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# OM Study of Effect of HIP and Heat Treatments on Microstructural Restoration in Cast Nickel-Based Superalloy, GTD-111

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## Abstract

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The present work aims at studying and searching for the effect of repairing conditions on microstructural restoration. It also seeks to investigate into the most suitable and practicable repairing condition, 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, cast nickel-based superalloy grade GTD-111 operated by Electricity Generating Authority of Thailand (EGAT). During solution treatment, coarse carbides and over-exposed coarse gamma prime precipitates, which had formed previously during service, would dissolve into the matrix. 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 ($\gamma'$) particles are arranged properly in a structure, which results in an optimum balance of tensile, fatigue, and creep properties.(1) Several previous research works(2-5) had been carried out to investigate these relationships of microstructure-mechanical properties. One of these processes is heat treatment, which solutioning in most cases is followed by a single or a double aging sequence to precipitate homogeneous distributions of either cuboidal or spherical gamma prime within the grain 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.(6-9) This allows for making 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 heat treatments for long-term exposed gas turbine blades, cast nickel-based superalloy grade GTD-111 after 50,000-hour service operated by Electricity Generating of Thailand (EGAT).

## Material and Experimental Procedure

The cast nickel-based superalloy in this study was GTD-111 with the following composition (wt%): 13.5%Cr, 9.5%Co, 4.75%Ti, 3.3%Al, 3.8%W, 1.53%Mo, 2.7%Ta, 0.09%C, 0.23%Fe, and 0.01%B and balance nickel. Rectangular plates, having a dimension of 1cm$^2$, were cut from the most severe degradation zone of turbine blades. The HIP condition is as follows: specimens were HIPed at a pressure of 100 MPa for 86.4 ks at 1473 K. Then the HIPed specimens were heat treated according to heat treatment conditions including solution treatment as well as primary and secondary precipitate aging treatments in vacuum furnace (see experimental heat-treatment details in Table 1). Heat-treated plates were cross-sectioned in order to analyse the microstructure in comparison to plates 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 $CuSO_4$, 50 ml HCl, and 50 ml $H_2O$. The microstructures of heat treatment samples were studied by scanning electron microscope with secondary electron mode and image analyser.

## 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 $L1_2$ γ’ intermetallic phase within dendrite core and in the interdendritic region. Carbides predominantly of MC type, borides,

**Table 1.** Heat treatment conditions applied to long term exposed GTD-111

| 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*** sulphur-carbide and γ-γ' eutectic, which formed during ingot solidification, were found in smaller volume fraction located along the interdendritic region. The chromium carbide ($M_{23}C_6$) and agglomerated gamma prime particles can also be seen. Coalescence of the gamma prime particles, as a result of long-term service, seems to continually occur, resulting in 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.

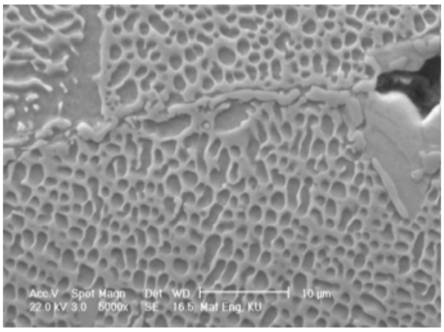

**Figure 1.** As-received microstructure after long-term service showing the coalescence of γ' particles, areas of γ-γ' eutectic and grain boundary carbides

### *Heat Treated Microstructure After HIP Process Investigated by OM*

All of the re-heat treatments after HIP process in long-term serviced turbine blade GTD-111 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, consist of the uniform distribution of fine gamma prime particles precipitating in the matrix as nearly cubic shape, Figures 2-4. It should be noted that each micro-structure contains only a single size of precipitated gamma prime particles which have sizes in the narrow range 0.15-0.3 μm. In these structures, it could be counted that the heating during HIP process practically worked as solutioning at 1473K 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, which previously existing coarse gamma prime particles were almost dissolved into the matrix. When precipitation (secondary) aging was applied, fine gamma prime particles re-precipitated in the matrix at a high volume fraction.

