THE SEM STUDY OF MICROSTRUCTURAL RESTORATION BY RE-HEAT TREATMENTS IN CAST SUPERALLOY TURBINE BLADE

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ŠTÚDIUM ZOTAVENIA MIKROŠTRUKTÚRY ODLIEVANEJ TURBÍNOVEJ LOPATKY ZO SUPERZLIATINY PO OPÄTOVNOM TEPELNOM SPRACOVANÍ VYUŽITÍM RIADKOVACEJ ELEKTRÓNOVEJ MIKROSKOPIE

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Abstract

The purpose of this study was to examine the effect of re-heat treatment conditions on the microstructural refurbishment and development in long-term serviced gas turbine blades, which were made of superalloy grade IN-738. The microstructures of cast polycrystalline nickel base superalloy, IN-738, operated by Electricity Generating Authority of Thailand (EGAT) for long-term services (70,000 hrs) were investigated by SEM in secondary electron mode after different re-heat treatment conditions. It is well-recognized that mechanical properties such as tensile and both low and high cycle fatigue at elevated temperatures as well as creep strengths are all strongly dependent on the morphology of the tested or serviced microstructures. This is concerning to grain structure, strengthened intermetallic precipitating γ' phase (in size, shape, area distribution and volume fraction), carbide type and its morphology as well as grain (dendrite, in case of casting superalloy) boundary morphology. Therefore, re-heat treatment was linked to the manner, in which rejuvenates microstructures approached in order to re-exploit the high temperature strength of the alloy. It was found that SEM micrograph of the exposed specimen after long-term service could be nearly recovered by a re-solution treatment followed by two-step aging treatments. During solution treatment, the coarse carbides and gamma prime (γ') precipitates were partially dissolved into the matrix. Then specimens were heat treated through series of aging resulting in uniformly dispersed precipitation of gamma prime particles, which is more uniform than those in the long-term exposed microstructure. However, it was also found that the higher solution annealing temperature resulted in less volume fraction of coarse gamma prime precipitates and less homogeneous in microstructure than the lower one.

Keywords: Microstructural Refurbishment, Rejuvenation, Re-Heat Treatment, Superalloys, Lifetime Extension, IN-738

Abstrakt

Cieľom tohto príspevku bolo stanoviť vplyv podmienok opätovného tepelného spracovania na obnovenie a zlepšenie mikroštruktúry dlhodobo prevádzkovo exponovanej lopatky plynovej turbíny zo superzliatiny IN-738. Použitím riadkovacieho elektrónového mikroskopu boli študované mikroštruktúrne charakteristiky odlievanej polykryštalickej niklovej superzliatiny IN-738 po rozdielnych spôsoboch opätovného tepelného spracovania. Niklová superzliatina bola predtým dlhodobo prevádzkovaná (70000 hodín) spoločnosťou Electricity Generating Authority of Thailand (EGAT). Je všeobecne známe, že mechanické vlastnosti, ako napätie v ťahu, nízkocyklová a vysokocyklová únava pri zvýšených teplotách, ako aj pevnosť pri tečení, veľmi úzko súvisia s mikroštruktúrou. Týka sa to štruktúry hraníc, spevňujúcej intermetalickej primárnej fázy gama (veľkosť, tvar, rozloženie a objemový podiel), typu karbidov a ich morfológii, ako aj morfológie hraníc zŕn, a v prípade liatych superzliatin dendritov. Z tohto dôvodu bolo opätovné tepelné spracovanie zvolené za spôsob, ktorý umožní uzdraviť mikroštruktúru a tým opätovne zabezpečiť pevnosť zliatiny za zvýšenej teploty. Riadkovacou elektrónovou mikroskopiou bolo zistené, že mikroštruktúra vzoriek, ktoré boli v prevádzke dlhodobo exponované, môže byť takmer úplne uzdravená použitím opätovného rozpúšťacieho žíhania a následného dvojstupňového procesu starnutia. Počas procesu rozpúšťacieho žíhania došlo v matrici materiálu k čiastočnému rozpusteniu hrubých karbidických častíc a precipitátov primárnej fázy gama (γ’). Rôzne spôsoby použitého starnutia mali za následok, že v mikroštruktúre tepelne spracovaných vzoriek došlo k zrovnomerneniu rozloženia disperzných precipitátov primárnej fázy gama v porovnaní s mikroštruktúrou vzoriek, ktoré boli dlhodobo exponované v prevádzke. Taktiež bolo zistené, že vyššia teplota rozpúšťacieho žíhania mala za následok menší objemový podiel hrubých precipitátov primárnej fázy gama a menej homogénnu mikroštruktúru ako nižšia teplota rozpúšťacieho žíhania.

