Journal of Metals, Materials and Minerals. Vol.15 No.2 pp.69-79, 2005

Effect of Heat Treatments after HIP Process on Microstructure Refurbishment in Cast Nickel-Based Superalloy, IN-738

Panyawat Wangyao [1*], Gobboon Lothongkum [2]
Viyaporn Krongtong [3], Saruti pailai [4] and Sureerat Polsilapa [5]

1 Metallurgy and Materials Science Research Institute (MMRI), Chulalongkorn University, Bangkok, Thailand
2 Metallurgical Engineering Dept., Faculty of Engineering, Chulalongkorn University, Bangkok, Thailand
3 National Metals and Materials Technology Center (MTEC), Pathumthani, Thailand
4 Dept. of Applied Physics, Faculty of Science, King Mongkut's Institute of Technology, Ladkrabang, Bangkok, Thailand
5 Materials Engineering Dept., Faculty of Engineering, Kasetsart University, Bangkok, Thailand

Abstract

The aim of this research work is to investigate the most convenient and practicable repair procedure, which could provide the appropriate microstructure characteristics in exposed turbine blades. The blades were cast from nickel-based superalloy IN-738 and were long-term exposed under conditions of land base gas turbine operation for 50,000 hours by EGAT of Thailand. The rejuvenation process includes the hot isostatic pressing (HIP) which is followed by a series of various heat treatment conditions involving either precipitation aging and or both solutioning and precipitation aging. The HIP was carried out at a pressure of 100 MPa for 5 hours at 1,200°C Then HIPed specimens were vacuum heat treated. The hot isostatic pressing could mostly heal any internal structural voids and cracks, which were generated during service. It was found that any microcrack was observed after the HIP process was exposed for 5 hours. However, microvoids were still found even after the HIP process but in very small amounts. Therefore, both the size and the amount of remained microvoids should be evaluated with respect considering both obtained virgin original material and after long-term exposed material. Furthermore, during solution treatment, coarse carbides and over-exposed gamma prime precipitates, which were formed and modified previously at the grain boundaries during service by the creep mechanism, are expected to the matrix. Then specimens were processed through a series of precipitation aging, which re-precipitates the strengthening phase to form the proper morphology in size and shape as well as distribution that is almost similar to the new one. Metallurgical examination of the microstructure had been performed by utilizing an optical microscope and a scanning electron microscope after hot isostatic pressing and heat treatment to evaluate the micro-defects elimination.

Keywords: Nickel base superalloy structure degradation Hot Isostatic Pressing (HIP), Rejuvenation, Re-heat treatment, Microstructural healing

---

∗ To whom correspondence should be addressed:
E-Mail: panyawat@hotmail.com Tel: (+662) 218 4233; Fax: (+662) 611 7586

Introduction

Nickel-based superalloys are structural materials with each chemical composition and structure, which have been developed to be utilized in high temperature applications. The microstructures and mechanical properties of these alloys can be related to their manufacturing processes. One of these processes is heat treatment, when solution treatment in most cases is followed by a single or a double aging sequence to precipitate homogeneous distributions of either cuboidal or spherical gamma prime in the grains interior as well as discrete grain boundary carbides(1). Full solution treatment or partial solution treatment temperatures including aging treatments have been developed and modified with respect to optimize the precipitation of gamma prime phase in matrix.

The mechanical property behaviors of superalloys are very strongly related to the alloy microstructures. The size morphology, volume fraction and distribution of gamma prime phase are vital to control the creep strength at high to intermediate 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 superalloy microstructures continually change with hold-time at the elevated temperatures. In the new, heat treated alloy, the gamma prime (γ') particles are arranged in a structure, which results in an optimum balance of tensile, fatigue, and creep properties(2) the mechanical properties are related to the microstructures. Therefore, many previous research works(3-8) had been carried out to investigate these relationships of microstructure-mechanical properties.

However, the use of these expensive materials is usually employed with power generation where it requires a repair process providing the re-establishment of the initial properties and the near original microstructure of the long-term used or damaged parts for the economic reasons. The heat-treatment processes for nickel-based superalloys continue to change in order to be optimized for numerous mechanical and physical properties(9-15). This allows making the selection of heat treatment parameters increasingly challenging.

