Influence of Solution Treatment Temperature on the Microstructure of Ni-based HR-120 Superalloy

Noraziana Parimin1,*, and Esah Hamzah2

1School of Materials Engineering, Universiti Malaysia Perlis, 02600 Arau, Perlis Malaysia
2Faculty of Mechanical Engineering, Universiti Teknologi Malaysia, 81310 Skudai, Johor, Malaysia

Abstract. The solution treatment process has been carried out on Ni-based HR-120 superalloy (Fe-40Ni-24Cr) at six different temperatures, namely 950°C, 1000°C, 1050°C, 1100°C, 1150°C and 1200°C. All samples were soaking for 3 hours at desired temperature, followed by water quench. The main objective of the solution treatment process is to vary the grain size of the alloy. The effect of solution treatment process on the alloy has been investigated in term of microstructure, grain size, phase present and hardness test, by using optical microscope, scanning electron microscope (SEM) equipped with energy dispersive x-ray (EDX) spectrometer, x-ray diffraction (XRD) technique and Vickers hardness test. As a results, the grain size of the solution-treated alloy were increase as the solution treatment temperature increase from 27.27 μm to 40.86 μm for solution-treated alloy at 950°C to 1200°C, respectively. Three precipitate phases were detected during phase analysis which are NbC, TiC and (Nb,Ti)C on the solution-treated alloy. In addition, as the grain size increase when the solution treatment temperature increase, the hardness value was decrease.

1 Introduction

Ni-based superalloys are often the choice in high temperature applications in aerospace, nuclear, chemical and power industries applications because of their excellent high temperature mechanical properties [1-4], where substantial cooling is needed. This extracts forfeit in terms of thermal efficiency. Materials that allowed higher operating temperatures, high temperature strength and excellent oxidation resistance, results in significant growths in efficiency. Heat resistant Fe-Ni-Cr superalloys can potentially meet this need. Our previous studies has found that the exposure of Fe-Ni-Cr alloy at several oxidizing temperatures recorded excellent oxidation resistance [5-7].

The excellent properties of heat resistant Fe-Ni-Cr superalloys at high temperature conditions is achieved by their good resistance to industrial environments and high strength economical alloy, providing cost-effective solutions to many maintenance problems in the heat-treating and industrial heating industries. Along with potential application and comparable materials composition, HR-120 superalloy were selected as candidate materials

* Corresponding author: noraziana@unimap.edu.my
for solution treatment study especially for thermal related applications. HR-120 superalloy, which is Fe-40Ni-24Cr alloy is a heat-resistant alloy with good oxidation resistance [8-10], and excellent strength at elevated temperature. It is used for heat treating equipment, waste incinerators, muffles, retorts and furnace components [11].

HR-120 superalloy is a solid-solution strengthened, austenitic and heat resistant alloy [12-13]. The alloy is solution-annealed at a temperature ranging from 1175 to 1230ºC and rapidly cooled [11]. The material is mainly composed of face center cubic austenitic grains with annealing twins [14]. Annealing twins occurs during cooling due to the change in crystal system because of re-arrangement of unstable crystal structure and transform into another more stable form. Annealing twins are fairly common in FCC metals [15]. The HR-120 superalloy is composed of austenitic phase with annealing twins which usually found in alloy with high nickel content [16-17]. HR-120 superalloy is a heat resistant alloys with excellent metallurgical stability at high temperature exposure. The comparable equiaxed austenitic structure is stable at prolonged high temperature with excellent oxidation resistant properties.

Extensive investigations have been conducted on heat treatment of Fe-Ni-Cr superalloy to optimize the influence of test parameters on physical and mechanical properties. Heat treatment process has great effect on the mechanical properties and the microstructure of superalloy. The effect of microstructure on superalloy has been widely studied including heat treatment, aging treatment, solution treatment, thermomechanical treatment and surface treatment [18-24]. In order to develop an understanding on the grain alteration through solution treatment, the solution treatment process on HR-120 superalloy, which is Fe-40Ni-24Cr alloy has been carried out. The finding from this research will give better understanding of the microstructure and hardness of the alloy.