Introduction

Nickel base superalloys are used within the industrial gas turbine (IGT) engine leading to the need of the high temperature components. These materials can be exposed to very severe operating conditions where high tensile strength, good fatigue and creep resistance as well as oxidation and hot corrosion resistances are all required. A range of nickel base superalloys, from dilute, solid solution strengthened alloys to the highly alloyed precipitation hardened materials are available for high temperature loading performance and environmental resistance. For optimal performance, high pressure/high temperature turbine blades are generally made of nickel base superalloys. Due to mechanical properties are related to the microstructures. Therefore, many previous research works [1 - 6] 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 [7 - 13]. This allows making the selection of heat treatment parameters becoming increasingly important. The heat treatment processes for precipitation-strengthened nickel base superalloys are very complicated even they have been developed for many years. Heat treating of nickel-base superalloys can involve solution heat treating cycles, stabilizing cycles, and aging cycles. Each thermal cycle was developed and designed for very specific metallurgical structure operation and control. Each heat treatment is heated to a single and uniform temperature.

Cooling from these isothermal exposures is much more complicated. The cooling process from solution heat treatment cycles leads to, largely, the beneficial effects of gamma prime precipitation and the detrimental effects of residual stresses. Solution heat treatment has function to dissolve gamma-prime and secondary carbide phases allowing the optimum re-precipitation of these phases in next step of cooling or aging. The precipitation temperatures determine not only the type but also the size distribution of precipitates. Multiple precipitation treatments are usually common in wrought alloys but uncommon in cast alloys, [14].

Inconel 738 is a γ' precipitation-strengthened nickel base superalloy, which are widely used as turbine blades in hot sections of gas turbine engines due to its outstanding strength properties at high temperature as well as excellent hot corrosion resistance. Inconel 738 contains substantial amount of Al and Ti together more than 6 wt. %, which provides precipitation strengthening of an ordered $L1_2$ intermetallic $Ni_3(Al,Ti)$ γ' phase. The size distribution and the type of γ' precipitate are affected by aging temperature. Usually, selection of a single aging temperature may result in obtaining optimal amounts of multiple precipitating phases. Alternatively, a double-aging treatment, which is able to produce different sizes and types of precipitate at different temperatures may be utilized, [15]. Aging treatments usually are sequentially lower. During aging, not only primary strengthening precipitates (γ') but also carbides and, perhaps (under unfavorable conditions), TCP phases can form during aging. A principle reason for a need of double-aging, in addition to γ' control is to precipitate carbides or control grain boundary carbide morphology, [16].

