Determination of Solution Temperature in an Ex-Service Ni-Based Turbine Blade

Mohammad Shademani , Mohammad Reza Rahimipour , Ali Sedaghat, Mohammad Farvizi *

Ceramic Division, Materials and Energy Research Center (MERC), P.O. Box 14155-4777, Tehran, Iran

ARTICLE INFO

Article history:

Received 27 December 2016
Accepted 29 August 2017
Available online 15 December 2017

Keywords:

Nickel- based superalloy
Rejuvenation heat treatment
Gamma prime precipitates
Microstructure

ABSTRACT

It is well-known that the harsh operational conditions of turbine blades lead to a gradual change in the microstructure of underlying Ni-based superalloy of blades. Accordingly, a rejuvenation process should be conducted on turbine blades superalloys to recover their microstructures. The first and main step of the rejuvenation process is the determination of appropriate solution condition in which ϒ′ precipitates dissolve in the ϒ matrix. In this study, different heat treatment conditions were applied on a Ni-based single crystal superalloy to determine its solution condition. The results of scanning electron microscopy (SEM) showed that with increasing the solution treatment temperature from 1275°C (3h) to 1330°C (3h), at first, ϒ′ precipitates at dendritic regions dissolve and complete solution of ϒ′ phase occurs only after increasing the solution temperature to 1350°C (3h). The full solution condition (1350°C-3h) will be used as the first step of the rejuvenation process of these blades in future studies.

1-Introduction

Nickel-based superalloys have good mechanical properties at high temperatures and, for this reason, they have found widespread application in different industries such as aircraft and gas and steam turbines. Two important phases that exist in Ni-based superalloys are ϒ matrix and ϒ′ precipitates with the composition of (Ni_3 (Al, Ti)) and with $L1_2$ structure which is coherently dispersed in the ϒ matrix [1, 2].

Generally, the creep rupture occurs in grain boundaries. As a consequence, for improvement of creep resistance in an Ni-based superalloy, cast single crystal blades have been developed [3]. In single crystal superalloys, the volume fraction of ϒ′ increased to 60-70% and the refractory metals such as (Mo, Ta, Nb, W) were added to these alloys [4]. The microstructure of turbine blade during working at raised temperatures and high stresses continuously changes. The ϒ′ precipitates become coarser, the morphology of cubic ϒ′ changes into an irregular morphology and finally, rafting occurs in these precipitates. Also, the volume fraction of ϒ′ phase decreases accordingly which leads to the deterioration of the superalloy mechanical properties and reduction of the remaining life of blades [5, 6]. The costs of replacement of blades are too high and, as a consequence, finding an appropriate method for repairing the blades is crucial [7]. The rejuvenation heat treatment can convert the degenerated microstructure of superalloys to an

* Corresponding author:
E-mail address: mmfarvizi@yahoo.com

optimum structure and increase the remaining life of turbine blades. The rejuvenation heat treatment includes: (i) solution treatment and (ii) aging treatment. The first and the dominant stage in the rejuvenation process is solution treatment in which ϒ´ precipitates dissolute in ϒ matrix and the superalloy structure is converted to a homogeneous structure [8-10]. Also, it is well-known that the pre-service of turbine blades may affect the heat treatment conditions [11]. On this basis, the determination of solution time and temperature of ϒ´ precipitates is an essential step for the rejuvenation process in ex-service turbine blades. In previous researches, the solution conditions of fresh Zhs32 Ni-based superalloys have been explored [12]. However, to the best of our knowledge, the solution conditions of ex-service Zhs32 blades have not been reported yet.

In the current study, the temperature and time of full solution of ϒ´ precipitates in an ex-service Ni-based single crystal superalloy were investigated. Various temperatures were examined for solution treatment and the final microstructures of the heat treated samples were studied by scanning electron microscopy (SEM).

