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Heat Treatment Effect on Microstructure and Mechanical Properties of Re-Containing Inconel 718 Alloy

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ABSTRACT

The effect of Rhenium additions to the standard Inconel 718 (ST IN718) alloy as well as solution and aging treatments on microstructure and hardness property were studied. The microstructure of Re-containing alloys has higher volume fraction of δ phase than standard alloy. Conventional solution treatment (CST) at 1273 K for 1 h precipitates a thin film of δ phase at the grain boundaries as well as needle-like in γ matrix; however, after modified solution at 1440 K for 3 h long, both types of δ phase precipitates entirely vanish from the microstructure. Small colonies of needle-like δ phase start to appear with aging at 1023 K for 4 h, after CST. Prolonging the aging time to 50 h, these colonies enlarge in size and spread in the matrix. XRD and TEM observations were used to identify the precipitation of hard γ'' and γ' phases. The changing in hardness measurements were evidence about the precipitation of these hard phases. CST have higher rate to increase in hardness with aging time comparing to modified solution specimens.

Keywords: Standard Inconel 718; Rhenium; Solution and Aging treatments; Laves and Delta phases; Hardness Property

1. Introduction

The strength of the commercial superalloys arises from a combination of hardening mechanisms, including contributions from solid-solution elements, particles, and grain boundaries. Solid solution hardening is a powerful method to improve the mechanical properties of this kind of alloys. In addition, Ni base superalloys always contain substantial alloying elements in solid solution to provide strength, creep resistance, and resistance to surface degradation. After suitable heat treatment processes, further hardened is provided by coherent stable intermetallic compounds such as γ' {$Ni_3(Al, Ti)$} and γ'' $Ni_3(Nb)$ [1]. The mechanical properties of Ni base superalloys have been optimized, especially the creep resistance, by introducing large amounts of refractory alloying elements such as W, Ta and Mo. Recently, the alloys designers made a major step by adding Rhenium (Re) with high level up to 6% to single crystal Ni base superalloys [2].

Re as a pure metal has a highest strength and superiority modulus of elasticity in comparison with the other elements in refractory elements group such as Mo, Nb, W and Ta [3-5]. Moreover, Re significantly retarding the coarsening rate of γ' phase at high temperatures or during heat treatments [6]. Also Re such as Mo, W, and Ta has a strong preference for occupying the Al sites in γ' (Ni_3Al) increasing γ' volume fraction. Additions of Re to Ni base superalloys is beneficial in producing alloys with a small negative lattice misfit parameter [7,8]. Atom probe investigations of Re-containing superalloys have reported pileup of Re at the γ/γ' interface and a unique phenomenon for Re among other alloying elements by forming clusters within the γ matrix and these clusters thought to be a more potent source of strengthening than other elements in solid solution [9-12].

In this study, we are trying to improve the mechanical properties of St IN718 alloy by adding various amounts of Re. Also the influence of different heat treatment processes on the mechanical properties of IN718 alloys will be investigated. Another aim of this research is to get the optimum conditions of heat treatment and Re content to reach the best mechanical properties of IN718 alloy.

2. Experimental Procedures

2.1. Chemical Composition and Melting Process

The same chemical composition as shown in **Table 1** has been used for Re-Containing and Standard Inconel 718 in the present work. The melting process for Standard and Re-Containing alloys were accomplished in a graphite resistance electric furnace under argon gas atmosphere.

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Then the melt was cooled down to a predetermined temperature (~40 K superheat for all alloys) and subsequently poured into a preheated ceramic mold under the air atmosphere, the mold preheating temperature being kept at 1273 K in all cases. The ceramic molds which were composed of facecoat slurry including a cobalt aluminate ($CoO-Al_2O_3$) were used to cast the Re-Containing and Standard IN718 ingots.