Materials and Experimental Procedures

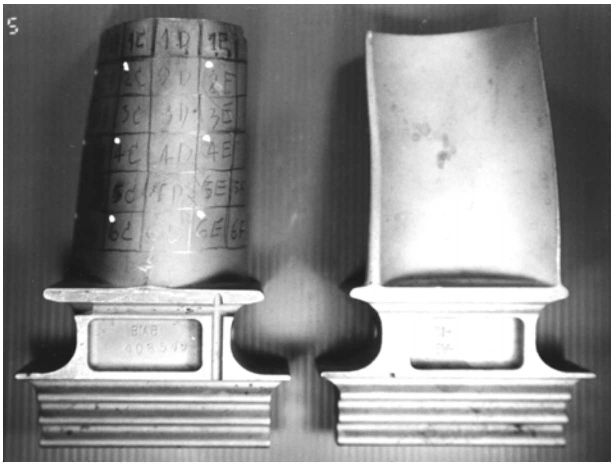

Fig.1 Cast nickel base superalloy, IN-738, turbine blade

The cast nickel base superalloy in this study was Inconel 738, with the following composition (by wt. %): Cr - 15.84, Co - 8.5, Ti - 3.47, Al - 3.46, W - 2.48, Mo - 1.88, Ta -1.69, Nb - 0.92, C - 0.11, Fe - 0.07, Zr - 0.04, B - 0.12 and balance nickel. About 1 cm^2 rectangular plates were cut from the most severe degradation zone of turbine blades, see Fig. 1. The plates were given different heat treatment conditions including solution treatment, 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 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 treatment samples were study by scanning electron microscope with secondary electron mode and Image Analyzer.

Table 1 Heat treatment conditions applied to long term exposed IN-738

No.	Solution Treatment	Primary precipitate aging	Secondary precipitate aging
1 ∗	1125°C/2 hrs. (AC)	-	845°C/24 hrs. (AC)
2	1125°C/2 hrs. (AC)	925°C/1 hr. (AC)	845°C/24 hrs. (AC)
3	1125°C/2 hrs. (AC)	1055°C/1 hr. (AC)	845°C/24 hrs. (AC)
4	1175°C/2 hrs. (AC)	-	845°C/24 hrs. (AC)
5	1175°C/2 hrs. (AC)	925°C/1 hr. (AC)	845°C/24 hrs. (AC)
6	1175°C/2 hrs. (AC)	1055°C/1 hr. (AC)	845°C/24 hrs. (AC)

∗ Standard Heat-Treatment condition

Experimental Results and Discussion

1. The microstructure of as-cast alloy

The microstructure of as-cast alloy generally consists of extensive precipitation of an 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 the previous work [17]. Microsegregation during ingot solidification also causes the formation of non-equilibrium γ- γ’eutectic, [18].

2. The microstructure of as-received alloy

Optical microscopy photographs, obtained from the transverse sections at about mid blade height of the airfoil, are shown in Fig.2 [19]. 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 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 work [20]. This is most probably due to the slow cooling rates.

From work [21], reported that the some of these 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 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 carbide 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.

The degree of degradation, as measured by the gamma prime particle size, increases with exposed time and service temperature. The primary gamma prime particle size varied from 1.0 to 1.4 μm and secondary gamma prime particle size was in the range of 0.15 to 0.35 μm, as reported by [21]. In this study, however, the primary gamma prime particle size was about 1.2 μm and secondary gamma prime particle size was about 0.25 μm. 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.

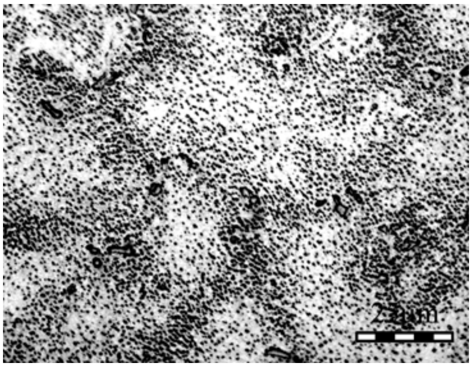

Fig.2 As-received microstructure after long-term service [19]

3. The microstructure of heat-treated alloy

According to the previous works [19, 22], repeating the standard heat treatment sequence does not always work well. The microstructures and properties were not fully recovered by this refurbishment treatment applied to these IN-738 blades. It is reported that the microstructure was only partially recovered by such simple re-heat treatment. However, when re-heat treatment condition, according to No. 1 (standard heat treatment), was applied to long-term exposed specimen in this study, the more homogeneous microstructure consisting of uniform dispersion of gamma prime precipitates are found. Both sizes of γ' precipitate particles are uniform and similar in size and shape, Fig. 3. This type of microstructure is previously expected and theoretically desired as the most optimized microstructure, which can provide very good mechanical properties at elevated temperatures, especially, for both short-term strength at elevated temperature and high creep resistance.

