Superalloys 718, 625, 706 and Derivatives 2005 *Edited by E.A. Loria*
TMS (The Minerals, Metals & Materials Society), 2005

STRUCTURE STABILITY STUDY ON A NEWLY DEVELOPED NICKEL-BASE SUPERALLOY—ALLVAC® 718PLUS™

Xishan Xie[1], Gailian Wang[1], Jianxin Dong[1], Chunmei Xu[1], Wei-Di Cao[2], Richard Kennedy[2]

[1]High Temperature Materials Research Laboratories,
University of Science & Technology Beijing; Beijing 100083, China
[2]ATI Allvac, an Allegheny Technologies Company,
2020 Ashcraft Avenue; Monroe, NC 28110, USA

Keywords: 718Plus, Stability, $Ni_3Al_{0.5}Nb_{0.5}$

Abstract

Recently a new Ni base superalloy to be used at 700°C has been developed by ATI Allvac designated as Allvac® 718Plus™. The critical criterion for this alloy is long time structure stability at 700°C that is closely related to the nature of phases and their stability in this alloy. In this study phase identification of 718Plus at as-heat treated and long-term thermal exposed conditions was conducted by various means.

Phase prediction of 718Plus was calculated by Thermo-Calc software and the detailed phase identification was conducted by SEM, TEM, EDS, SAD and XRD. Quantitative determination of different phases were conducted by electrolytic phase isolation and followed by micro-chemical analyses. The results show that alloy 718Plus is predominantly γ' strengthened alloy with very good structure stability at 700°C. The formation of δ phase occurred during heat treatment and thermal exposure. A new phase $Ni_3Al_{0.5}Nb_{0.5}$ with HCP structure was found. The phase precipitation behavior in combination with good mechanical properties makes 718Plus a very good candidate superalloy to be used at 700°C.

Introduction

The use of alloy 718 as a moderate temperature superalloy, especially for disk applications in aircraft and gas turbine engines has increased significantly in recent years as it has high yield and ultimate tensile strengths and exhibits excellent creep resistance up to 650°C [1~3]. Alloy 718 is also competitively priced due to the fact that the alloy contains no cobalt and has a relatively high content of iron. However, the instability of main strengthening phase γ″ at high temperature has limited the highest service temperature of IN718 only below 650°C[4~6]. Now the challenge is coming to alloy 718 that the service temperature is very desirable to increase significantly, e.g. 50°C, while at the same time its perfect combination of mechanical properties and moderate price should be retained.

Many studies have been made to improve the elevated temperature properties of alloy 718 by chemical composition modification, including changes in both major and minor elements. The earliest research to increase the microstructural stability was done by R. Cozar and A. Pineau in 1973[7]. They raised the ratio of (Al+Ti)/Nb and the sum (Al+Ti+Nb) in atomic percentage and developed a compact morphology of γ″ + γ′ heat treatment. In recent twenty years, Xishan Xie and Jianxin Dong at UST Beijing and other researchers such as Keh-Minn Chang also engaged in the major elements modifications such as Al, Ti, Nb adjustment [8~13] and Co, Ta [14,15]

additions. The attribute of major elements modification was to enhance the microstructure and mechanical property stability. Xishan Xie, Zhuangqi Hu and Wenru Sun in China as well as Wei-Di Cao and R. Kennedy of Allvac in USA also did a lot of work in minor elements changes. The results show that the increase of P and B leads to a significant improvement of stress rupture life with unchanged tensile strength [16~24]. Despite these efforts, there is no commercial alloy in manufacture today which can increased the temperature capability of 718 while maintaining its many attributes.

Recently, Wei-Di Cao and R. Kennedy published their research work on a new Ni base superalloy named alloy 718Plus[25-28]. The differences of alloy 718Plus chemistry compared to alloy 718 are the increase in sum of Al+Ti, the ratio of Al/Ti, the addition of W and especially Co to replace Fe. This newly developed alloy 718Plus appears to have increased temperature capability of alloy 718 by 55°C and excellent thermal stability. It also has a good combination of mechanical properties and moderate price as well as good workability.

In spite of the development in alloy 718Plus, the strengthening mechanism is still not clear. In this paper, alloy 718Plus and alloy 718 have been studied to evaluate the effect of chemical composition adjustment on microstructure changes as well as the structure stability at high temperature.

Materials and Experimental Procedure

The chemical composition of alloy 718Plus used in this study is shown in Table I. For comparison, the composition of alloy 718 is also listed. The differences of alloy 718Plus chemistry compared to alloy 718 are the increase of Al+Ti content, Al/Ti ratio, the addition of Co and W, replacing half amount of Fe, and the addition of small amount of P. All these two alloys were vacuum induction (VIM) and vacuum arc-remelted (VAR). Ingots were homogenized after conditioning. Homogenized ingots were then forged and heat-treated. The heat treatment for alloy 718 was the standard routine while for alloy 718Plus, a small adjustment had been taken (Table II). For accelerated evaluation on the long-term thermal stability at 704°C, experimental thermal exposure was conducted at higher temperature 760°C for shorter times 100, 350 and 500 hrs.

Table I. Chemistries of Alloys 718 and 718Plus (wt.%)

Alloy	C	S	P	Cr	Ni	Mo	W	Fe	Co	Nb	Ti	Al	B
718Plus	0.028	<0.0003	0.013	17.42	Bal.	2.72	1.04	9.66	9.13	5.48	0.71	1.46	0.005
718	0.025	<0.0003	0.008	17.90	Bal.	2.86	0.03	18.08	0.16	5.22	1.01	0.49	0.004