2-Experimental procedure

The chemical composition of the nickel–based cast single crystal superalloy (ZhS32) was determined by means of an Oxford Foundry Master Quantometer device. The weight percentages of the elements of the superalloy are shown in Table 1. As can be seen in Fig.1, samples with the dimensions of 1×1 cm^2 from the center part of the serviced turbine blade were prepared by using Electrical Discharge Machine (EDM). The aluminide diffusion coating of turbine blades was removed by a chemical solution composed of 9 ml HNO_3, 2 ml HCl, 0.37 gr $CuSO_4$, 0.025 gr, $FeCl_3$, and 9ml H_2O. After electrical discharge machining, the samples were ground to a 2500 grit with the SiC papers followed by polishing using diamond paste. The specimens were chemically etched with a marble solution of 20 gr $CuSO_4$, 50 ml HCl and 100 ml H_2O.

Microstructural observations of the samples were performed with a TESCAN scanning electron microscope (VEGA II LMU/XMU). The geometrical parameters such as the particle size and volume fraction of ϒ´ were measured utilizing Image-pro Plus software by the linear intercept method. The solution heat treatment was performed on the specimens in an argon atmosphere furnace.

The heat treatment details are presented in Table 2. For solution treatment, the samples were heated at a rate of 10 °C/min. After reaching the peak temperature, the samples were hold for 3h and then water quenched.

In order to estimate the ϒ′ solvos temperature and to design the solution heat treatment conditions, differential scanning calorimetry (DSC) analysis was performed on the ex-service blades. The results showed that the ϒ′ solvos temperature is about 1285°C which is slightly higher than the reported solution temperature of new Zhs32 superalloy (1275°C) [11]. As a consequence, the temperature of 1275°C was chosen as the lower limit of solution treatment. The upper limit of solution treatment of the ex-service blades was the first temperature in which ϒ′ precipitates completely dissolved throughout the sample.

For detection of full solution temperature of ϒ´, microstructural analysis was performed on specimens. After mechanical polishing, the samples were electrochemically etched in a 20% oxalic acid (DC voltage=4 v, time=5 s).

Fig. 1. Ex-service turbine blade.

Table 1. Chemical composition of the Ni-based cast single crystal superalloy.

C	Cr	Co	Ta	W	Mo	Ti	Al	Nb	Hf	Re	Ni
0.15	5.09	9.27	4.01	8.50	1.29	0.061	5.86	1.87	0.03	4	bal

Table 2. Solution heat treatment schedules for the Ni–based cast single crystal superalloy.

No.	Solution temperature (°C)	Holding time (h)	Cooling
1	1275	3	Water Quenching (WQ)
2	1285		
3	1295		
4	1330		
5	1350		

3-Results and discussion

3-1-Microstructural evolution of the Ni-based superalloy

In Fig. 2a, microstructure of the Ni-based superalloy prior to service has been demonstrated. It can be seen that two kinds of ϒ′ precipitates exist in Ni-based superalloys: (i) primary ϒ′ with cuboid morphology and (ii) secondary ϒ′ precipitates with spherical morphology. The primary ϒ′ precipitates nucleate at high temperatures at the triple junctions and pin the γ grain boundary and reach the equilibrium composition. Due to higher nucleation temperature in primary gamma prime precipitates, these precipitates can reach to larger sizes and higher volume fractions in the microstructure. However, due to changing diffusivities and build up of supersaturation in the γ matrix, secondary γ′ precipitates nucleate at lower temperatures and due to limited growth, these precipitates maintain their spherical morphology and non-equilibrium compositions. The result is a bimodal distribution of γ′ precipitates in the ϒ matrix. The γ′ precipitates are the most important strengthening phase in the Ni-based superalloys. The coexistence of γ′ precipitates with spherical and cuboid morphologies will yield better mechanical performance. In a typical Ni-based superalloy, fine and homogenously dispersed secondary γ′ precipitates provide creep resistance while coarse primary γ′ precipitates improve the ductility of the alloy. As a consequence, a bimodal configuration of primary and secondary gamma prime precipitates is favored [13].

