Effect of hafnium addition and heat treatment process on the microstructure and mechanical properties of Inconel 718 superalloy

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ABSTRACT

The effects of hafnium (Hf) addition and heat treatment process on the mechanical properties of the Inconel 718 superalloy were studied. The microstructure of samples was observed with scanning electron microscope (SEM). Their phases were analyzed with X-ray diffraction (XRD) and element analysis with electro-probe micro-analysis (EPMA). The results show that a new phase, Ni_5Hf and the MC phase which fused with Hf have formed during the post heat treatment. Moreover, the samples with 1.5 wt% Hf addition and then proceeded with T8 aging heat treatment (1140℃/2hr/AC→R.T.→780℃/10hr/FC→600℃/12hr/AC→R.T.) have a hardness of HRc36, the yield strength of 932 MPa and the tensile 1108 MPa, respectively. Compared to the raw Inconel 718 sampless, the hardness, yield and tensile strength have enhanced for 20%, 5% and 4.3%, respectively after the optimal Hf addition and post heat treatment.

Keywords: Inconel 718 superalloy, Hafnium, Ni-Hf phase diagram

添加鉿元素及熱處理程序對鑄造 Inconel 718 鎳基超合金機械性質之影響

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摘　要

本研究探討添加鉿元素及熱處理條件對鑄造 Inconel 718 鎳基超合金機械性質之影響，並藉由顯微組織觀察及成分分析了解其影響機械性質的原因。 結果顯示，鑄造 Inconel 718 鎳基超合金添加 Hf 元素後，以 XRD 及 EPMA 分析，可得知 Ni_5Hf 及含 Hf 的 MC 相析出；此外當添加 1.5 wt% Hf，進行 1140℃持溫 2 小時固溶處理後空冷至室溫，再升溫至 780℃持溫 10 小時第一階段時效處理爐冷至 600℃持溫 12 小時第二階段時效處理空冷至室溫後，其硬度值為 HRC 36，降伏強度及抗拉強度分別為 932 MPa 及 1108 MPa，較未添加 Hf 元素試片分別提升 20%、5% 及 4.3%。

關鍵詞: Inconel 718 鎳基超合金，Hafnium，Ni-Hf 相圖

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Ⅰ. INTRODUCTION

Inconel 718 is a nickel-based superalloy strengthened mainly by the precipitation of Ni_3Nb (γ”) phase and $Ni_3(Al,Ti)$ (γ’) phase. It has been widely used to a variety of high temperature application such as the nuclear, plastics and petroleum industries, and the pipeworks for chemical transportation for its good corrosion resistance, wear resistance and creep properties. However, it can not be used at temperature higher than 650℃ for a long period owing to the fast coarsening of the main strengthening γ” phase (a tetragonal Ni_3Nb phase) and the formation of the harmful phase such as δ phase which will reduce the mechanical properties of Inconel 718 [1,2].

The research of Loewenkamp et al. indicates that adding rare earth elements such as tantalum (Ta) can improve the high temperature (above 650℃) properties of Inconel 718 superalloy [3]. Another research by Sims et al. also reveals that the addition of minor Hafnium (Hf) element could improve the high and room temperature mechanical properties of the nickel-based superalloys such as MAR-M 246, MAR-M 200 and MAR-M 247 [4]. It also indicates that the addition of Hf could strengthen the matrix of nickel-based superalloys by forming hafnium carbide (HfC) which also has the effect on crack growth prevention. In MAR-M 200 superalloy, the high temperature mechanical properties could be enhanced by adding 1.78 at% Hf. The oxidation resistance and grain size reduction effect could be enhanced when added with an amount less than 1 at% of Hf [5,6].

In Inconel 100 superalloy, HfC could be segregated in grain boundaries and thus has the grain boundary strengthening effect [7,8]. Moreover, when the added amount of Hf is smaller than 0.8 at%, Hf and Ni would form NiHf solid solution, and Ni_5Hf phase would precipitate when the amount of Hf is higher than 0.8 at%. When the added amount of Hf was higher than 2 at% and under the heat treatment process of heating to 1060℃ for 25 hr and then aging at 605, 695℃, respectively, the Ni_5Hf phase nucleated and precipitated in the grain boundaries of Ni-matrix and then grow up into the inside of grains. On the contrast, the Ni_5Hf phase only precipitated in the grain boundaries when heated to 1060℃ for 25 hr and aged at 505℃. The research of Zhang et al. indicated that Ni_7Hf_2 phase would form in Ni-based superalloy with addition of Hf element [9].

As we know that the Ni_5Hf and Ni_7Hf_2 phases have both high melting point of 1190℃ and 1240℃, respectively, as shown in the Ni-Hf binary phase diagram in Fig. 1 [10]. If they can be precipitated in Inconel 718 superalloy by adding of Hf element, the high temperature properties of Inconel 718 superalloy have a chance to be improved. In this study, we select the composition located in the γ and Ni_5Hf coexistent domain according to the 1130℃ isothermal Ni-Al-Hf ternary phase diagram [9] (as shown in Fig. 2). The variations of microstructure and mechanical properties of Inconel 718 superalloy after adding different amount of Hf and undergoing different heat treatment processes was discussed.