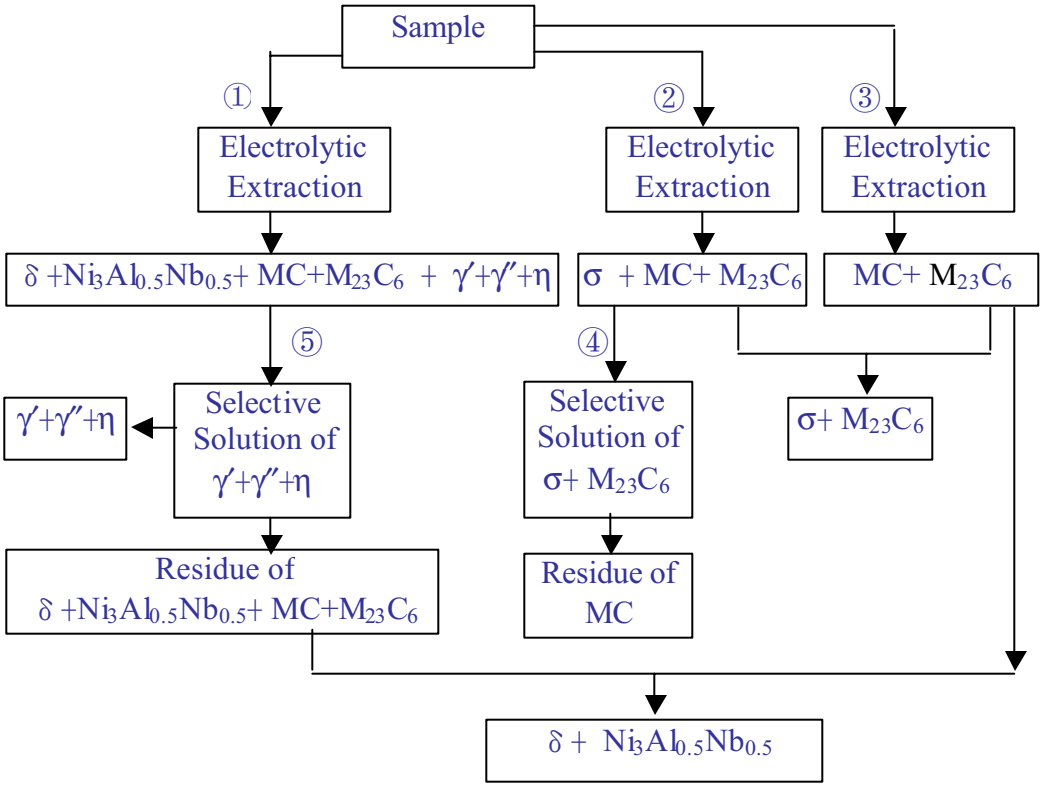
Table II. Heat Treatment Conditions

718Plus	954°C /1 hr/AC + 788°C/2 hrs/FC 55°C/hr to 649°C/8 hrs/FC
718	954°C /1 hr/AC + 718°C/8 hrs/FC 55°C/hr to 621°C/8 hrs/FC

In order to predict the phases in the alloy, Thermo-Cal software was used to calculate the phases and their precipitation temperatures. Then SEM was used to observe the morphology and distribution of all the phases. XRD, TEM, EDS and SAD were used to identify and confirm the phases observed. All the phases were quantitatively determined by physical-chemistry phase analyses. The sizes of the strengthening phases were detected by X-ray small angle scattering goniometer.

The precipitated phases for XRD and physical chemistry phase analyses were extracted electrolytically from the samples in different solutions for different phases[29]. The current

density and the time for extraction also depend on the phases and the solutions. Figure 1 shows the method of phase extraction and separation used for XRD and micro-chemical quantitative phase analyses.



Solutions for electrolytic extraction:

①1%$(NH_4)_2SO_4$+2% Citric acid +H_2O, T=5~10℃, t=1h,i=0.02~0.025A/cm^2

②3.6%ZnCl2+5%HCl+1%Tartaric acid+Methanol, T=-5℃,t=1~2h, i =0.10 A/cm^2

③ 4%Sulfosalicylic acid+1%LiCl+5% Glycerine + Methanol
T=-10~-7℃, t=1~2h, i =0.1 A/cm^2

Solutions for phase separation:

④200ccH_2SO_4+200ccH_2O+20gTartaric acid, reflux for t=2~3h, δ +α-Cr dissolved

⑤ 5%H_2SO_4+7%Tartaric acid +H_2O, in boil bath for t=3h, γ′+γ″ dissolved

Figure 1. Procedure of Phase Extraction and Separation

Experimental Results

Microstructure, As-Heat-Treated Condition

The average grain size of alloy 718 and alloy 718Plus are 80μm and 33μm, respectively. Figure 2 shows the low magnification SEM pictures of both alloys at as heat-treated conditions.

Figure 3a shows the typical microstructure of alloy 718, with its familiar δ phase, distributed mainly at grain boundaries and also in grains. The strengthening phases γ″ and γ′ are very fine and the sizes of γ″/γ′ are still difficult to resolve at 5000×. The microstructure of Alloy 718Plus is different from that of Alloy 718 (Figure 3b). Besides the δ phase, there are also plate-like precipitates distributed not only at grain boundaries but also in the grains. Moreover, the fine strengthening phase particles in the grains can be resolved at the deep etching conditions. It reveals that the fine precipitates are mostly in globular morphology, which is different from disk-

like γ″ phase in Alloy 718. The globular morphology of precipitates reminds us that those precipitates are mainly γ′ strengthening particles.

