COMPARING REJUVENATED MICROSTRUCTURES AFTER HIP PROCESS AND DIFFERENT HEAT TREATMENTS IN CAST NICKEL BASE SUPERALLOYS, IN-738 AND GTD-111 AFTER LONG-TERM SERVICE

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POROVNANIE ZOTAVENÝCH MIKROŠTRUKTÚR PO PROCESE IZOSTATICKÉHO LISOVANIA ZA TEPLA A ROZDIELNYCH SPÔSOBOCH TEPELNÉHO SPRACOVANIA ODLIEVANÝCH NIKLOVÝCH SUPERZLIATIN IN-738 A GTD-111 PO DLHODOBEJ PREVÁDZKE

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Abstrakt

Cieľom príspevku je porovnať vplyv podmienok výsledného tepelného spracovania na proces zotavenia mikroštruktúry dvoch dlhodobo exponovaných odlievaných polykryštalických niklových superzliatin. Príspevok je taktiež pokusom získať vhodnejšie a praktickejšie podmienky obnovy mikroštruktúrnych charakteristík superzliatin IN-738 a GTD-111 cestou ich zotavenia využitím metódy izostatického lisovania za tepla a následne rozdielnymi podmienkami tepelného spracovania. Niklové superzliatiny boli predtým dlhodobo prevádzkované spoločnosťou Electricity Generating Authority of Thailand (EGAT). Metódou izostatického lisovania za tepla je možné odstrániť odstrániť niektoré vnútorné póry a trhliny (využitím spekania), ktoré sa vytvorili počas prevádzky. Bolo zistené, že po dlhšom čase procesu izostatického lisovania za tepla (5 hodín), nebola pozorovaná v štruktúre materiálu žiadna mikrotrhlina. Hoci je nutné podotknúť, že mikropóry boli pozorované dokonca aj po 5 hodinách procesu izostatického lisovania za tepla pri teplote 1200°C, ale vo veľmi malom množstve. Preto je potrebné študovať veľkosť a množstvo zvyškových mikropórov, tak získaného originálneho materiálu, ako aj materiálu po dlhom čase prevádzkovania. Okrem toho, počas rozpúšťacieho žíhania, hrubé karbidy a primárne precipitáty gamma, ktoré sa vytvorili počas prevádzky na hraniciach zŕn creepovým mechanizmom sa môžu rozpustiť v matrici materiálu. Následne boli vzorky spracované sériou procesov starnutia, pri ktorých opätovne vyprecipitovala spevňujúca fáza majúca požadovanú veľkosť, tvar ako aj rozloženie, takmer rovnaké ako mala pôvodná vzorka. Na kontrolu odstránenia mikrodefektov z mikroštruktúry materiálu po procese izostatického lisovania za tepla a následného tepelného spracovania bola využitá riadkovacia elektrónová mikroskopia.

Abstract

The present work has an aim to compare the effect of final heat treatment conditions of rejuvenation process on microstructures of two long-term serviced cast polycrystalline nickel base superalloys. The work has also an attempt to possibly obtain the most suitable and practicable repair-condition, which could provide the desired microstructural characteristics by rejuvenation method of hot isostatic pressing (HIP) followed by various heat treatment conditions for long-term serviced gas turbine blades, casting nickel base superalloys grade IN-738 and GTD-111, operated by Electricity Generating Authority of Thailand (EGAT). The hot isostatic pressing could mostly heal any internal structural voids and cracks (by means of sintering), which were generated during service. It was found that no any microcrack was observed after longer time of HIP process such as for 5 hours HIP time. However, microvoids were still found even after 5 hours of HIP process at 1200°C but in very small amount. Therefore, both size and amount of remain microvoids should be considered comparing to both obtained original material and after long-term service one. Furthermore, during solution treatment, coarse carbides and over-exposed gamma prime precipitates, which formed previously at the grain boundaries during service by creep mechanism, would dissolve into the matrix. Then specimens will be 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 scanning electron microscope after hot isostatic pressing and heat treatment to evaluate the micro-defects elimination.