2.2. Heat Treatment

The ingots were cut horizontally away from the bottom surface by 25 mm. Then a cylindrical disk with thickness of 2 mm was obtained from the bottom surface again. This disk divided to small pieces to be the required specimens for different heat treatment process. The solution heat treatment was applied to the as cast Standard and Re-Containing IN718 alloys. Two types of solution treatment were carried out, the first one, standard solution treatment, was accomplished at 1273 K for 1 h [13-15], while the other one was made at 1440 K for 3 h (modified solution treatment), both were followed by water quenching. Solution treatment was followed by aging heat treatment at two levels of temperatures; 953 K and 1023 K for 2 h to 150 h.

2.3. Hardness Test

Hardness measurements are very important in this kind of research because hardness and tensile strength are clearly related to each other and in turn to the microstructure. Therefore the Vickers hardness was measured with Akashi Hardness Tester Machine (Akashi Co. Ltd.) under a load of 30 Kg. The mean value over ten measurements was evaluated.

2.4. Identification of Microstructure

To investigate the phases presents in the microstructure, Electron Probe Micro Analyzer (EPMA) was used. Shimadzu EPMA-1600 was used to confirm about the precipitation of δ phase. Both Super Back Scattered Electron (SBSE) and Reflected Electron (RE) images were very helpful for recognizing and differentiating between various phases such as Ni_2Nb (Laves), NbC and Delta phase.

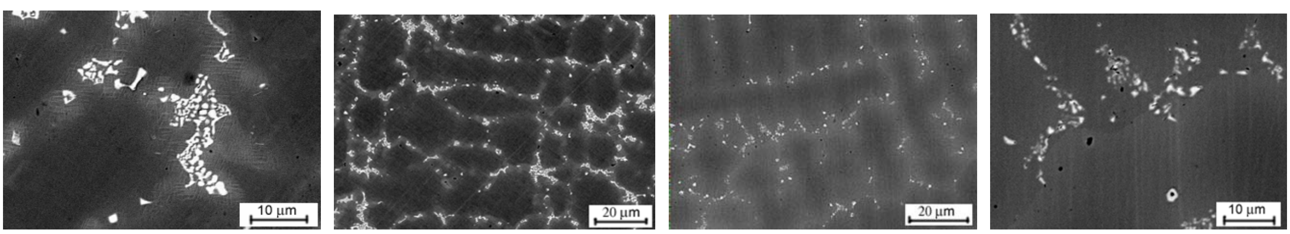
3. Results and Discussion

3.1. Microstructure of Solution Treated Alloys

Figure 1 shows the microstructures of cast standard IN718 alloy after solution treatments at 1273 K for 1 h (standard) and at 1440 K for 3 h (modified). The differences between the two microstructures for standard and modified solution treatment are in volume fraction of Ni_2Nb phase and δ phase (Ni_3Nb). The microstructure of St IN718 alloy after standard solution treatment, **Figures 1(a)** and **(b)**, has a high volume fraction of Ni_2Nb phase in comparison with the microstructure of St IN718 alloy after modified solution treatment, **Figures 1(c)** and **(d)**. As the standard solution treatment has low temperature and shorter time than modified one, therefore, the dissolution of the eutectic phase is less than that for modified solution treatment. Moreover, δ phase is appeared in the standard solution microstructure of St IN718 alloy surrounded the Ni_2Nb phase while there is no evidence on the existence of δ phase in the modified solution microstructure of St IN718 alloy.

Table 1. Chemical composition of specimens (mass%).

% Alloy	C	Nb	Ti	Cr	Fe	Ni	Mo	Al	Re
St In718	0.06	4.88	0.95	19.45	18.39	52.65	3.06	0.56	-
2.4 Re-In718	0.059	4.76	0.93	18.98	17.83	51.39	2.99	0.55	2.40
3.5 Re-In718	0.058	4.71	0.92	18.77	17.63	50.81	2.95	0.54	3.50
6.0 Re-In718	0.056	4.59	0.89	18.28	17.17	49.49	2.88	0.53	6.00



(a) (b) (c) (d)