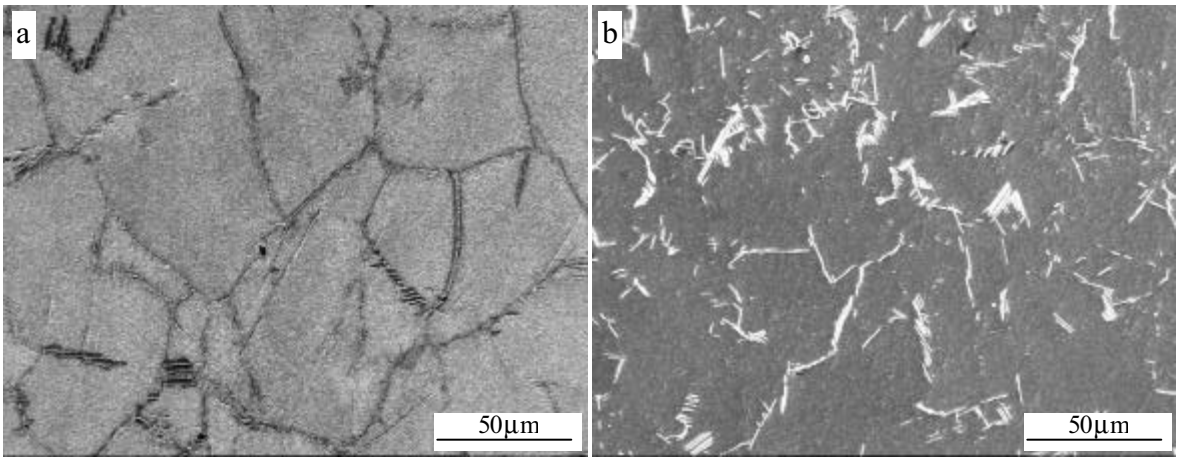


Figure 2 Grain Structure of (a) Alloy 718 and (b) Alloy718plus
at as Heat-treated Conditions

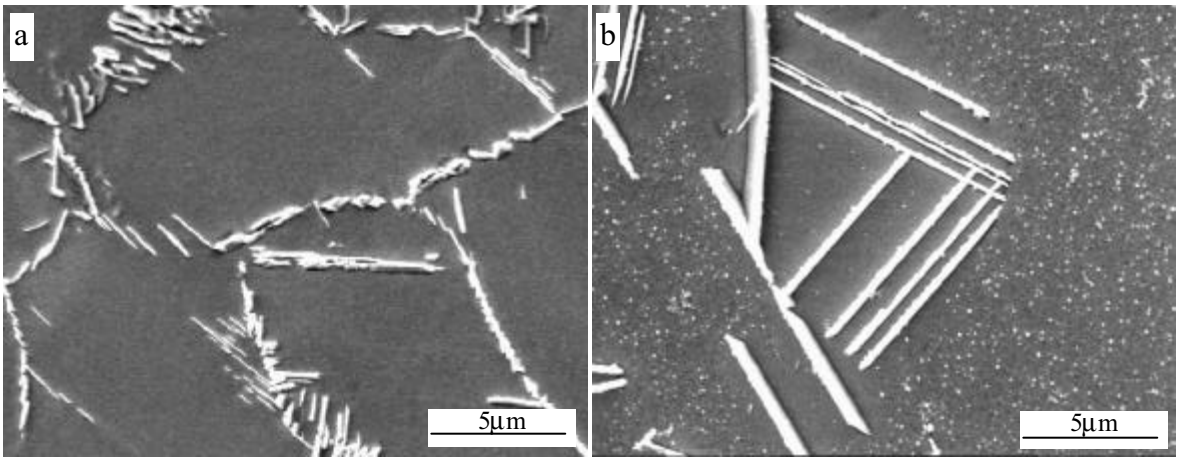
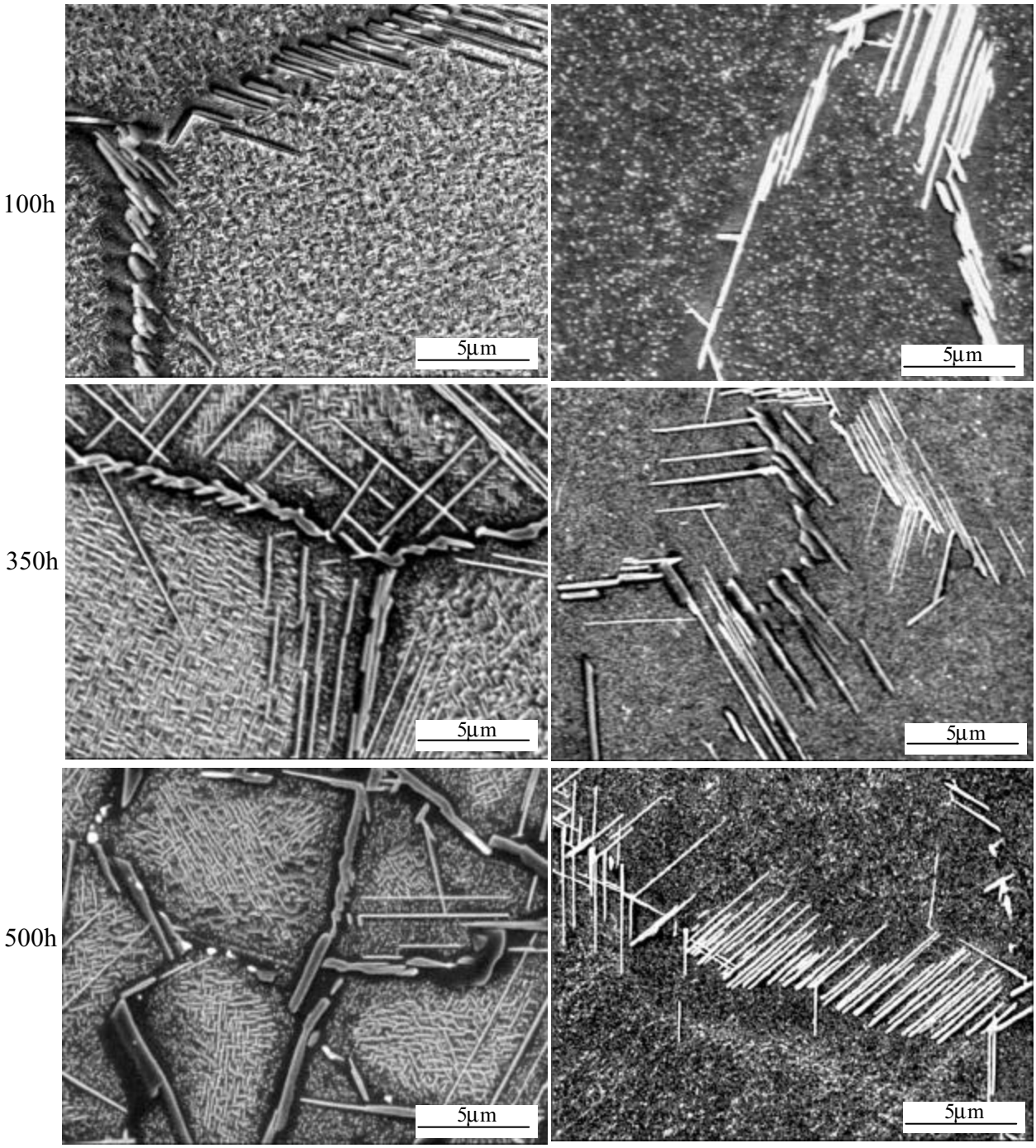


