RE-HEAT TREATMENTED MICROSTRUCTURES AND GAMMA PRIME PARTICLE COARSENING BEHAVIOR AT 1000˚C OF CAST NICKEL BASE SUPERALLOY, IN-738

Wangyao P.[1], Polsilapa S.[2], Sopon P.[2], Panich N.[1], Chuankrerkkul N.[1]
[1] Metallurgy and Materials Science Research Institute (MMRI), Chulalongkorn University, Bangkok, Thailand; Email: panyawat@hotmail.com
[2] Materials Engineering Dept., Faculty of Engineering, Kasetsart University, Bangkok, Thailand

ŠTÚDIUM VÝVOJA MIKROŠTRUKTÚRY A HRUBNUTIA PRIMÁRNYCH ČASTÍC GAMMA PRI 1000˚C V ODLIEVANEJ NIKLOVEJ SUPERZLIATINE IN-738 PO OPÄTOVNOM TEPELNOM SPRACOVANÍ

Wangyao P.[1], Polsilapa S.[2], Sopon P.[2], Wanichsamphan J.[1], Chuankrerkkul N.[1]
[1] Metallurgy and Materials Science Research Institute (MMRI), Chulalongkorn University, Bangkok, Thailand; Email: panyawat@hotmail.com
[2] Materials Engineering Dept., Faculty of Engineering, Kasetsart University, Bangkok, Thajsko

Abstrakt

Skúšky dlhodobej izotermálnej expozície boli aplikované na odlievanú niklovú superzliatinu značky IN-738 pre hodnotenie procesu hrubnutia primárnych častíc gamma po rozdielnych podmienkach tepelného spracovania za účelom obnovy a získania vhodnej mikroštruktúry. V dôsledku rozdielnych spôsobov tepelného spracovania pri dlhodobých skúškach boli získané rozdielne typy mikroštruktúr. Tieto rozdielne mikroštrukturálne charakteristiky môžu taktiež mať za následok rozdielnu teplotnú stabilitu primárnej fázy gamma pri dlhodobej expozícii zliatiny. Skúšky dlhodobej teplotnej expozície (až do 2500 hodín) boli urobené pri vysokých teplotách (1000°C) s cieľom sledovať proces hrubnutia primárnych častíc gamma. Na charakterizáciu zhrubnutia primárnych častíc gamma v závislosti od doby ohrevu boli vzorky študované metódami metalografie. Vo všetkých študovaných prípadoch boli primárne častice gamma hrubšie v porovnaní s počiatočným stavom po opätovnom tepelnom spracovaní. Zhrubnutie primárnych častíc gamma bolo pozorované buď v ich zaobľovaní alebo v ich spoločnej aglomerácii. Miera zhrubnutia primárnych častíc gamma narastá s predlžovaním času ohrevu. Pri aplikovaných teplotách ohrevu sa veľkosť primárnych častíc gamma zväčšila, pričom podiel sekundárnych častíc gamma mierne poklesol. Režimy opätovného tepelného spracovania pri 1175°C/ 2 hodiny (AC), 1055°C/ 1 hodina (AC) a 845°C/ 24 hodín (AC) poskytli najväčšiu fázovú stabilitu prejavujúcu sa minimálnou rýchlosťou hrubnutia po dlhodobej teplotnej expozícii pri teplote 1000˚C.

