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

TTT DIAGRAM OF A NEWLY DEVELOPED NICKEL-BASE SUPERALLOY– ALLVAC® 718PLUS™

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Keywords: 718Plus, TTT Diagram, δ Phase, γ' Phase, γ" Phase

Abstract

A new nickel-base superalloy designated as Allvac® 718Plus™ has been developed by ATI Allvac to be used at 700°C. For phase characterization, heat treatment selection and structure stability evaluation in high temperature range, the determination of TTT diagram for alloy 718Plus is not only a basic research issue but also an important matter for the alloy application at high temperatures. The conventional alloy 718 has been also studied for comparison. Both alloys were solution treated at 1040°C for full solution of precipitated phases except stable carbide MC. After solution treatment both alloy samples were heat treated at 649 to 954°C for the times from 3-6 minutes till 100 hrs. Phase identification was mainly by means of SEM, XRD and partially TEM and SAD. Hardness test was also conducted on the samples as the indication of γ" and γ' precipitation hardening. The experimentally determined T-T-T diagrams and discussion on the phase formation in alloys 718 and 718Plus are described in this paper.

Introduction

The first T-T-T diagram for wrought alloy 718 was presented by Eiselstein in 1965[1]. However, the composition of early material used by Eiselstein as well as the thermal history have changed over the years and some of the phases Eiselstein reported are not currently found. Since the pioneering work by Eiselstein, many investigators have produced TTT diagrams determined on alloys solution treated to be strain free.[2-6] All these latter studies differed from the former one by the fact that a brittle Laves phase was not found to precipitate at short holding time as reported by Eiselstein and another fact that the delta phase formed in shorter times than that reported by Eiselstein. Recently V. Beaubois *et al.*[7] further studied short term precipitation kinetics of delta phase in strain free 718 alloy. This precipitation has been studied on both as received and solution-treated conditions in the temperature range 650 to 1020°C and for times up to 10 h. The results indicate that the start of precipitation of delta phase in alloy 718 in the high temperature range where forming is carried out seems to proceed at higher temperatures and shorter times than reported in most previous work.

From all published literature on alloy 718, it is clear that the beginning of precipitation and the type of precipitation is dependent on the nature of the starting material, i.e. the degree of residual strain, the amount of delta produced by the solid solutioning step, and probably the most important is the degree of Nb segregation left after the processing cycles. Obviously it is very dependent on the experimental conditions.

Allvac® 718Plus™ alloy is a new Ni-base superalloy developed at ATI Allvac, and it has high strength at high temperature, good structure stability compared with conventional 718 alloy and can be used up to 704°C [8-11]. To tailor the microstructure for properties, an understanding of phase stability with time and temperature is essential. Therefore, this investigation was initiated to determine the T-T-T diagram for alloy 718Plus, which is not only a basic research but also an important matter for the alloy application at high temperatures. Alloy 718 has been also studied for comparison.

Experimental Method and Materials

The alloy 718Plus used in this study was supplied by ATI Allvac as 20 mm RCS rolled bars. Alloy 718 offered by Fushun Steel Company has also been studied for comparison. Chemical composition of the materials is reported in Table I. Samples 10× 10× 10 mm were cut from the as-rolled bars, then solution treated for 1 hour at 1040°C and water quenched for full dissolution of precipitated phases except stable carbide MC. After solution treatment both alloy samples were heat treated in the temperature range of 649°C to 954°C for 3-6 minutes to 100 hours and water quenched. Phase identification was mainly conducted by means of SEM, XRD and partially TEM and SAD. Hardness tests were also conducted on the samples as the indication of γ'' and γ' precipitation hardening.