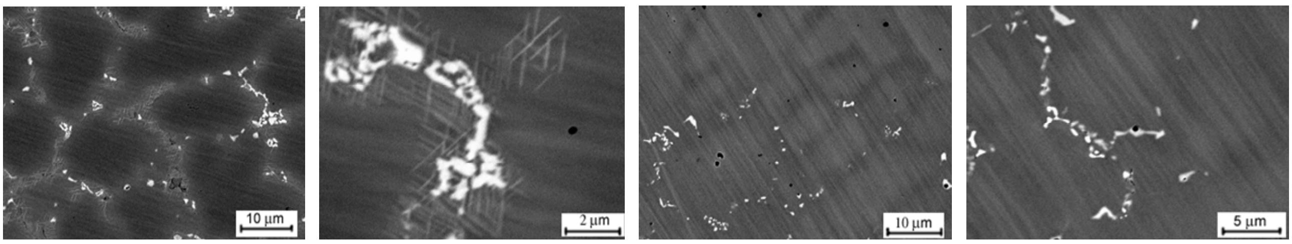
Figure 1. (a) and (b) microstructure of St IN718 after solution at 1273 K/1h, (c) and (d) microstructure of St IN718 after solution at 1440 K/3h.

3.2. Microstructure of Aged Alloys

After aging at 1023 K for 50 h, the volume fraction of Ni_2Nb phase in the standard and modified solution microstructure of St IN718 alloys decreases and the δ phase ratio increases in case of St IN718 alloy, as shown in **Figure 2**. The δ phase precipitates as needle-like morphology in interdendritic region near eutectic Ni_2Nb, **Figure 2(a)**. The aged microstructure of modified solution microstructure has a lower ratio of Ni_2Nb phase in addition to the complete absence of δ phase from the microstructure.

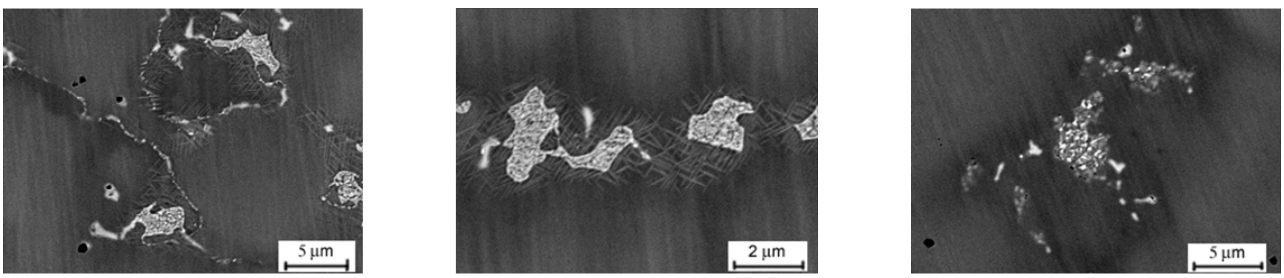
The aged microstructures at 1023 K for 50 h for standard and modified solution of 6% Re-containing IN718 alloy are given in **Figure 3**. **Figures 3(a)** and **(b)** show the aged microstructure of standard solution treated 6% Re containing IN718 alloy, which contains Ni_2Nb and δ phase. This figure shows the δ phase precipitates in both needle and plate like shapes. The needle type δ phase precipitates surround the Ni_2Nb as in case of St IN718 alloy, while the plate like δ phase precipitate along the grain boundaries and this morphology of δ phase was not found in microstructure of modified 6% Re-containing IN718 alloy, **Figure 3(c)**.