Figure 3 Microstructures by SEM of (a) Alloy 718 and (b) Alloy718plus
at as Heat-treated Conditions

Microstructure, 760°C Long Time Age

After thermal exposure at 760°C for 100hrs, the strengthening phases γ″ and γ′ in alloy 718 become resolvable. After 350hrs or longer time aging till 500hrs, the strengthening phases γ″ and γ′ coarsen significantly. In alloy 718Plus, the morphology of strengthening phase is different from those in alloy 718. It is clear that there are not disk-like precipitates of γ″ phase. The main strengthening precipitates of alloy 718Plus are in globular morphology and those globular morphology particles do not coarsen significantly after 760°C long time thermal aging till 500hrs, (see Figure 4). These precipitates in alloy 718Plus should be γ′ phase.

The TEM morphologies of strengthening precipitates in alloy 718 and alloy 718Plus are shown in Figures 5 and Figure 6. The disk-like strengthening phase γ″ in alloy 718 grows very fast at 760°C for 100hrs and changes in very large size (several hundred nanometers) in 500hrs, see Figure 5. In result alloy 718 losses strengthening effect. However, there are totally different pictures in alloy 718Plus (see Figure 6). The globular γ′ precipitates characterize very stable behavior at 760°C. The sizes of γ′ particles are still in tens of nanometers. It means this strengthening effect of stable γ′ precipitates remain after 760°C exposure.



Alloy 718 Alloy 718plus

Figure 4 Microstructures by SEM of Alloy 718 and Alloy718plus after 760℃ Long Time Aging

Phase Identification

Just as mentioned in SEM structure of alloy 718Plus, there are plate-like precipitates distributed in the alloy. In order to identify these phases, XRD was conducted. Figure 7 and Table III show the XRD patterns and phase identification of Alloy 718Plus at as heat-treated condition and after thermal aging for 350hrs. Considering the long plate phase may not be δ phase, it must be noticed that all peaks for δ phase are also fit for η and a new phase $Ni_3Al_{0.5}Nb_{0.5}$. δ phase has an orthorhombic crystal structure which is different from the HCP crystal structure of η-Ni_3Ti and

$Ni_3Al_{0.5}Nb_{0.5}$. η-Ni_3Ti is a Ti rich phase but $Ni_3Al_{0.5}Nb_{0.5}$ is rich in Nb and Al. However, both phases characterize with HCP structure. In order to certify these phases in alloy 718Plus, it is necessary to conduct the selected area diffraction (SAD) in combination with the EDS on TEM to confirm its crystallographic structure and chemical composition.

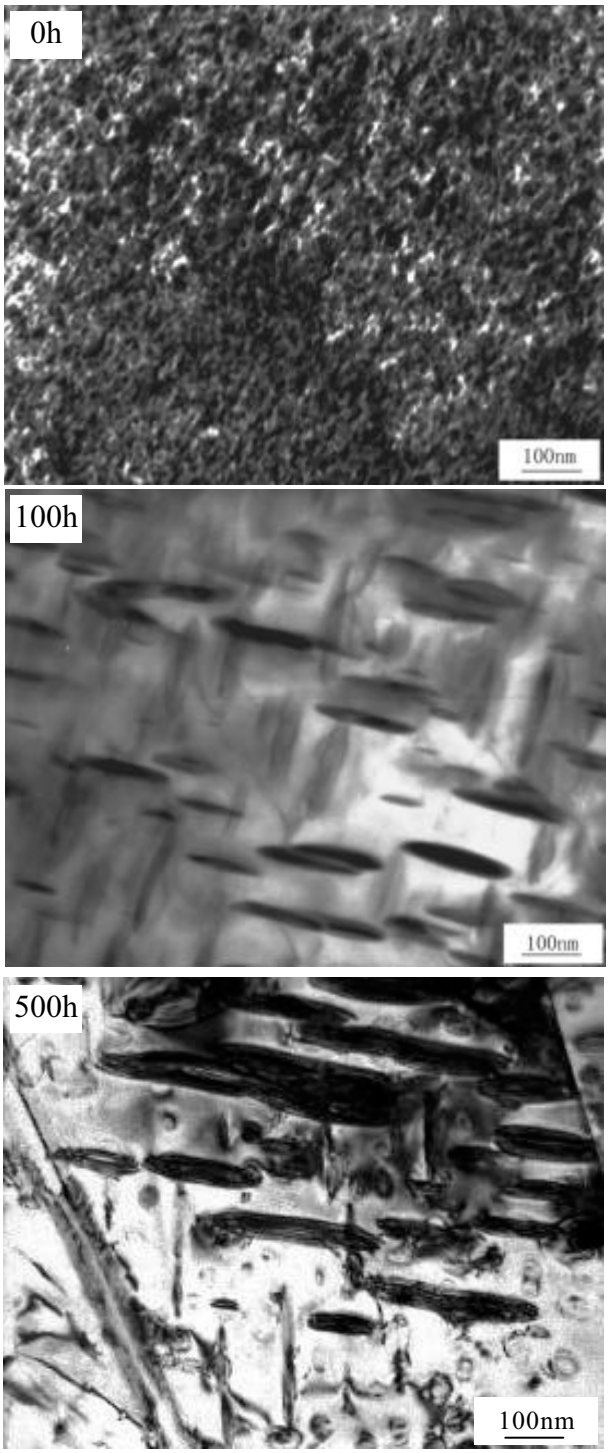


Figure 5 The Coarsening of Strengthening Phases γ″ and γ′ in Alloy 718 after 760℃

