Vol. 10 №. 3 Trans. Nonferrous Met. Soc. China Jun. 2000

Article ID: 1003 - 6326(2000)03 - 0340 - 05

Effect of thermal exposure on precipitation behavior and hardness of alloy 718 ①

GENG Lin(耿 林)[1], ZHENG Zhen-zhu(郑镇洙)[1], Na Yong-Sang[2], Park Nho-Kwang[2]

1. *School of Materials Science and Engineering, Harbin Institute of Technology, Harbin 150001, P. R. China*

2. *Korea Institute of Machinery and Materials, 66 Sangnamdong, Changwon, Kyungnam 641-010, Korea*

Abstract: The precipitation and grain growth behavior during isothermal treatment of hot-extruded alloy 718 was investigated. It is found that the hardness of the hot-extruded alloy 718 increases with holding time at 600 ~ 700 ℃, but slightly decreases at 800 ℃. When the exposure temperature reaches above 900 ℃, a rapid decrease in hardness of alloy 718 occurs before the holding time of 0.3 h. Above the δ solvus temperature, γ' and γ'' phases are dissolved directly into the matrix without δ precipitation, resulting in a rapid grain growth.

Key words: precipitation; grain growth; superalloys; isothermal treatment **Document code: A**

1 INTRODUCTION

Alloy 718 is widely used to make gas turbine parts where the major requirements are high strength, good creep resistance and low-cycle fatigue (LCF) strength. These properties are extremely sensitive to the microstructure such as precipitates and grain size. The precipitation of the intermetallic phases in Inconel 718 has been extensively studied[1~3]. Age hardening in this alloy is brought about mainly by the homogeneously nucleated γ'' precipitates which are coherent with the austenite matrix. Some strengthening is also brought about by the γ' precipitates. Cozar and Pineau[4] have observed that in alloy 718 a physical association between γ' and γ'' particles often results in the evolution of a "compact morphology" in which cuboidal γ' particles are coated on the six faces with the γ'' phase, and this "compact morphology" will enhance the microstructural stability of the alloy. δ phase is considered to be an important parameter for creep rupture resistance[5,6]. Desvallees et al[5] have found that the platelike δ phases are harmful to creep rupture life because they are preferential sites for cavity growth. It was also found by Moll et al[6] that controlled precipitation of the δ phase at the grain boundaries is believed to have a beneficial effect on the stress rupture ductility.

Grain size has been proved to be another important factor which influences the mechanical properties and deformation behavior of the Ni-base superalloy[7~9]. In general, fine grain size can make the tensile strength and LCF properties increase[7]. However, grain size must not be too fine, because the creep resistance is reduced by fine grains[8]. It was also pointed by Park et al[9] that grain size is one of the factors which influence the deformation modes of the Ni-base superalloy. The deformation tends to be dominated by shearing with increasing grain size.

In this research, the precipitation and grain growth behavior during isothermal treatment of a hot-extruded alloy 718 was investigated. Annealing temperatures are in the range of 600 ℃ to 1 150 ℃, which are commonly used for age hardening, solution treatment and high-temperature annealing.

2 EXPERIMENTAL

The commercial alloy 718 used for this investigation is in hot-extruded state. The chemical composition is listed in Table 1. The hot extruded alloy 718 was isothermally treated at temperatures from 600 ℃ to 1 150 ℃ for different times from 0.3 h to 72 h followed by oil quench.

Table 1 Chemical composition of experimental alloy (%)

C	Cr	Mo	Co	Ti
0.027	17.90	2.88	0.33	0.98
Al	**Fe**	**Nb**	**B**	**Ni**
0.47	17.4	5.38	0.004	Balance

The grain sizes were obtained by measuring the mean diameter of about 20 of the largest grains out of about 200 grains on optical microscope, because the largest cross-sectional diameter among the cross sections of a grain can give the real grain size. The precipitation behavior was analyzed by hardness measurement and observed by JXA-8600 SEM and JEM-2000

① **Received date:** Apr.7, 1999; **accepted date:** Jul.5, 1999

TEM. The micro-hardness was measured by an Ogital Micro Hardness Tester with a load of 5 N and a time of 10 s. Because the error range of HV value for 12 times of measurement was no more than 10, the average value was used for analysis. Thin foils for TEM observation were prepared using a twin-jet polisher in an 1∶9 mixture of perchloric acid and ethanal at - 35 ℃ and 30 V.