2 Methodology

The materials used in this study is a Ni-based HR-120 superalloy, which is Fe-40Ni-24Cr alloy (ASTM-B-409: ASME-SB-409, UNS N08120). The chemical composition of this alloy has been analyzed using optical emission spectrometer (OES), presented in Table 1.

Table 1. Chemical composition of Fe-40Ni-24Cr alloy in weight percent (wt. %) with balance Fe.

| Alloy / Element | Ni  | Cr  | C   | Al  | Ti  | Si  | Mn  | P   | Cu  | N   | Nb |
|----------------|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|----|
| Fe-40Ni-24Cr   | 40  | 24  | 0.048 | 0.080 | 0.029 | 0.441 | 0.702 | 0.014 | 0.114 | 0.20 | 0.44 |

The alloy were undergoing a solution treatment process at six different temperature, namely 950°C, 1000°C, 1050°C, 1100°C, 1150°C and 1200°C, soaking for 3 hours followed by water quench. The solution-treated samples was hot mounted in acrylic transparent resin and was ground using silicon carbide grinding paper P320, P500, P600, P800 and P1000 grit followed by polishing using diamond paste of 9 μm and 3 μm, and then final polishing with colloidal silica 0.04 μm to obtain a smooth and mirror like finish. The polished sample was etched in solution of 2 parts glycerol (C_{3}H_{8}O_{3}), 3 parts hydrochloric acid (HCl) and 1 part nitric acid (HNO_{3}) for 5-120 seconds to reveal the microstructure. The average grain diameter was measured based on at least 5 optical micrographs using Heyn linear intercept procedure ASTM E112. The solution-treated samples were characterized in terms of microstructure, grain size, phase analysis and hardness test. The microstructure of solution-treated samples were examined using optical microscope model NIKON equipped with i-Solution image analysis software and scanning electron microscope (SEM) coupled with energy dispersive x-ray (EDX) analyzer model JOEL JSM-6460 LA and PHILIPS XL40. The phase analysis
was examined using x-ray diffraction (XRD) technique model SIEMENS coupled with BRUKER D5000 XRD instrument software. The hardness test was examined using Vickers hardness tester model Matsuzawa-DVK II series.

3 Results and Discussion

3.1 Microstructure of Solution-Treated Alloy

Fig. 1 shows the optical micrographs of the Fe-40Ni-24Cr alloy for all solution-treated samples. The optical image shows an austenitic matrix and primary precipitates of carbides present at the grain. In the Fe-40Ni-24Cr alloy, the addition of titanium, niobium and carbon have a tendency to form simple MC-type carbides, which are TiC, NbC or (Nb,Ti)C during high temperature exposure [24-26]. Fig. 2 shows SEM image of as-received Fe-40Ni-24Cr alloy with two area of EDX analysis. The large precipitates detected at spectrum 1 indicates the enrichment of element Nb and C as presented in chemical composition of EDX analysis as shown in Table 2. This nearly spherical morphology suggested the formation of NbC precipitate. Whereas, EDX analysis at spectrum 2 indicates the presence of major element Nb, Ti and C. The results suggested that at this area composes of precipitates of TiC and/or (Nb,Ti)C.

![Optical micrographs of solution-treated Fe-40Ni-24Cr alloy at different temperature](image)

Fig. 1. Optical micrographs of solution-treated Fe-40Ni-24Cr alloy at different temperature: (a) 950°C, (b) 1000°C, (c) 1050°C, (d) 1100°C, (e) 1150°C and (f) 1200°C.