After aging for 100 h at 1023 K, the microstructure of modified 6% Re containing IN718 alloy has lower volume fraction of Ni_2Nb phase and still no precipitations of δ phase, (see **Figure 4**). In general, as the aging time prolongs the solution of eutectic Ni_2Nb increases. Dissolution of Ni_2Nb phase leads to increase the available Nb atoms to combine with Ni again to precipitate as a γ'' hard phase in the matrix. This is the interpretation of the cause of increasing hardness values versus the aging time. At the same time the prolonging time for aging at 1023 K, increase the probability for precipitation of Re clusters as it is a diffusion dependence process leading to elevate the hardness levels. **Figures 5** and **6** illustrate the line analysis for Ni_2Nb, δ and NbC phases in aged St IN718 alloy, standard solution treatment. In **Figure 6**, the Ni content in Ni_2Nb phase is lower while the Nb content is higher than in γ matrix. In case of δ phase, the Ni content is higher in comparison with γ matrix.



(a) (b) (c) (d)

Figure 2. (a) and (b) microstructure of St IN718 (1273 K/1h), (c) and (d) microstructure of St IN718 (1440 K/3h), (aged at 1023 K for 50 h).



(a) (b) (c)

Figure 3. (a) and (b) microstructure of 6% Re-IN718 (1273 K/1h), (c) microstructure of 6% Re-IN718 (1440 K/3h), (aged at 1023 K for 50 h).

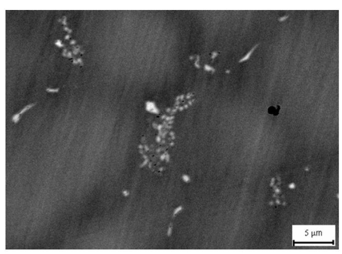


Figure 4. Microstructure of 6% Re-IN718 (1440 K/3h), (aged at 1023 K for 100 h).

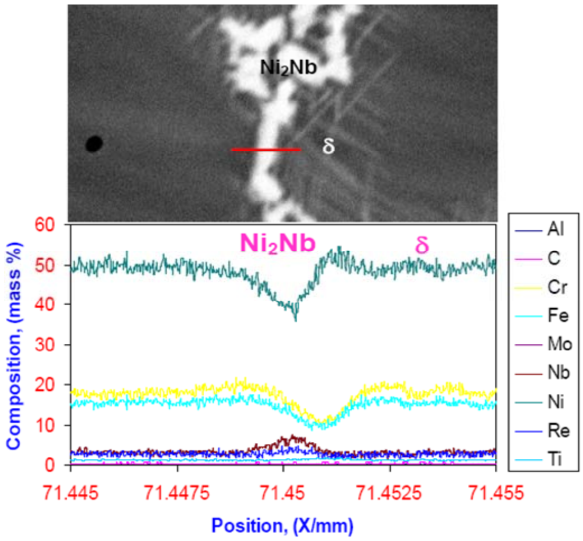


Figure 5. Line analysis through Ni_2Nb (Laves) and (Ni_3Nb) Delta phase (St IN718, 1023 K/50h - 1273 K/1h).

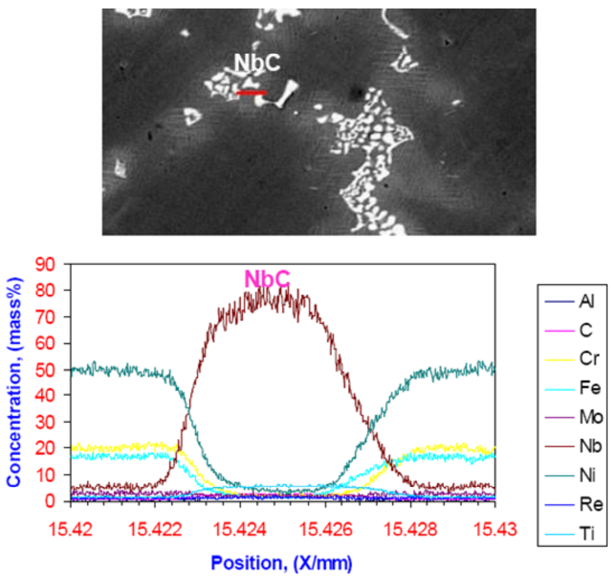


Figure 6. Line analysis through NbC (St IN718, 1023 K/50h 1273 K/1h).