3 RESULTS AND DISCUSSION

The initial material used here is hot-extruded alloy 718. Because dynamic recrystallization happened during the hot extrusion, the hot-extruded alloy 718 shows a fine and equiaxed grain structure with an average grain size of 12 μm, as shown in Fig.1(a). It is also shown that many twins exist in the structure, which are considered to be formed during cooling after hot plastic deformation. The twins were believed to promote the δ phase precipitation[10]. Discrete γ'/γ'' precipitates were found in the structure as shown in Fig.1(b), which were formed during cooling from hot extrusion temperature. It is difficult to distinguish γ' and γ'' phases because of the ambiguities in imaging extremely fine precipitates. It was pointed out by Cozar and Pineau[4] that γ' precipitation precedes γ'' precipitation in alloy 718 only when the ratio of the combined atomic concentration of titanium and aluminum to the atomic concentration of niobium is greater than 0.8. In case of the alloy used in this work, the value of this ratio is only 0.65. Therefore, one can expect that such a sequence of precipitation does not occur in this alloy and γ' precipitation does not precede γ'' precipitation.

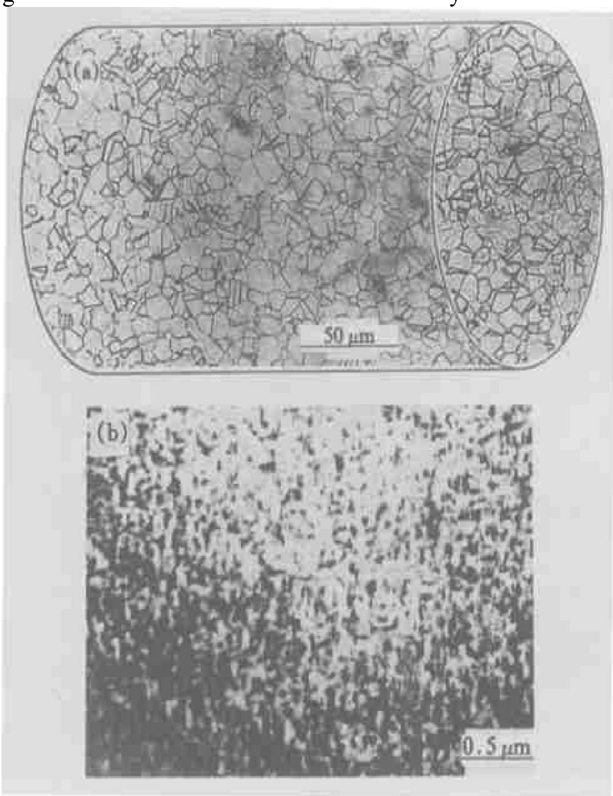

Fig.1 Microstructures of hot-extruded alloy 718
(a) —Optical photograph; (b) —TEM image

The major strengthening phases in alloy 718 are γ' and γ'' phases. The hot-extruded alloy used here is not in the fully heat-treated condition, though large amounts of γ'/γ'' phases have been precipitated. Therefore, further aging is necessary for the as-extruded alloy 718 to get peak hardness. Fig.2 shows the hardness of the hot extruded alloy 718 isothermally treated in the temperature range from 600 ℃ to 1 150 ℃ for different durations. The grain sizes of alloy 718 measured under the same conditions are shown in Fig.3. It can be seen that the grain size changed a little in the temperature range from 600 ℃ to 900 ℃, so it is concluded that the change in hardness shown in this temperature range is mainly due to the precipitation behavior of alloy 718.