Abstract

Long-term isothermal exposure tests were conducted on cast nickel base superalloy grade IN-738 alloy to evaluate the variation of gamma prime particle coarsening behavior after different rejuvenated heat treatment conditions as attempts to restore the useful microstructure. Different microstructures due to various heat treatments were obtained for long-term tests. These different microstructural characteristics should also provide the different behaviors in long-term phase stability of gamma prime phase. The long-term exposure tests (up to 2,500 hours) were conducted at high temperatures (1000˚C) to assess gamma prime particle coarsening behavior. Metallographic work had been performed on isothermally aged samples to characterize gamma prime coarsening as a function of heating time. At all investigated heating times, the gamma prime particles in all tested specimens were coarser than the initial re-heat-treated ones. The gamma prime particles appeared to coarsen more into round shape or agglomerated between them comparing to those of each re-heat treatment. The degree of coarsening, as evidenced by gamma prime particle size, increased with increasing heating time. At the given heating temperature, the gamma prime particle size increased, and the area fraction of the secondary gamma prime particle slightly decreased with increasing heating time. The re-heat-treated microstructure obtained after heat treatment at 1175°C/ 2 hrs. (AC), 1055°C/ 1 hr. (AC) and 845°C/ 24 hrs. (AC) provided the most precipitated phase stability in term of minimum coarsening rate after long-term exposure at 1000˚C.

Keywords: rejuvenation, re-heat treatment, microstructural repair, gamma prime particle coarsening, nickel base 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 - 6]. 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 [7 - 10].

Due to mechanical properties are related to the microstructures, many previous research works [11 - 14] 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 numerous mechanical and physical properties. This allows making the selection of heat treatment parameters increasingly challenging. This work has an aim to investigate the effect of various re-heat-treated microstructures on long-term phase stability of precipitated gamma prime phase concerning to coarsening behavior. The most proper re-heat treatment condition should provide the optimum microstructure, which is the most similar to initial one as well as express the most phase stability of precipitated strengthening gamma prime particles after long-term exposure.

The present study is aimed to determine the most suitable and practicable repair condition, which provides the best microstructural characteristics and stability by rejuvenation method of various heat treatments for long-term exposed gas turbine blades.

2. Material and experimental procedure

The cast nickel base superalloy in this study was IN-738, obtained from after 50 000-hour service operated by Electricity Generating of Thailand (EGAT). The chemical composition of the IN-738 used in this study is shown in Table 1.

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

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

Rectangular plates, having a dimension of 1×1 cm, were cut from the most severe degradation zone of turbine blades. 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. These re-heat-treated specimens of each condition were then long-term heated at 1000ºC for 500, 1000, 1500, 2000 and 2500 hours in order to investigate the coarsening behavior of gamma prime particles after various re-heat treatments.

All sectioned samples after each aging were grinded and 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 an optical microscope (OM), a scanning electron microscope (SEM) with a secondary electron mode and an Image Analyzer. Size and volume fraction of gamma prime particles were determined.

Table 2 Heat treatment conditions applied to long term exposed for 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

3. Results and discussion

3.1 As-received microstructure

Optical micrographs 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. 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. 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.

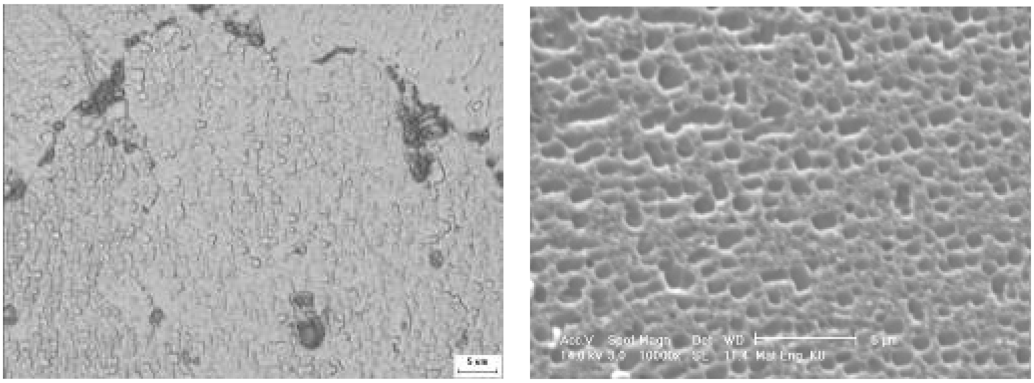

Fig.1 As-received microstructure after long-term service showing the coalescence of γ’ particles, areas of γ-γ’ eutectic and grain boundary carbides, OM (left) and SEM (right)