At elevated temperatures, the stability of all phases in the superalloy microstructure is very important as the occurrance of microstructure changes can result in the deterioration of creep and fatigue strengths and ductility with γ' phase size increasing with time and temperature and complex carbide reaction. The alloy's standard heat treatment is not always effective when applied to the long-term serviced microstructure to reestablish mechanical properties as well as to the welded superalloy components or HIPed superalloy parts. For example, the heat treatment cycle used for the most common industrial turbine rotating blade material, a cast polycrystalline IN-738 has the following steps: Solution treatment at 1,120°C for 2-4 hours followed by a rapid gas quench (25°C to 55°C /minute) to below 650°C and) precipitate aging at 845°C for 24 hours followed by a rapid gas quench to room temperature. The reason why the standard heat treatment does not often work well is that the γ' solution temperature for the alloy is in the range of 1,175°C to 1,190°C(2). Therefore, the alloy needs to be solution treated at a higher temperature for of 1,200°C to fully restore the degraited microstructure after long-term service. However, if hot isostatic pressing (HIP) is applied before the reheat treatment then an effective way to incorporate the high temperature cycle is needed to restore the serviced microstructure. The high temperature used at during the HIP process also assists to homogenize the microstructure.

Therefore the high-temperature strength of the alloy depends strongly on γ' properties. According to previous work(18), the rejuvenation process provides blades to double and in some cases, triple the lifetime as compared to the original ones. For alloys such as IN 738, IN 792, U 500, X-750 and the newer alloys such as GTD 111, GTD 111DS, R80DS, and IN 939, which are used in many land base gas turbines, have been rejuvenated and successfully returned to service according to the previous information of LIBURDI Engineering Company Limited, Canada. In each case, the blades were creep life expired when received for processing and then gave reliable service after rejuvenation. The aim of this research work is to investigate and obtain the most suitable and practicable repair-conditions, which provides the improved microstructural characteristics by the rejuvenation method of hot isostatic pressing (HIP) and followed by various heat treatments of long term exposed gas turbine blades, casting nickel-based superalloy after 50,000-hours of service operated by Electricity Generating Authority of Thailand (EGAT).

Materials and Experimental Procedure

The cast nickel-based superalloy IN-738 is used as a blade material in the first row of the high pressure stage of gas turbines. The alloy contains refractory elements such as Mo, W, Ta, Cr and Co to prevent local hot corrosion[17]. The alloy has a multi-phase microstructure consisting of FCC γ matrix, bimodal γ' precipitates (primary and secondary), γ-γ' eutectic, carbides and a small amount of deleterious phases such as σ, δ, η and Laves. The total weight percent of γ' in IN-738 superalloy is higher than 60%[18]. The cast nickel-based superalloy in this study was IN-738 (see chemical composition in Table 1)

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

About 1cm^2 rectangular plates were cut from the most severe degradation zone trailing edge of turbine blades. HIP treatment: of specimens was conducted at a pressure of 100 MPa for 5 hours at 1200ºC, and then HIPed specimens were heat treated according to heat treatment conditions including solution treatment, primary and secondary precipitation aging treatments in a vacuum furnace, see experimental heat-treatment details in Table 2. Heat treated plates were cross sectioned in order to observe the microstructures compared to those of the parallel grinded and polished surface of the turbine blades. All sectioned samples were polished using standard metallographic techniques and were subsequently etched in marble etchant, which has the following chemical composition as following 10 g. $CuSO_4$, 50 ml HCl, and 50 ml H_2O. The microstructures of heat treated samples were studied with a scanning electron microscope with secondary electron mode and Image Analyzer.

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

No.	Solution Treatment	Primary precipitate aging	Secondary precipitate aging
1 *	-	-	845°C/ 24 hr. (AC)
2	-	925°C/ 1 hr. (AC)	845°C/ 24 hr. (AC)
3	-	1055°C/ 1 hr. (AC)	845°C/ 24 hr. (AC)
4	1125°C/ 2hr. (AC)	-	845°C/ 24 hr. (AC)
5	1125°C/ 2hr. (AC)	925°C/ 1 hr. (AC)	845°C/ 24 hr. (AC)
6	1125°C/ 2hr. (AC)	1055°C/ 1 hr. (AC)	845°C/ 24 hr. (AC)

∗ Standard Heat-Treatment condition

Results and Discussion

As-received Microstructure

Optical microscopy micrographs of structures, obtained from the transverse sections at about mid blade height of the airfoil, are shown in Figure 1. The microstructure of as-cast alloy generally consists of an extensive precipitation of ordered $L1_2$ γ' intermetallic phase within a dendrite core and in the interdendritic region. Carbides / carbonitrides predominantly MC type, borides, sulphur-carbide and γ-γ' eutectic which form during ingot solidification are found in smaller fractions located along the interdendritic region as well, according to works[19]. Microsegregation during ingot solidification causes the formation of non-equilibrium γ- γ'eutectic. The chromium carbide ($M_{23}C_6$) and agglomerated gamma prime and secondary gamma prime particles can be seen in structure. Coalescence of the primary and secondary gamma prime particles, as a result of long-term service, seems to occur resulting in larger and rounded particles. It was also reported that eutectic gamma prime islands as well as elongated gamma prime partials are observed as well in the works[20-21]. This is most probably due to too much thermal and stress exposure.