Keywords: Hot Isostatic Pressing (HIP), Rejuvenation, Re-heat treatment, Microstructural Repair, Cast Nickel-Based Superalloy

1. Introduction

Nickel base superalloys are structural materials with chemical composition and structure, which have been developed to be utilized at high temperature applications. The microstructures and mechanical properties (for both low and high temperatures) can be related to their manufacturing processes. One of these processes is heat treatment, which 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 within 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 to optimize the completed precipitation of gamma prime phase in matrix.

The size, 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 mechanical property behaviors of superalloys are very strongly related to the alloy microstructures. The superalloy microstructures continually change with 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]. Due to mechanical properties are related to the microstructures. Therefore, many previous research works [3 - 10] had been carried out to investigate these relationships of microstructure-mechanical properties. However, the use of these expensive materials requires a repair process providing the re-establishment of the initial properties and the original microstructure of the long-term used or damaged parts for the economic reason. The heat-treatment processes for nickel-base superalloys continue to change in order to optimize for numerous mechanical and physical properties. This allows making the selection of heat treatment parameters increasingly challenging.

At elevated temperatures, the stability of all phases in superalloy microstructure is very important as the occurring of microstructure changes can result in lower creep and fatigue strengths and ductility with γ' phase increases with time and temperature and complex carbide reaction. However, the alloy's standard heat treatment does not always work properly when applied to the long-term serviced microstructure to re-establish mechanical properties as well as to the welded superalloy components or HIPed superalloy parts. For example, practical heat treatment cycles used for the most common industrial turbine rotating blade material, a cast polycrystalline IN-738 is the following step: 1) Solution treatment at 1120^{o}C for 2 - 4 hours followed by a rapid gas quench (25^{o}C to 55^{o}C/minute) to below 650^{o}C and 2) Precipitation aging at 845^{o}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 range from 1175^{o}C to 1190^{o}C [2]. Therefore, the alloy need to be solution treated at 1200^{o}C to fully restore the microstructure after long-term service. However, if hot isostatic pressing (HIP) is applied before the re-heat treatment then an effective way to incorporate the high temperature cycle needed to restore the serviced microstructure. The high temperature during HIP process also assists to homogenize the microstructure.

The cast nickel-base superalloys IN-738 and GTD-111 are generally used as a blade material in the first row high-pressure stage of gas turbines. The alloys contain refractory elements such as Mo, W, Ta, Cr and Co to prevent local hot corrosion [3]. The alloy has a multi-phase microstructure consisting 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 and GTD-111 superalloys is higher than 60 % [11 - 12]. Therefore the high-temperature strength of the alloys depend strongly on γ' properties. According to previous work [13], 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 landed-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 expiry when received for processing and then giving reliable service after rejuvenation.

The aim of this research work is to determine the most suitable and practicable repair-condition, which provides the best microstructural characteristics by rejuvenation method of hot isostatic pressing (HIP) followed by various heat treatment for long term exposed gas turbine blades, casting nickel base superalloy after 50 000-hour service operated by Electricity Generating Authority of Thailand (EGAT).

2. Material and Experimental Procedure

The cast nickel base superalloys in this study were IN-738 and GTD-111 (see the chemical composition in Tables 1 and 2)

Table 1 Chemical composition of IN-738 (wt.%)

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

Table 2 Chemical composition of GTD-111 (wt.%)

Ni	Cr	Co	Ti	Al	W	Mo	Ta	C	Fe	B
Bal.	13.5	9.5	4.75	3.3	3.8	1.53	2.7	0.09	0.23	0.01

About 1 cm^2 rectangular plates were cut from the most severe degradation zone of turbine blades. The HIP conditions is following: specimens were HIP at 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 vacuum furnace, see experimental heat-treatment details in Table 3. Heat treated plates were cross sectioned in order to observe microstructure comparing 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 chemical composition as following 10 g $CuSO_4$, 50 ml HCl, and 50 ml H_2O. The microstructures of heat-treated samples were studied by scanning electron microscope with secondary electron mode.