The degree of degradation, as measured by the gamma prime particle size, increases with exposed time and service temperature. In this study, the coarse gamma prime particle size was approximately 1.2 micron. 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 The microstructures of heat-treated alloy

According to the previous works [15-18], repeating the standard heat treatment sequence does not always work well. The structure 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 was obtained. The size of γ’ precipitate particles are uniform and similar in size and shape, Fig. 2. This type of microstructure is previously expected and theoretically desired as the most optimized microstructure, which could 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. 3), primary precipitate aging at 925°C for 1 hour resulted in an early precipitation of very fine gamma prime γ' particles, with higher average area of each particle 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, resulted in the coarsening of these very fine precipitates during secondary aging leading to the lower final area fraction of gamma prime phase. However, some of coarse irregular γ' particles could not further grow up during secondary aging at 845°C.

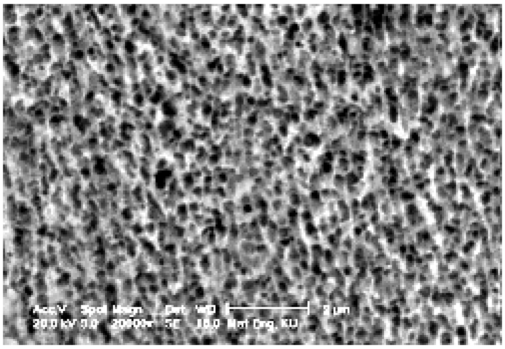

Fig.2 After standard heat treatment at 1125°C/ 2 hrs. (AC) and 845°C / 24 hrs (AC); Condition No. 1

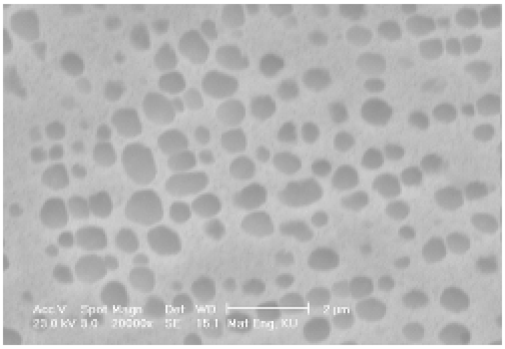

Fig.3 After heat treatment at 1125°C/ 2 hrs. (AC), 925°C/ 1 hr. (AC) and 845°C/ 24 hrs. (AC); Condition No. 2

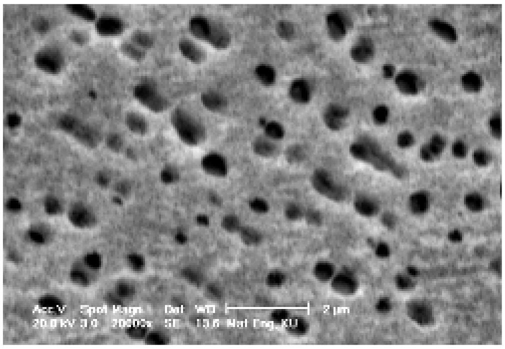

Fig.4 After heat treatment at 1125°C/ 2 hrs. (AC), 1055°C/ 1 hr. (AC) and 845°C/ 24 hrs. (AC); Condition No. 3

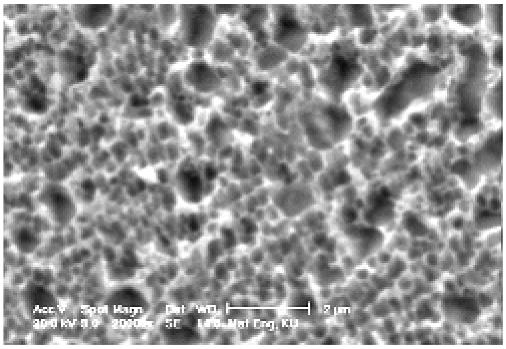

Fig.5 After heat treatment at 1175°C/ 2 hrs. (AC) and 845°C/ 24 hrs. (AC); Condition No. 4