Work[2], reported that some of the grain boundary carbides had precipitated during long-term service at elevated temperatures (760°C to 982°C), where chromium carbide could precipitate. Usually, in the non-degraded root sections, the carbides are discontinuous along the grain boundaries and provide grain boundary strength. However, coarsening of these precipitates and the formation of continuous grain boundary carbide and/or gamma prime phases normally lowers ductility or toughness of the blade alloy. This can lead to lower creep strength and impact resistant of the blades.

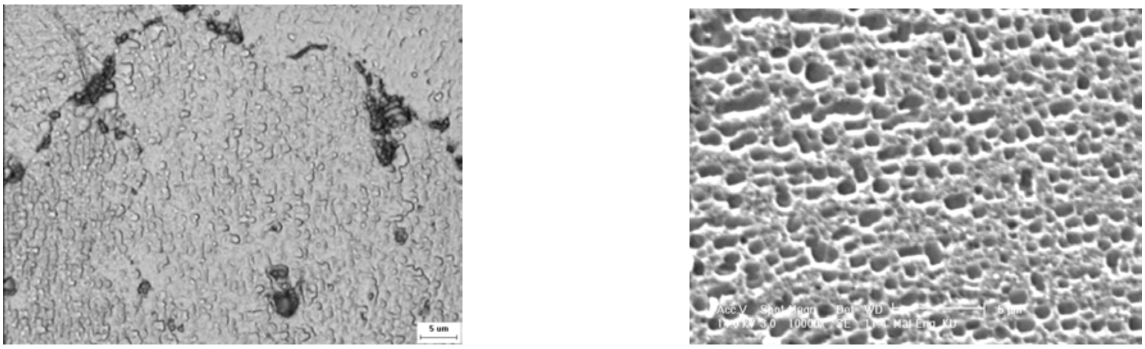

Figure 1. As-received microstructure after long-term service showing the coarsening of γ' particles, areas of γ-γ' eutectic and grain boundary carbides, Light microscopy (LM) (Left) and SEM (Right)

The degree of degradation, as measured by the gamma prime particle size, increases with exposure time and service temperature. The primary gamma prime particle size varied from 1.0 to 1.4 micron and secondary gamma prime particle size was in the range of 0.15 to 0.35 micron, as reported by previous work[2]. In this study, however, the coarse gamma prime particle size was about 1.2 micron. The primary gamma prime particles have spheroidized and secondary gamma prime coarsened in the airfoil samples. This type of microstructure is theoretically expected to have low efficiency to block dislocation movements during loading at high temperatures resulting in lower creep resistance. Therefore, it is needed to recover the microstructures to the same or similar as the original one by simple re-heat-treatment processes.

The SEM observation of all specimens after long-term service was supposed to confirm the progress of internal voids, as shown in Figure 2. The SEM analysis of un-etched specimens shows the presence of micro-voids located both in the matrix and at the carbides, which had been developed during service under long-term stress and temperature. The diameter size and amount of micro-voids depends on the location of the turbine blade. It was found that the zone of the airfoil tips, upper parts of the turbine blade, consist of a greater amount and bigger diameter size of micro-voids. This was because of the effect of the much higher temperature the blade was exposed to during operation supporting the higher rate of diffusion for internal void nucleation and growth.