Fig. 3 shows the average grain sizes of solution-treated Fe-40Ni-24Cr alloy at different solution treatment temperatures. Results indicate the grain size increased with increase in solution treatment temperature. Solution-treated sample at 950°C for Fe-40Ni-24Cr alloy resulted finest grain size which is 27.27 μm, followed with linear increase in size up to 40.86 μm for solution-treated sample at 1200º. The grain size of as-received Fe-40Ni-24Cr alloy was 38.13 μm. The as-received sample had been annealed by the material supplier at the temperature of 1177º during manufacturing process. Effect of grain size coarsening is primarily caused by the carbon content in the grain boundary carbides dissolve into the alloy matrix, which leads to atypical grain growths occurred, thus reducing the pinning effect and
the obstacle of grain growth. Similar observation was recorded by other researcher [23, 27], which discovered the irregular grains growth at solution-treated temperature above 1150ºC for Fe-23Ni-12Cr and Fe-38Ni alloy.

Fig. 2. SEM image of the as-received Fe-40Ni-24Cr alloy.

Table 2: EDX analysis of the as-received Fe-40Ni-24Cr alloy (atomic %).

| Spectrum | Nb  | Ti  | C    |
|----------|-----|-----|------|
| 1        | 53.78 | -   | 46.22 |
| 2        | 16.16 | 67.54 | 16.30 |

Fig. 3. Effect of solution treatment on the grain size of Fe-40Ni-24Cr alloys.

3.2 Phase Analysis of Solution-Treated Alloy

Fig. 4 shows the x-ray diffraction patterns of solution-treated samples of Fe-40Ni-24Cr alloy at 1100ºC. This sample was chosen to represent other samples due to the similar pattern of all solution-treatment samples. The most intense peaks belong to the base alloy austenite phases. In addition, less intense peaks were also identified which belong to the NbC, TiC and (Nb,Ti)C phases. The NbC and (Nb,Ti)C peak were overlap on each other. The mixed presence of TiC and (Nb,Ti)C precipitates is supported by EDX analysis (Fig. 2, spectrum 2) which recorded the enrichment of Ti, Nb and C elements, indicating mixed existence of both carbides. According to other researchers [23-26], addition of Ti, Nb and C in the alloy system would favour a formation of MC carbides during high temperature exposure which are, TiC, NbC and/or (Nb,Ti)C.
3.3 Hardness of Solution-Treated Alloy

The hardness test results of solution-treated Fe-40Ni-24Cr alloy is shown in Fig. 5. The results indicate that increasing in the solution treatment temperatures was decrease the hardness of the alloy. The hardness value of Fe-40Ni-24Cr alloy was highest at solution-treated 950°C which is 207.66 HV followed by linear decrease of the hardness until solution-treated at 1200°C. However, the decrease in hardness value at higher solution treated temperature at 1100°C to 1200°C is small compared to lower solution treatment temperatures from 950°C to 1050°C showing large value range. The decrease in hardness value as temperature increases is due to the grain become larger. Similar observation has been recorded by other researcher [27]. The hardness value for as-received Fe-40Ni-24Cr alloy is 193.67 HV. Hardness is a resistance to plastic deformation, usually by indentation. Solution treatment process at high temperature will recovered great amount of imperfection and dislocation. When sufficient thermal energy is supplied during recovery, its allow the arrangement of dislocation into lower energy configuration, hence lowered the internal energy of the recovered metal. Prolong the exposure alloy at a temperature at which recrystallization take place will cause a new grain structure transforms completely. At this stage, the ductility of alloy usually increase significantly. Then, the grain growth would take place by the grain boundary migration through atomic diffusion across boundary resulting coarsening of grains. Consequently, as the grain enlarge, the capability of metal to resist plastic deformation will decrease due to reducing of grain boundary area which act as an obstacle for permanent plastic deformation, hence resulting lower hardness value.

![Fig. 4. XRD pattern of solution treatment of Fe-40Ni-24Cr alloy at 1100°C.](image)

![Fig. 5. Effect of solution treatment on the hardness of Fe-40Ni-24Cr alloys.](image)
4 Summary

The solution treatment process on Fe-40Ni-24Cr alloy produced varies grain size. Fine grain size recorded at solution-treated 950ºC which is 27.27 μm, whereas, coarse grain size recorded at solution-treated 1200ºC, which is 40.86 μm. Three precipitate phases were detected consists of NbC, TiC and (Nb,Ti)C. The hardness of the solution-treated samples was decreasing, as the solution treatment temperature increase.