3.3. Hardness Measurements

Hardness gives a good indication about the precipitations of hard phases such as γ'' and γ'. Therefore after different heat treatments conditions for solution and aging processes, the hardness of as cast standard and Re-containing IN718 alloys was measured to find out the relationship between heat treatment and precipitation of hard phases in the microstructure.

3.3.1. Standard Solution and Aging at 953 K

The standard and Re-Containing IN718 alloys have been applies for the solution heat treatment at 1273 K for 1 h then followed by aging process at 953 K for different durations of time. The hardness values for standard and Re- containing IN718 alloys are shown in **Figure 7**. Generally, as the aging time increases the hardness value for both standard and Re-containing IN718 alloys increases as well. The hardness measurements for standard IN718 alloy always are lower than that for Re-containing IN718 alloys, as shown in **Figure 7**. Additionally as the Re co- ntent in IN718 alloy increase the hardness also increase when compared after the same aging time. The maximum hardness was obtained by adding 6% Re to standard IN718 alloy while the standard alloy has the lowest hardness value in comparison with Re-containing alloys, at the same conditions. For instance, after 150 h aging time, the hardness for standard IN718 alloy is about 320 Hv and increased to 335 Hv for 2.4% Re containing alloy. By increasing the Re content to 3.5%, the hardness increased to 346 Hv and finally the hardness reach about 369 Hv with 6% Re-containing IN718 alloy.

This increment in hardness value for standard IN718 alloy depend mainly on the precipitation of hard γ'' and γ' phases. As the aging time prolonged, the ratio of these hard phases increased, which in turn increase the hardness level. While in Re-containing IN718 alloys, the increase in the hardness value could be related to the existence of both hard γ'' and γ' and the precipitation of nano-scale of Re cluster (1 nm) in γ matrix [16]. This could be the clue for the hardness difference between standard and Re-containing IN718 alloys. Additionally by increasing the Re content in IN718 alloy the Re cluster ratio increases and in turn the hardness level is increased. **Figure 8** demonstrate the formation of rhenium pentamer, which consists of five atoms of Re combine together into one cluster [17].

3.3.2. Standard Solution and Aging at 1023 K

After standard solution heat treatment process, as the aging temperature increases from 953 K to 1023 K, the hardness level increases either. As shown in **Figure 9**, the hardness measurements for standard and Re-containing

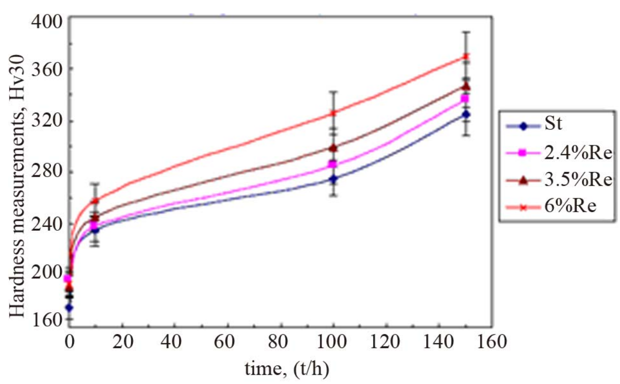


Figure 7. Hardness for aged standard and Re-containing IN718 alloys at 953 K at different aging time (standard solution).

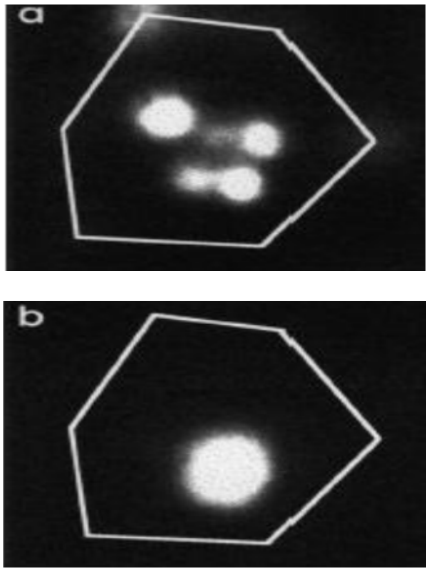


Figure 8. Formation of rhenium pentamer five Re atoms (a) have combined into one cluster (b) [16].