The increase of hardness with holding time at 600 ℃ and 700 ℃ indicates the further increase in the amount of the γ'/γ'' precipitates. A hardness peak appears at 700 ℃, 24 h, resulting from the coarsening of the γ'/γ'' precipitates. When the hot-extruded alloy 718 is isothermally treated at 800 ℃, δ phases begin to appear. When the exposure time is only 0.3 h, small size δ phases appear at grain boundaries and the γ'/γ'' phases are still very fine, as shown in Fig.4(a). When the time increases to 10 h, coarsened δ phases can be clearly observed by SEM (Fig.4(b)), while γ'/γ'' phases have grown to an average diameter about 0.1 μm which is enough in size for TEM image as shown in Fig.5(a). After 72 h exposure at

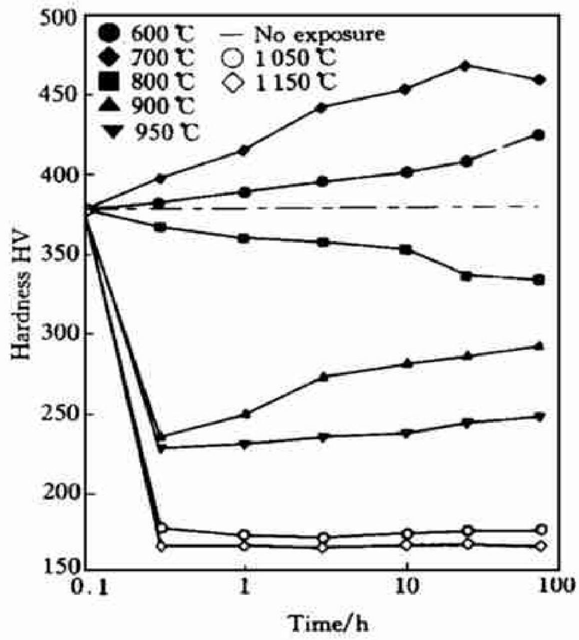

Fig.2 Micro hardness of hot-extruded alloy 718 isothermally treated at temperatures from 600 ℃ to 1 150 ℃ for different times

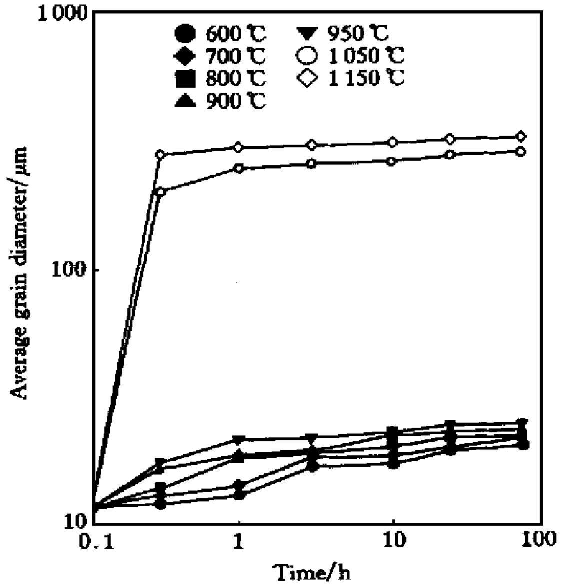

Fig.3 Effect of isothermal treatment on grain size of hot-extruded alloy 718

800 ℃, some larger size needle shaped δ precipitates are formed mainly at grain boundaries (Fig.4(c)), and a large amount of γ'/γ'' precipitates at intragranular sites are also found by both SEM (Fig.4(c)) and TEM (Fig.5(b)). However, in this case the size of the γ'/γ'' precipitates increases to about 1 μm in diameter. γ' free zones are also found in Fig.4(c) and Figs.5(a) and (b), indicating that the larger size needle shaped δ precipitates grow at the expense of the γ'' phases. It has been suggested by Sundararaman et al[10] that the γ'' precipitates were replaced by δ precipitates on prolonged aging for alloy 718 at high temperatures (e.g. 900 ℃). However, for the as-extruded alloy 718 used here, this phenomenon happens at a lower temperature (100 ℃ lower than the value in Ref.[10]). From the SEM and TEM investigations, it is concluded that the slight decrease of the hardness at 800 ℃ shown in Fig.2 is mainly due to the increase of size by the coarsening of γ'' phase and the decrease of the amount of γ'' phase by the γ''