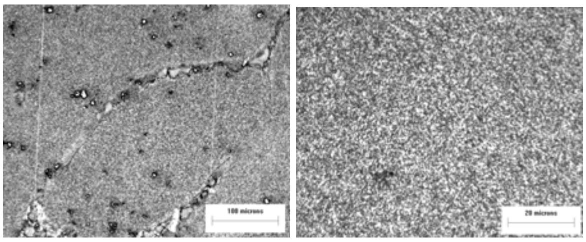

**Figure 2.** After heat-treatment at 1118 K for 86.4 ks (AC); Condition No. 1

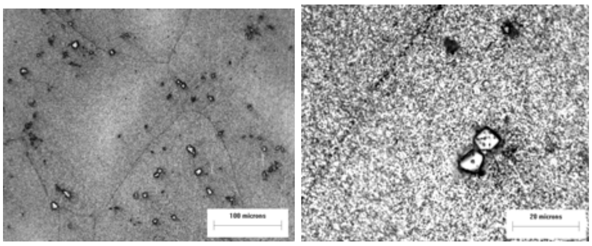

**Figure 3.** After heat-treatment at 1198 K for 3.6 ks (AC), and 1118 K for 86.4 ks (AC) ; Condition No. 2

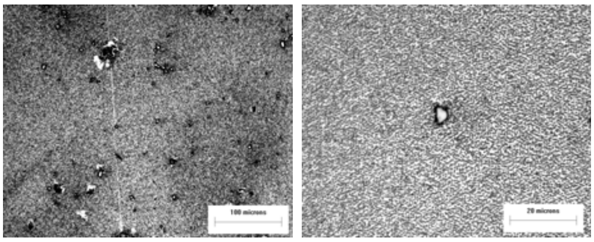

**Figure 4.** After heat-treatment at 1328 K for 3.6 ks (AC), and 1118 K for 86.4 ks (AC) ; Condition No. 3

The solution treatment at 1398 K for 7.2 ks had more effect on microstructure characteristics. Solutioning at this temperature provided morphology with more coarsening γ' phase precipitation, as shown in Figures 5-7. The average diameter size of γ' particles was about 0.35 μm for coarse particles (see more details by SEM analysis in previous work). (10) Furthermore, this solutioning resulted in more cubic shape of coarse γ' particles. The solutioning at 1398 K for 7.2 ks caused more dissolution into the matrix of previous or residual coarse $\gamma'$ particles. After final aging at 1118 K for 86.4 ks with or without primary aging, coarse $\gamma'$ particles could uniformly reprecipitate into the matrix with coarser size and higher volume fraction of total $\gamma'$ precipitated phase compared to those final microstructures according to conditions No. 1-3. The addition of primary aging (at both 1198 K and 1328 K for 3.6 ks) only had a slight effect on the microstructural characteristic investigated by OM. It should be noted that microstructure after heat treatment condition No. 6 consists of needle-like phase (white) located in grain boundary carbides. This might be sigma $(\sigma)$ phase that occurred in the vicinity of MC or $M_{23}C_6$ carbides.

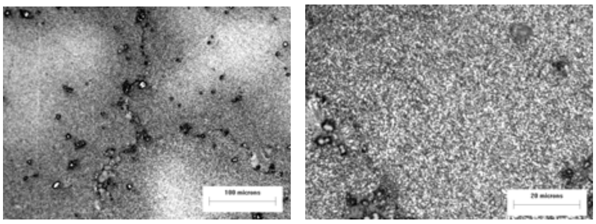

**Figure 5.** After heat treatment at 1398 K for 7.2 ks (AC) and 1118 K for 86.4 ks (AC) ; Condition No. 4

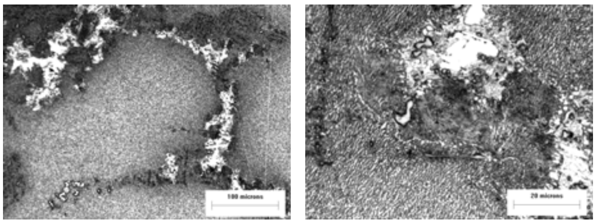

**Figure 6.** After heat treatment at 1398 K for 7.2 ks (AC), 1198 K for 3.6 ks (AC), and 1118 K for 86.4 ks (AC) ; Condition No. 5

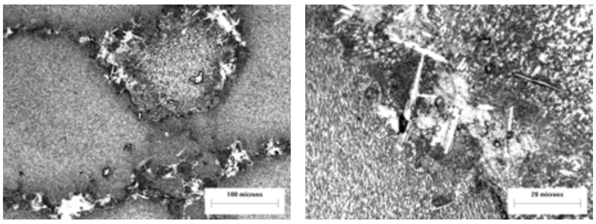

**Figure 7.** After heat treatment at 1398 K for 7.2 ks (AC), 1328 K for 3.6 ks (AC), and 1118 K for 86.4 ks (AC) ; Condition No. 6

Figures 8-10 show the effect of solution treatment at 1448 K for 7.2 ks on final microstructures. Compared to microstructures with the lower solutioning temperature (at 1398 K) of conditions No. 1-3, it was found that this higher temperature of solutioning provided the coarser average size of precipitated $\gamma'$ particles. However, the volume fractions of coarse $\gamma'$ particles in these final microstructures with 1448 K-solutioning were less than those of final microstructures according to conditions No. 1-6. The dispersed coarse $\gamma'$ particles in matrix should be the result of $\gamma'$ precipitation during long-term aging after solutioning. The primary aging at 1198K for 3.6Ks resulted in a significant effect on the microstructures but only resulted in a coarser shape of $\gamma'$ particles compared to microstructures without primary aging. However, when primary aging at higher temperature of 1328K for 3.6Ks was applied, the $\gamma'$ precipitation could take place rapidly, resulting in higher volume fraction and coarser size of both coarse and fine $\gamma'$ particles after final aging.

