INVESTIGATION ON HIGH THERMAL STABILITY AND CREEP RESISTANT MODIFIED INCONEL 718 WITH COMBINED PRECIPITATION OF γ'' AND γ' *

Xishan Xie, Qiang Liang, Jianxin Dong, Weigang Meng and Zhichao Xu
University of Science and Technology Beijing, Beijing 100083, China

Mengzhe Chen
Beijing Laboratory of Electron Microscopy
Chinese Academy of Sciences
Beijing 100080, China

Fugang Wang, Yu Cai and Junshan Zhang
Dalian University of Technology, Dalian 116024, China

Ning Wang, E. Andrieu and A. Pineau
Centre des Materiaux, Ecole des Mines, B. P. 87, 91003 Evry Cedex
URA CNRS DO866, France

Abstract

One of the recent advances in INCONEL 718 development is intended to find a modified Alloy 718 with high thermal structure stability and creep resistance to be used beyond the ceiling temperature of 650℃. Twelve heats of modified Alloy 718 with chemical composition variation were designed to study the effect of Al, Ti, Nb and Mo on γ'' and γ' precipitation morphology and on microstructure stability and high temperature mechanical properties above 650℃. Detail TEM and high resolution electron microscopy (HREM) study has shown that associated precipitation and compact morphology of γ'' and γ' in modified 718 Alloys characterize with higher thermal stability than separate precipitation of γ'' and γ' in conventional INCONEL 718. Creep rupture life depends not only on Nb + Al + Ti content but also on coefficient $k = \sqrt{(Al+Ti/Nb)^2 + (Al/Ti)^2} \cdot Nb$. Long time aging stability study at 650, 700 and 750℃ for 5000 hrs has shown that the modified Alloy 718 with associated precipitation and compact morphology of γ'' and γ' expresses excellent thermal structural stability.

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Introduction

γ'' (bct DO_{22} structure) and γ' (fcc Ll_2 structure) are typical strengthening phases in nickel-base superalloy. The unique strengthening effect of γ'' -Ni_3Nb and γ'-Ni_3(TiAlNb) helps Alloy 718 to have wide industrial application. Because of the stability limitation of γ'' the highest service temperature of Alloy 718 can reach 650℃only. However, the stability of γ'strengthening phase in nickel-base superalloys is much higher than 650℃. Basic idea of this research is intended to find a combination of high strengthening effect of γ'' and good stability of γ' in modified INCONEL 718 to be allowed alloy to use at temperature higher than 650℃.

Since 1973 R. Cozar and A. Pineau (1) published a pioneer paper on modified Alloy 718 with compact morphology of γ'' and $\gamma'$to be possible on higher thermal stability as shown in age hardening test results. After 15 years a series of papers (2—9) modifying the chemical composition with variation of Al, Ti and Nb contents in Alloy 718 has shown that mechanical properties beyond 650℃ can be improved with either the non-compact or the compact morphology of γ'' and γ' obtained by appropriate heat treatment. However, long time thermal stability study from the point of view of strengthening mechanisms and creep resistance has not appeared yet. This investigation concentrates on TEM analyses of γ'' and γ' precipitation morphology and their long time structure stability beyond 650℃ and follows with creep tests in point of view from high temperature strengthening effect. The first phase goal of this research tries to get fundamental understanding on stability improvement that helps modified Alloy 718 development.

Materials and Experimental Procedure

Twelve heats of experimental alloys were melted in 25 and 50kg VIM furnaces. All VIM ingots were homogenized by 2 step high temperature treatment (1st step for Laves phase solution and 2nd step for homogenization of Nb), Then ingots were forged down to 40×40mm billets and finally hot rolled for 18mm diameter bars (except 2 heats of powder metallurgy prepared modified 718 alloys). Chemical composition of 12 modified 718 alloys are devided in 3 groups and listed in Table I.

Group A consists of 3 alloys with different levels of Nb (5.08, 4.38 and 3.44%). Alloy 11 and 13 compose of higher contents of Ti and Al for γ' strengthening precipitation. Alloy 15 was designed with high content of Mo (7%) for strong solid solution strengthening. Group B consists of 7 alloys with 3 levels of Nb (~5.5, 5.1 and 4.75%) and higher contents of Al, Ti and higher ratios of Al+Ti/Nb and Al/Ti also. All 7 heats were micro-alloyed with~50ppm Mg for hot workability and ductility improvement. Alloy 10 was considered as conventional Alloy 718. Group C consists of 2 alloys and specially prepared by powder metallurgy in IMPHY, France. Alloy 975 was considered as Super 718 with high content of Nb (5.39%) and normal content of Ti (~ 1%) and Al (~0.5%). Alloy 976 (similar to Alloy 4 in Group B) was also specially designed for associated precipitation and compact morphology of γ'' and γ' and therefore high thermal stability with medium level of Nb (~5%) and high contents of Al (1.05%) and Ti (1.22%) and also high atomic sum of Al+Ti+Nb and atomic ratios of Al+Ti/Nb and Al/Ti for keeping a good combination of γ'' and γ'precipitation.

