# Effect of Temperature Dropping During Solution Treatment During Rejuvenation Heat Treatment on Final Microstructures in Cast Nickel Base Superalloy, Grade Inconel-738

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## Abstract

Generally, the rejuvenation heat treatment of degraded turbine blades, which were made of cast nickel based superalloy, Grade Inconel 738, usually consist of solution treatment condition with temperature range of 1125°C- 1205°C for 3 hours and then following with double aging processes, which include primary aging at 1055°C for 1 hour and secondary aging at 845°C for 24 hours. However, in practical working conditions of reheat treatment processed, the possible change of temperature during solution treatment can be obtained by error or malfunction of heating furnace, which usually provides the temperature dropping down. To simulate this effect, the dropping temperature during solution treatment was chosen to decrease till the level of 845°C, which usually happens in practices then immediately heating up again to solution treatment. The maximum number of temperature droppings during the single solution treatment is up to 3 times. From the received results, it was found that the effect of temperature dropping during solution treatment has extremely influenced on the final rejuvenated microstructures.

Keywords : Nickel base superalloy, Rejuvenation heat treatment, Inconel-738, Temperature dropping, Solution treatment

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# Introduction

At the present time, nickel-based superalloys are widely used as gas turbine blade materials in hot section of engines in electricity generating industries. Superior corrosion, fatigue and creep resistance properties at elevated temperature are the main reasons to use these superalloys comparing to other alloys. The studied turbine blades were produced of nickel based superalloys, Grade IN-738, which can be operated under high temperature pressure gas with severe corrosion and load.<sup>(1-3)</sup> However, the microstructures would continuously degrade and finally influence on mechanical strengths when they are used at elevated temperature for long term.<sup>(4-5)</sup>

The rejuvenation of degraded material can be done by reheat treated for purpose of the refurbishment the microstructures to have similar or same mechanical properties as before. Benefits of rejuvenation heat treatment can save the materials cost for replacing.<sup>(6-8)</sup> Nevertheless, in many rejuvenation processes, it could have some problems about the temperature controlling of vacuum furnace, which sometimes the controlled temperature could automatically shut off when the temperatures are over the furnace limit. Therefore, the final microstructures were not expected for requirements. This was due to the changing of microstructure has relation to temperature and time in solution treatment process.<sup>(9)</sup>

This research was studied about the effect of temperature dropping during solution process in rejuvenation heat treatment and following long-term simulated service at high temperatures on final microstructures of nickel based superalloys grade Inconel-738 to evaluate the final microstructure and the phase stability.

# **Materials and Experimental Procedures**

The nickel-based superalloys, grade Inconel-738 with 50,000 hours serviced lifetime of blades in hot section of gas turbine engine was studied in this work. The chemical composition is shown in Table 1.

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Elements	Ni	Cr	Со	Ti	Al	С	W	Мо	В	Nb	Та	Zr
Weight Percent (%wt.)	62.8 6	16.7 1	8.37	3. 5	2.38	2.0 8	1.68	1.1	0.57	0.4	0.3	0.01

Table 1. The chemical composition of nickel-based superalloys, grade Inconel-738.

First step, the material was prepared before rejuvenation, which the specimens were cut size of 1x1x1 cm<sup>3</sup> in process with 12 condition, as shown reheating programs in Figure 1-4 for Programs 1-4, respectively. Furthermore, rejuvenated heat treatment includes solution treatment, primary aging and secondary aging, respectively.

**Program 1** (Figure 1) : The solution treatment process as follows.

- A3 Solution treatment at  $1125^{\circ}$ C/3h then air cooling + 2 Aging processes
- B3 Solution treatment at  $1155^{\circ}$ C/3h then air cooling + 2 Aging processes
- C3 Solution treatment at 1185°C/3h then air cooling + 2 Aging processes

**Program 2** (Figure 2) : The solution treatment process as follows.

- A12 Solution treatment at 1125°C/1h + Cool down to 845°C + Heat up to 1125°C/2h + 2 Aging processes
- B12 Solution treatment at 1155°C/1h + Cool down to 845°C + Heat up to 1155°C/2h + 2 Aging processes
- C12 Solution treatment at 1185°C/1h + Cool down to 845°C + Heat up to 1185°C/2h + 2 Aging processes



Figure 1. Rejuvenated heat treatment process with conditions of specimens A3, B3 and C3