In cast Ni-based superalloys, $M_{23}C_6$, M_6C and MC carbides exist in the microstructure [14]. Generally, in the Ni-based superalloys with high weight percentages of tungsten and molybdenum elements, M_6C-type carbides are formed with an acicular morphology [15]. In Fig. 2(b), M_6C carbides with a typical acicular morphology have been demonstrated. The results of EDS analysis taken from these carbides show that Nickel, Cobalt, Tungsten and Chromium are the main elements which exist in the M_6C carbide structure. Previous results performed on Ni-base cast single-crystal superalloys have shown that the M_6C carbides are formed in the temperature range of 816-1038°C [14].

3-2-Determination of solution temperature in the serviced ZhS32 superalloy

In Ni-base cast single-crystal superalloys, higher amounts of tungsten, rhenium, and tantalum elements exist in the microstructure which will elevate their solution temperature. For a new ZhS32 turbine blade, the homogenization temperature has reported to be about 1275°C [16-19]. In Fig. 3, microstructure of the serviced superalloy after solution treatment at 1275°C for 3h has been presented. It can be seen that ϒ′ precipitates in dendritic and inter-dendritic regions did not dissolve, indicating that operational conditions of turbine blade change the solution conditions of the underlying superalloy. Also, as observed in Fig.3 (b), coarsening of ϒ′ precipitates in inter dendritic region occurred and their size increased from 0.601 μm^2 (initial size) to 2.19 μm^2 after solution at 1275°C for 3h. With increasing the solution temperature to 1285°C for 3h (Fig. 4), similar to the sample solutionized at 1275°C, coarsening of ϒ′ precipitates occurs. In dendritic and inter-dendritic regions, the ϒ′ phase starts to dissolve. With the aid of the results obtained from image analysis (table 3), it can be recognized that the volume fraction of the ϒ′ phase in this sample reduced in comparison with the sample solution treated at 1275°C.