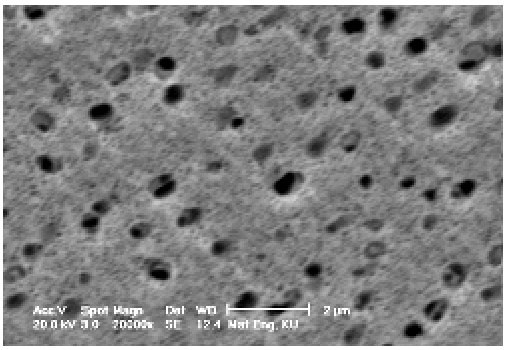

Fig.6 After heat treatment at 1175°C/ 2 hrs. (AC), 925°C/ 1 hr. (AC), and 845°C/ 24 hrs. (AC); Condition No. 5

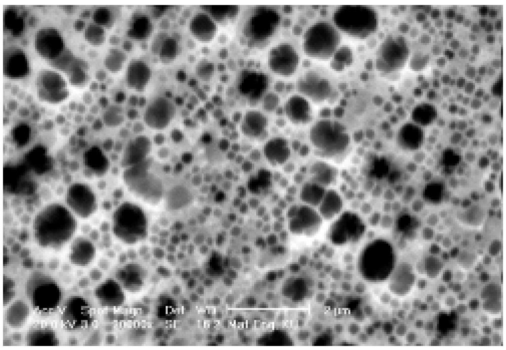

Fig.7 After heat treatment at 1175°C/ 2 hrs. (AC), 1055°C/ 1 hr. (AC), and 845°C/ 24 hrs. (AC); Condition No. 6

In Figure 4, the microstructure after heat treatment according to program No. 3 shows the more uniform dispersion of both coarse and very fine gamma prime precipitates. The microstructure is quite similar to microstructure of sample No. 2 in Fig. 3. The coarse 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 with lower total volume fraction of γ' phase after secondary aging comparing to the heat-treated microstructure of programs No. 1 and 2, which has higher amount of both primary and secondary precipitated particles, which were aged at 925°C.

Figures 5 - 7 show the effect of the highest solutioning temperature on final microstructures. This highest solutioning temperature provided the microstructure with much lower driving force for coarse γ' phase precipitation during aging resulting in very low volume fraction of coarse γ' precipitated particles. The inserted primary aging could not strongly influence to the size, distribution and volume fraction of coarse γ' particles but do greatly to the very fine one. The final microstructure according to program No. 6 is probably expected to have better characteristics for long-term mechanical properties at elevated temperatures than those of specimens according to programs No. 1 - 5. The γ' particle morphology produced in the secondary aging treatment is good for rupture life. Especially, under creep conditions, the stable particles would become rafting or coarsening slowly resulting in longer lifetime. Double aging treatment is commonly used not only to control size distribution of γ' particles, but also to control grain boundary morphology. Such kind of microstructure could probably provide good rupture and/or creep resistance in some degree. 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 primary aging at temperature of 1055°C produced more uniform and coarser of very fine rounded γ' particles.