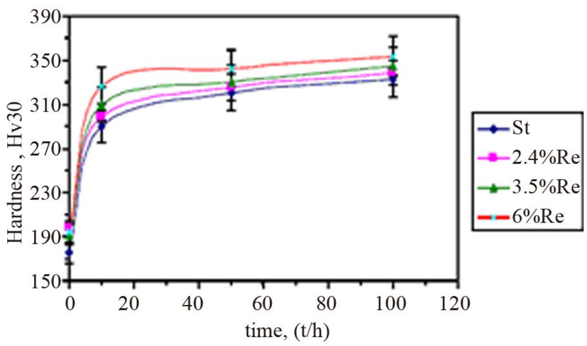


Figure 9. Hardness for aged standard and Re-containing IN718 alloys at 1023 K at different aging time (standard solution).

IN718 alloys are increased with the prolongation of aging time. At any condition of aging heat treatment, the hardness of standard IN718 alloy is the lowest and increases by increasing Re additions to 2.4% and 3.5% and the maximum hardness is obtained with 6% Re-containing IN718 alloy. In this figure, it could be noticed that the hardness of solution IN718 alloys is increased rapidly just after aging at 1023 K for 10 h. For example, the hardness of standard IN718 alloy after solution treatment is 174 Hv and increased after aging at 1023 K for 10 h to 289 Hv. While the hardness of 6% Re-containing IN718 alloy after solution only is about 200 Hv and increased to 325 Hv after the same aging conditions.

After 100 h aging time, the hardness for standard IN718 alloy is about 330 Hv and for 6% Re-containing IN718 alloy reaches 355 Hv. While after the same duration time of aging but at aging temperature of 953 K, the hardness of standard IN718 alloy is 274 Hv and for 6% Re-containing IN718 alloy is 325 Hv. For the standard IN718 alloy the hardness is related to the precipitation of γ'' and γ' phases while in case of Re-containing IN718 alloys, in addition to the precipitation of γ'' and γ' phases, the precipitation of Re clusters in γ matrix is also affect the hardness values. In addition, the Re pile up adjacent to γ' reject the γ' coarsening during heat treatment resulting an improve in hardness measurements [18].

3.3.3. Modified Solution and Aging at 1023 K

Figure 10 illustrates the relationship between hardness measurements of standard and Re-containing IN718 alloys and aging duration time. Standard and Re-containing alloys had been applied for solution heat treatment first at 1440 K for 3 h, then aged at 1023 K. In comparison with **Figure 9**, the increasing rate of hardness level after 10 h in **Figure 10** is lower than that in **Figure 9**. As the modified solution treatment has higher temperature and longer time than for standard solution one, the diffusion for the eutectic, Ni_2Nb, phase is more affected by the modified solution and in turn the ratio of Nb element that diffuse in matrix is higher than in standard solution process. Additionally, Nb is very important and main element in hard γ'' (Ni_3Nb) phase, which is a vital source of increasing hardness level for IN718 alloys. Based on the earlier, the gathering of the required Nb atoms to precipitate γ'' takes longer time in the modified solution treatment than in standard solution one. This needed duration of time just before precipitations of γ'' is called "incubation period".

The longer time and higher temperature in modified solution than standard solution lowering the volume fraction of Ni_2Nb phase in microstructure, which means increasing the ratio of Nb element in matrix leading to expanding the volume fraction of γ'' in the microstructure. Therefore, the hardness level of IN718 alloys with modi fied solution treatment after aging at 1023 K is higher than that for IN718 alloys with standard solution one. After 100 h aging time at 1023 K, the hardness for modified solution standard IN718 alloy is 345 Hv and for 6% Re-containing IN718 alloy is 394 Hv while in case of standard solution standard IN718 alloy is 330 Hv and for 6% Re-containing IN718 alloy is only 355 Hv.