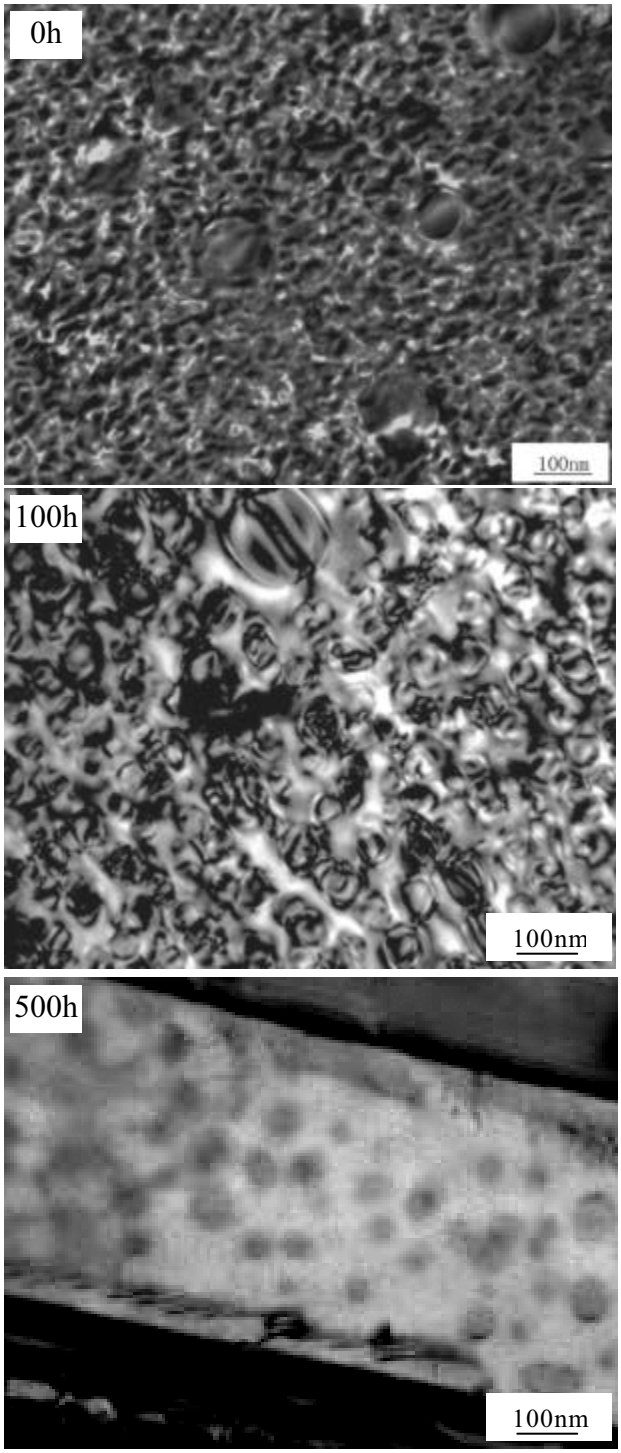


Figure 6 The Coarsening of Strengthening Phase γ′ in Alloy 718plus after 760℃ Aging

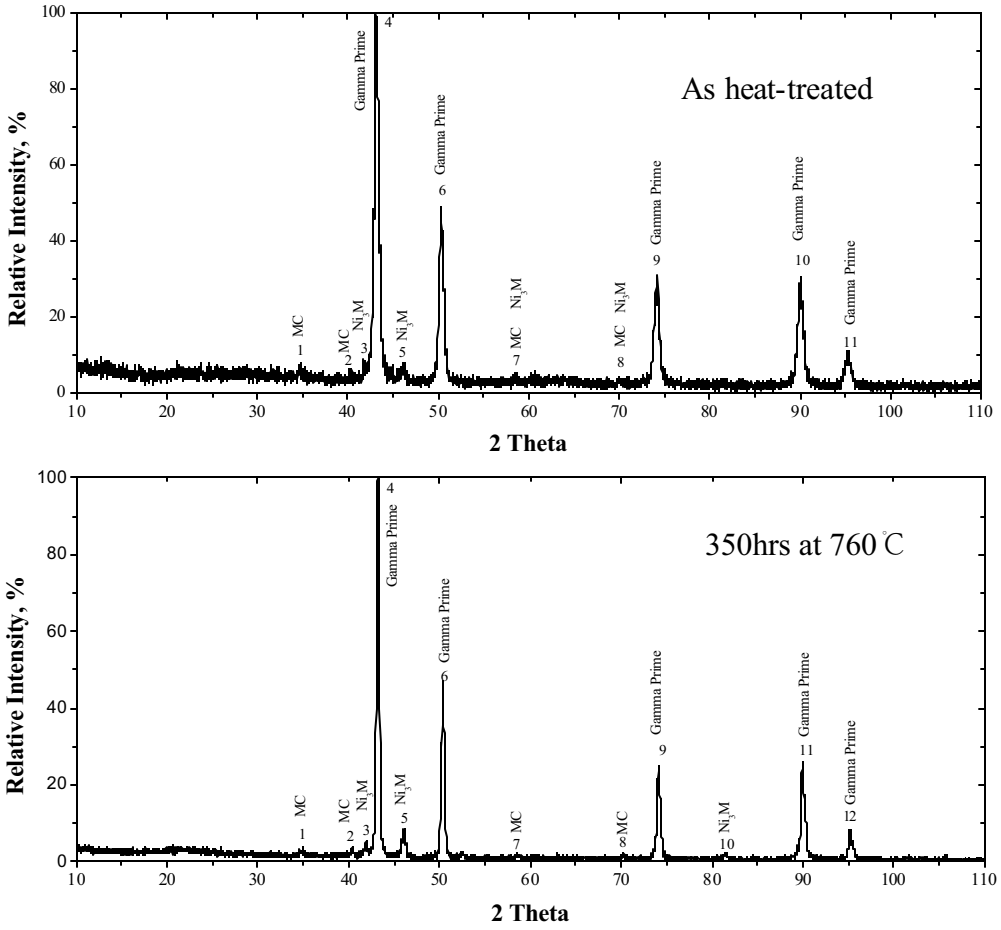


Figure 7 XRD of Alloy 718plus
Residues for XRD were extracted in solution 1%$(NH_4)_2$ SO_4+2% Citric acid +H_2O
at condition of T=5~10℃, i=0.025A/cm^2, for t=1h
γ-matrix was dissolved but γ′,η,δand MC were extracted