According to heat treatment of program No. 2 (Fig. 4), it was found that the morphology is very similar to that of the standard heat-treated one, which consists of bimodal morphology. Primary precipitate aging at 925°C for 1 hour resulted in an early precipitation of gamma prime γ', which its volume fraction is less than that of the microstructure according to program No. 1. After secondary precipitate aging, elements needed to form γ' precipitate would diffuse into the former gamma prime precipitates more causing the coarsening of the precipitates during secondary aging. Thus, large irregular γ' particles are created at 845°C. Such kind of microstructure could probably provide good rupture and/or creep resistance. As it is already well known that creep strength of alloys by γ' precipitation is a function of γ' particle size. It can be concluded that temperature of 925°C as primary aging provided more uniform and coarser rounded γ'.

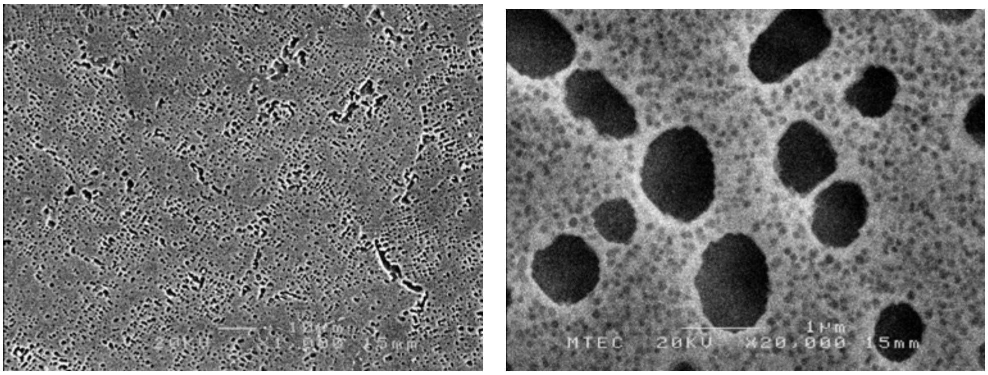

Fig.3 After standard heat treatment at 1125°C/2 hrs. (AC) and 845°C/24 hrs. (AC); Condition No. 1

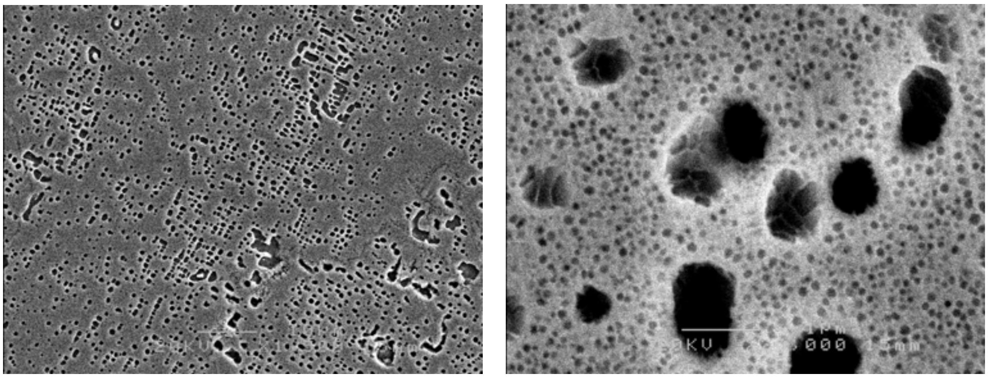

Fig.4 After heat treatment at 1125°C/2 hrs. (AC), 925°C/1 hr. (AC), and 845°C/24 hrs. (AC); Condition No. 2

In Figures 5 and 6, the microstructures after heat treatment according to program No. 3 show the more uniform dispersion of gamma prime precipitates. The microstructure is very similar to microstructure of sample No. 1 in Fig. 3. The gamma precipitates are in rounded or cubic shape at the proper size. Effect of primary precipitate aging at 1055°C for 1 hour resulted in more uniform precipitation of coarse gamma prime particles including higher volume fraction after secondary aging comparing to the heat treated microstructure of programs No.1 and 2, which has lower amount of primary precipitate particles, which were aged at 925°C.