Table I. Chemical Composition (wt%)

Alloy	C	Cr	Mo	W	Co	Fe	Nb	Al	Ti	Ni
718	0.035	18.42	2.96	-	-	19.35	5.05	0.55	0.95	Bal
718Plus	0.028	17.42	2.72	1.04	9.13	9.66	5.48	1.46	0.71	Bal

Results and Discussion

Hardness

Figure 1 shows Time-Temperature-Hardness (TTH) curves for alloy 718 and alloy 718Plus specimens exposed in the temperature range of 649 to 954 for 3-6 minutes to 100 hours. The Vickers hardness for alloys 718 and 718Plus is 225HV and 223HV, respectively, after solution treatment at 1040°C for 1 hour. γ'/γ'' start to precipitate in the matrix when the Vickers hardness exceeds 225HV and 223HV, respectively, during the aging treatment. γ'/γ'' solvus in Figure 1 have been obtained from Thermo-Calc thermodynamic calculation. It can be seen that the precipitation hardening process in alloy 718Plus is a little bit faster than that in alloy 718.

Evolution of Microstructure with Time and Temperature

Figure 2 shows optical and SEM pictures of alloy 718 after solution treatment. The average grain size of alloy 718 is 44 μm, equivalent to ASTM 6. After solution treatment at 1040°C for 1 hr, alloy 718 contains mainly MC carbides (Nb and Ti carbides) and no additional precipitation could be observed by SEM on exposure at 649°C up to 100 hrs.

Figure 3 shows photographs of alloy 718 specimens exposed at 732°C for 0.5, 1 h, and 100 hours. No additional precipitation could be observed on exposure at 732°C for 0.5 hour. With increasing exposure time to 1 hour, grain boundary precipitates can be observed. At 100 h exposure, grain boundaries are essentially covered with precipitates and the matrix has a mottled appearance typical of overaged γ''/γ'. Alloy 718 specimens exposed at 816°C for 0.5 h shows grain boundary precipitates only and intragranular mottled overaged γ''/γ' also appear with increasing exposure time to 50 h, Figure 4. Grain boundary precipitates have appeared after exposed at 850°C for 0.1 h and intragranular mottled overaged γ''/γ' is observed after exposure for 5 h and 30 h, Figure 5.