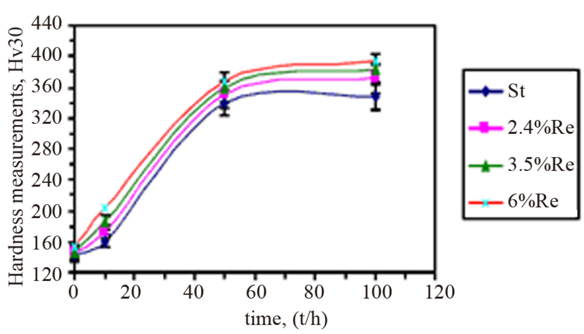


Figure 10. Hardness for aged standard and Re-containing IN718 alloys at 1023 K at different aging time (modified solution).

4. Conclusions

1) Comparing to the conventional solution treatment alloys, modified solution treatment alloys have higher levels of hardness for the Standard and Re-containing IN718 alloys.

2) The microstructure of standard IN718 alloy has a precipitation of δ phase with morphology of needle shape surrounding Ni_2Nb phase after standard solution while no precipitation of δ phase after modified solution.

3) The microstructure of Standard IN718 alloy that aged at 1023 K for 50 h with modified solution has lower volume fraction of Ni_2Nb phase than that with conventional solution alloys and has no precipitation of δ phase while standard solution alloys has needle type.

4) Aging at 953 K increases the hardness of Standard and Re-containing IN718 alloys, which was solution treated at 1273 K for 1 h (conventional solution), as the aging time increases the hardness increases. This is originating from the precipitation of the hard phases such as γ', γ'' and Re clusters.

5) The hardness level for Re-containing IN718 alloys is higher than that for Standard IN718 alloys after any condition of heat treatment. The precipitation of Re clusters in addition to the precipitation of γ', γ'' phases in Re-containing IN718 alloys could be the reason.

6) By increasing the aging temperature from 953 K to 1023 K, the hardness increases for both Standard and Re-containing IN718 alloys, with conventional solution, when compared after the same aging time.

7) 6% Re-containing IN718 alloy that aged at 1023 K for 50 h with conventional solution treatment has higher volume fraction of Ni_2Nb phase in comparison with modified solution treated alloys. Additionally, 6% Re-containing IN718 alloy with conventional solution has precipitation of two types of δ phase; needle shape and plate like along the grain boundaries while modified solution alloy has no evidence of δ phase precipitation.

REFERENCES

[1] T. S. Chester, S. S. Norman and C. H. William, "Superalloys II," John Willey & Sons, Inc., New York, 1976.

[2] P. Caron and T. Khan, "Evolution of Ni-Based Superalloys for Single Crystal Gas Turbine Blade Applications," *Aerospace Science and Technology*, Vol. 3, No. 8, 1999, pp. 513-523. doi:10.1016/S1270-9638(99)00108-X

[3] Aerospace Structural Metals Handbook, Code 5201, 1963; Code 5206, 1966; Code 5208, 1967; Code 5209, 1971; Code 5211, 1973; US Department of Defense, Mechanical Properties Data Center.

[4] Y. S. Song, W. F. Gao, C. Wang, X. W. Lei and H. L. Wang, "Effect of Heat Treatment Technology on Microstructure, Mechanical Property and Corrosion Resistance of Nickel-Base Alloy Inconel 718," *Journal of Materials Engineering*, Vol. 6, 2012, p. 37.

[5] T. Carneiro and H. S. Moura, "Electron Beam Melting and Refining of Niobium at CBMM," Paper Presented at the Electron Beam Melting and Refining, State of the Art 1998, Bakish Materials Corporation, Englewood, 11 December 1998, pp. 110-125.