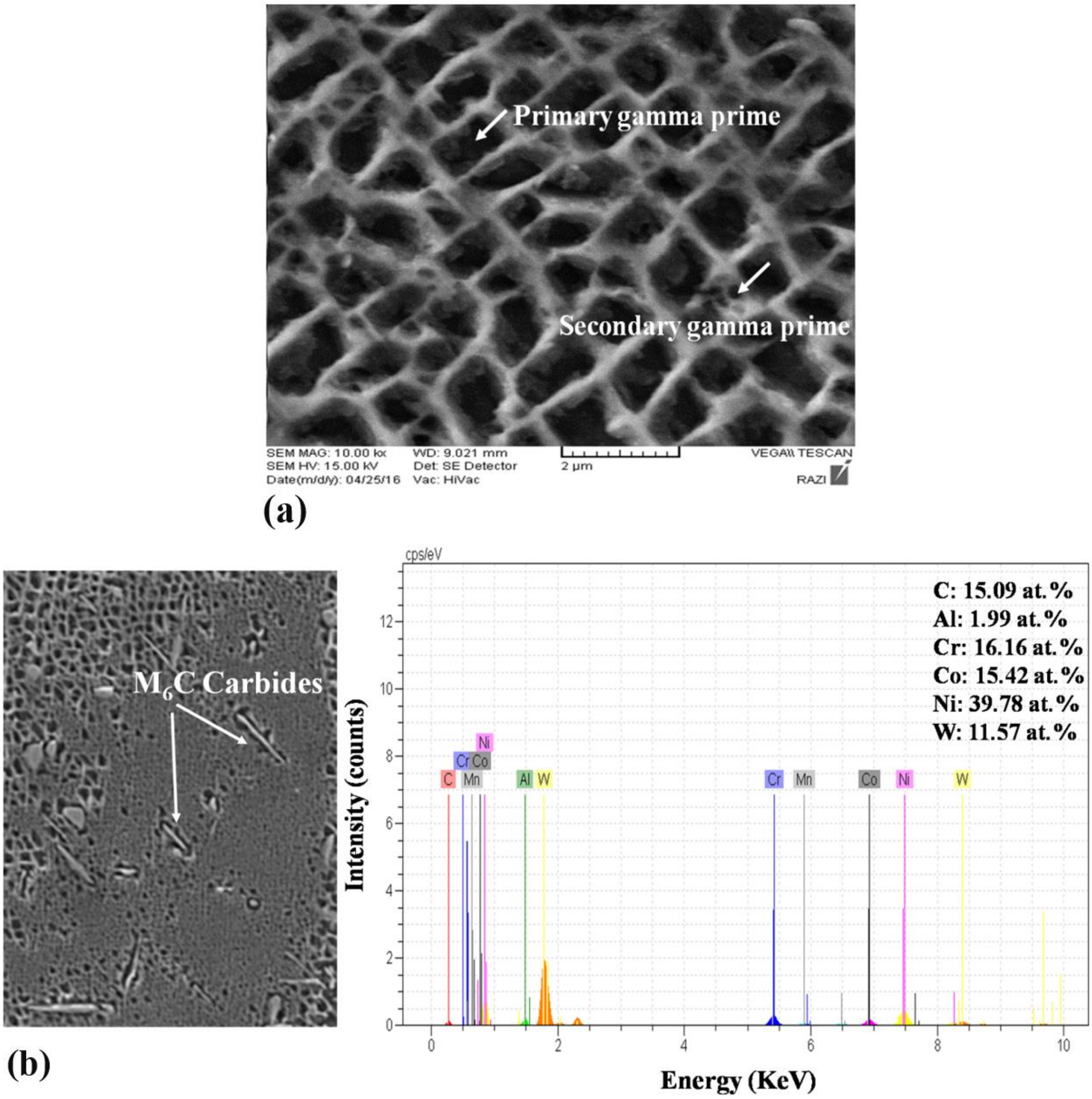


Fig. 2. (a) Microstructure of primary and secondary gamma primes in pre-serviced Zhs32 superalloy, (b) SEM micrograph shows the acicular morphology of M_6C carbides and EDS results obtained from this phase.