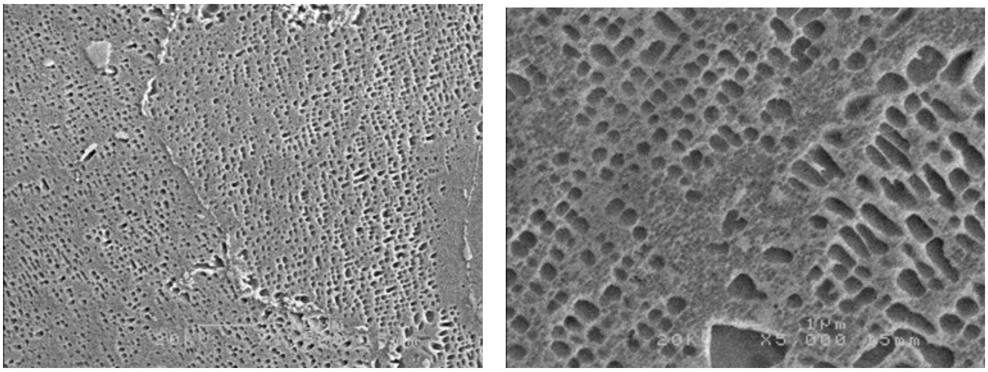

Fig.5 After heat treatment at 1125°C/2 hrs. (AC), 1055°C/1 hr. (AC), and 845°C/24 hrs. (AC); Condition No. 3

It should be noted that such higher temperature of primary aging might not provide the early precipitation of γ’ as occurring in the case of the lower temperature. Therefore, the coarser γ’ precipitates do not appear after secondary aging as in program No. 2. The sufficient high temperature of primary aging at 1055°C could be considered as a carbide stabilization heat treatment. Such high temperature could not only optimize the size and morphology of γ’ but also decomposition of the coarse, as-cast MC carbides, into fine grain boundary carbides. However, it was also found that the diameter sizes of very fine gamma prime particles are coarsening than those obtained from heat treatment conditions No. 1 & 2.

Furthermore, after secondary aging at 845°C for 24 hours then air-cooling, medium size gamma prime precipitates are very stable and growing very slowly. This final microstructure is expected to have better characteristics for short-term mechanical properties at elevated temperatures than those of specimens according to program No. 1 and No. 2. The γ’ particles produced in the secondary aging treatment is good for tensile strength as well as for rupture life according to the previous works, [23 - 24]. Especially, under creep conditions, the stable particles should become rafting or coarsening slowly resulting in longer lifetime. Double aging treatment is not only used commonly to control the size distribution of γ’ but also to control grain boundary morphology.

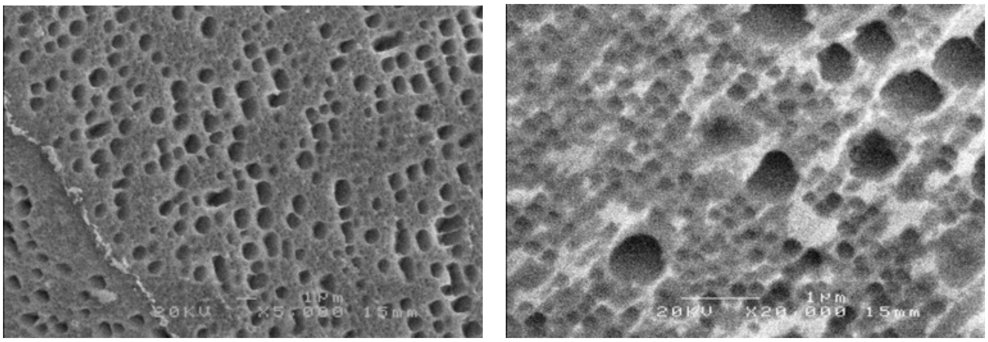

Fig.6 After heat treatment at 1125°C/2 hrs. (AC), 1055°C/1 hr. (AC), and 845°C/24 hrs. (AC); Condition No. 3

According to the results of program No. 4, No. 5 and No. 6, it should be noted that the higher solution annealing temperature could influence significantly the final microstructures in each condition, see Figures 7 - 10. In all cases, it was found that microstructures are very non-homogeneous consisting of partial-coarser γ’ precipitate dispersion and contain lower volume fraction of coarse gamma prime precipitates than those of programs No. 1 - No. 3. It should be noted that at higher solutioning temperatures of 1175°C for 2 hours, γ’ forming elements would be dissolved more into the matrix and carbides than solutioning at lower temperatures of 1125°C for 2 hours, resulting in the precipitation of more stable and coarser of fine γ’ particles during aging.