Table III. Phase Identification by XRD for Alloy 718Plus after 350hrs' Thermal Aging

No.	I/I_0	2 Theta	d	Gamma Prime	NbC	Ni_3M		
						Delta-Ni_3Nb	Eta-Ni_3Ti	$Ni_3Al_{0.5}$ $Nb_{0.5}$
1	3.3	35	2.56		2.563vs	2.56(10)	2.56(10)	
2	3.78	40.42	2.23		2.22s	2.22(40)	2.21(20)	2.219(10)
3	5.05	41.96	2.15			2.12(70)	2.13(50)	2.146(40)
4	100	43.26	2.09	2.090vs			2.07(50)	2.090(35)
5	8.05	46.14	1.97			1.97(100)	1.95(100)	1.961(100)
6	47.1	50.38	1.81	1.810s		1.80(10)		
7	1.96	58.64	1.57		1.57m	1.56(10)		
8	1.75	70.3	1.34		1.339m	1.30(70)	1.330(20)	1.334(5)
9	25.43	74.12	1.28	1.280s	1.282w			1.281(20)
10	2.04	81.46	1.18			1.20(40)		1.179(15)
11	26.43	89.94	1.09	1.091s		1.09	1.087(50)	1.092(25)
12	8.77	95.22	1.04	1.045w			1.046(20)	1.046(5)

Figures 8a and 8b show the long plate precipitates, occasionally in lamellar structure morphology, at grain boundaries and in the grains. These plates look different from δ-Ni_3Nb phase and similar to η-Ni_3Ti phase in superalloys with high content of Ti. The selected area diffraction pattern of the long plates is shown in Figures 8 d and e, which is identified as hexagonal structure equivalent to HCP η-Ni_3Ti. However, chemical composition analysis result shows that the long plate phase is not enriched in Ti and still characterizes with high content of Nb. Quantitative composition determination via EDS shows this long plate phase can be formulated as $(Ni_{0.797}Cr_{0.047}Co_{0.087}Fe_{0.060}W_{0.002}Mo_{0.008})_3(Nb_{0.460}Al_{0.278}Ti_{0.119}Cr_{0.142})$, which can be identified as HCP $Ni_3Al_{0.5}Nb_{0.}$ See Figure 8c.