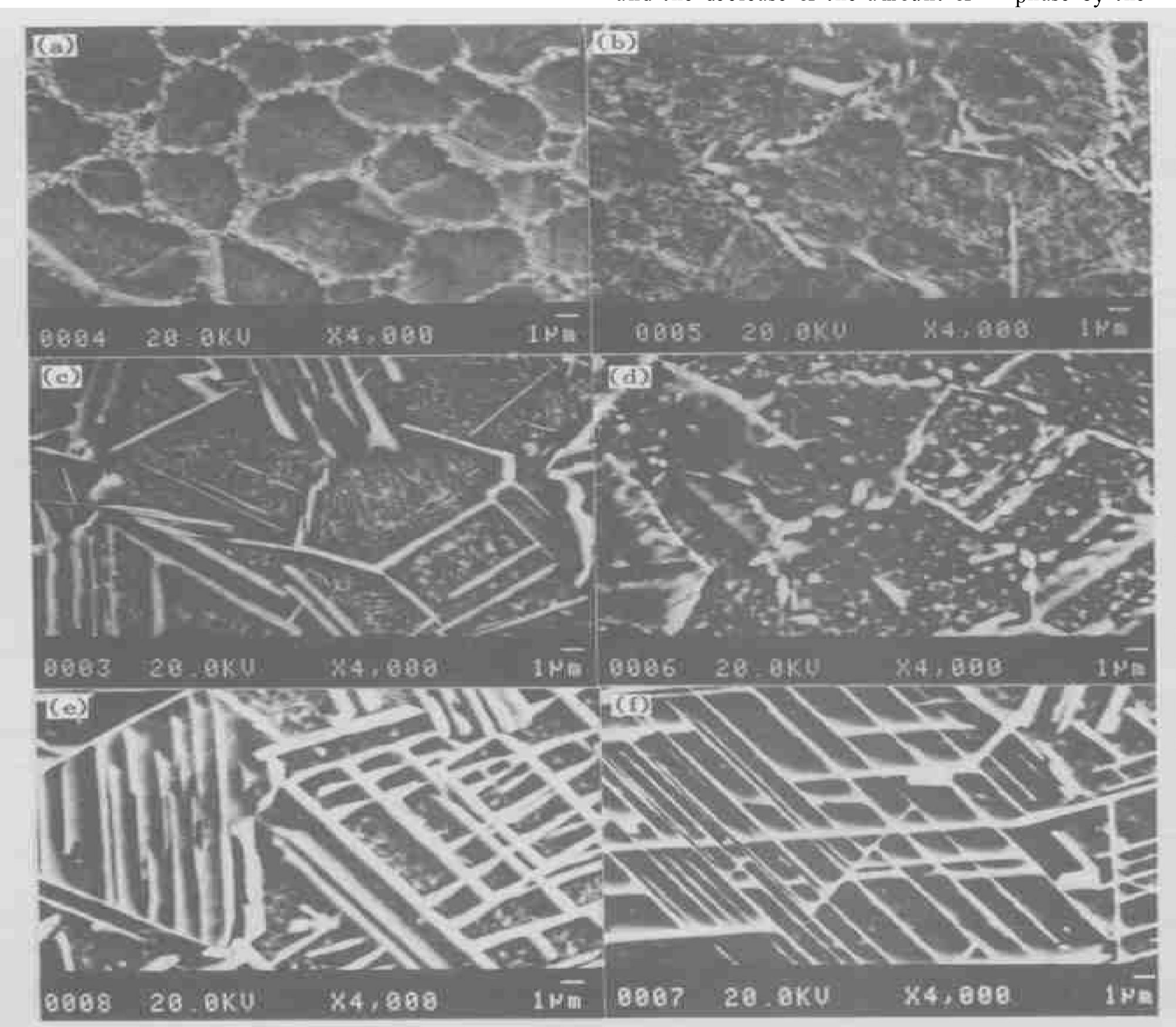

Fig.4 SEM photographs showing microstructure of alloy 718 exposed at 800 for 0.3 h(a), 10 h(b) and 72 h(c) and at 900 ℃ for 0.3 h(d), 10h(e) and 72 h(f)