However, the groups of coarse γ’ precipitate particles were found in some areas of microstructures, where the finer γ’ precipitate particles locate among them. It should be noted that these coarse γ’ particles could possibly be from either the rest of non-completely dissolved coarse γ’ particles, which might have slightly different chemical composition providing more high temperature stability during solutioning at higher temperature. In the another hand, these γ’ particles, which did grow in faster rate due to the difference in chemical composition in both γ’-phase and γ-matrix resulting in higher difference in lattice mismatch comparing to those of other zones during aging.

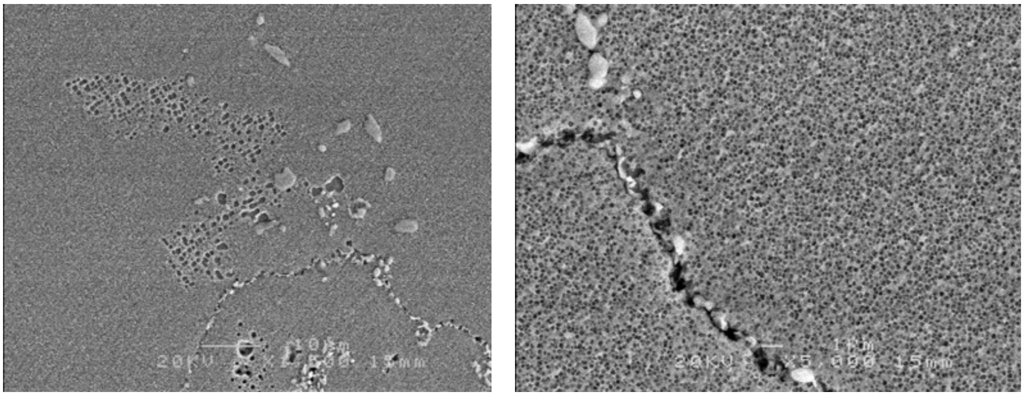

Fig.7 After heat treatment at 1175°C/2 hrs. (AC) and 845°C/24 hr. (AC); Condition No. 4 (low magnifications)

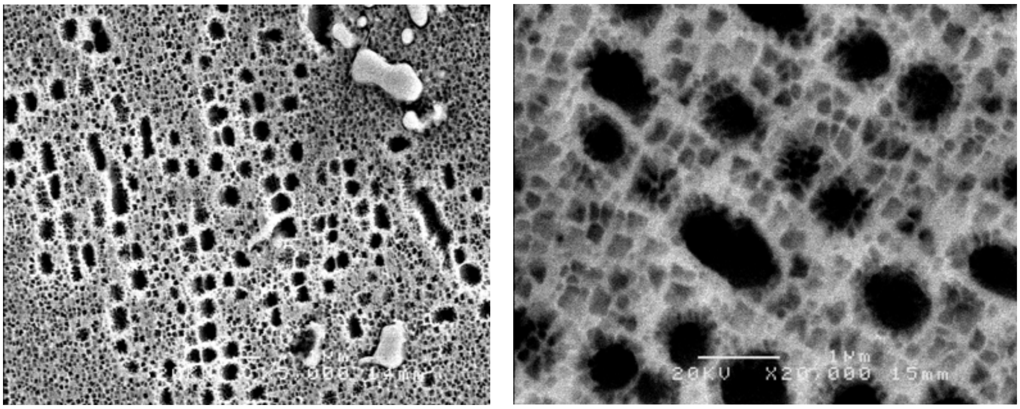

Fig.8 After heat treatment at 1175°C/2 hrs (AC) and 845°C/24 hrs. (AC); Condition No.4 (high magnifications)