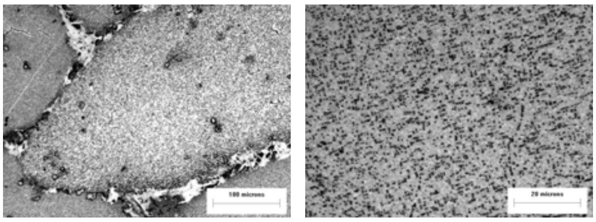

**Figure 8.** After heat treatment at 1448 K for 7.2 ks (AC) and 1118 K for 86.4 ks (AC) ; Condition No. 7

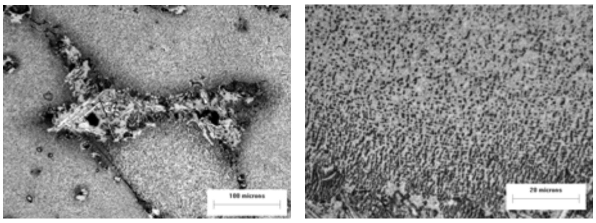

**Figure 9.** After heat treatment at 1448 K for 7.2 ks (AC), 1198 K for 3.6 ks (AC), and 1118 K for 86.4 ks (AC) ; Condition No. 8

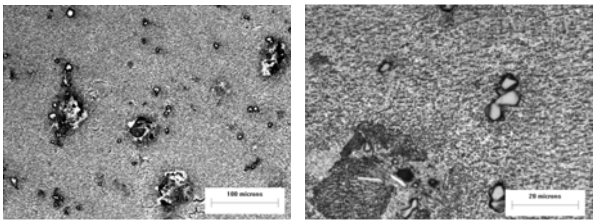

**Figure 10.** After heat treatment at 1448 K for 7.2 ks (AC), 1328 K for 3.6 ks (AC), and 1118 K for 86.4 ks (AC) ; Condition No. 9

The solutioning at the highest tested temperature of 1478 K for 7.2 ks could result in very high dissolution of previous or residual γ’ particles after HIP process into the matrix. The obtained microstructures after this solutioning were expected to be very stable dissolved elements in the matrix. Therefore, during the next step long-term aging at 1118 K for 86.4 ks, the γ’ particles could uniformly re-precipitated in single size and round shape in some areas far away from grain boundaries. It should be noted that coarse γ’ particles were also observed in areas close to grain boundaries (Figure 11).

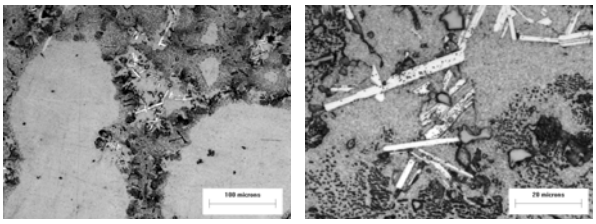

**Figure 11.** After heat treatment at 1478 K for 7.2 ks (AC) and 1118 K for 86.4 ks (AC) ; Condition No. 10

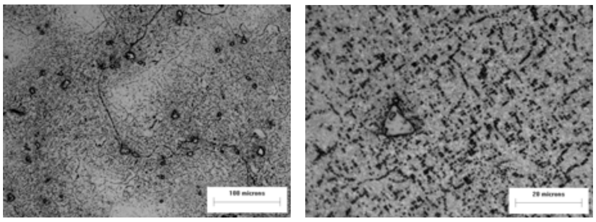

**Figure 12.** After heat treatment at 1478 K for 7.2 ks (AC), 1198 K for 3.6 ks. (AC), and 1118 K for 86.4 ks (AC) ; Condition No. 11

However, when inserting primary aging process between solutioning and final aging (or secondary aging), this could lead to more uniform precipitation of γ’ particles during primary and secondary agings, Figures 12-13. These heat treatment conditions provided the maximum particle sizes, especially of coarse γ’ particles in cubic shape. However, it should be pointed out that the volume fractions of coarse γ’ particles were lowest in comparison with those of other heat treatment conditions. The higher temperature (1328K) of primary aging in condition No. 12 resulted in more slight precipitation of γ’ particles than the lower primary aging temperature of 1198 K in condition No. 11.

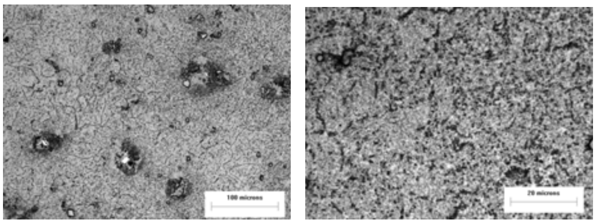

**Figure 13.** After heat treatment at 1478 K for 7.2 ks (AC), 1328 K for 3.6 ks (AC), and 1118 K for 86.4 ks (AC) ; Condition No. 12

## Conclusion

1. The most proper solutioning temperature in this experimental program that should be employed after HIP process is 1398 K, which could provide the final microstructures with highest volume fractions with proper shape and size of coarse γ’ particles.

2. The addition of primary aging could assist in a more uniform distribution of both coarse and very fine γ’ particles as well as increase volume fractions compared with 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.

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