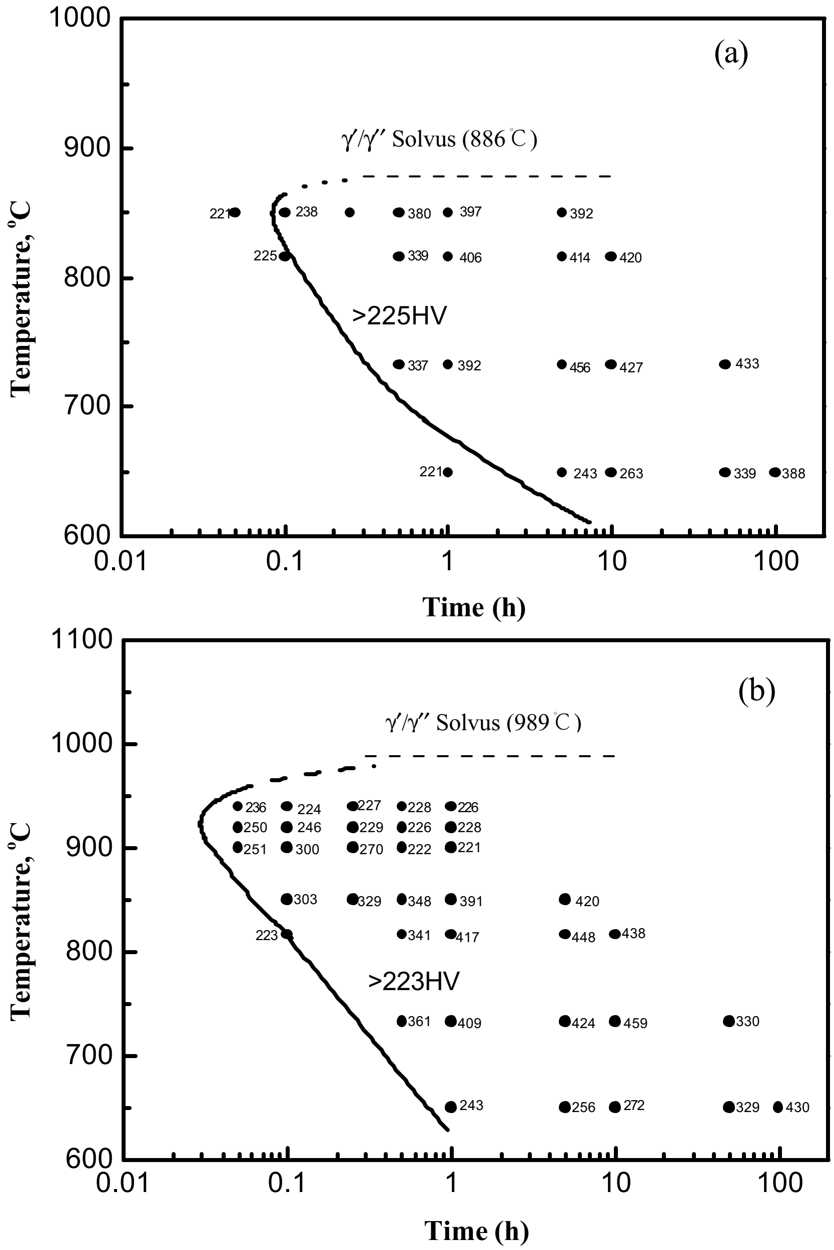

Figure 1 Time-Temperature-Hardness Plots of the Materials Solution Treated at 1040℃ for 1h and Exposed in the Temperature Range of 649℃~954℃ for the Time of 3-6 Minutes to 100h.
(a) Conventional Alloy 718 (b) Modified Alloy 718Plus

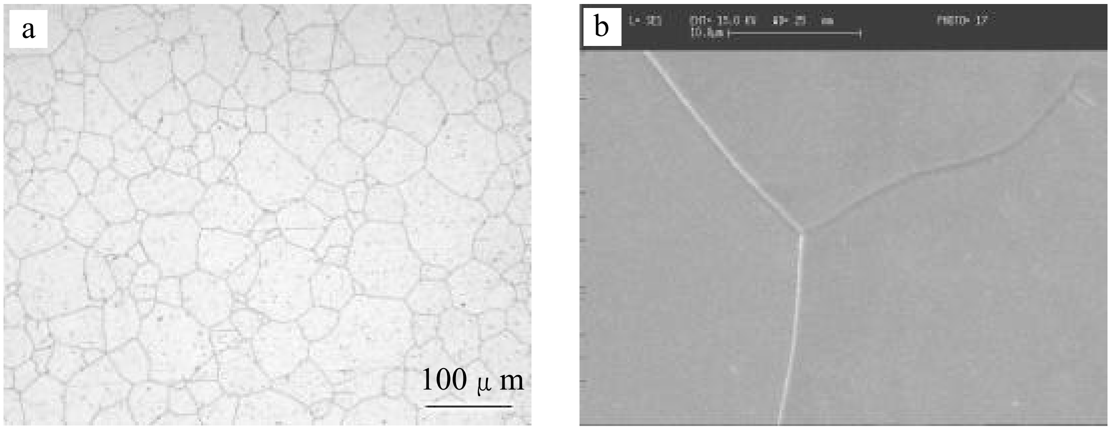

Figure 2 Optical (a) and SEM (b) Pictures of Alloy 718
after Solution Treatment (1040°C/1h) Prior to Thermal Aging

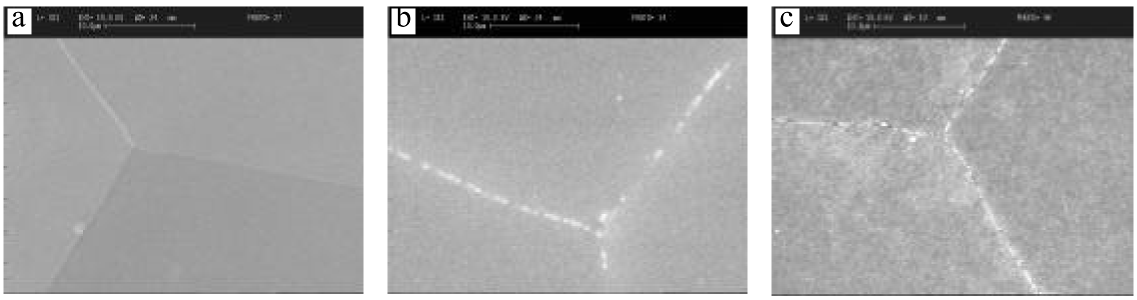

Figure 3 Microstructure of Alloy 718 Specimens Exposed at 732°C
for (a) 0.5 h, (b) 1 h, (c) 100 h