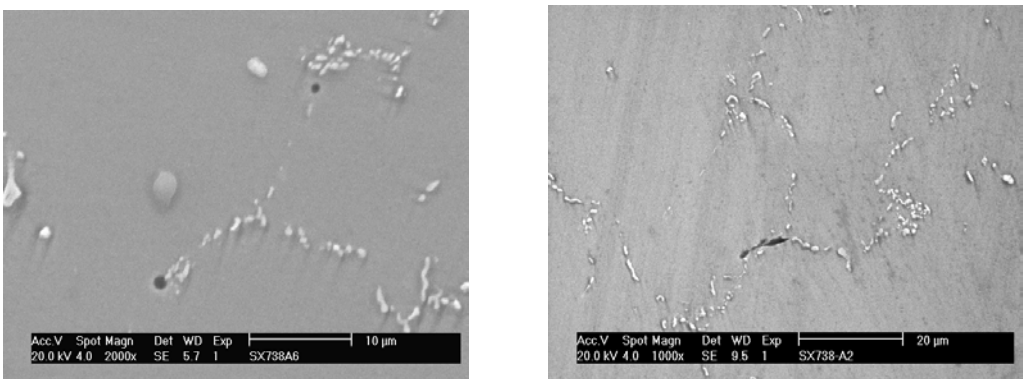

Figure 2. As-received microstructure after long-term service showing microvoids

- **HIPed microstructure**

The HIP process temperatures were selected so that the alloy yielded or crept in compression under the action of the applied pressure. The result is elimination of internal voids (porosity) and/or microcracks as well as nearly full densification of the alloy. HIP is able to almost remove internal voids and promote diffusion bonding across the surfaces of the void, which is replaced by continuous material. Higher temperature and longer HIP time show the smaller internal void diameter. As it was already known that the higher temperature and longer time during the HIP process provides the significant beneficial effect of void healing in the sintering process. The role of applied temperature and time is to increase the diffusion rate and diffusion time to take place across the interface for local yielding and creep, which can increase the real area of contact.

Nevertheless, this information is probably not enough to indicate that these HIP conditions are not proper to close all microvoids completely. It should be noted that HIP dose not only reduces the microvoid diameters, which should be counted, but also the amount of remaining microvoids. It was found that the amount of remaining microvoids decreased drastically when HIP time was increased. Therefore, by SEM investigation of specimens, it was found that the microvoids were very rarely to be detected. This fact is very important to consider about the advantages of the HIP process for the refurbishment of superalloy components. Figure 3 shows the etched microstructure of the HIPed specimen with the uniform dispersion of less coarse gamma prime particles, which previously were partially dissolved into the matrix during HIP at high temperature. It should be noted that the previous coarse gamma prime particles could not be solutioned at 1200°C for 5 hours.

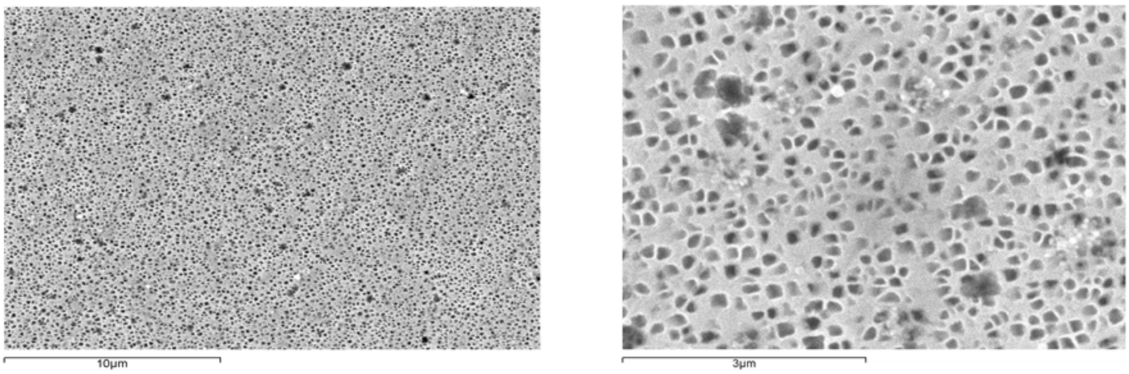

Figure 3. HIPed Microstructure showing partial dissolved gamma prime particles

- Heat-treated microstructure after HIP

The microstructures of the HIPed specimen followed with the secondary aging are shown in Figure 4. The heat treated microstructure according to program schedule No.1 contains a the homogeneous distribution of very fine γ’ particles precipitated in the matrix as a nearly cubic (butterfly) shape. It should be noted that the microstructure consists of only a single size of precipitated γ’ particles, which have the size in the range of 0.2-0.4 µm. In this structure, it could be considered that heating during the HIP process is contributing to solution treatment (at 1,200°C for 5 hours) which was followed by simple secondary aging (at 845°C for 24 hours). The previous precipitated γ’ particles could almost be dissolved into the matrix during the HIP process. Therefore, when secondary aging was applied then the γ’ precipitates could reprecipitate again in a very fine size of uniform distribution.