3.3 Microstructures after long-term exposures

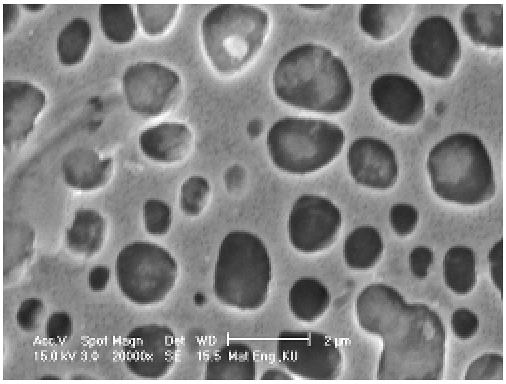

Fig.8 Condition No. 1 after long-term exposure at 1000ºC for 2500 hours

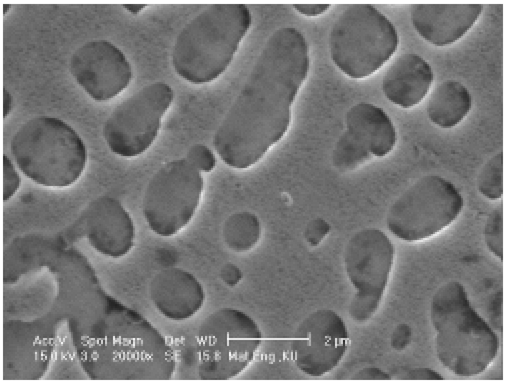

Fig.9 Condition No. 2 after long-term exposure at 1000ºC for 2500 hours

The aging at high temperature resulted in drastically coarser gamma prime particles comparing to the initially received re-heat treated specimens, see Figs. 8 - 13. The gamma prime particles greatly coarsened more into round shape than those in each initially re-heat-treated specimen. In the alloy, the degree of coarsening, as determined by gamma prime particle size, increased with the increasing heating time. At the aging temperature, the size of gamma prime particles increased, and the volume fraction of secondary gamma prime particle decreased with increasing aging time. It was also observed that an increasing aging temperature resulted in lower amount of secondary gamma prime precipitation. During long-term aging, secondary gamma prime particles would dissolve into the matrix and then diffused to agglomerate with the primary gamma prime particles.