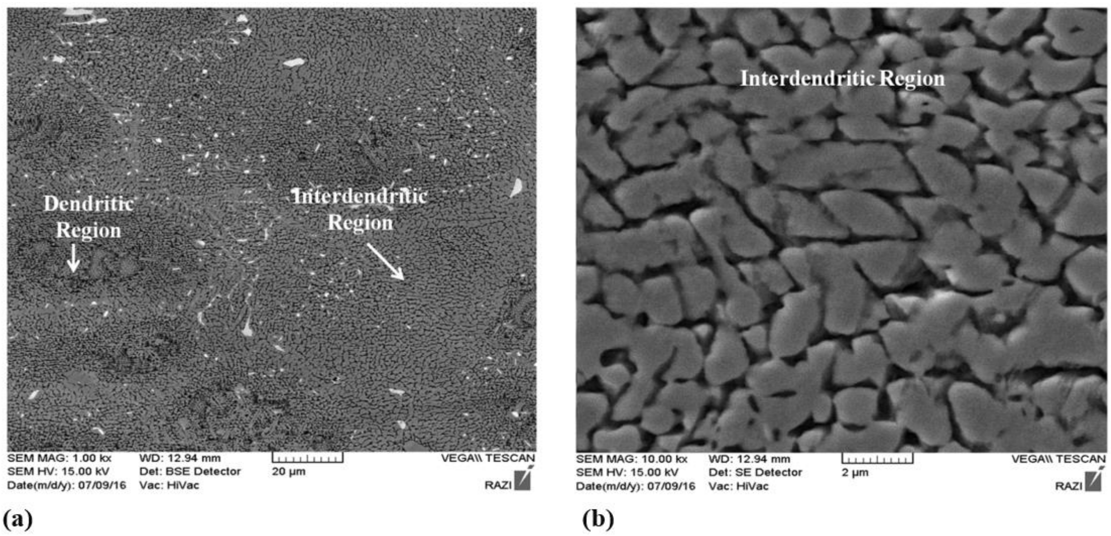
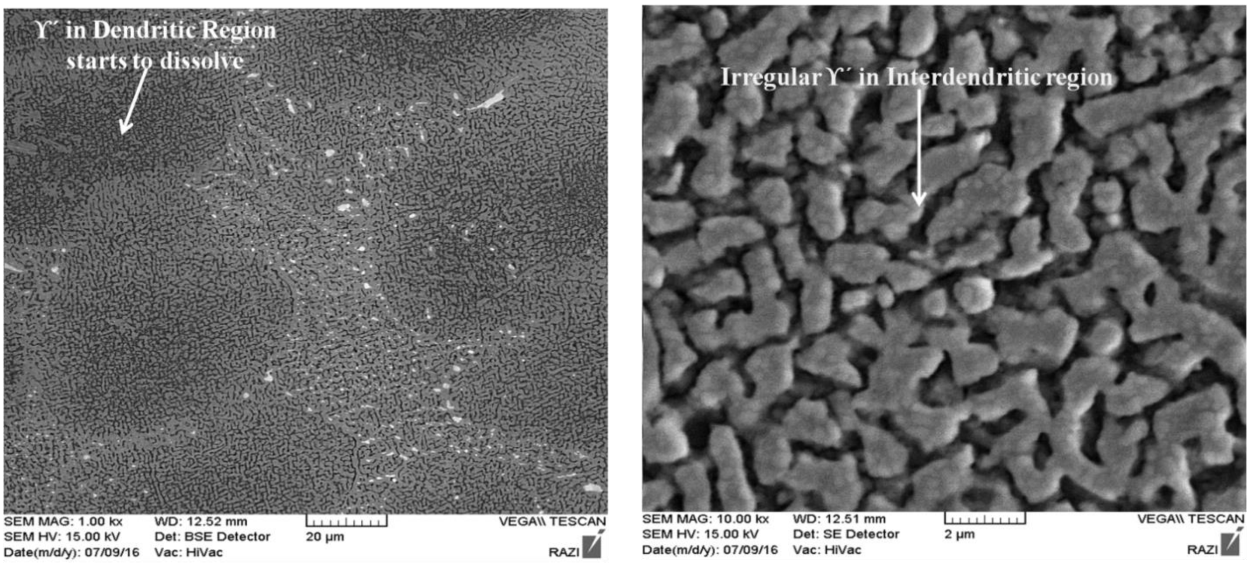


Fig. 3. SEM micrographs of the serviced superalloy after solution treatment at 1275°C for 3h.