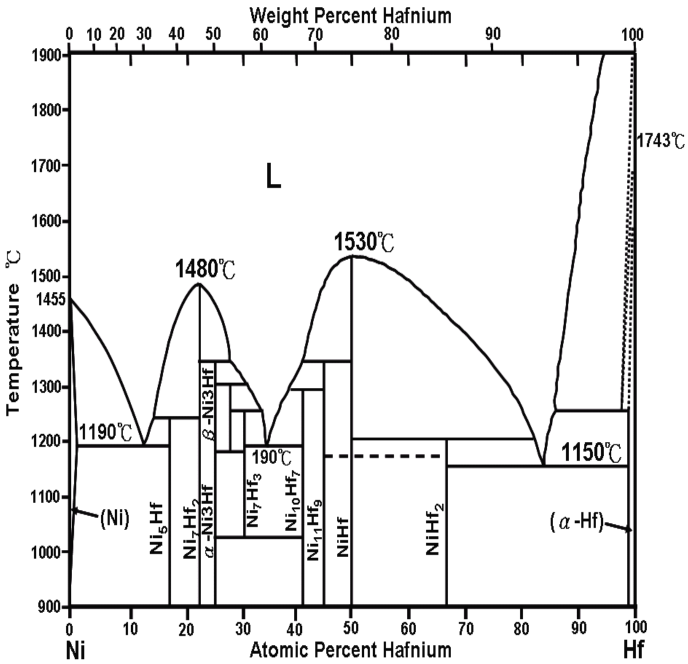


Fig. 1. Ni-Hf binary phase diagram [10].

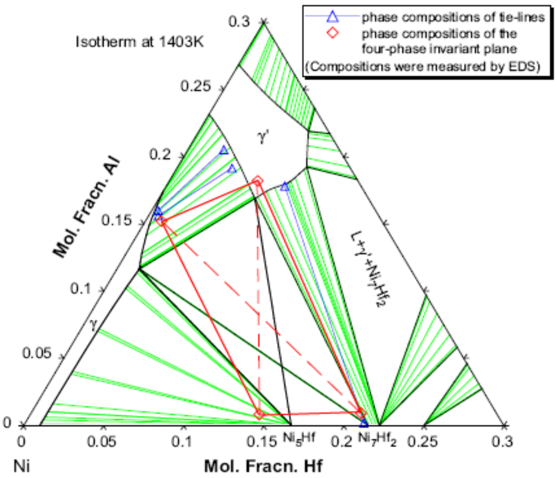


Fig. 2. Isothermal Ni-Al-Hf ternary phase diagram at 1130℃ [9].

Ⅱ. EXPERIMENTAL

The Inconel 718 superalloy round bar with diameter of 70 mm and length of 140 mm produced by IHI Company Japan was chosen as the raw materials. The chemical compositions of IHI Inconel 718 superalloy are listed in Table 1. These raw Inconel 718 bars were re-melted and added with 1, 1.5, and 2 wt% Hf using the DUVAC (VIM electrode and VAR ingot) processes. The remelted ingots were then post heat treated with different parameters and prepared to do the tensile test and hardness test according to ASTM standards. The microstructures of these samples were analyzed with optical microstructure observation and X-Ray diffraction (XRD). Their compositions were analyzed with electro probe micro analysis (EPMA).

The tensile test of samples added with different amount of Hf and underwent different post heat treatment were performed at room temperature to obtain the properties of the yield strength, tensile strength, and percentage of elongation. The tensile specimens were prepared according to ASTM standard as shown in Fig. 3. Tensile tests were performed using a MTS 810 universal material testing machine with maximum load of 100 ton. The parameters of tensile test were list in Table 2.

The HRC hardness tests were performed with a MRK-M2 apparatus produced by Matsuzawa SEIKI Company. Each value plotted represents the mean value over 10 measurements.

The double aging post heat treatment was performed under 2.1×10^{-2} torr vacuum. According to the Ni-Hf binary phase diagram (Fig. 1) and the added amount of Hf, we set the temperature of solution treatment as 1140 ℃ to form Ni_5Hf phase (melting point: 1190 ℃). Meanwhile, a lower solution temperature was set at 960℃ as a contrast to compare the microstructure and mechanical properties of the specimens prepared with different post heat treatment process. Fig. 4 shows the procedure of heat treatment in this work. The sample number and heat treatment conditions are listed in Table 3.

The optimal heat treatment parameters of Inconel 718 are performed by using Taguchi method, the results revealed that the T3 and T8 process had a better hardness and strength, respectively. Therefore, the heat treatment of T3 and T8 process are chosen in this study and the related parameters are listed as below:

T3:
960℃/3hr/AC→R.T.→780℃/14hr/FC→680℃/12hr/AC→R.T.

T8:
1140℃/2hr/AC→R.T.→780℃/10hr/FC→600℃/12hr/AC→R.T.

The heat treated specimens were cut into cylinder samples with 15 mm φ × 5 mm and then hot mounted, ground and polished to prepare for the metallographic observation. Before optical observation, the samples were etched with a corrosive composed of 40 ml HCl, 40 ml CH_3OH and 2 g of $CuCl_2$ [4]. The microstructure examinations were carried out using HITACH S-3000 and JEOL JSM-6500 scanning electron microscopy (SEM). X-ray diffraction (XRD) investigations were conducted using a Bruker D8 diffractometer with Cu Kα radiation (λ=0.15406 nm). Chemical compositions were analyzed using JOEL JXA-8500F electro probe microanalysis (EPMA).