Figure 2. Rejuvenated heat treatment conditions of specimens A12, B12 and C12

**Program 3** (Figure 3): The solution treatment process as follows.

- A21 Solution treatment at  $1125^{\circ}C/2h + Cool down to 845^{\circ}C + Heat up to <math>1125^{\circ}C/1h + 2 Aging processes$
- B21 Solution treatment at 1155°C/2h + Cool down to 845°C + Heat up to 1155°C/1h + 2 Aging processes
- C21 Solution treatment at 1185°C/2h + Cool down to 845°C + Heat up to 1185°C/1h + 2 Aging processes

**Program 4** (Figure 4): The solution treatment process as follows.

- A111 Solution treatment at 1125°C/1h + Cool down to 845°C + Heat up to 1125°C/1h + Cool down to 845°C + Heat up to 1125°C/1h + 2 Aging processes
- B111 Solution treatment at 1155°C/1h + Cool down to 845°C + Heat up to 1155°C/1h + Cool down to 845°C + Heat up to 1155°C/1h + 2 Aging processes
- C111 Solution treatment at 1185°C/1h + Cool down to 845°C + Heat up to 1185°C/1h + Cool down to 845°C + Heat up to 1185°C/1h + 2 Aging processes



Figure 3. Rejuvenated heat treatment process with conditions A21, B21 and C21



Figure 4. Rejuvenated heat treatment conditions of specimens A111, B111 and C111

Then the specimens were prepared with cutting, grinding, polishing and etching with SiC papers and marble etchant for microstructure analysis by Scanning Electrons Microscopy (SEM). Finally, which were measured hardness by micro-vickers hardness test.

### **Results and Discussion**

# 1. Microstructure of as-receive specimens

The as-received microstructure after serviced operation by The Electricity Generator Authority of Thailand (EGAT) for 50,000 hours are shown in Figure 5. It was found that there are various sizes of the coarse  $\gamma'$  particles, precipitating nearby throughout on the  $\gamma$  matrix. Furthermore, the very fine  $\gamma'$  particles was also found, precipitating around the bond of coarse  $\gamma'$  particles.



Figure 5. Microstructures of as-received specimen after long-term serviced operation.

# 2. Microstructures of A3, B3 and C3

The microstructures of rejuvenated heat treatment specimens according to conditions A3, B3 and C3 (solution treatment temperatures at 1125, 1155 and 1185°C, respectively) are shown in Figure 6a)-6c). After the primary and secondary aging, the solution treated at the lowest temperature (1125°C) can not completely dissolve all of the  $\gamma$ ' particles into  $\gamma$  matrix. Therefore, it was still many

the coarse  $\gamma'$  particles remain in the  $\gamma$  matrix. Conversely, microstructure shows the uniform fine  $\gamma'$  particles in matrix at highest solution temperature. This was due to that the solutioning treatment with the highest temperature can completely dissolved the coarse  $\gamma'$  particles and uniformly re-precipitated after the both aging processes on  $\gamma$  matrix. Furthermore, it also shows the same size and similar cubic shape of  $\gamma'$  particles.



Figure 6. Microstructures of specimens after rejuvenated heat treatment of program 1.

## 3. Microstructures of A12, B12 and C12

Figures 7a)-7c) show microstructures of specimens after rejuvenated heat treatment with conditions A12, B12 and C12 (solution treatment temperatures at 1125, 1155 and 1185°C, respectively), respectively. It was due to that  $\gamma$ ' particles can be a little bit dissolved into the  $\gamma$  matrix in the first heating cycle because the solutioning time was just 1 hour. Furthermore the duration of temperature dropping and reheating after the first hour of

solution treatment, it could re-precipitate the fine  $\gamma'$  particles in the matrix. Thus this effected on increasing in sizes and numbers of  $\gamma'$  particles again. In addition, the next 2 hours of final solutioning duration was not long enough to dissolve these occurred coarse  $\gamma'$  particles to the  $\gamma$  matrix. However when the solutioning temperature was increased, size of  $\gamma'$  particles, it resulted in finer, which is very similar and uniform as figure 6c).



Figure 7. Microstructures of specimens after rejuvenated heat treatment of program 2.