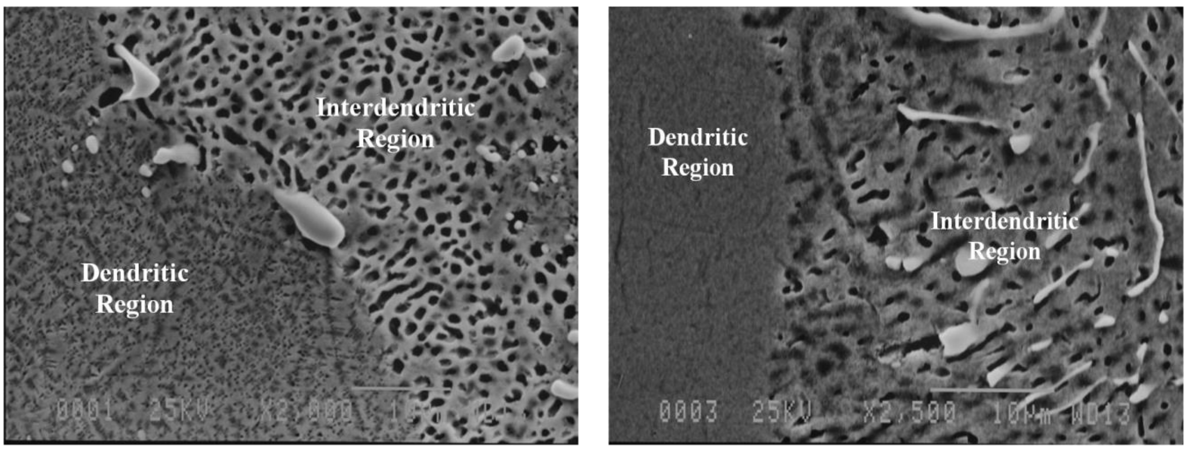
(a) **(b)**

Fig. 4. SEM micrographs of the serviced superalloy after solution treatment at 1285°C for 3h.

Table 3. The results of image analysis after heat treatment.

Heat treatment procedure	ϒ´ particle size(um^2)	ϒ´ volume fraction%
1275°c / 3hr	2.19	85.57
1285°c / 3hr	2.353	72.45
1295°c / 3hr	0.931	17.25
1330°c / 3hr	0.757	6.5
Base Sample	0.601	42.33

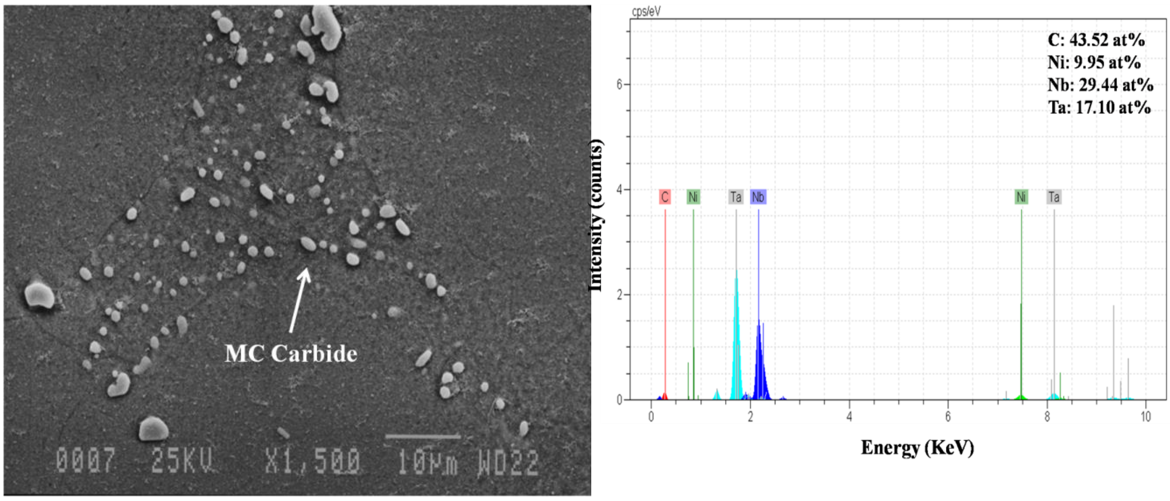
With further increment of the solution temperature to 1295°C for 3h (Fig. 5(a)), the volume fraction and size of ϒ´ precipitates considerably reduced. In solution treatment at 1330°C for 3h (Fig. 5(b)), the ϒ´ precipitates in dendritic regions completely dissolve and in inter-dendritic regions and only a small amount of ϒ´ precipitates will remain. According to the studies conducted by Sidhu and colleagues [20], precipitation of fine secondary ϒ´ particles occurs predominantly within the dendrite cores and to a lesser extent in the interdendritic regions. Also, it is well known that secondary ϒ´ precipitates nucleate at lower temperatures in comparison with primary ϒ´ phases. As a consequence, during solution treatment, the rate of ϒ´ particles dissolution in dendritic regions is more than that in inter-dendritic regions. This issue can be clearly observed in Fig. 4(a) and Fig. 5(a,b).



(a) **(b)**

Fig. 5. SEM micrographs of the serviced superalloy after solution treatment at (a) 1295°C for 3h, (b) 1330°C for 3h.