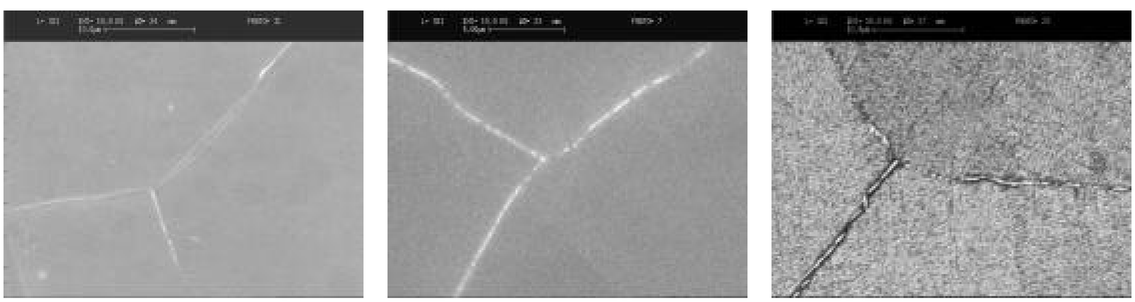

Figure 4 Microstructure of Alloy 718 Specimens Exposed at 816°C
for (a) 0.1h, (b) 0.5h, (c) 50h

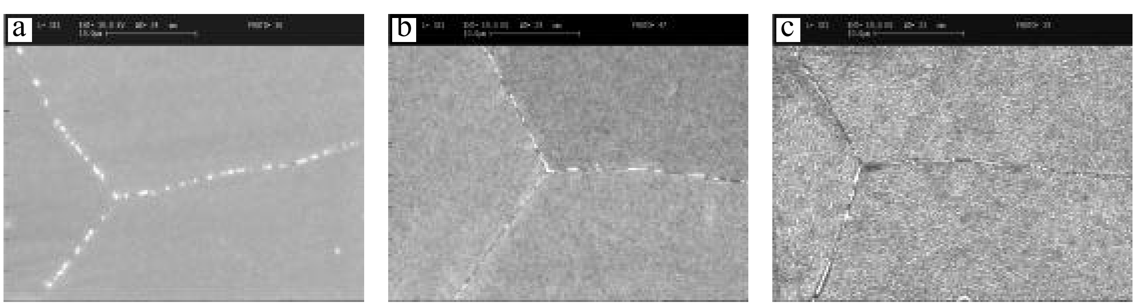

Figure 5 Microstructure of Alloy 718 Specimens Exposed at 850°C
for (a) 0.1 h, (b) 5 h, (c) 30 h

Figure 6 Microstructure of Alloy 718 Specimens Exposed at 900℃
for (a) 0.1 h, (b) 5 h, (c) 10 h

Figure 7 Microstructure of Alloy 718 Specimens Exposed at 954℃
for (a) 0.1 h, (b) 10 h, (c) 50 h

An exposure at 900°C for 0.1 h reveals grain boundary precipitates only, Figure 6a. However, with increasing exposure time at 900°C to 5 h and 10 h, inter- and intragranular plate-like precipitates have been observed, Figure 6b and 6c. Absence of intragranular mottled appearance suggests that γ''/γ' precipitates are in solution at 900°C. An exposure at 954°C for 0.1 h reveals grain boundary precipitates only, Figure 7a. With increasing exposure time at 954°C to 10 h and 50 hours clearly show inter- and intragranular plate-like precipitates, Figure 7b and 7c. Obviously the volume fraction and size of the plate-like precipitates for 50 h exposure are larger than those of 10 h exposure.