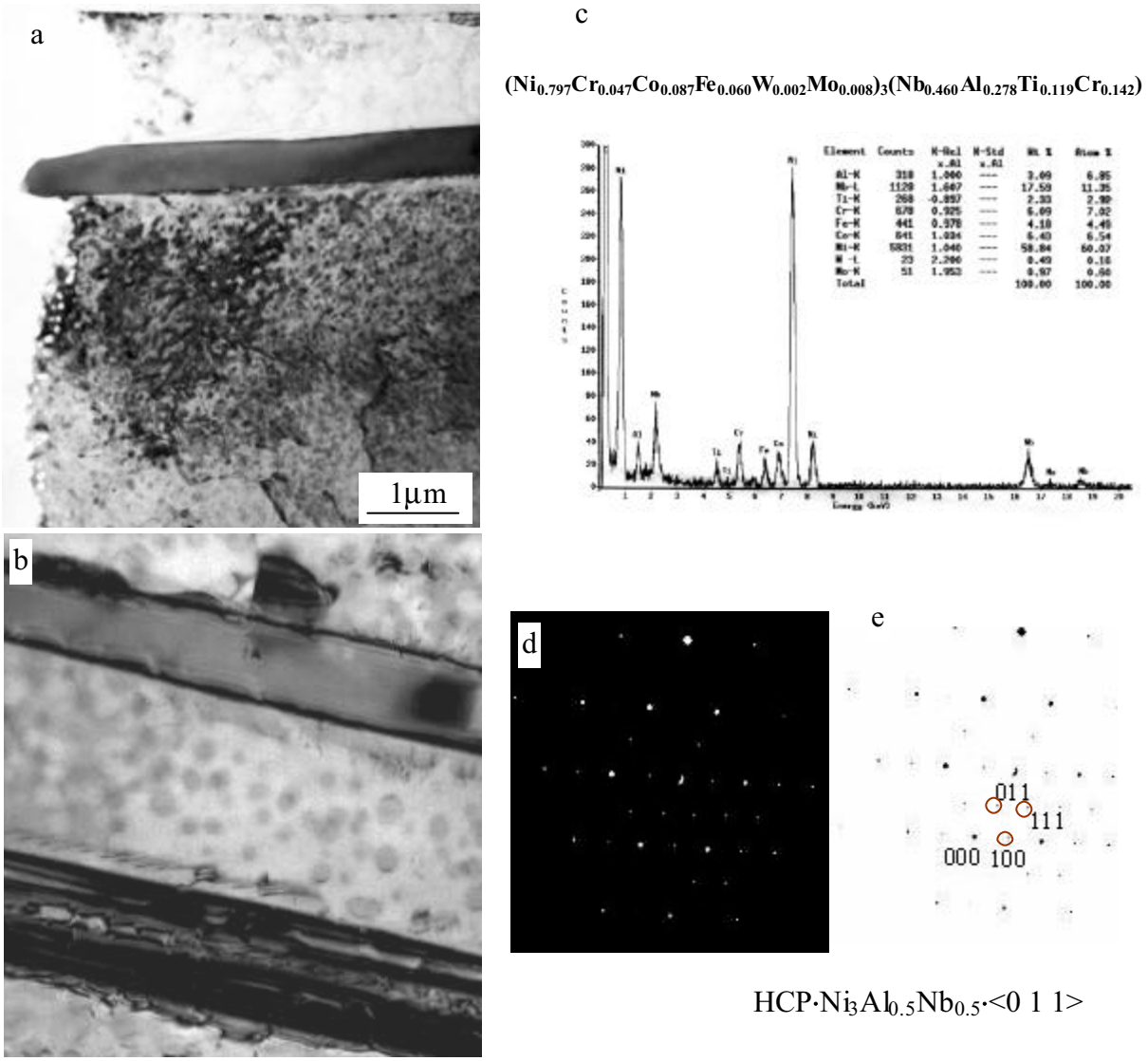


Figure 8. TEM Analyses for phase identification of Alloy 718plus HCP phase is $Ni_3Al_{0.5}Nb_{0.5}$

(a) Morphology of $Ni_3Al_{0.5}Nb_{0.5}$ (as heat-treated)

(b) Morphology of $Ni_3Al_{0.5}Nb_{0.5}$ (760℃/500hrs)

(c) EDS of $Ni_3Al_{0.5}Nb_{0.5}$

(d) SAD of $Ni_3Al_{0.5}Nb_{0.5}$

(e) Phase identification of SAD patter in (d)

Quantitative Determination of Phases

All precipitated phases have been quantitatively analyzed by physical-chemistry analyses method. Figure 9 shows the fraction of δ + $Ni_3Al_{0.5}Nb_{0.5}$ and strengthening phases γ″+γ′ (for alloy 718) or γ′ (for alloy 718Plus) after long time aging at 760°C. It can be seen that the amount of strengthening phase in alloy 718Plus at as heat-treated condition is about 21% while is only 15% in alloy 718. The δ-Ni_3Nb phase in alloy 718 and δ + $Ni_3Al_{0.5}Nb_{0.5}$ phases in alloy 718Plus are almost equivalent in about 0.5%. After 500hrs long time aging at 760°C, alloy 718Plus can still keep a high fraction of strengthening phase (~20%), but alloy 718 only contain a low level of γ″+γ′ (~8%) and a very large amount of δ phase (~12%) forms. However, the amount of δ + $Ni_3Al_{0.5}Nb_{0.5}$ phases in alloy 718Plus grows slowly and remains in the level of about 1%.

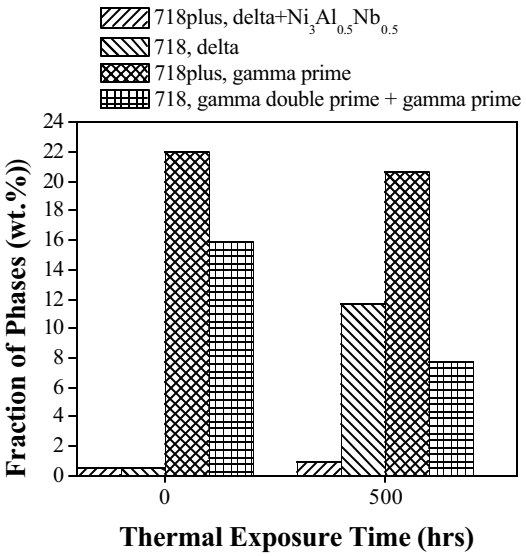


Figure 9 The Amount of phases in Alloy 718 and Alloy 718plus

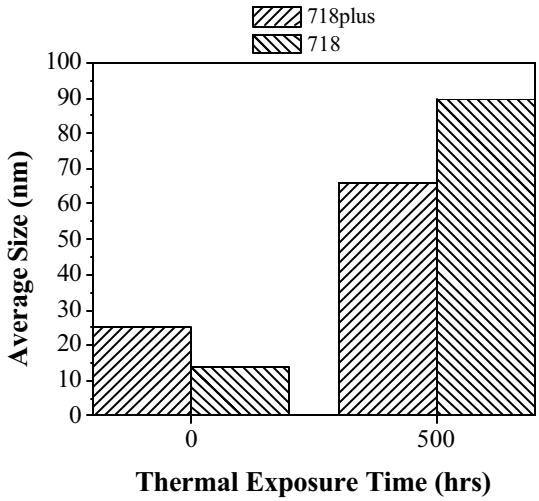


Figure 10 The size of strengthening phases in Alloy 718 (γ″/γ′) and Alloy 718plus (γ′)

The size of strengthening phases increases in both alloys after long time thermal exposure (Figure 10), but more significantly in alloy 718. In alloy 718, the average size of γ″+γ′ grows from about 15nm at as heat-treated condition to almost 100nm after 500hrs long time aging at 760°C as indicated in Figure 10 and the main strengthening phase γ″ grows to about 200nm in estimation (see Figure 5). However, in alloy 718Plus, the main strengthening phase γ′ coarsens slowly (see Figure 6) and the average size of γ′ is still about 70nm as indicated in Figure 10. These important quantitative phase analyses results convince us that alloy 718Plus has a superior stable microstructure in comparison with alloy 718.

General Discussion

Structure Characterization

The new alloy 718Plus has a different microstructure in comparison to conventional alloy 718. The strengthening phase of alloy 718Plus is γ′ rather than γ″/γ′, where γ″ is the main strengthening phase in alloy 718. The amount of γ′ in alloy 718Plus is much more than that of γ″+γ′ in alloy 718 at as heat-treated and after long time aging conditions (Figure 9). Figure 11 shows the relationship of calculated phase amount with aging temperature. The calculated data in Figure 11 is in accordance with our experimental results in Figure 9. The calculated temperature for γ′ solvus in alloy 718Plus (989°C) is higher than that in alloy 718 (879°C) and γ′ is more stable than γ″+γ′ in alloy 718. The δ phase formation temperature in alloy 718Plus (1060°C) is

also higher than that in alloy 718 (1026°C). Moreover, there is a new phase $Ni_3Al_{0.5}Nb_{0.5}$ formed in alloy 718Plus as our experiment indicated. This phase cannot be calculated by Thermo-Calc software because there is not such a phase in Thermo-Calc database.