The authors would like to thank the Ministry of Education Malaysia for the research fund under the Fundamental Research Grant Scheme (FRGS) (Project Code. FRGS/1/2016/TK05/UNIMAP/02/4).

References

1. C.N. Athreya, K. Deepak, D.I. Kim, B. Boer, S. Mandal, V.S. Sarma, J. Alloys & Comp. 778 (2019)
2. D. Saber, I.S. Emam, R. Abdel-Karim, J. Alloys & Comp. 719 (2017)
3. J. Jiang, G. Xiao, Y. Wang, Y. Liu, J. Alloys & Comp. 784 (2019)
4. Z. Hao, R. Cui. Y. Fan, J. Lin, Int. J. of Mech. Sci. 150 (2019)
5. N. Parimin, Jemahri, IOP Conf. Ser.: Mater. Sci. Eng. 701 (2019) 012062
6. N. Parimin, E. Hamzah, A. Amrin, Solid State Phenomena 280 (2018)
7. N. Parimin, Z. Zulnuraini, S.A.C. Sakdun, Solid State Phenomena 280 (2018)
8. N. Parimin, E. Hamzah, IOP Conf. Ser.: Mater. Sci. Eng. 701 (2019) 012022
9. N. Parimin, Singh, IOP Conf. Ser.: Mater. Sci. Eng. 701 (2019) 012063
10. N. Parimin, E. Hamzah, A. Amrin, J. of Adv. Reser. In Fluid Mech. & Ther. Sci. 49 (2018)
11. HAYNES International, HAYNES® HR-120® alloy, (H-3125l) (U.S.A: Haynes International, 2019)
12. X. Ledoux, S. Mathieu, M. Vilasi, Y. Wouters, P. Del-Gallo, M. Wagner, Oxid. Met. 80 (2013)
13. L. Couture, F. Ropital, F. Grosjean, J. Kittel, V. Parry, Y. Wouters, Oxid. Met. 80 (2013)
14. Y. He, L. Chen, P. Liaw, R. Medaniels, C. Brooks, R. Seeley, D. Klarstrom, Int. J. of Fatigue 24 (2002)
15. F.C. Campbell, Elements of Metallurgy and Engineering Alloys (Ohio: ASM International, 2008)
16. K.E. Tillous, J. Dulcy, T. Belmonte, Oxid. Met. 82 (2014)
17. L.J. Chen, P.K. Liaw, H. Wang, Y.H. He, R.L. McDansels, L. Jiang, B. Yang, D.L. Klarstrom, Mech. of Mater. 36 (2004)
18. A. Munitz, S. Saltov, G. Gutmann, N. Deromieu, M. Nahmany, Mater. Sci. & Eng. A 742 (2019)
19. C.Li, M. Seyring, X.Li, Y. Zhong, Z. Ren, M. Rettenmayr, Met. & Mater. Trans. A 50 (2019)
20. A. Munitz, L. Meshi, M.J. Kaufman, Mater. Sci. & Eng. A 689 (2017)
21. L. Tan, J.T. Busby, H.J.M. Chichester, K. Sridharan, T.R. Allen, J. of Nuc. Mater. 437 (2013)
22. L. Tan, X. Ren, K. Sridharan, T.R. Allen, Cor. Sci. 50 (2008)
23. D. Cai, Y. Mei, N. Pulin, L. Wenchang, Mater. Lett. 57 (2003)
24. I.A. Sustaita-Torres, S.H. Rodríguez, M.P. Guerrero-Mata, M. Garza, E. Valdés, F. Deschaux-Beaume, R. Colás, Mater. Chem. & Phy. 133 (2012)
25. B. Piekarski, Mater. Charac. 61 (2010)
26. R. Dehmolaei, M. Shamanian, A. Kermanpur, Mater. Charac. 59 (2008)
27. J.F. Zhang, Y.F. Tu, J. Xu, J.S. Zhang, J.L. Zhang, J. Iron & Steel Res. Int. 15 (2008)