Table 3 Heat treatment conditions applied to long-term exposed IN-738 and GTD-111

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

∗ Standard Heat-Treatment condition

3. Results and Discussion

3.1 As-received Microstructure

Optical microscopy photographs, obtained from the transverse sections at about mid blade height of the airfoil, are shown in Fig. 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/carbonitrides predominantly MC type, borides, sulphur-carbide and γ-γ' eutectic which form during ingot solidification are found in smaller volume fraction locating along the interdendritic region as well, according to works [14].

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. Coalescence of the primary and secondary gamma prime particles, as 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 [15 - 16]. This is most probably due to the slow cooling rates. From work [2], reported that the some of these grain boundary carbides had precipitated during long-term service at elevated temperatures (760^{o}C to 982^{o}C), where chromium carbide could precipitate. Usually, in the undegraded 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 carbides and/or gamma prime phases normally lowers ductility or toughness of blade alloy. This can lead to lower creep strength and impact resistant of the blades.

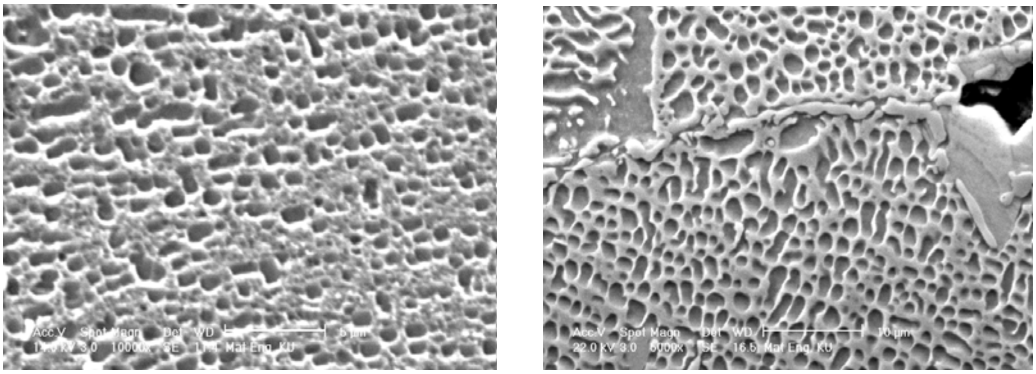

Fig.1 As-received microstructure after long-term service showing the coalescence of γ’ particles, areas of γ-γ’ eutectic and grain boundary carbides, IN-738 (Left) and GTD-111 (Right)

The degree of degradation in both alloys, as measured by the gamma prime particle size, increases with exposed time and service temperature. In this study, however, the coarse gamma prime particle size was about 1.2 and 2.5 μm for IN-738 and GTD-111, respectively. The airfoil microstructure shows significant degradation in service comparing to the microstructure of the root section. 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 microstructure to the same as or similar to the original one by simple re-heat treatment processes.