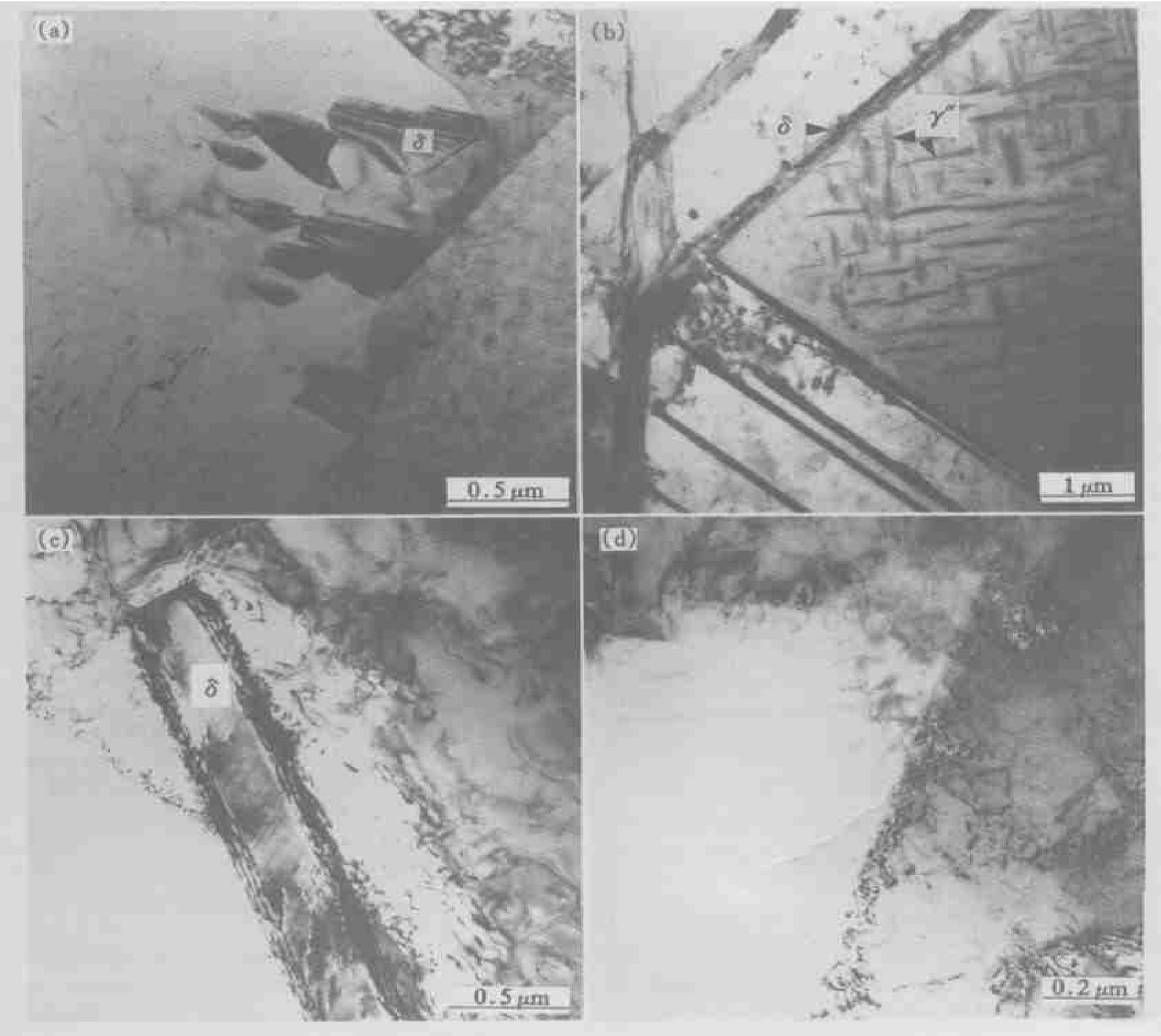

Fig.5 TEM images showing microstructure of isothermally treated alloy 718 at 800 ℃ for 10 h(a), 800 ℃ for 72 h(b), 950 ℃ for 72 h(c) and 1 050 ℃ for 72 h(d)

→ δ transformation.

It was found by TEM observation that the rapid decrease in hardness of the alloy 718 after exposure at 900 ℃ and 950 ℃ for 0.3 h is the result of dissolution of γ′ and γ″ phases existing in the as-extruded alloy 718. It can be concluded from the results of this experiment that the γ′ solvus temperature of the hot-extruded alloy 718 is lower than 900 ℃. TEM analysis also indicates that almost no γ″ phase exists in the γ matrix after exposure at 900 ℃ for only 1 h. It is believed that a great amount of γ″ precipitates have been dissolved into the matrix or transformed into stable γ phases that nucleate and grow fast at both grain boundaries and intragranular sites. In fact, some large size γ phases were observed at grain boundaries of the specimens exposed for only 0.3 h at 900 ℃, as shown in Fig.4(d). Dong et al[11] have also found the γ″ dissolution and the γ″ → δ transformation in alloy 718 exposed at 800 ℃ for more than 75 h. Moreover, it can be seen from Figs.4(d), (e) and (f) that the amount of δ phase increases with time at 900 ℃. Therefore it is believed that the slight increase of the hardness for prolonged exposure at 900 ℃ is due to the further precipitation of δ phase. When the specimens were exposed at 950 ℃, the nucleation rate of δ precipitates was lower but its size was bigger than that at 900 ℃ (Fig.5(c)), so the hardness did not vary significantly.