#### 4. Microstructures of A21, B21 and C21

The obtained microstructures of specimens after rejuvenated heat treatment with conditions A21, B21 and C21 (solution treatment temperatures at 1125, 1155 and 1185°C, respectively) are shown in Figures 8a)-8c). From figure 8, 7c), it was found that the highest solution temperature could influenced on more highly dissolve almost coarse  $\gamma'$  particles into the  $\gamma$  matrix. In these cases, there was also re-precipitating during the first duration of solutioning temperature dropping. However, the next 1- hour solutioning cycle was not enough to completely dissolve the coarse  $\gamma'$  particles to finally obtain the uniform particles dispersion and same size. Hence, this caused the coarse  $\gamma'$  particles still remaining and being larger in size than that of particles of figure 7c).



Figure 8. Microstructures of specimens after rejuvenated heat treatment of program 3.

#### 5. Microstructures of A111, B111 and C111

Figures 9 a) - c) show rejuvenated microstructures after reheat treatment with solution temperature dropping according to conditions A111, B111 and C111, respectively. it was found that, two times of temperature dropping influenced to extremely reprecipitation on  $\gamma$  matrix with larger  $\gamma'$  particles than those of figure 8a)-8c). Furthermore, the last 1 hour time of solution treatment process was not enough also to dissolve  $\gamma'$  particles into the  $\gamma$ matrix again. And these figures were found those have the largest  $\gamma'$  particles in  $\gamma$  matrix of all programs.



Figure 9. Microstructures of specimens after rejuvenated heat treatment of program 4.

#### 6. Image analysis of rejuvenated microstructures

Figures 10 a) and b) show percentage area of coarse and fine  $\gamma'$  particles respectively, in microstructures from rejuvenated heat treatment condition with temperature dropping during solution treatment. From these results, it was found that the higher **temperature of solution treatment** resulting in the decreasing of coarse  $\gamma'$  particles percentage area but the percentage area of fine  $\gamma'$  particles was increased in the  $\gamma$  matrix. This was due to that the higher temperature of solution treatment, the coarse  $\gamma'$  particles could be me dissolved into the  $\gamma$  matrix. After both aging processes, the fine  $\gamma'$  particles can be observed if they were not completely dissolved.

The durations of temperature dropping during solution treatment process can be understood, if there was a dropping in initial solutioning duration, it would result in decreasing of coarse  $\gamma'$  particles in final size and density increasing of fine  $\gamma'$ particles. Due to the existed duration of temperature dropping could reprecipitate new  $\gamma'$  particles and accererate the coarsening of the coarse  $\gamma'$  particles, which could lead to larger size of them. Furthermore, the effect of final heating time was very important on  $\gamma'$  particles dissolvability. It was found that the longer soaking time could more dissolve coarse  $\gamma'$  particles.

Moreover, if the number of temperature dropping cycles was. This would provide the increasing in coarse  $\gamma'$  particles density and the decreasing in fine particles density resulting in the undesired final microstructures. The numbers of temperature dropping cycles could be also related with last duration of heating time, which influence the find microstructure.



Figure 10. Relationship between reheat treatment conditions and percentage area of  $\gamma$ ' particles.

## 7. Microhardness Results

Figure 11 shows the results of microhardness (HV) of the specimens from various rejuvenated heat treatment conditions with temperature dropping during solution treatment. It was found that, the

hardness values are in the range of 380-450 MPa. In addition, it should be noted that the microstructures of specimens A12, B1, A21, and B21with bimodal structure provided the higher hardness than those of the single  $\gamma$ ' structures.



Figure 11. Relationship between heat treatment conditions and microhardness.

# Conclusions

- 1. Temperature dropping during solution treatment has damage to the final repaired microstructures.
- 2. Higher solution temperature could more completely dissolve  $\gamma'$  particles into the  $\gamma$  matrix, which has an effect on decreasing percentage area of coarse particles and fine particles increase. Therefore, the following duration time of next solutioning cycle must be long enough to dissolve all coarse  $\gamma'$  particles.
- 3. The initial temperature dropping during solution treatment has less detrimental effect than occurring was the end. The duration of final time and temperature of fine cycle have more effect on dissolve the  $\gamma'$  particles than the previous cycles.
- 4. More numbers of temperature dropping during solution treatment resulted in less homogeneous microstructure of rejuvenated microstructures.
- 5. The acceptable microstructure of rejuvenated heat treatment with temperature dropping during solution treatment is only condition C12 (Solutioning of temperature 1185°C).
- 6. Bimodal structures provide higher hardness than those of single structure.

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