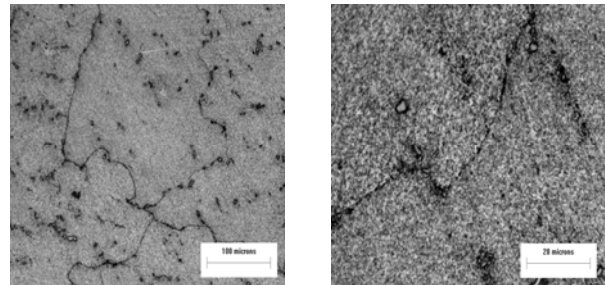


Figure 10. After heat-treatment at 1448 K for 7.2 ks (AC), 1328 K for 3.6 ks (AC), and 1118 K for 86.4 ks (AC); Condition No. 9

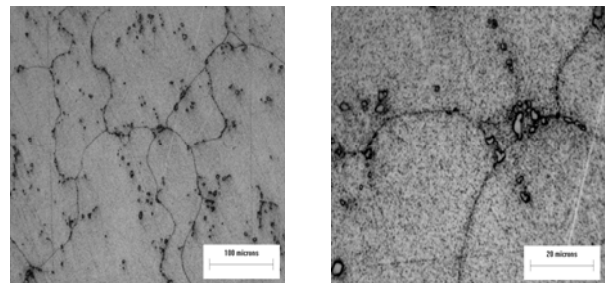


Figure 11. After heat-treatment at 1478 K for 7.2 ks (AC) and 1118 K for 86.4 ks (AC) ; Condition No. 10

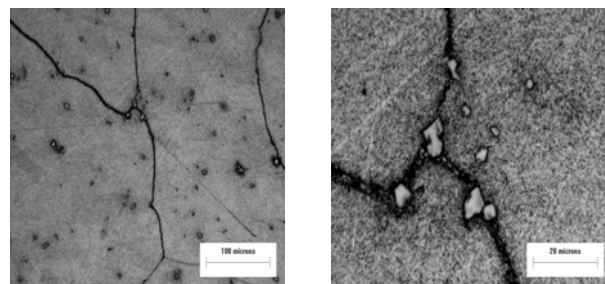


Figure 12. After heat-treatment at 1478 K for 7.2 ks (AC), 1198 K for 3.6 ks (AC), and 1118 K for 86.4 ks (AC); Condition No. 11

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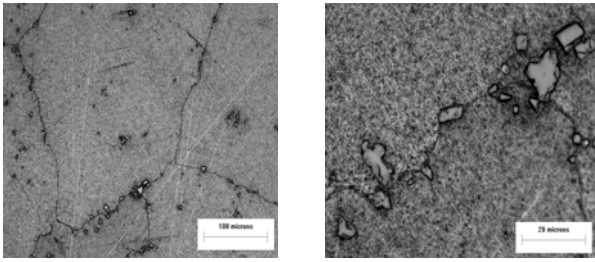


Figure 13. After heat-treatment at 1478 K for 7.2 ks (AC), 1328 K for 3.6 ks (AC), and 1118 K for 86.4 ks (AC); Condition No. 12

Conclusion

1. The most proper solutioning temperature in this experimental program that should be applied after HIP process is 1398 K. This could provide the final microstructures made up of highest volume fractions with proper shape and size of fine γ' particles.

2. The addition of primary aging could assist in more uniform distribution of both coarse and very fine γ' particles as well as the increase of volume fractions compared to those without primary aging. However, applying the primary aging after very high solutioning temperatures (of 1448 K and 1478 K) would lead to fast and abnormal precipitation, resulting in partially coarse γ' particles, conditions No. 7-12.

3. The most proper heat treatment conditions after HIP process for the alloy should be conditions No. 4 (standard heat treatment), 5, and 6 due to their highest volume fractions of γ' particles, which were nearly 60%, as well as coarse γ' particles that precipitated uniformly and densely. However, mechanical testing, especially at elevated temperatures such as creep, fatigue and thermal fatigue should be performed in further works to evaluate and confirm the relationship between the rejuvenated microstructures and mechanical properties.

Acknowledgement

This research work was financially supported by the postdoctoral research grant (No. MGR 4880088) from The Thailand Research Fund (TRF) and The Commission of Higher Education

of Thailand. A special thank is also extended to Electricity Generating Authority of Thailand (EGAT), Nonthaburi, Thailand, for material support and technical help.

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