Fig.2 also shows even more rapid decrease in hardness of the hot-extruded alloy 718 when exposed at 1 050 ℃ and 1 150 ℃ for only 0.3 h. The rapid decrease of hardness is due to the dissolution of both γ′ and γ″ phases, as noted in the specimens heat-treated at 900 ℃ and 950 ℃. It is also worth noting that the rapid grain growth temperature of Ni based superalloys is affected by many factors, such as precipitates and starting grain size[12,13]. It has been proved that γ′ particles effectively inhibit the grain growth after primary recrystallization of Ni based superalloys which do not contain Nb[12,13]. The present experimental results indicate that both γ' and γ'' phases had already disappeared when the as-extruded alloy 718 was exposed at 900 ℃ or 950 ℃ for 0.3 h, but rapid grain growth did not occur in this temperature range. It can be seen from Figs.4(a) and (d) that small δ particles and big needle shaped δ phases appear at grain boundaries for the as-extruded alloy 718 exposed at 800 ℃ or 900 ℃ for 0.3 h. Therefore, δ phases play the most important role in limiting the rapid grain growth of alloy 718 at 800 ~ 950 ℃. Above 1 050 ℃, both γ' and γ'' phases were dissolved directly into the matrix and no δ phases were precipitated, so rapid grain growth occurred. The rapid grain growth temperature of the hot-extruded alloy 718 used here is corresponding to the solvus temperature between 950 ℃ and 1 000 ℃[14,15]. The hardness of alloy 718 exposed at 1 050 ℃ and 1 150 ℃ for a longer time (more than 0.3 h) remains constant because there is no precipitates (Fig.5(d)) and the grain size is virtually constant (Fig.3).