Finally, with solution treatment at 1350°C for 3h and water quenching (Fig. 6(a)), the ϒ′ precipitates in both dendritic and inter-dendritic regions dissolve, showing that full solution of this phase occurred in this condition. Also, the results of EDS analysis (Fig. 6(b)) taken from carbides confirm that the MC-type carbides which contain refractory metals remain after solution treatment in the microstructure [21]. During solution treatment, the MC-carbides are partially dissolved and their morphology changes from initial blocky shape to semi-spherical particles. It has been shown that the formation of spherical and discrete MC-carbides is beneficial for the enhancement of creep resistance of the Ni-based superalloys [22]. This solution treatment condition (1350°C for 3h) will be used as the first step of rejuvenation process in future works.



(a) **(b)**

Fig. 6. (a) SEM micrograph and (b) EDS spectra taken from MC carbides after solution treatment at 1350°C for 3h.

4-Conclusion

During operation of turbine blades, microstructure of the underlying superalloys continuously changes which deteriorates its properties. As a consequence, an intermediate rejuvenation process should be performed to recover its microstructure. The first step of the rejuvenation process is solution treatment. Microstructural changes and segregation of elements during operational conditions will cause the solution condition of the serviced turbine blade to differ from that of the virgin blades. In this study, various solution treatments were conducted on the serviced Ni-base cast single crystal superalloy (ZhS32). The result of DSC analysis performed on the ex-service blade showed that the ϒ′ solvos temperature is about 1285°C which is slightly higher than the reported solution temperature of virgin Zhs32 superalloy. Microstructural analysis performed by SEM showed that the solution treatment at 1275°C for 3h did not yield the dissolution of ϒ′ phase and further temperature increment required for full solution. The SEM images taken from solutionized microstructure at 1285°C and 1295°C proved that the coarsening of ϒ′ precipitates occurred and that the volume fraction of ϒ′ precipitates reduces with increasing the solution temperature. Also, it was observed that the rate of ϒ′ particles dissolution in dendritic regions is more than that in inter-dendritic regions which is attributed to the higher nucleation ratio of secondary ϒ′ phases in dendritic cores. The full solution of ϒ′ phase occurred at the temperature of 1350°C for 3h in which all of the ϒ′ precipitates dissolve and a supersaturated solid solution with MC-type carbides remains.

References

[1]Y. Zhou, Z. Zhang, Z. Zhao, Q. Zhong, "Morphological evolution of γ′ precipitates in a nickel-based superalloy during various solution treatments", Rare Metals, Vol. 31, 2012, pp. 221-226.

[2]J. Sluytman, A. Suzuki, A. Bolcavage, R. Helmink, D. Ballard, T. Pollock, "Gamma Prime Morphology and Creep Properties of Nickel Based Superalloys with Platinum Group Metal Additions (Preprint)", In.: DTIC Document; 2008.