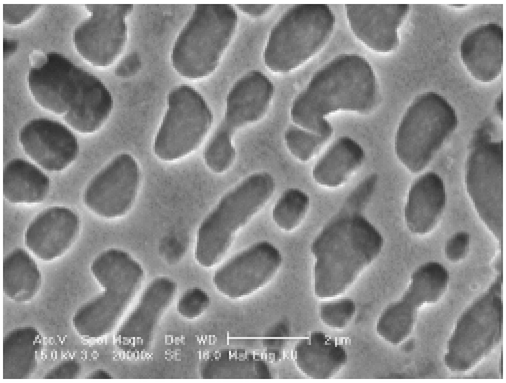

Fig.10 Condition No. 3 after long-term exposure at 1000ºC for 2500 hours

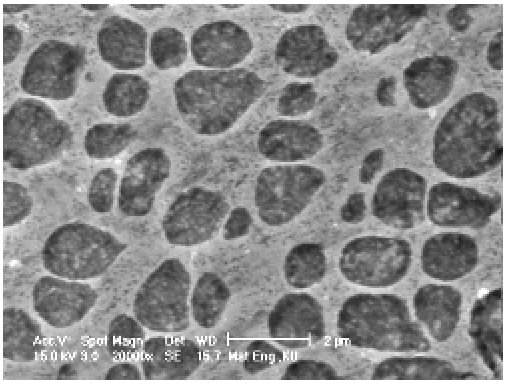

Fig.11 Condition No. 4 after long-term exposure at 1000ºC for 2500 hours

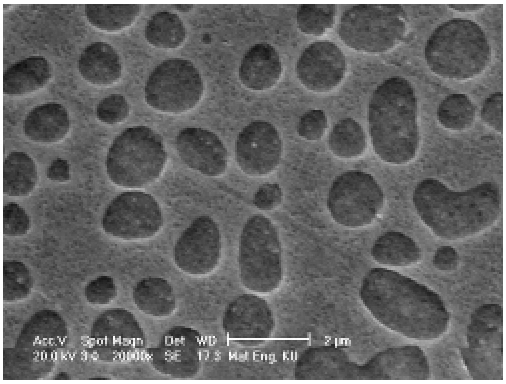

Fig.12 Condition No. 5 after long-term exposure at 1000ºC for 2500 hours

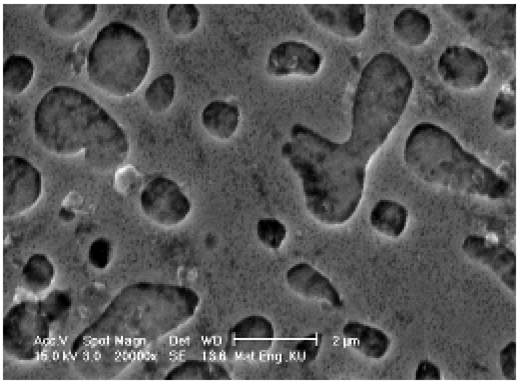

Fig.13 Condition No. 6 after long-term exposure at 1000ºC for 2500 hours

In long-term aging at 1000ºC, it was found that the microstructure under re-heat treatment condition No. 6 provided the most stable microstructure as compared to those under other reheat treatment conditions. However, it should be noted that the starting size of primary gamma prime particles after re-heat treatment condition No. 6 was the most coarsening one, Fig. 14. This should influence in lower kinetic of gamma prime particle coarsening during long-term exposed at this temperature. Nevertheless, it was also found that the volume fraction of this re-heat-treated specimen was the lowest one, Fig. 15. Therefore, it could be concluded that all reheated microstructures were not to show any beneficial characteristic for long-term using at such high temperature of 1000˚C, where all specimens already showed the continually degraded microstructures.

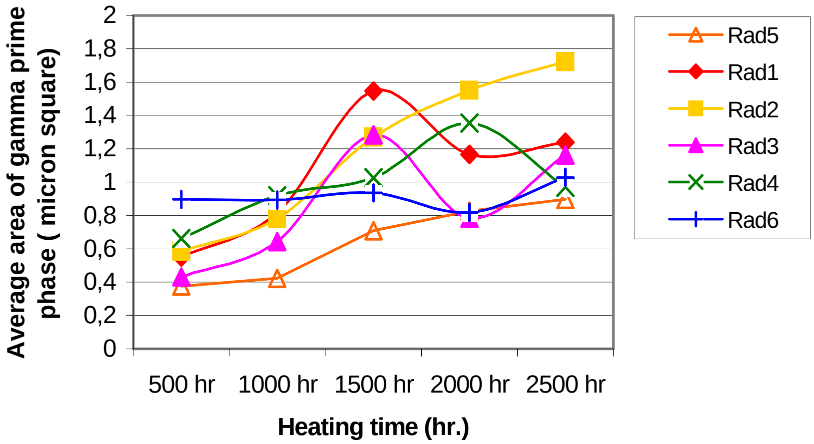

Fig.14 The relationship between heating time and average size of gamma prime particles

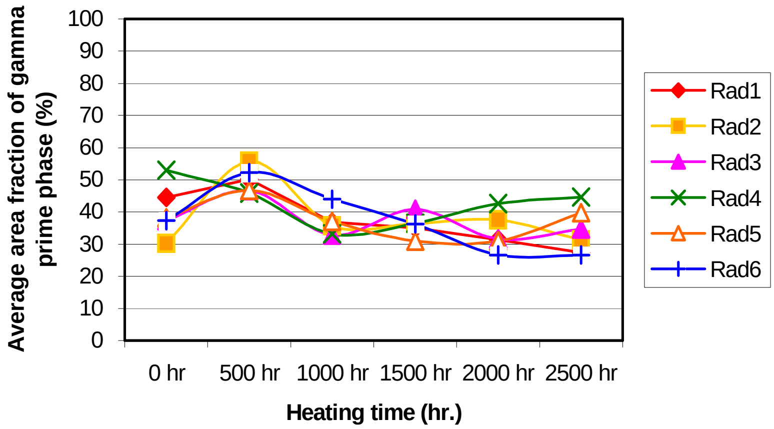

Fig.15 The relationship between heating time and average area fraction of precipitated phase

Conclusions

1. The re-heat-treated microstructure according to program No. 6 provided the most precipitated phase stability in term of minimum coarsening rate after long-term exposure at 1000˚C.
2. In almost re-heat-treated microstructures, average area fractions of gamma prime phase after heating for 2500 hours slightly decreased comparing to initial ones.

Acknowledgement

The authors would like to express special thank to Ing. Weerasak Homkrajai, Electricity Generating Authority of Thailand (EGAT) for all useful advices, technical discussions and material supports.