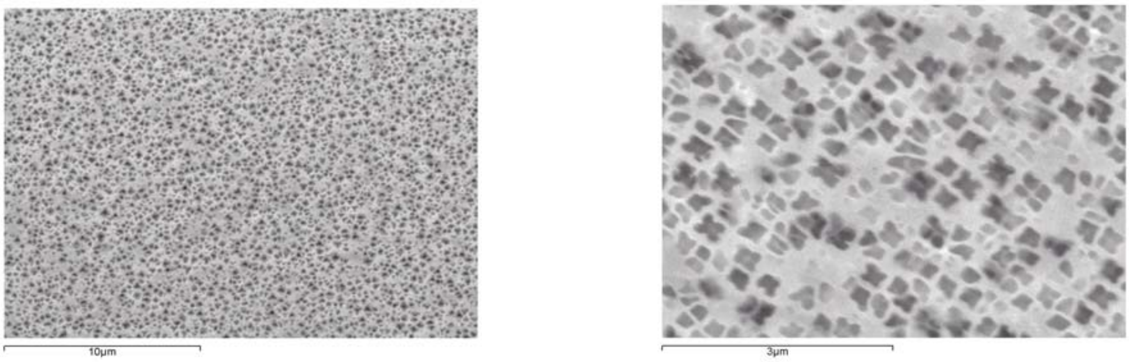

Figure 4. After heat-treatment at 845°C / 24 hr. (AC); Condition No. 1

Figure 5 shows the heat-treated microstructures according to schedule No. 2 with the additional primary aging at 925^{o}C for 1 hours. The microstructures are very similar to the heat-treated microstructure of schedule No.1. The received microstructure consists of a uniform distribution of fine precipitated γ’-precipitate of uniform size in the range of 0.25-0.4 µm. However, comparing microstructures, it can be seen that the added primary aging at 925^{o}C for 1 hours could result in a more uniform precipitation of γ’ precipitates with a higher volume fraction and γ’ particles becoming more cubic in shape.

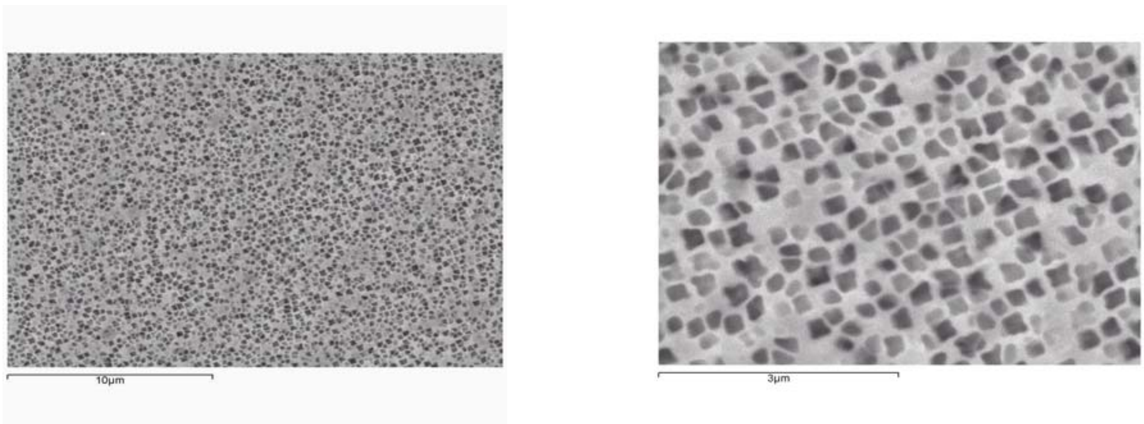

Figure 5. After heat-treatment at 925°C / 1 hr. (AC), and 845°C / 24 hr. (AC); Condition No. 2

Figure 6 shows the heat treated microstructure according to schedule No. 3 primary aging inserted at 1,055°C for 1 hour between solutioning and secondary aging compared to the microstructure of heat treatment schedule No. 2. It could be noted that this inserted primary aging with a higher temperature resulted in a more uniform distribution of finer γ' particles. Furthermore, these γ' particles also became closer to a cubic shape with a single size in the range of 0.2-0.4 µm.