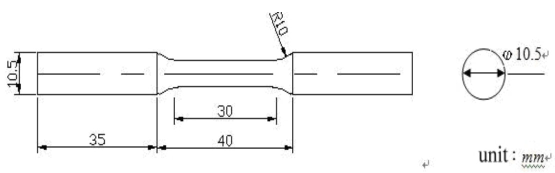


Fig. 3. Dimensions of the tensile specimen.

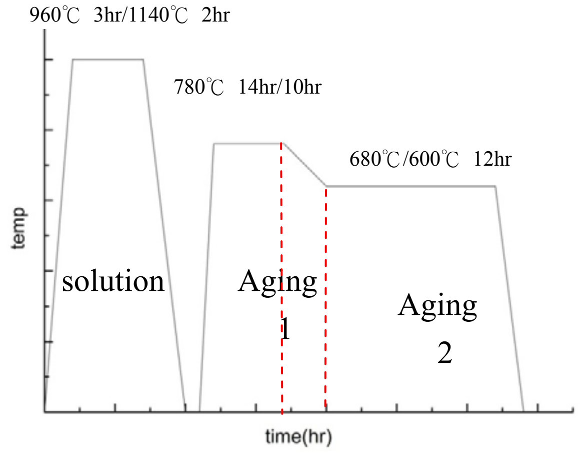


Fig. 4. Heat treatment scheme

Table 1. Chemical compositions of the raw Inconel 718 superalloy

Element	Ni	Cr	Nb+Ta	Mo	Ti	Al	Co	Fe
Wt%	bal	20.11	4.83	3.073	0.85	0.61	0.21	Rem.
Element	Mn	Si	P	C	B	Cu	S	
Wt%	<0.01	0.04	0.002	0.04	0.004	0.01	0.001	

Table 2. The parameters of tensile test

Parameter	Value
Initial Speed (mm/min)	0.20
Normal Gage Length (mm)	25
Secondary Speed (mm/min)	0
Strain Change Point (%)	2

Table 3. Illustration of the designation of sample number and heat treatment procedure

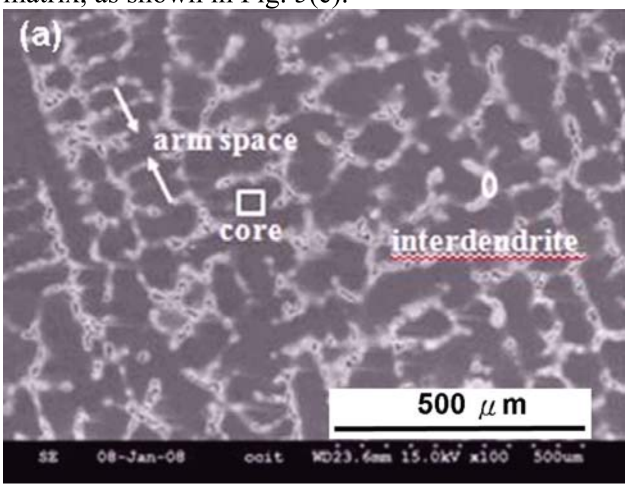
Sample NO.	Hf content (wt%)	Heat treatment condition
H_1	1.0	None
H_2	1.5	
H_3	2.0	
H_{31}	1.0	960℃/3hr/AC→R.T.→ 780℃/14hr/FC→680℃/ 12hr/AC→R.T.
H_{32}	1.5	
H_{33}	2.0	
H_{81}	1.0	1140℃/2hr/AC→R.T.→ 780℃/10hr/FC→600℃/ 12hr/AC→R.T.
H_{82}	1.5	
H_{83}	2.0	

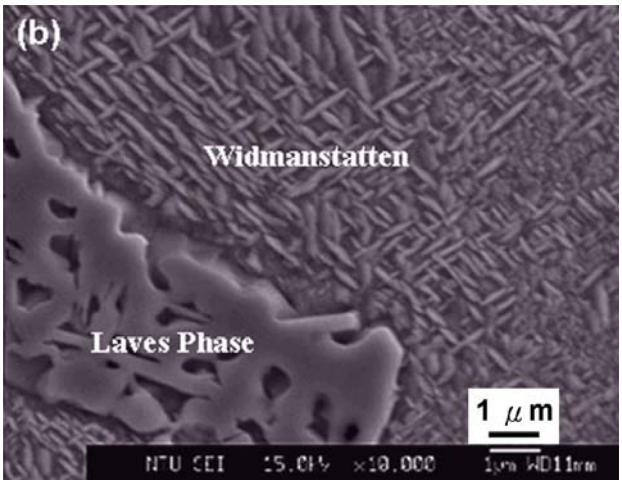
Ⅲ. RESULTS AND DISCUSSION

3.1 The microstructures

3.1.1 Microstructure of raw Inconel 718 superalloy

The typical microstructure morphology of as-cast Inconel 718 superalloy is shown in Fig.5. Fig. 5(a) indicates that the inter-dendritic structures (white areas) formed in the casting work-piece. The distance between dendrite layers was called as arm space. The detailed microstructures at higher magnification are shown in Fig. 5(b) and (c). In Fig.5(b), Nb element segregated in grain boundaries and formed porous Laves phase and Widmanstatten structure. In addition to them, the γ’+γ” phases and needle-like δ phases were also shown in matrix, as shown in Fig. 5(c).