3.2 HIPed microstructure

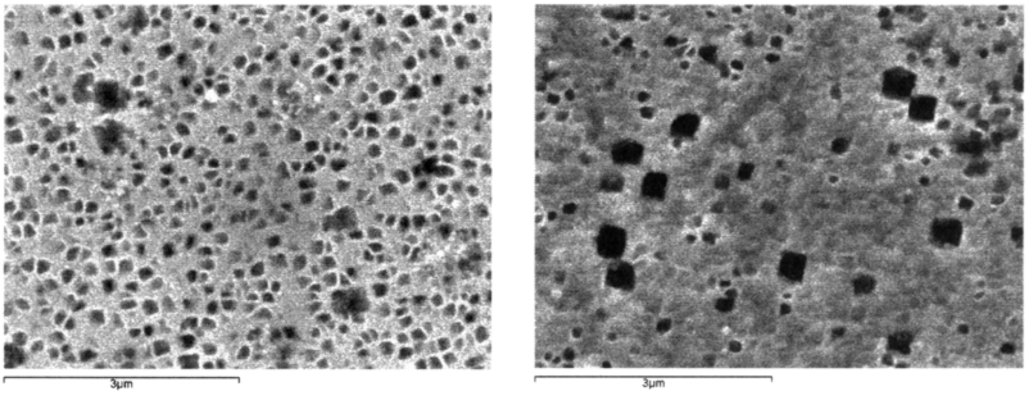

Fig.2 HIPed microstructure showing partial dissolved gamma prime particles, IN-738 (Left) and GTD-111 (Right)

It was found that the amount of remain microvoids decreased drastically when HIP time was increased. Therefore, by SEM investigation in specimens, it was found that the microvoids were very rarely to be detected. This fact is very important to consider about the advantage of HIP process for refurbishment of superalloy components. Figure 2 shows the etched microstructure of HIPed specimen with the uniform dispersion of smaller 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 completely at 1200°C for 5 hours for both alloys.

3.3 Heat-treated microstructure after HIP

The microstructures of HIPed specimen followed with the only secondary aging shown in Fig. 3. The heated treated microstructure according to program No.1 contains the homogeneous distribution of very fine γ' particles precipitating in the matrix as nearly cubic shape. It should be noted that the microstructure contains with only single size of precipitated γ' particles, which have the size in the range of 0.2 - 0.4 μm. In both cases, it could be considered that heating during HIP process also working as solution treatment at 1200°C for 5 hours and then followed by simple secondary aging at 845°C for 24 hours. The previous precipitated γ' particles could nearly be dissolved into matrix in some degree during HIP process. Therefore, when secondary aging was applied then the γ' particles could reprecipitate again in very fine size with uniform distribution.

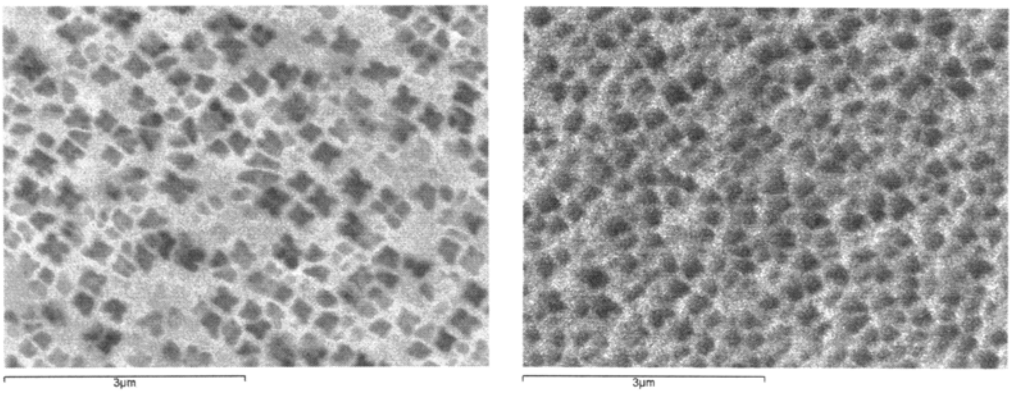

Fig.3 After heat treatment at 845°C/ 24 hrs. (AC); Condition No. 1, IN-738 (Left) and GTD-111 (Right)

Figure 4 shows the heat-treated microstructures according to the program No. 2 with the additional primary aging at 925°C for 1 hour. The microstructures are very similar to heat-treated microstructure of program No. 1. In both cases, the received microstructures consist of uniform distribution of precipitated γ'-particles as in single size in the range of 0.25 - 0.4 μm. However, comparing microstructures according to program No. 1, it can be seen that the added primary aging at 925°C for 1 hour could result in more uniform precipitation of γ' particles with slightly higher volume fraction and γ' particles becoming more cubic shape.