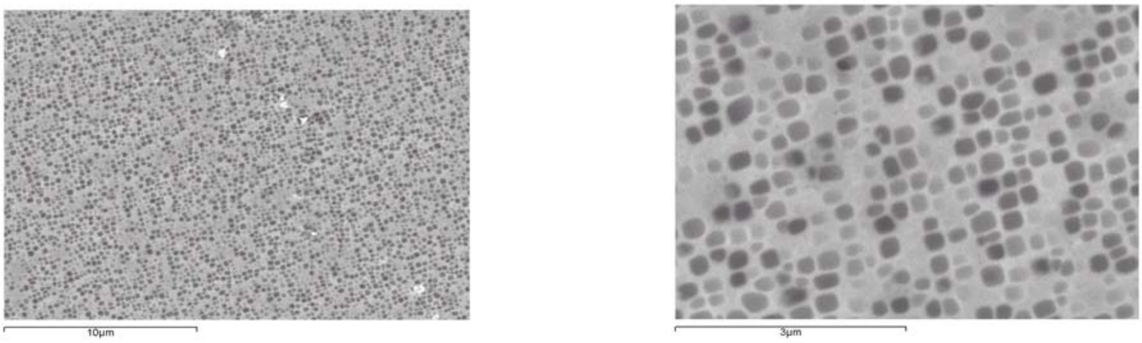

Figure 6. After heat-treatment at 1055°C/ 1 hr. (AC), and 845°C/ 24 hr. (AC); Condition No. 3

Figures 7-9 show microstructures resulting from the HIP followed by the additional solutioning prior to secondary aging and/or with primary and secondary aging. The additional solutioning provides the different of microstructure characteristics compared to those of heat-treated microstructures without solutioning. Heat treated microstructures according to schedule No. 4 shows the heterogeneous distribution of single sized precipitated γ' particles after secondary aging at 845°C for 24 hours Figure 7. The additional solutioning step could result in the early step precipitation of very fine γ' particles which could continue to grow in coarser cubic particles during secondary aging. In microstructures, there were not observed the fine precipitated particles.

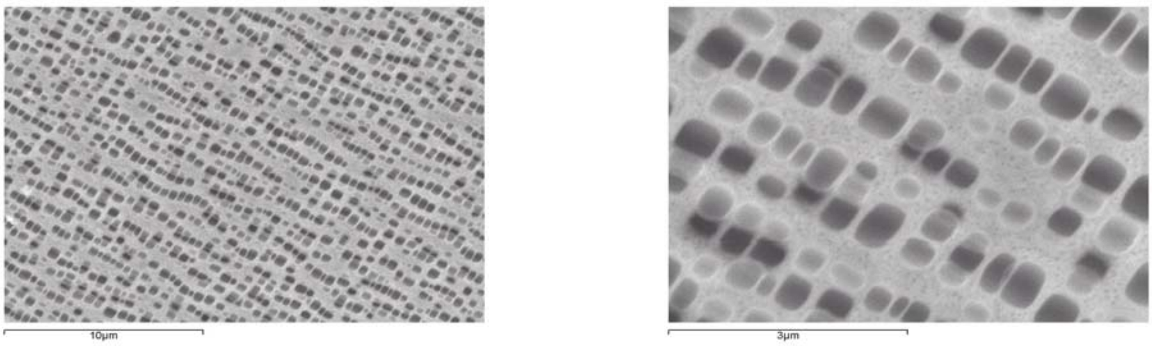

Figure 7. After heat-treatment at 1125°C / 2hr. (AC) and 845°C / 24 hr. (AC); Condition No. 4

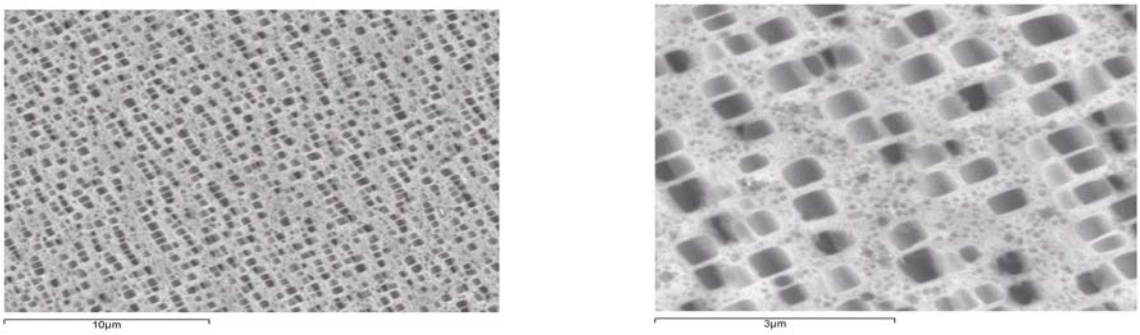

Figure 8. After heat-treatment at 1125°C/ 2hr. (AC), 925°C / 1 hr. (AC), and 845°C / 24 hr. (AC); Condition No. 5