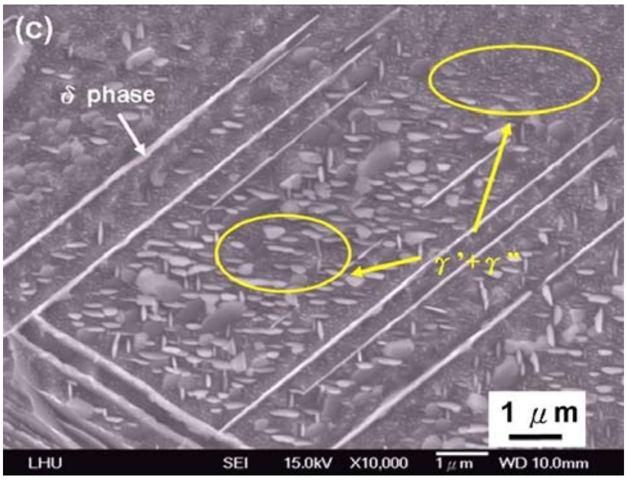


Fig. 5. The typical microstructure in casting Inconel 718 superalloy (a) inter-dendritic structure (b) Laves phase and Widmanstatten structure (c)δ,γ'and γ"phase.

3.1.2 Microstructure of the Inconel 718 after addition of Hf element

Figure 6(a) and (b) show the microstructures of Inconel 718 superalloy added with 1 wt% and 2 wt% Hf respectively, before post heat treatment. Several precipitated phases can be observed in the inter-dendritic structure, and the needle-like δ phase was not found as compared to the as-cast Inconel 718 sample. We infer that the addition of Hf element would restrain the precipitation of δ phase. In addition, two different type of microstructure with gray and white areas were observed in back scattering electron (BSE) mode observation. Their chemical compositions were analyzed with EPMA and listed in Table 4. It indicates that the Hf concentration in precipitated "white phase" (6.88 at%) is higher than that of "gray phase" (2.56 at%), and the concentration of Nb in both phases is higher than the Hf concentration. Since the concentration of Hf and Al element in "white phase" was about 6.88 at% and 0.55 at% respectively, in light of the Ni-Al-Hf ternary phase diagram and the research reported by Zhang et al. [9], we infer that the "white phase" should be Ni_5Hf. The composition of "gray phase" is similar to the composition of traditional Laves phase [11] but with some dissolved Hf and higher carbon content. Therefore, the "gray phase" should be a modified Laves phase. The red circle in the top left area of the Fig. 6 (a) is the composite microstructure of γ + Ni_5Hf and Laves phase. The precipitation phase in the red circles underneath should be the metal carbide (MC) type carbide in which the M is Nb, Ti or Hf element.

Figure 7 shows the low magnification microstructure observations of the raw Inconel 718 and those added with different content of Hf before post heat treatment. In these SEM images, the inter-dendrite structures formed during casting process with different cooling rate still existed in the samples after adding different amount of Hf. The variation of arm space of the inter-dendrites and the tensile strength of above specimens are listed in Table 5. By comparing arm space of the inter-dendrite structure of each specimen and their mechanical properties, it indicates that the arm space decreased with the increase of added Hf content, but there shows no apparent effect of the Hf content on the mechanical properties of Inconel 718.

In traditional casting of Inconel 718 superalloy, Nb is readily easy to segregate during the cooling process. In order to know whether Hf has also the same effect of segregation as Nb has, the line scan mode of EPMA was used to examine the dispersion of Hf and Nb in the samples added with 2 wt% Hf before and after post heat treatment.

The variation of Hf and Nb elements along the "AB" line the the matrix and inter-dendrite of Inconel 718 in Fig. 8 and 9 were examined by EPMA. Figure 8 shows that the content of both Hf and Nb in inter-dendrite are higher than matrix and the segregation of Nb is evident. Moreover, the content of Hf in the precipitation phase (as shown in the point ① and ② in Fig. 8) is higher than in other positions. This result indicates that the Hf element concentrated on the interface between the matrix and inter-dendrite before heat treatment. The microstructure of the specimens after T8 (1140℃/2hr/AC→R.T.→780℃/10hr/FC→600 ℃/12hr/AC→R.T.) post heat treatment is shown in Fig. 9. It indicates that more Nb and Hf segregated into inter-dendrite areas. The content of Hf in positions ① and ② are higher than other areas, and the content of Nb in gray phase (see position③) is also higher than other areas. Therefore, we deduce that the Nb and Hf elements have segregated in the inter-dendrite areas and form MC carbides, Laves phase and Ni_5Hf phase after heat treatment.