Figure 8 shows optical and SEM pictures of alloy 718Plus after solution treatment. The average grain size of 718Plus alloy is 79 μm, equivalent to ASTM 4. Solution treated (1040°C/1h) alloy 718Plus contains mainly MC carbides (Nb and Ti carbides) and no additional precipitation could be observed by SEM on exposure at 649°C till 100h.

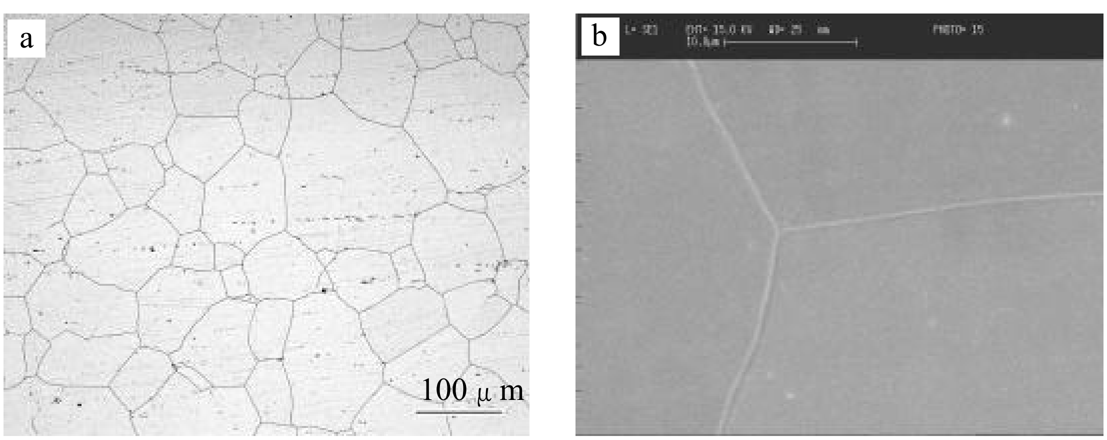

Figure 8 Optical (a) and SEM (b) Pictures of Alloy 718Plus
after solution treatment prior to thermal aging

Figure 9 shows photographs of the alloy 718Plus specimens exposed at 732°C for 0.5, 1 h, and 100 hours. No additional precipitation could be observed on exposure at 732°C for 0.5 hour. With increasing exposure time to 1 hour, grain boundary precipitates have been observed. At 100 h exposure, grain boundaries are essentially covered with precipitates and the matrix reveals a mottled appearance typical of overaged γ'/γ". Alloy 718Plus specimen exposed at 816°C for 0.5 h shows grain boundary precipitates only and intragranular mottled overaged γ'/γ" also appear after 50 h exposure, Figure 10. Grain boundary precipitates have appeared after exposure at 850°C for 0.1 h and intragranular mottled overaged γ'/γ" also appear after exposure for 30 h, Figure 11.

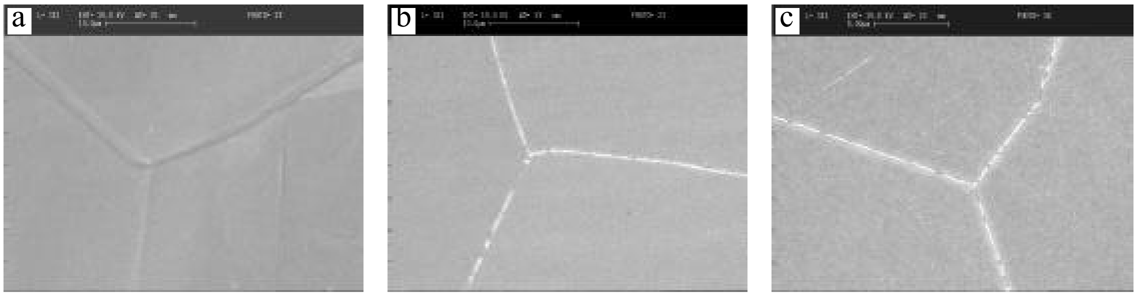

Figure 9 Microstructure of Alloy 718plus Specimens Exposed at 732℃
for (a) 0.5 h, (b) 1 h, (c) 100 h