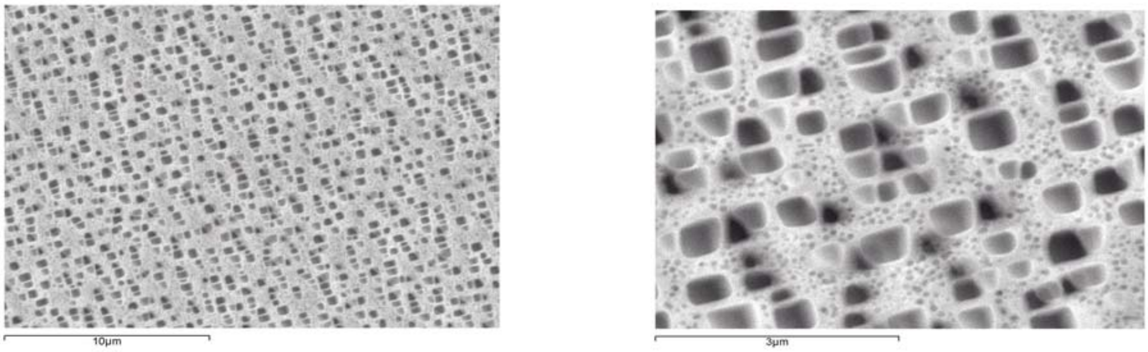

Figure 9. After heat-treatment at 1125°C/ 2hr. (AC), 1055°C/ 1 hr. (AC), and 845°C/ 24 hr. (AC); Condition No. 6

When the inserted primary aging was applied between the solutioning and the secondary aging step, the very fine precipitates of γ' particles found the coarse cubic γ' particles; as shown in Figures 8 and 9. This might be due to that the inserted primary aging allowed the microstructures to precipitate these very fine γ' particles in a very small size before secondary aging and continuing to grow in some degree during the last aging. However it could be seen that the heat treated microstructure according to schedule No.4 is in a slightly higher volume fraction of coarser γ' particles compared to those of schedules No.5 and 6.

Figure 8 shows the HIPed microstructure after primary aging at 925°C for 1 hours and then secondary aging applied at 845°C for 24 hours, The resulting structure consists of a homogeneous distribution of bimodal precipitated γ'-particle sizes. The microstructure consists of the coarser cubic γ'-particle with size in the range of 0.2-0.5 µm and the very fine γ' particle. Such micro-structures are also found in heat-treated micro-structure according to schedule No. 6 with a higher temperature during primary aging (1,055°C/1hr). However, the volume fraction of γ' particles is slightly less than that of heat-treated micro-structures according to schedule No.5. This might be driven by the lower primary aging temperature which is a more proper aging temperature, and could provide slightly more reprecipitated γ' particles than when employing the higher one.

Conclusion:

1. The heat-treated microstructures (after HIP) without the solutioning step show the homogeneous distribution of finer precipitated γ' particles compared to those heat treated microstructures with solution treatment.

2. The inserted primary aging provide more uniform precipitation of γ' particles as well as these γ' particles becoming closer to the cuboidal morphology cubic shape (especially in higher temperature aging) when considering heat treated microstructures of schedule No. 1-3.

3. The additional solution treatment according to schedules No.4-6, results in the homogeneous distribution of precipitated γ' particles however a of coarse cubic shape. The inserted primary aging results in the uniform precipitation of γ' precipitated particles among coarser cubic γ' particles.

4. Heat-treated microstructures according to schedule No.4-6 should be more proper for long term service, especially in creep load conditions. Microstructures according to schedules 5 and 6 should provide the better and balanced combination of tensile and creep strength due to the bimodal γ' particles.

Acknowledgement

This research was financial supported by the grant of Thai Research Fund (TRF), Bangkok, Thailand. Special thanks is also extended to Electricity Generating Authority of Thailand (EGAT), Nonthaburi, Thailand for material supports and technical helps.

References

(1) Zrnik, J., Strunz P., Vrchovinsky, V., Muransky, O., Novy, Z. and Wiedenman, A. 2004. *Mater.Sci. Eng.* **A 387-789** : 728-733.

(2) Daleo, J. A., Ellison, H. A. and Boone, D.H. 2002. *J. Eng. . Gas Turbines .Power*. **124** (July) : 571-579.