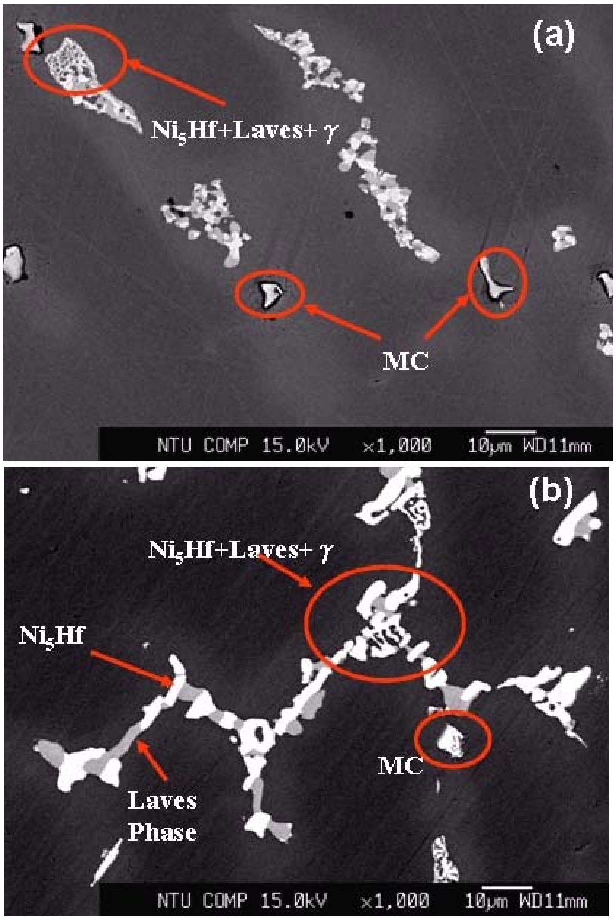


Fig. 6. SEM images showing the microstructures of the Inconel 718 superalloy added with different amount of Hf: (a) 1 wt% Hf ; (b) 2 wt% Hf.

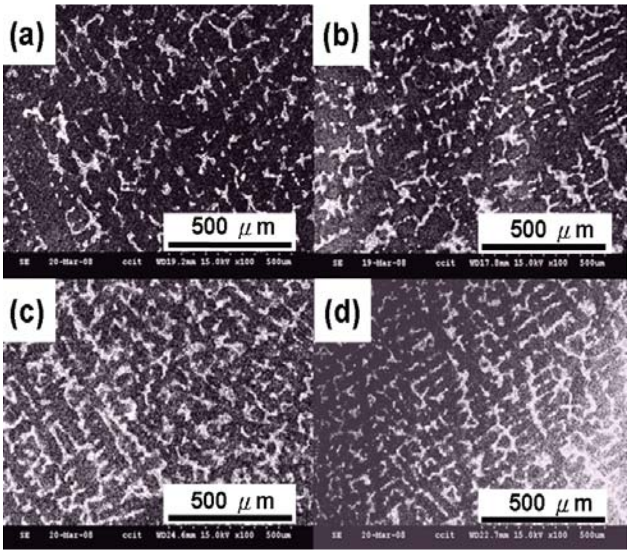


Fig. 7. SEM images showing inter-dendrite structure of raw Inconel 718 and different additions of Hf content: (a) raw Inconel 718; (b) 1 wt% Hf; (c) 1.5 wt% Hf; (d) 2 wt% Hf.

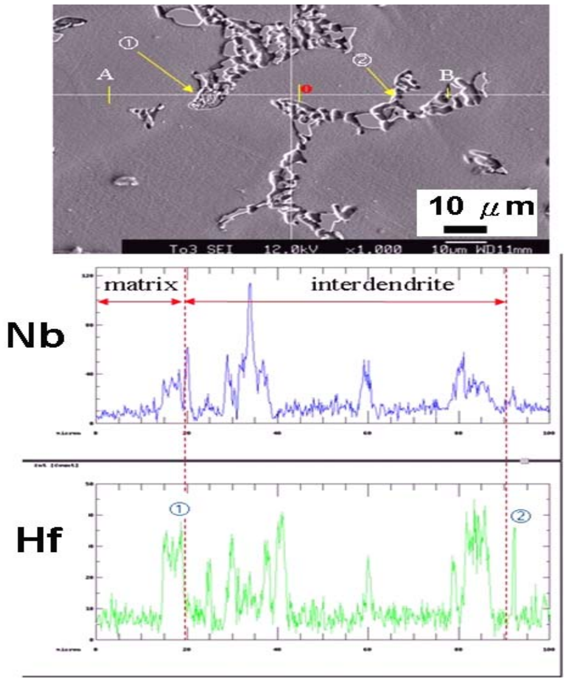


Fig. 8. The distribution of Nb and Hf content along the line AB passing through the matrix and inter-dendrite of Inconel 718 added with 2 wt% Hf before post heat treatment.

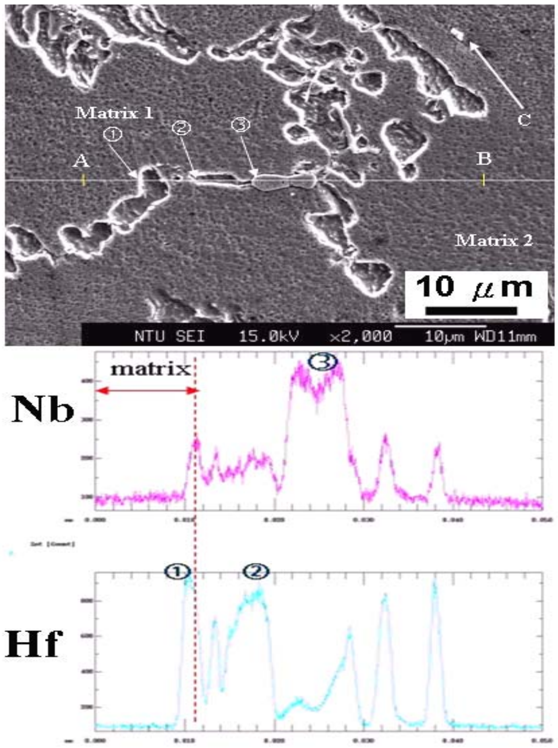


Fig. 9. The change of Nb and Hf element between matrix and inter-dendrite of Inconel 718 added with addition 2 wt% Hf after T8 post heat treatment.