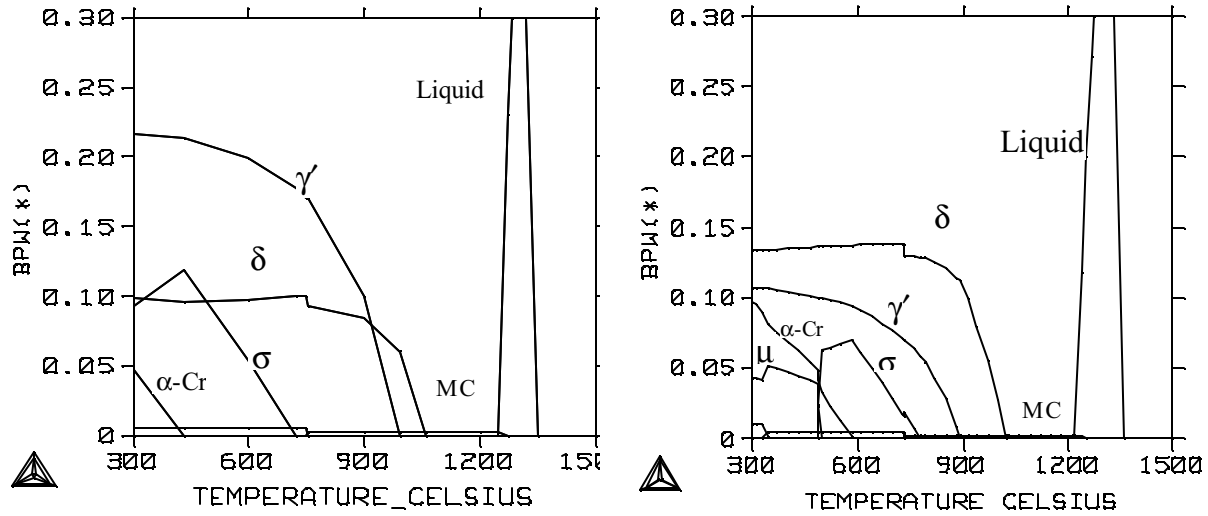


Figure 11 Weight Faction of Phases vs Temperature Calculated by Thermo-Calc in Alloy 718plus (left) and Alloy 718 (right)

Structure Stability

The microstructure instability of alloy 718 is due to the fact that the main strengthening phase γ″coarsens rapidly and also transforms to an equilibrium phase δ-Ni_3Nb after high temperature long time aging. Figure 9 shows the tremendous decreasing of γ″+γ′ and the increasing of δ in alloy 718 after 500hrs thermal aging at 760°C. In alloy 718Plus, however, the main strengthening phase is γ′ which has a very small misfit with the γ-matrix. The γ′ phase shows a lower tendency of coarsening and, therefore, is more stable than γ″.

Strengthening Effect

The high fraction of γ′ strengthening phase and its stability after 760°C long time aging bring the new alloy 718Plus satisfactory mechanical properties (in not only tensile but also stress rupture properties) and good stability. The existence of long plate phase $Ni_3Al_{0.5}Nb_{0.5}$ in lamellar morphology does not show harmful effect on stress rupture properties. It comes from the fact that the lamellar phase of $Ni_3Al_{0.5}Nb_{0.5}$ can harmoniously deform with the matrix at stress rupture condition. Sometimes, $Ni_3Al_{0.5}Nb_{0.5}$ has a lamellar morphology that often makes people worry about its promotion for crack initiation. However, the experimental result of the longitudinal section of stress rupture failed specimen (Figure 12) shows that the cracks do not initiate at the lamellar structure of $Ni_3Al_{0.5}Nb_{0.5}$. It is really clear that this phase can deform in conjunction with the matrix.

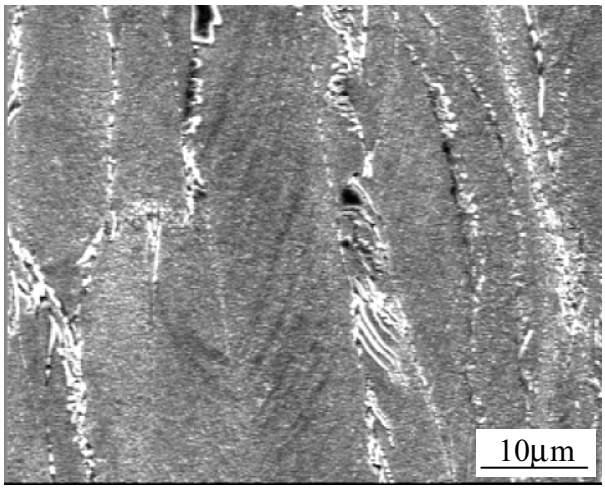


Figure 12 $Ni_3Al_{0.5}Nb_{0.5}$ Lamellar Structure Deforms Coordinately with Matrix in Alloy 718plus

Conclusions

1. The main strengthening phase of alloy 718Plus is γ′, which is different from γ″+γ′ in alloy 718. In addition to the phases of γ′ and δ, a new phase HCP $Ni_3Al_{0.5}Nb_{0.5}$ has been found in alloy 718Plus. Sometimes, $Ni_3Al_{0.5}Nb_{0.5}$ can form as lamellar precipitation at grain boundaries and in grains. However, the existence of $Ni_3Al_{0.5}Nb_{0.5}$ has no harmful effect on mechanical properties.

2. The unique high temperature mechanical properties in alloy 718Plus are developed by the high fraction and high stability of main strengthening phase γ′ in comparison with γ″+γ′ in alloy 718

3. From viewpoint of structure stability and unique mechanical properties, the newly developed alloy 718Plus is a good candidate superalloy to be used at 700°C.

Acknowledgement

The University authors are grateful to ATI Allvac for financial support of the structural stability study on alloy 718Plus.

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