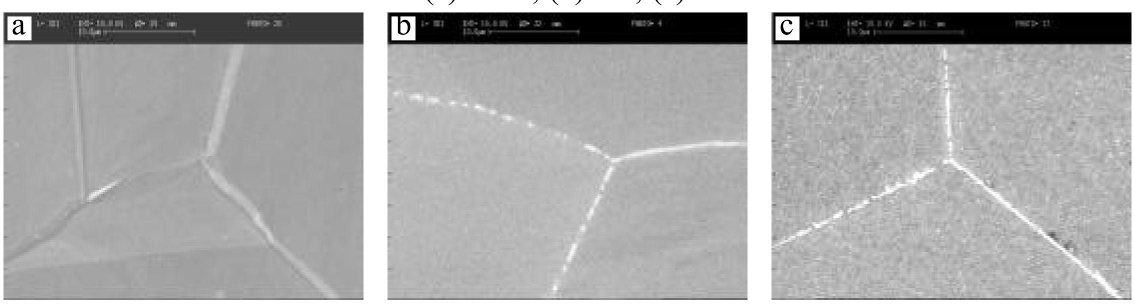

Figure 10 Microstructure of Alloy 718plus Specimens Exposed at 816℃
for (a) 0.1 h, (b) 0.5 h, (c)50 h

Figure 11 Microstructure of Alloy 718plus Specimens Exposed at 850℃
For (a) 0.1 h, (b) 10 h, (c) 30 h

An exposure at 900°C for 0.1 h reveals grain boundary precipitates only, Figure 12a. However, with increasing exposure time at 900°C to 1 h and 10 h, intragranular plate-like precipitates have been observed, Figure 12b and 12c. The alloy 718Plus specimen exposed at 954°C for 0.1 h shows grain boundary precipitates only and exposures for 10 h and 50 h resulted in grain boundary precipitates, intragranular mottled overaged γ'/γ" and intragranular plate-like precipitates. Volume fraction and size of the plate-like precipitates for 50 h exposure are more and larger than 10 h exposure. Presence of intragranular mottled appearance suggests that γ'/γ" precipitates are not solutioned in γ-matrix in the temperature range of 649-954°C, Figure 13.

Figure 12 Microstructure of Alloy 718plus Specimens Exposed at 900℃
For (a) 0.1 h, (b) 1 h, (c) 10 h

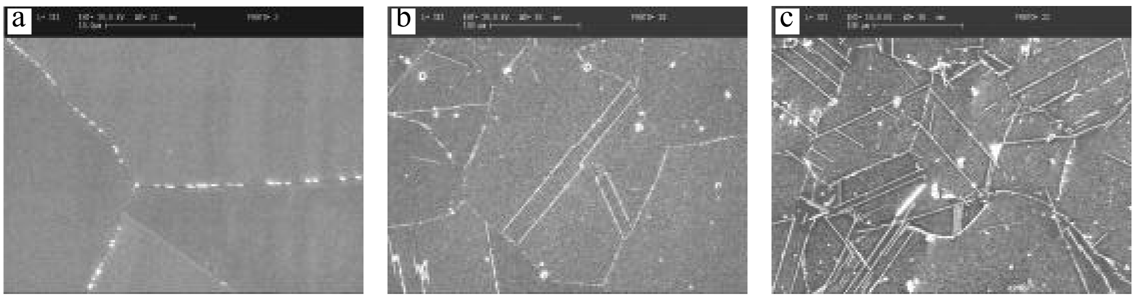

Figure 13 Microstructure of Alloy 718plus Specimens Exposed at 954℃
For (a) 0.1 h, (b) 10 h, (c) 50 h