Table 4. Chemical compositions of various precipitations

Element Content (at%)	Al	Cr	C	Ti	Fe	Nb	Ni	Mo	Hf
White phase	0.55	3.72	12.90	1.29	6.08	9.30	58.97	0.24	6.88
Gray phase	0.51	14.74	10.03	1.26	13.24	15.06	39.30	3.25	2.56
Conventional Laves	0.42	14.11	2.79	1.95	12.12	17.10	47.52	3.96	-
MC-Carbide	0.00	0.86	44.49	5.80	0.79	41.51	2.08	0.59	3.88

Table 5. The effect of added content of Hf on the arm space and mechanical properties of Inconel 718 superalloy

Element(wt%)	Arm Spacing Size (μm)	Y.S. (MPa)	UTS (MPa)
Inconel 718	39.5±5	592	852
Inconel 718–1% Hf	38.8±5	543	856
Inconel 718–1.5% Hf	35.1±5	559	802
Inconel 718–2% Hf	35.6±5	563	803

3.1.3 Microstructure of Hf added Inconel 718 superalloy after heat treatment

Figure 10 (a) shows the BSE SEM image of Inconel 718 superalloy added with 1 wt% Hf after T3 (960℃/3hr/AC → R.T. → 780℃/14hr/FC → 680℃/12hr/AC → R.T.) post heat treatment with sample number of H_{31}. Since the γ” phase transformed into the stable δ phase at 960℃ solution temperature, the needle-like δ phases formed around the precipitation phase. Figure 10(b) shows the SEM image of the specimen after T8 (1140℃/2hr/AC→780℃/10hr/FC→600℃/12hr/AC→R.T.) post heat treatment with sample number of H_{81}. In contrast to the H_{31} sample shown in Fig.10 (a), there is no needle-like δ phase formed around the precipitation phase. It is because that the δ phase would fully dissolve into matrix under the 1140℃ solid solution temperature. The phenomenon of different precipitation amount of δ phase caused by low (960℃) and high (1140℃) solution temperature was also reported on the research of Cai et al. [15]. The similar result was also revealed in the study of heat treatment temperature (under 940 and 1040℃) on mechanical properties of Inconel 718 [16].

In addition, by comparing the microstructure of H_{31} and H_{81}, we found that the amount of gray phase in sample H_{31} marked with red ellipse is more than that on sample H_{81}. We infer that the Laves phase re-dissolved into matrix and the Nb content in matrix of the sample heat treated with T8 process having higher solution temperature has increased.

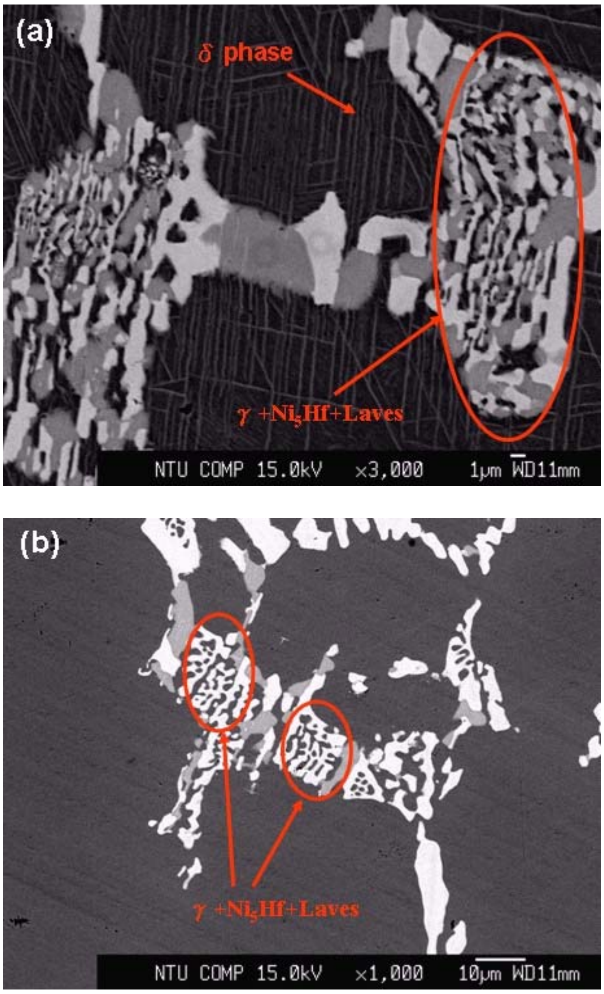


Fig. 10. SEM images showing various precipitates in Inconel 718 added with 1 wt% Hf after different heat treatment process (a) T3 (b) T8.