(3) Zrník, J., Horňak, P., Pinke, P., Žitňanský, M. 1996. *Creep Fatigue Characteristics of Single Crystal Nickel Base Superalloy CMSX 3, Metals Alloys Technologies*. (Kovine zlitine technologije), 3-4, Ljubljana, Slovenia. : 179 – 183.

(4) Zrník, J., Wang, J.A., Yu, Y., Peijing, L. and Horňak, P. 1997. Influence of cycling frequency on cycling creep characteristics of nickel base single crystal superalloy. *Mater. Sci. . Eng*. **A234-236** : 884 – 888.

(5) Wangyao, P., Nisaratanaporn, E., Zrník, J., Vrchovinský, V., Horňak, P. 1997. High temperature properties of wrought nickel base superalloy in creep - fatigue conditions. *J.Met. Mater. Miner*. **7 (1)** : 1 – 12.

(6) Zrník, J., Wangyao, P., Vrchovinský, V., Horňak, P. and Mamuzič, I. 1997. Deformation behavior of Wrought Nickel Base Superalloy in Conditions of Thermomechanical Fatigue. *Metalurgija (Metallurgy)* **36** (4) : 225 – 228.

(7) Zrník, J., Huňady, J., Horňak, P. 2002. Nickel Based Single Crystal Superalloy CM 186 – potential Material for Blades of Stationary Gas Turbine. *Metalurgija (Metallurgy).* **41** (3) : 232.

(8) Zrník, J., Strunz, P., Horňak, P., Vrchovinský, V. and Wiedenmann, A. 2002 Microstructural changes in long-time thermally exposed Ni-base superalloy studied by SANS. *Appl. Phys.* **A 74** : S1155-S1157.

(9) Zrník , J., Semeňák, J., Wangyao, P., Vrchovinský, V., Horňak, P. 2003. The Analysis of Low Cycle Fatigue Behaviour in a Nickel Based Superalloy. *J.Met. Mater. Miner.* **12 (2)** : 33 – 40.

(10) Zrník, J., Jenčuš, P., Lukáš, P., Horňak, P., Wangyao, P.2004. Stress Evolution in Nickel Based Single Crystal Superalloy Subjected to Thermal Cycling, *J. Met. Mater. Miner*. **13 (2)** :. 25 – 31.

(11) Wangyao, P., Zrník, J., Nisaratanaporn, E., Vrchovinský, V., Horňak, P. 2004. The Study of Isothermal and Anisothermal Deformation Behaviors of Wrought Polycrystalline Nickel Based Superalloy. *J.Met. Mater. Miner.* **13 (2)** : 55 – 63.

(12) Zrník, J., Strunz, P., Vrchovinský, V., Muránsky, O., Horňak, P. and Wiedenmann, A. 2004. Creep deformation and microstructural examination of a prior thermally exposed nickel base superalloy. Key Eng. Mater. **274 - 276**, : 925 – 930.

(13) Jenčuš, P., Zrník, J., Lukáš, P. and Horňak P.2004. Thermal fatigue aspects in nickel base single crystal superalloy. *Acta Metall. Slovaca.* **10** : 487 – 493.

(14) Zrník, J., Strunz, P., Vrchovinský, V., Muránsky, O., Horňak, P. and Wiedenmann, A. 2004. Evaluation of structure stability in thermally exposed nickel base superalloy. *Acta Metall. Slovaca.* **10** : 448 – 453

(15) Zrník, J., Semeňák, J., Horňak, P. and Vrchovinský, V. 2004. Lifetime behaviours of the wrought nickel base superalloy subjected to low cyclic fatigue with holds. *Kovové Materiály – Metallic Materials.* prijaté do tlače **15** : 12.

(16) Sajjadi, S.A., Nategh, S., and Guthrie, R.I.L. 2002. *Mate. Sci. Engi.* **A 325** : 484-489.

(17) Nategh S. and Sajjadi S. A. 2003. *Mater. Sci. Eng.* **A 339:** 103-108.

(18) www.liburdi.com

(19) Hoffelner, W., Kny, E., Stickler, R., and McCall, W. J. Z. 1979. Werktoff . **10** : 84.

(20) Ojo, O. A., Richards, N. L. and Chaturvedi M. C. 2004. *Scripta Materilia*. **50** : 641-646

(21) Sawaminathan, V. P., Cheruvu, N. S., Klien, J. M. and Robinson, W. M. 1998. *The American Society of Mechanical Engineers (ASME)* June.