[6] J. P. Gu, C. Beckermann and A. F. Giamei, "Motion and Remelting of Dendrite Fragments during Directional Solidification of a Nickel-Base Superalloy," *Metallurgical and Materials Transactions A*: *Physical Metallurgy and Materials Science*, Vol. 28, No. 7, 1997, p. 1533.

[7] H. Murakami, H. Harada and H. K. D. H. Bhadeshia, "The Location of Atoms in Re- and V-Containing Multicomponent Nickel-Base Single-Crystal Superalloys," *Applied Surface Science*, Vol. 76-77, 1994, pp. 177-183. doi:10.1016/0169-4332(94)90340-9

[8] H. Harada, A. Ishida, Y. Murakami, H. K. D. H. Bhadeshia and M. Yamazaki, "Atom-Probe Microanalysis of a Nickel-Base Single Crystal Superalloy," *Applied Surface Science*, Vol. 67, No. 1-4, 1993, p. 299.

[9] J. He, S. Fukuyama and K. Yokogawa, "A Stacking Fault with a Series of Cross Fringes in Inconel 718 Ni-Base Superalloy," *Scripta Metallurgica et Materiala*, Vol. 31, No. 10, 1994, p. 1421.

[10] D. Blavette, P. Caron and T. Khan, "An Atom Probe Investigation of the Role of Rhenium Additions in Improving Creep Resistance of Ni-Base Superalloys," *Scripta Metallurgica*, Vol. 20, No. 10, 1986, pp. 1395-1400. doi:10.1016/0036-9748(86)90103-1

[11] S. Chambreland, A. Walder and D. Blavette, "Early Stages of Precipitation of γ'-Phase in a Nickel Base Superalloy: An Atom-Probe Investigation," *Acta Metallurgica*, Vol. 36, No. 12, 1988, p. 3205.

[12] N. Wanderka and U. Glatzel, "Chemical Composition Measurements of a Nickel-Base Superalloy by Atom Probe Field Ion Microscopy," *Materials Science and Engineering*: *A*, Vol. 203, No. 1-2, 1995, pp. 69-74. doi:10.1016/0921-5093(95)09825-9

[13] G. Appa Rao, M. Srinivas and D. S. Sarma, "Effect of Thermomechanical Working on the Microstructure and Mechanical Properties of Hot Isostatically Pressed Superalloy Inconel 718," *Materials Science and Engineering A*, Vol. 383, No. 2, 2004, p. 201.

[14] W. C. Liu, F. R. Xiao, M. Yao, Z. L. Chen, Z. O. Jiang and S. G. Wang, "Relationship between the Lattice Constant of ϒ Phase and the Content of δ phase, γ'' and γ' Phases in Inconel 718," *Scripta Materialia*, Vol. 37, No. 1, 1997, pp. 59-64. doi:10.1016/S1359-6462(97)00064-X

[15] C. Salama and M. Abdellaoui, "Structural Characterization of the Aged Inconel 718," *Journal of Alloys and Compounds*, Vol. 306, No. 1-2, 2000, pp. 277-284. doi:10.1016/S0925-8388(00)00789-1

[16] G. Muralidharan, R. G. Thompson and S. D. Walack, "Analysis of Precipitation in Cast Alloy 718," *Ultramicroscopy*, Vol. 29, No. 1-4, 1989, pp. 277-283. doi:10.1016/0304-3991(89)90255-6

[17] J. T. Goldstein and G. Ehrlich, "Atom and Cluster Diffusion on Re(0001)," *Surface Science*, Vol. 443, No. 1-2,

1999, pp. 105-115.
doi:10.1016/S0039-6028(99)00950-4

[18] P. J. Warren, A. Cerezo and G. D. W. Smith, "An Atom Probe Study of the Distribution of Rhenium in a Nickel-Based Superalloy," *Materials Science and Engineering A*, Vol. 250, No. 1, 1998, pp. 88-92.
doi:10.1016/S0921-5093(98)00541-3