3.2 X-ray diffraction analysis

Figure 11. shows the X-ray diffraction patterns of raw Inconel 718 sample and those added with different content of Hf. The raw

Inconel 718 sample has a typical XRD pattern marked as O, in which the diffraction peaks occurred at 2θ = 43.38°, 50.49°, 74.35°, and 90.82° indicating the diffraction of (111), (200), (220), and (311) planes of γ matrix. A small diffraction peak near the (111) diffraction peak of γ matrix can be observed and it was identified as the diffraction peak of δ phase. However, the diffraction peak of δ phase disappeared and the diffraction intensity of γ matrix become weaker after the addition of Hf, which is in accordance with the preceding discussion of the microstructures. There are several diffraction peaks of new phases near the (111) peak of γ phase and these peaks are diffracted from the MC carbide, Ni_5Hf, and Laves phase through the comparison with JCPDS database. In addition, the quantity of Laves phase increase with increase of Hf content.

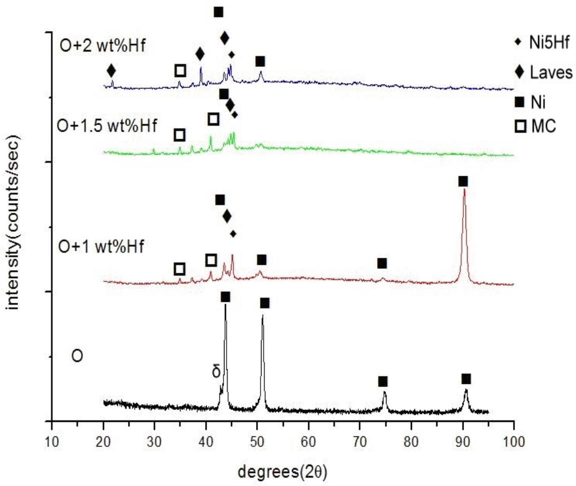


Fig. 11. X-ray diffraction patterns of the raw Inconel 718 and those added with different amount of Hf.

3.3 Mechanical properties

3.3.1 Hardness test

The hardness variations of Inconel 718 added with different content of Hf and underwent different heat treatment are shown in Figure 12. The hardness increases with the increase of Hf content before post heat treatment, and the sample added with 2 wt% Hf raises the hardness to 9% (from 20 HRC to 25 HRC) than the original value. When underwent T3 post heat treatment, the hardness of the raw Inconel 718 samples raised from HRc20 to HRc38 and kept at the hardness around HRc 37 to 38.5 as the addition of Hf from 1.0wt% to 2.0wt%. The samples underwent T8 post heat treatment shows that the hardness of raw Inconel 718 samples raised from HRc20 to HRc30 and increased gradually to about HRc36.8 as the Hf addition increased to 1.5wt% and then drop to HRc35 when the Hf addition was 2.0wt%.

Figure 12 also shows that the hardness of raw Inconel 718 samples underwent T3 post heat treatment is obviously higher than those underwent T8 heat treatment. It is due to the fact that the solution temperature of T3 heat treatment process is 960℃, the γ” phase will transform into stable δ phase and form a great quantity of δ phase (i.e. Widmanstatten structure in Fig. 10) during T3 aging process. However, the samples added with 1.0 to 1.5wt% Hf and underwent T3 or T8 post heat treatment show no big difference in hardness.

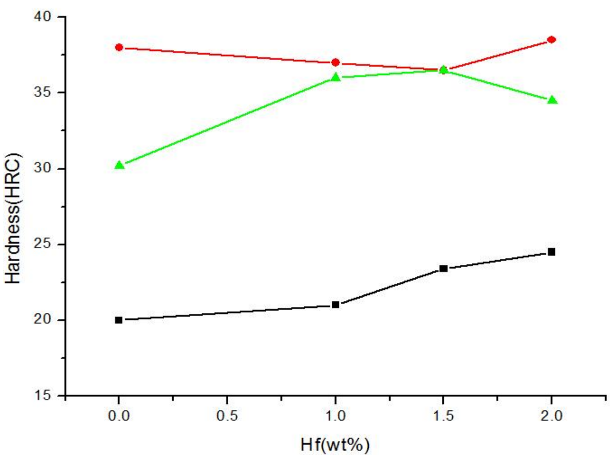


Fig.12. The effect of different content of added Hf and post heat treatment on the hardness of Inconel 718 superalloy (■-before heat treatment; ●-after T3 post heat treatment; ▲-after T8 post heat treatment).

3.3.2 Tensile test

Figure 13(a)-(c) show the test curves of various mechanical properties (yield strength, ultimate tensile strength and percentage elongation) of Inconel 718 samples added with different content of Hf and underwent different post heat treatment. The yield strength of samples with different Hf content does not change obviously before post heat treatment, but the yield strength increased obviously when the samples underwent T3 and T8 post heat treatment. In the T3 samples, the Inconel 718 added with 1.5 wt% Hf has a maximum yield strength around 850 MPa, and the yield strength has enhanced 5% comparing to those samples without adding of Hf (about 887 MPa). The samples of T8 post heat treatment have the highest yield strength about 932 MPa, which is also located at the samples added with 1.5 wt% Hf, as shown in Fig. 13(a). It also indicates that the yield strength slightly increased with the increase of Hf content for those samples underwent different post heat treatment process. Moreover, the literatures also indicate that the lump MC carbides appeared within the grain boundaries will improve the stress rupture and prevent the grain-boundaries sliding of Inconel 718 [12-14].

Figure 13 (b) shows the tensile strength variation of the samples added with different Hf content. In the raw Inconel 718 samples, T3 and T8 post heat treatment can apparently increase the tensile strength of the samples in spite that the T8 has more increment than the T3 process. However, the response of the sample's tensile strength to the addition of Hf and different post heat treatment has the same tendency to that of the yield strength.