Figure 5 shows the heat-treated microstructure according to program No. 3 with an inserted primary aging at 1055°C for 1 hours before the secondary aging comparing to microstructure of heat treatment program No. 2, it could be noted that this inserted primary aging with higher temperature resulted in more uniform distribution of finer γ' particles, especially in microstructure of GTD-111. Furthermore, these γ' particles also became closer to cubic shape with the single size. Both microstructures are the most similar to each other after this heat treatment conditions and 0.2 - 0.4 and 0.3 - 0.6 μm for IN-738 and GTD-111, respectively.

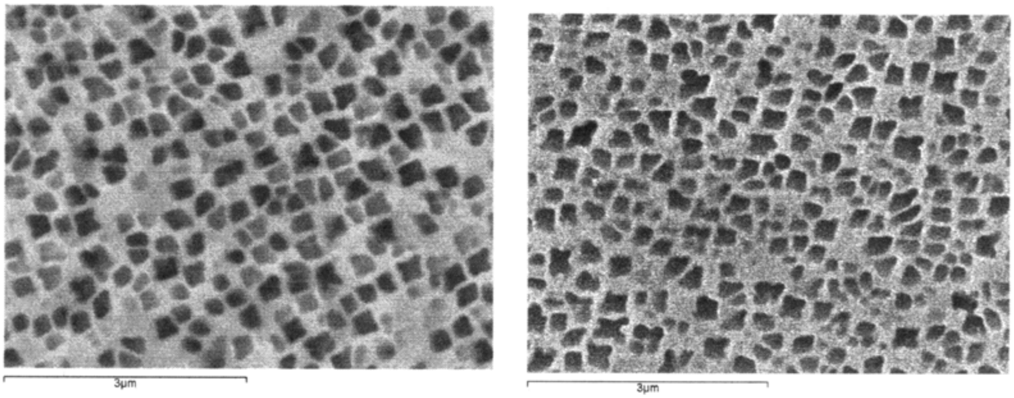

Fig.4 After heat treatment at 925°C/ 1 hr. (AC), and 845°C/ 24 hrs. (AC); Condition No. 2, IN-738 (Left) and GTD-111 (Right)

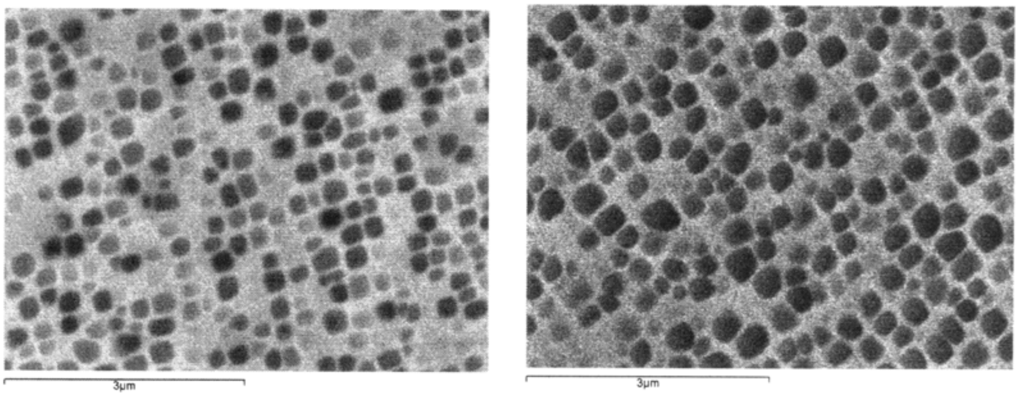

Fig.5 After heat treatment at 1055°C/ 1 hr. (AC), and 845°C/ 24 hrs. (AC); Condition No. 3, IN-738 (Left) and GTD-111 (Right)

Figures 6 - 8 show the HIPed microstructures following with the additional solutioning before the secondary aging and/or primary and secondary aging. The additional solutioning step could strongly provide the difference of microstructure characteristics comparing to those heat-treated microstructures without any solutioning. Heat-treated microstructure according to program No. 4 shows the homogeneous distribution of precipitated coarse γ' particles after secondary aging at 845^{o}C for 24 hours, Fig. 6. The additional solutioning step could result in the early step precipitation of very fine γ' particles which could continue to precipitate in coarser cubic particles during secondary aging. Very fine precipitated γ' particles were very difficult to be detected clearly in these microstructures.