[3] C.T. Sims, N.S. Stoloff, and W.C. Hagel, Superalloys II, John Wiley & Sons, New York, NY, 1987.
[4] E. Kablov, N. Petrushin, M. Bronfin, A. Alekseev, "Specific features of rhenium-alloyed single-crystal nickel superalloys", Russian Metallurgy (Metally), Vol. 2006, 2006, pp. 406-414.
[5] H. Li, L. Zuo, X. Song, Y. Wang, G. Chen, "Coarsening behavior of γ′ particles in a nickel-base superalloy", Rare metals, Vol. 28, 2009, pp. 197-201.
[6] W. Panyawat, C. Nuthida, S. Polsilapa, "Gamma Prime Phase Stability after Long-Term Thermal Exposure in Cast Nickel Based Superalloy IN-738", Chiang Mai J. Sci., Vol. 36, 2009, pp. 312-319.
[7] L. Rettberg, M. Tsunekane, T. Pollock, "Rejuvenation of Nickel-Based Superalloys GTD444 (DS) and René N5 (SX)", in Superalloys 2012 (eds E. S. Huron, R. C. Reed, M. C. Hardy, M. J. Mills, R. E. Montero, P. D. Portella and J. Telesman), John Wiley & Sons, Inc., Hoboken, NJ, USA.
[8] M.R. Reyhani, M. Alizadeh, A. Fathi, H. Khaledi, "Turbine blade temperature calculation and life estimation-a sensitivity analysis", J. Propul. Power, Vol. 2, 2013, pp. 148-161.
[9] E. Lvova, "A comparison of aging kinetics of new and rejuvenated conventionally cast GTD-111 gas turbine blades", J. Mater. Eng. Perform., Vol. 16, 2007, pp. 254-264.
[10] M. Jahangiri, M. Abedini, "Effect of long time service exposure on microstructure and mechanical properties of gas turbine vanes made of IN939 alloy", Mater. Design, Vol. 64, 2014, pp. 588-600.
[11] M. Retima, S. Bouyegh, H. Chadli, "Effect of the heat treatment on the microstructural evolution of the nickel based superalloy", Metalurgija, Vol. 17, 2011, pp. 71-77.
[12] N. Protasova, I. Svetlov, M. Bronfin, N. Petrushin, "Lattice-parameter misfits between the γ and γ′ phases in single crystals of nickel superalloys", Phys. Met. Metallogr., Vol. 106,2008, pp. 495-502.
[13] S.S. Hosseini, S. Nategh, A-A. Ekrami, "Microstructural evolution in damaged IN738LC alloy during various steps of rejuvenation heat treatments", J. Alloys Compd., Vol. 512, 2012, pp. 340-350.
[14] G. Pigrova, A. Rybnikov, "Carbide phases in a multicomponent superalloy Ni-Co-W-Cr-Ta-Re", Phys. Met. Metallogr., Vol. 114, 2013, pp. 593-595.
[15] Z-h. Yu, L. Lin, J. Zhang, "Effect of carbon addition on carbide morphology of single crystal Ni-based superalloy", Trans. Nonferrous Met. Soc. China, Vol. 24, 2014, pp. 339-345.
[16] H. Li, X. Song, Y. Wang, G. Chen, "Characterization of γ′ precipitates in a nickel base superalloy quenching from aging temperature at different rates" Rare Metals, Vol. 29, 2010, pp. 204-208.
[17] L. Liu, T. Jin, N. Zhao, Z. Wang, X. Sun, H. Guan, Z. Hu, "Microstructural evolution of a single crystal nickel-base superalloy during thermal exposure", Mater. Letter., Vol. 57, 2003, pp. 4540-4546.
[18] J. Yang, Q. Zheng, X. Sun, H. Guan, Z. Hu, "Relative stability of carbides and their effects on the properties of K465 superalloy", Mater. Sci. Eng. A, Vol. 429, 2006, pp. 341-347.
[19] Y.G. Bykov, T. Zakharova, E. Monastyrskaya, A. Fishgoit, A. Dushkin, A. Demidov, "Mechanical properties of high-temperature alloy ZhS32 at 1150–1250° C", Met. Sci. Heat Treat., Vol. 48, 2006, pp. 41-43.
[20] R.K. Sidhu, O.A. Ojo, M.C. Chaturvedi, "Microstructural Response of Directionally Solidified Rene 80 Superalloys to Gas-Tungsten Arc Welding", Metall. Mater. Trans. A, Vol. 40, 2009, pp. 150-162.
[21] K. Al-Jarba, G. Fuchs, "Carbon-containing single-crystal nickel-based superalloys: Segregation behavior and carbide formation", JOM, Vol. 56, 2004, pp. 50-55.
[22] A. Jafari, S. M. Abbasi, A. Rahimi, M. Morakabati, M. Seifollahi, "The effects of solution treatment on the microstructure of the cast Ni-based IN100 superalloy", Metall. Mater. Eng., Vol. 21, 2015, pp. 167-181.