Figure 13 (c) indicates that while the T3 and T8 post heat treatments can improve the strength of the Inconel 718 samples, the percentage elongation decreased with both heat treatment. Moreover, although the T8 heat treated samples have the highest strength among all the samples having the same composition but undergoing different heat treatment, they still have higher percentage elongation than those of T3 heat treated samples. It suggests that the T8 heat treatment may be a more suitable process to improve the strength and still keeps medium ductility of the Inconel 718 alloys. The highest percentage elongation (about 13%) occurs at the sample added with 1 wt% Hf without undergoing any post heat treatment.

Combining the results of preceding mechanical properties, the optimum adding amount of Hf and post heat treatment procedure for Inconel 718 was carried out at Hf content of 1.5 wt% and T8 post heat treatment. At this condition, the modified Inconel 718 samples can obtain better mechanical properties such as good hardness of HRc36, high yield strength of 932 MPa, high ultimate tensile strength of 1108 MPa and fair percentage elongation of 7.4 %.

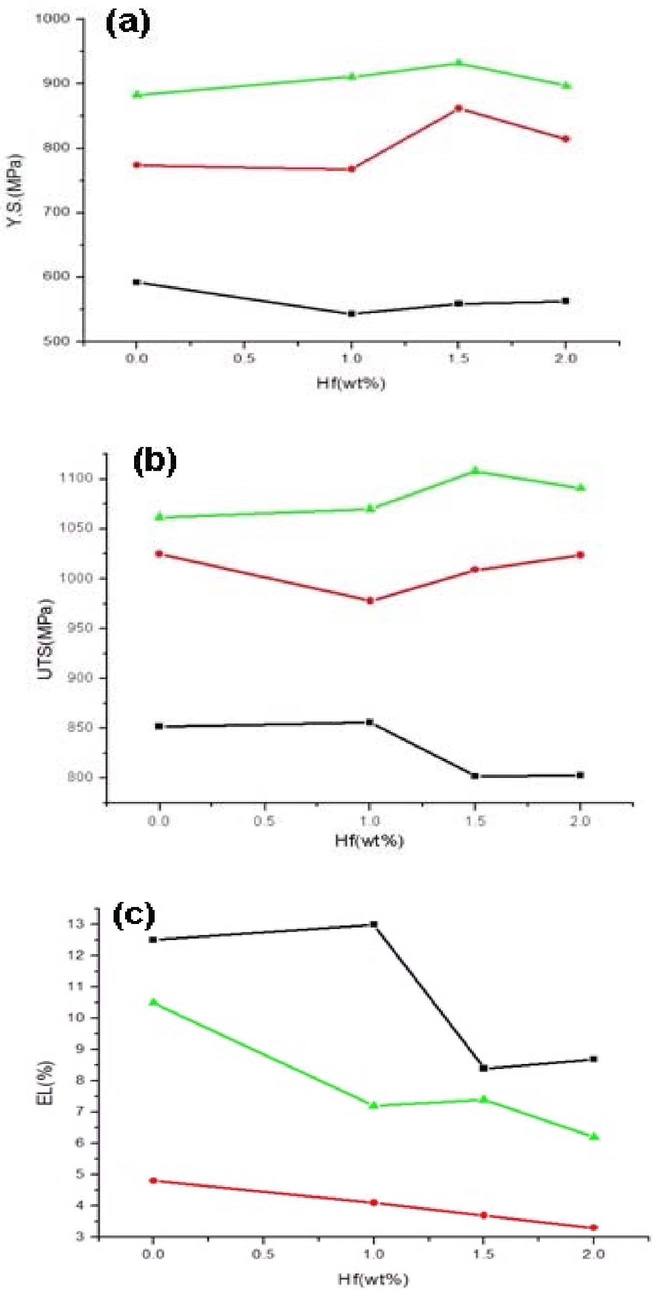


Fig.13. The effect of different content of added Hf and post heat treatment on the mechanical properties of Inconel 718 superalloy (■-before heat treatment; ●-after T3 post heat treatment; ▲-after T8 post heat treatment): (a) yield strength; (b) tensile strength; (c) percentage elongation.

Ⅳ. CONCLUSIONS

We have studied the influence of added Hf content and different post heat treatment procedure on the morphology and mechanical properties of Inconel 718 superalloy. Based on the aforementioned results, the following conclusions can be drawn.

1. The hardness of samples after T3 post heat treatment is higher than those after T8 heat treatment because a large number of δ phase and Widmanstatten structure formed under lower solution temperature of the T3 condition.
2. The arm space of Inconel 718 lowered with the increase of added content of Hf. The smallest arm space occurred at the samples added with 1.5 wt% Hf. The SEM observation and EPMA analysis indicate that Ni_5Hf phase formed in the matrix after adding of different amount of Hf. The precipitation of Ni_5Hf phase may improve the high temperature mechanical properties of Inconel 718 because of its high melting point.
3. The samples added with 1.5 wt% Hf and then conducted with T8 post heat treatment have a hardness of HRc36, the yield strength of 932 MPa and the tensile strength of 1108 MPa. Compared with the raw Inconel 718 superalloy, the hardness, yield strength and tensile strength have been enhanced for 20%, 5% and 4.3%, respectively.

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