The major precipitated phases found in alloy 718 in this study by isothermal treatments up to 100 h are γ', γ'', and δ phases. While the phases found in alloy 718Plus are γ', γ'', δ, and $Ni_3Al_{0.5}Nb_{0.5}$ phases[12]. The difference between alloys 718 and 718Plus is that the main strengthening phase in alloy 718 is γ'', but the main strengthening phase in alloy 718Plus is γ'. Moreover, a new plate-like $Ni_3Al_{0.5}Nb_{0.5}$ with HCP structure has been found in alloy 718Plus. The grain boundary precipitates EDX analyses of the specimens for both alloy 718 and alloy 718Plus solution treated at 1040°C for 1 h and aged at 954°C for 5 h shows definite enrichment in Nb and Ni. The residues from the HCl-Methanol electrolytic extraction on the same specimens contain δ and MC (Nb and Ti carbide). It confirms that the grain boundary precipitates are δ-Ni_3Nb. Moreover, selected TEM analyses were conducted to confirm the presence of plate-like $Ni_3Al_{0.5}Nb_{0.5}$ phase[12] in addition to plate-like δ phase in alloy 718Plus after aging at 900°C and 954°C for some times.

Time-Temperature-Transformation Diagrams

Based on the structure analyses and hardness tests, two TTT diagrams for alloys 718 and 718Plus are presented in Figure 14. Changes in hardness (Figure 1) correlate well with the γ'/γ'' precipitation. γ'/γ'' start to precipitate in the matrix when the Vickers hardness exceeds 225HV and 223HV for alloy 718 and alloy 718Plus respectively during the aging treatment. γ'/γ'' and δ solvus in Figure 14a and 14b have been obtained from Thermo-Calc thermodynamic calculation. Figure 14a shows the TTT curve for alloy 718 and the nose of the TTT curve for γ'/γ'' precipitation occurs at about 850°C for less than 0.1 h and the nose of the TTT curve for grain boundary δ precipitation occurs at about 900°C for much less than 0.1 h. It is found that δ phase precipitation is much faster than reported in most previous work. The right part of Figure 14a also shows the large plate-like δ precipitation curve. The large plate-like δ phase can form at higher temperature range after several hours aging. Figure 14b shows the TTT curve for alloy 718Plus and the nose of the TTT curve for γ'/γ'' precipitation occurs at about 900°C for less than 0.1 h and the nose of the TTT curve for δ precipitation occurs at about 900-950°C for less than 0.1 h. The plate-like Ni_3M precipitation can form at 900°C for only 1hr thermal aging. It can be seen that the TTT curves of γ'/γ" and δ precipitation for alloy 718Plus have shifted to the higher temperature for shorter time than for conventional alloy 718. γ'/γ" and δ solvus (989°C and 1060°C) for alloy 718Plus obtained from Thermo-Calc thermodynamic calculation are much higher than that for conventional alloy 718. It explains that the precipitation hardening phases in alloy 718Plus are more stable at higher temperature than that in alloy 718.