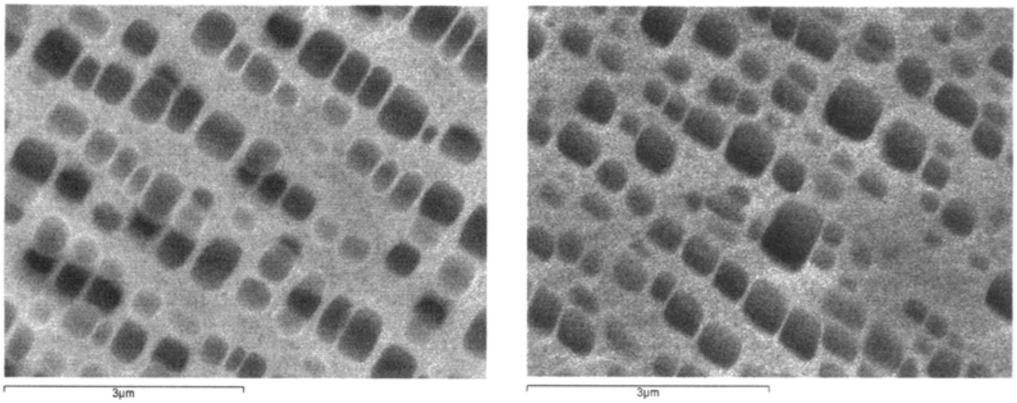

Fig.6 After heat treatment at 1125°C/ 2 hrs. (AC) and 845°C/ 24 hrs. (AC); Condition No. 4, IN-738 (Left) and GTD-111 (Right)

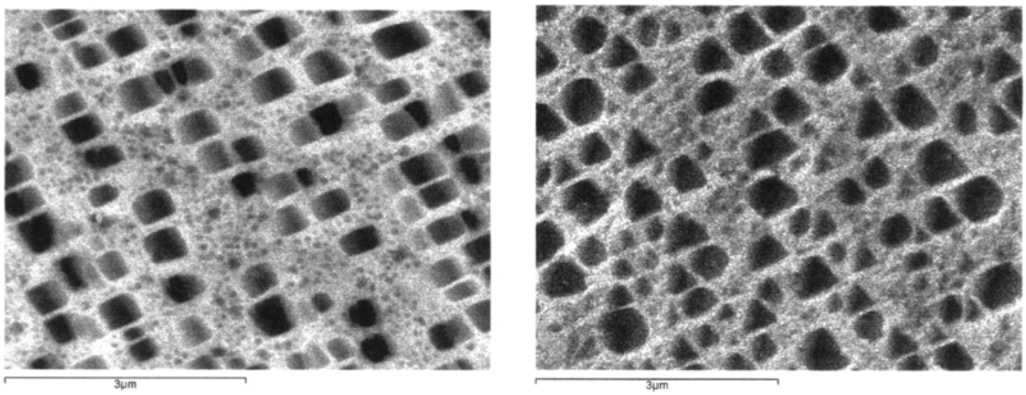

Fig.7 After heat-treatment at 1125°C/ 2 hrs. (AC), 925°C / 1 hr. (AC), and 845°C / 24 hrs. (AC); Condition No. 5, IN-738 (Left) and GTD-111 (Right)