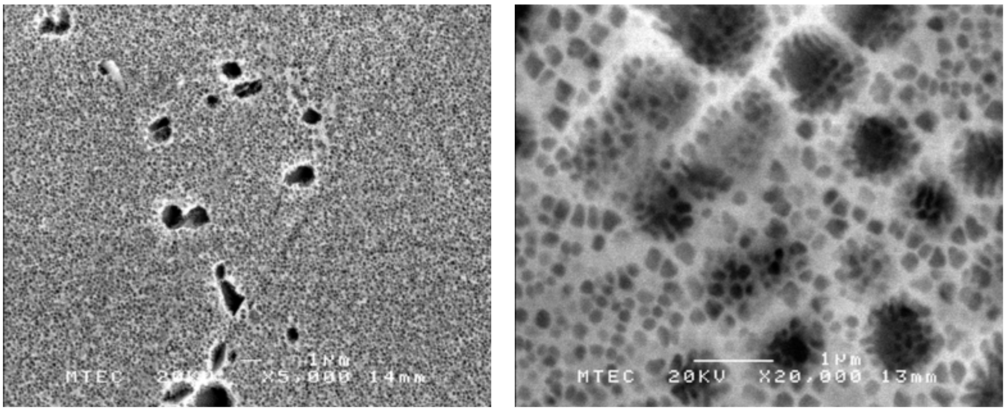

Fig.9 After heat treatment at 1175°C/2 hrs. (AC), 925°C/1 hr. (AC), and 845°C/24 hrs. (AC); Condition No. 5

The obtained results of particle size of all re-heat treatment conditions are summarized in Table 2. It could be summarized that the inserted aging before secondary aging after higher solutioning temperature did not show any significant effect on microstructures.

Table 2 Gamma prime particle size after heat treatments

Heat treatment program	Primary γ’ particle size (μm)	Secondary γ’ particle size (μm)
1	0.725-0.85	0.11
2	0.9-1.1	0.12
3	0.95	0.208
4	1.15	0.167
5	1.1	0.161
6	1.12	0.164

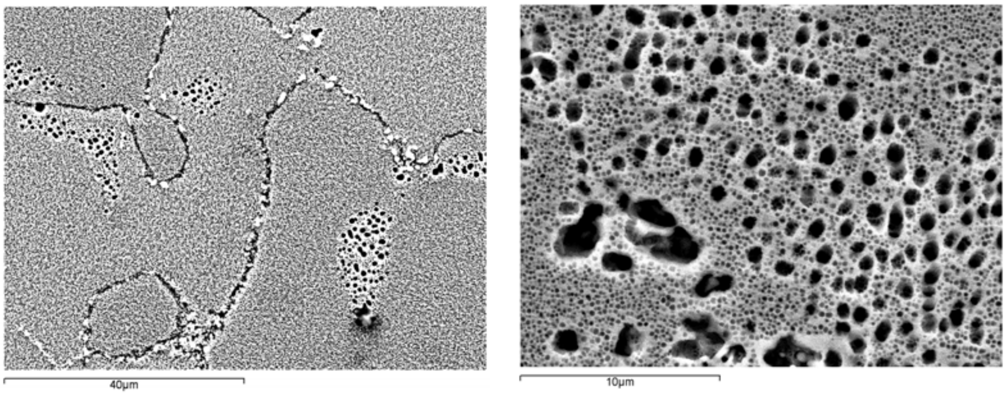

Fig.10 After heat treatment at 1175°C/2 hrs. (AC), 1055°C/1 hr. (AC), and 845°C/24 hrs. (AC); Condition No. 6

Conclusions

1. After various re-heat treatment conditions, the microstructures according to the program No.1 and 3 seems to be the optimized microstructures for both short-time tensile and fatigue properties at elevated temperatures as well as creep rupture properties.
2. For the other programs (No. 2), the microstructures with the over-aging of γ’ precipitates are expected to provide only creep rupture strength in some degree unless these γ’ particles tend to become rafting or coarsening earlier than the more stable γ’ particles of programs No. 1 and 3.
3. The higher temperature of solutioning provided the only lower energy of driving force for uniform precipitation and growth of γ’ particles in proper size comparing to those of programs No.1 - No.3.

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