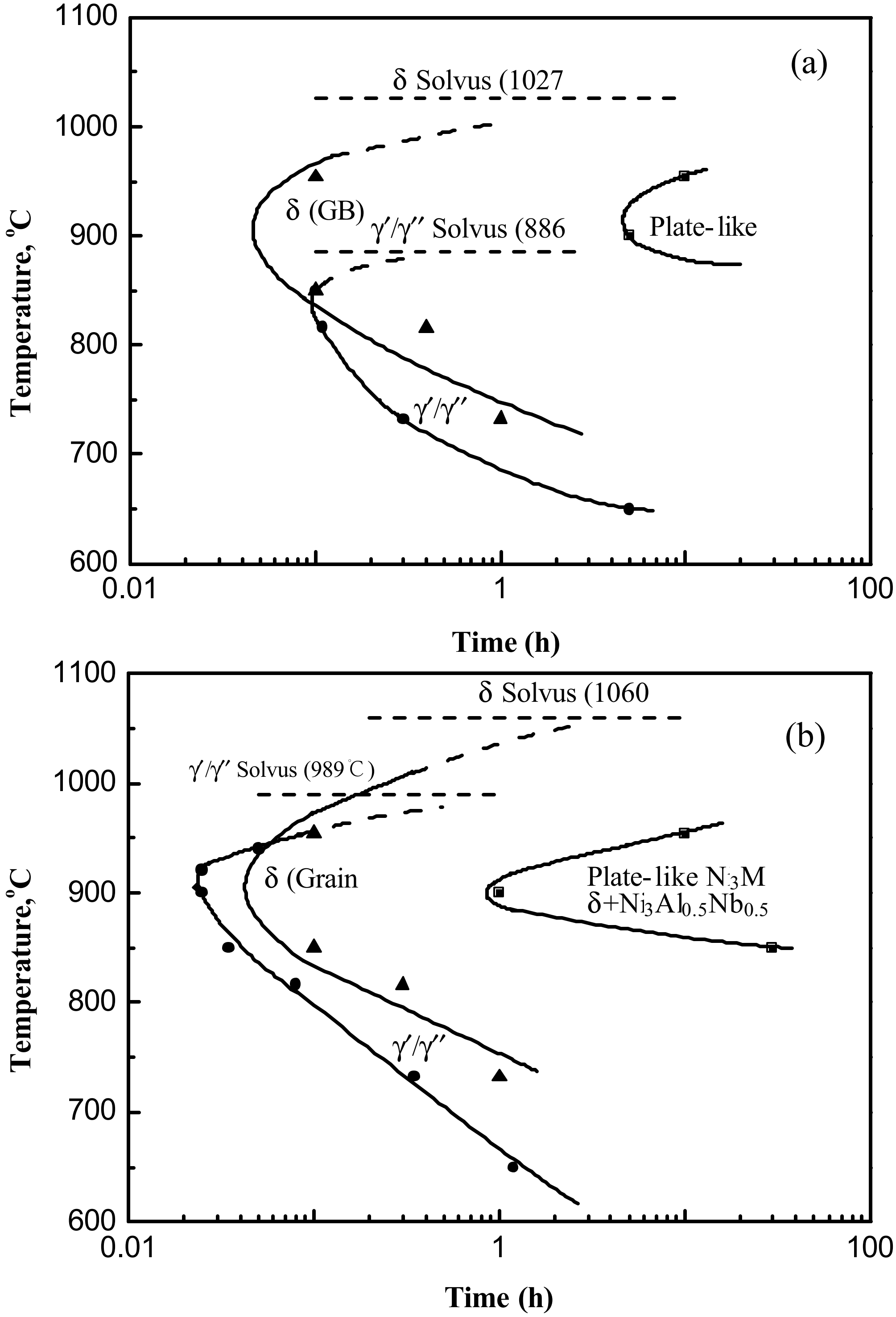

Figure 14 Time-Temperature-Transformation Curves
(a) Conventional Alloy 718, (b) Alloy 718Plus

Conclusions

1. In alloy 718 only γ', γ", and δ phases can be found in addition to the primary MC (Nb and Ti carbide) phases, while in alloy 718Plus additional plate-like Ni_3M ($Ni_3Al_{0.5}Nb_{0.5}$) phase with HCP structure has been found. The morphology of $Ni_3Al_{0.5}Nb_{0.5}$ phase is similar to the δ phase but difficult to differentiate from δ phase. No α-Cr, σ phases were found in this study, which are often found when stress is applied or in very long time exposures.

2. The start of precipitation of γ'/γ" and δ phases in alloy 718 seems to proceed in shorter times than reported in most previous work. Meanwhile, the start of precipitation of γ'/γ" and δ phases in alloy 718Plus proceeds at higher temperatures and shorter times than those in conventional alloy 718.

3. In comparing the phase formation curves in alloy 718Plus with conventional alloy 718, all curves move to higher temperature and shorter times. It is probably the effect of high aluminum content (1.45%Al) in alloy 718Plus which promotes γ' precipitation and also may lead to the formation of the $Ni_3Al_{0.5}Nb_{0.5}$ in comparison with alloy 718.

Acknowledgement

The authors would like to thank ATI Allvac and Fushun Steel Company for the materials used in this study. The financial support of ATI Allvac is most appreciated.

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