Literature

[1] Jones R. M. F., Jackman L.A.: The structural evolution of superalloy ingot during hot working, JOM, 1999, pp. 27 - 31

[2] Howson T. E., Couts W. H.: Thermomechanical Processing of Superalloys in Superalloys, Supercomposites and Superceramics, edited by Tien, J. K. & Caulfield, T., Academic Press, Inc., 1989, pp. 183 - 213

[3] Symonds C. H.: Hot working Chapter in The Nimonic Alloys & Other-Base High Temperature Alloys, edited by Betteridge, W. & Heslop, J., Edward Arnold, 1974, pp. 129-149

[4] Jackman L. A.: Forming and Fabrication of Superalloys, in Superalloys Source Book, ASM, 1984, pp. 217 - 233

[5] Couts W. H. jr.: Mechanical Processing in The Superalloys, edited by Sims, C. T. and Hagel, C. W., John Wiley & Son Inc. (Publisher), 1972, pp. 451 - 478

[6] Decker R. F., Sims C. T.: The Metallurgy of Ni-base alloys in The Superalloys, edited by Sims, C. T. & Hagel, W. C., John Wiley & Son Inc. (Publisher), 1972, pp. 36 - 62

[7] Tamura M.: Alloying effect on hot deformation in Superalloys, Supercomposites and Superceramics, edited by Tien, J. K. & Caulfield, T., Academic Press, Inc., 1989, pp. 215 - 233

[8] Courtney T. H.: Mechanical Behavior of Materials, McGraw-Hill (Publishers) Ltd., 1990

[9] Dieter G. E.: Mechanical Metallurgy, McGraw-Hill (Publishers) Ltd., 1988

[10] Zrník J., Fujda M., Seliga T.: Low cycle fatigue and premature failure of nickel base superalloy, Engineering Mechanics, Vol. 11, 5, 2004, pp. 329 - 333

[11] Zrník J., Fujda M.: Porušovanie niklovej superzliatiny v podmienkach nízkocyklovej únavy, In: „Fraktografia 2000“, Stará Lesná, 2000, pp. 301 - 313

[12] Zrník J., Fujda M.: Cyklické poškodenie niklovej konštrukčnej superzliatiny, In: „Degradácia vlastností konštrukčných materiálov únavou“, Rajecké Teplice, 2001, pp. 54-61

[13] Zrník J., Štrunz P., Horňak P., Vrchovinský V., Wiedenmann A.: Microstructural changes in long-time thermally exposed Ni-base superalloy studied by SANS, Journal of Appl. Phys. A, 74, 2002, S1155 - S1157

[14] Zrník J., Štrunz P., Vrchovinský V., Muránsky O., Horňak P., Wiedenmann A.: Creep deformation and microstructural examination of a prior thermally exposed nickel base superalloy, Key Engineering Materials Vols. 274 - 276, 2004, pp. 925 - 930

[15] Daleo J. A., Ellison H. A., Boone D. H.: Journal of Engineering for Gas Turbines and Power, 2002, Vol. 124, pp. 571 - 579

[16] Wangyao P., Korath T., Harnvirojkul T., Homkrajai W., Effect of re-heat treatment conditions on microstructural refurbishment of nickel base superalloy turbine blade after long-term serviced, Journal of Metals, Materials and Minerals, Vol. 14, No. 1, 2004, pp. 49-59

[17] Wangyao P., Lothongkum G., Krongtong V., Pailai S., Polsilapa S., Effect of heat treatments after HIP process on microstructure refurbishment in cast nickel base superalloy, In-738, Journal of Metals, Materials and Minerals, Vol. 15, No. 2, 2005, pp. 69-78

[18] Wangyao P., Krongtong V., Homkrajai W., Tuengsook P., Panich N., The relationship between reheat-treated microstructures and hardness in cast nickel base superalloy, GTD-111, Journal of Metals, Materials and Minerals, Vol. 16, No. 1, 2006, pp. 55-62