4 CONCLUSIONS

1) When the hot-extruded alloy 718 was exposed at 600 ~ 700 ℃, the γ'/γ'' phases were further precipitated, resulting in the increase of hardness with holding time.

2) When the isothermal treatment was carried out at 800 ℃, δ phases were precipitated by the expense of γ'' phases. The decrease in amount and increase in size of the γ'' phases led to a slight decrease of hardness with holding time.

3) When the exposure temperature was above 900 ℃, a rapid decrease in hardness of alloy 718 occured after holding for a short time (less than 0.3 h), resulting from the dissolution of γ' phases and a sharp decrease in the amount of γ'' phases which were dissolved into the matrix or replaced by the fast forming δ phase. The γ' solvus temperature of the hot-extruded alloy 718 used in this work is below 900 ℃. Above the δ solvus temperature, γ' and γ'' phases will be dissolved directly into the matrix without δ precipitation, resulting in a rapid grain growth.

REFERENCES

[1] Jena A K and Chaturved M C. Roll of alloying elements in the design of nickel base superalloys [J]. J Mater Sci, 1982, 16: 555.

[2] Sundararaman M, Mukhopadhyay P and Banerjee S. Precipitation of the σ-Ni_3Nb phase in two nickel base superalloys [J]. Metall Trans, 1988, 19A: 453.

[3] Sundararaman M, Mukhopadhyay P and Banerjee S. Some aspects of the precipitation of metastable intermetallic phases in Inconel 718 [J]. Metall Trans, 1992, 23A: 2015.

[4] Cozar R and Pineau A. Morphology of γ' and γ'' precipitates and thermal stability of Inconel 718 type alloys [J]. Metall Trans, 1973, 4: 47.

[5] Desvallees Y, Bouzidi M, Bois F, et al. Delta phase in Inconel 718: Mechanical properties and forging process requirements [A]. Loria E A ed, Superalloys 718, 625, 706 and Various Derivatives [C]. TMS, 1994: 281.

[6] Moll L H, Maniar G M and Muzyka D R. Heat treatment of 706 alloy for optimum 1 200 °F stress-rupter properties [J]. Metall Trans, 1971, 2: 2153.

[7] Loria E A. Recent developments in the progress of superalloy 718 [J]. JOM, 1992: 33.

[8] Loria E A. The status and prospects of alloy 718 [J]. J Metals, 1988, (7): 36.

[9] Park Nho-Kwang, Kim Byung-Hoon and Lee Sang Lae. Deformation modes of a Ni-base superalloy under comperssion [J]. Scripta Metall Mater, 1993, 29: 117.

[10] Sundararaman M, Mukhopadhyay P and Banerjee S. Precipitation and room temperature deformation behavior of Inconel 718 [A]. Loria E A ed, Superalloys 718, 625, 706 and Various Derivatives [C]. TMS, 1994. 419.

[11] DONG Jian-xin, XIE Xi-shan and ZHANG Shou-hua. Coarsening behavior of γ'' precipitates in modified Inconel 718 superalloy [J]. Scripta Metall Mater, 1995, 33: 1933.

[12] Mino K, Nakagawa Y G and Ohtomo A. Abnormal grain growth behavior of an oxide dispersion strengther superalloy [J]. Metall Trans A, 1987, 18A: 777.

[13] Kusunoki K, Sumino K, Kawasaki Y, et al. Effects of the amounts of γ' and oxide content of the secondary recrystallization temperature of nickel base superalloys [J]. Metall Trans A, 1990, 21A: 547.

[14] Radavich J G. Metallography of alloy 718 [J]. J Metals, 1988, (7): 42.

[15] Burke M G, Mager T R, Miglin M T, et al. The effects of thermal treatment on SCC of alloy 718: A structure-properties study [A]. Loria E A ed, Superalloys 718, 625, 706 and Various Derivatives [C]. TMS, 1994. 763.

(Edited by PENG Chao-qun)