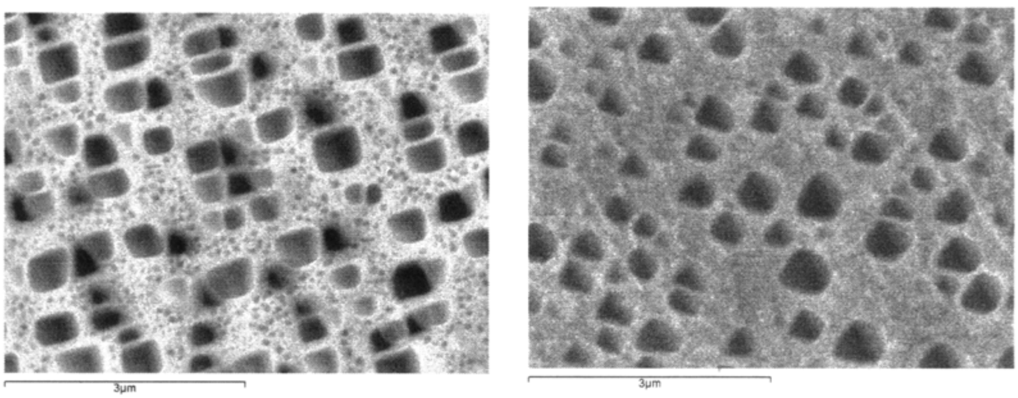

Fig.8 After heat treatment at 1125°C/ 2 hrs. (AC), 1055°C/ 1 hr. (AC), and 845°C/ 24 hrs. (AC); Condition No. 6, IN-738 (Left) and GTD-111 (Right)

When the inserted primary aging was applied between solutioning and secondary aging step, the very fine precipitation of γ' particles were obviously found among the coarse cubic γ' particles; as shown in Figs. 7 and 8, especially in case of IN-738. This might be due to that the inserted primary aging allowed microstructures to precipitate these very fine γ' particles in 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 program No. 4 is slightly higher in volume fraction of coarse γ' particles comparing to those of programs No. 5 and 6.

Figure 7 (Left) shows the HIPed microstructure of IN-738 followed with primary aging at 925^{o}C for 1 hour and then secondary aging was applied at 845^{o}C for 24 hours, which consists of homogeneous distribution of bimodal precipitated γ'-particle sizes. The microstructure consists of the coarser cubic γ'-particle with its size in the range of 0.2-0.5 µm and the very fine γ' particle as in near-round shape. Such microstructure is also found in heat-treated microstructure, Fig. 8 (Left) according to program No. 6 with the higher temperature during primary aging (1055^{o}C/ 1hr.). However, the volume fraction of γ' particles is slightly less than that of heat-treated microstructure according to program No. 5. This might be due to that the lower primary aging temperature is more proper aging temperature, which could provide slightly more reprecipitated γ' particles than utilizing the higher one.

In the case of GTD-111 according to programs No. 5 and 6 (Fig. 7 (Right) and Fig. 8 (Right), respectively), it should be noted that the inserted primary aging after solutioning had significant and negative effect on microstructure and could result in the lower driving force for next secondary aging to produce the uniform precipitation of cubic coarse γ' particles. In these cases, the higher temperature of primary aging provides the lower volume fraction of coarse γ' particles in more rounded-shape and smaller size.

4. Conclusions

1. The heat-treated microstructures (after HIP) without solutioning step show the homogeneous distribution of finer precipitated γ' particles comparing to those heat-treated microstructures with solution treatment.
2. The inserted primary aging provides more uniform precipitation of fine γ' particles as well as these γ' particles becoming closer to the cubic shape (especially in higher temperature aging) when considering heat treated microstructures of programs No.1 - 3.
3. The additional solution treatment in heat treatments of programs No. 4 - 6, results in the homogeneous distribution of precipitated γ' particles with coarse cubic shape. The inserted primary aging results in the uniform precipitation of very fine γ' particles among coarser cubic γ' particles, especially in case of IN-738.
4. Heat-treated microstructures according to program No. 4 should probably be the most proper for long-term service, especially in creep load condition for both alloys. Microstructures of IN-738 according to programs 5 and/or 6 should provide the combination of tensile and creep strength due to the bimodal γ' particles.

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