Table I Chemical Composition of Experimental Alloys

Group	Alloy	wt%										at%		
		C	Cr	Fe	Ni	Mo	Nb	Al	Ti	B	Mg	Al+Ti / Nb	Al / Ti	Al+Ti +Nb
A	11	0.075	19.01	19.53	bal.	2.98	5.08	0.73	1.14	0.008	—	0.93	1.14	6.09
	13	0.063	19.05	20.23	bal.	3.02	4.38	0.72	1.13	0.007	—	1.07	1.13	5.62
	15	0.057	19.01	17.72	bal.	7.04	3.44	0.68	0.595	0.011	—	1.01	2.01	4.37
B	1	0.02	19.06	18.70	bal.	3.04	5.47	0.98	1.02	0.0034	0.0065	0.98	1.71	6.75
	2	0.03	18.94	18.56	bal.	3.08	5.51	1.15	1.14	0.0024	0.0075	1.12	1.79	7.24
	4	0.02	19.12	18.76	bal.	3.04	5.09	0.98	1.17	0.0024	0.0056	1.11	1.49	6.65
	6	0.02	19.04	18.30	bal.	2.75	5.06	1.25	1.39	0.0025	0.0057	1.38	1.60	7.45
	7	0.02	18.74	19.25	bal.	3.04	4.72	0.86	1.19	0.0029	0.0056	1.12	1.28	6.19
	9	0.02	19.04	19.31	bal.	3.04	4.75	1.28	1.37	0.0015	0.0055	1.49	1.66	7.28
	10	0.02	18.88	18.36	bal.	3.00	5.14	0.46	1.03	0.0033	0.0039	0.70	0.79	5.44
C	975	0.04	18.00	18.80	bal.	2.97	5.39	0.54	1.02	0.0044	trace	0.71	0.94	5.76
	976	0.036	17.65	18.80	bal.	3.00	5.00	1.05	1.22	0.0037	trace	1.20	1.53	6.80

Age hardening study on Alloys 11, 13 and 15 was conducted with 950℃/1h/WC solution treatment and followed by aging at 710, 730, 760, 780, 800 and 820℃ for 1, 3, 8, 12 and 20 hrs. For stability study the specimens after 950℃/1h/WC+760℃/8h/WC were long time aged at 730℃ for 200 hrs again.

Creep tests on Group B Alloys were taken at 700℃ and 500, 550 and 600 Mpa. All the specimens were heat treated as follows:

1000℃/1h/AC+700℃/8h→F. C. 50℃/h+600℃/8h/AC.

Alloy 975 and 976 powder products were HIPed at 1150℃, 1000bar for 4hrs and followed by 1180℃ homogenization treatment for 72 hrs. For fully solution of γ'', γ' and δ phases the solid solution treatment temperature was chosen as 1025℃. Multi-step aging was designed for special reason, 1st step T℃/h for precipitation ~ 200Å average size of γ' and followed with certain cooling rate to promote γ'' nucleation directly at γ' phase; last step was chosen as 650℃/16h/AC for getting fully aged structure. Long time aging of Alloy 975, 976 and 976 with appropriate heat treatment (designated as 976M) was conducted at 650, 700 and 750℃ for 5000hrs. Hardness test was chosen as the simplest indication of strengthening effect. Microstructure and γ'', γ' morophology observation was mainly conducted on thin foil TEM with bright and dark field images. Typical compact morphology of γ'' and γ' was analysed by high resolution electron microscopy (HREM).

Results and Discussion

Age hardening

Age hardening curves of Alloy 11,13 and 15 at 710, 730, 760, 780, 800 and 820℃ are shown in Fig. 1. All the alloys possess typical age hardening effects. However, all three Alloys do not reach peak hardness at 710-730℃ aging for 20hrs. All the Alloys reach the peak hardnesses at 760℃ for 8hrs. Age hardening effect is promoted by raising temperature. However, Alloys characterize serious softening when the aging temperature is beyond 800℃. Age hardening effect is clearly dependent on Nb content. Age hardening curves are very close for Alloys 11 and 13, although 2 Alloys contain different levels of Nb (Alloy 11-5.08%Nb, Alloy 13-4.38%Nb). Age hardening effect is much lower in Alloy 15 than in Alloys 11 and 13 because of its very low content of Nb (3.44%), although it contains very high Mo (7%).

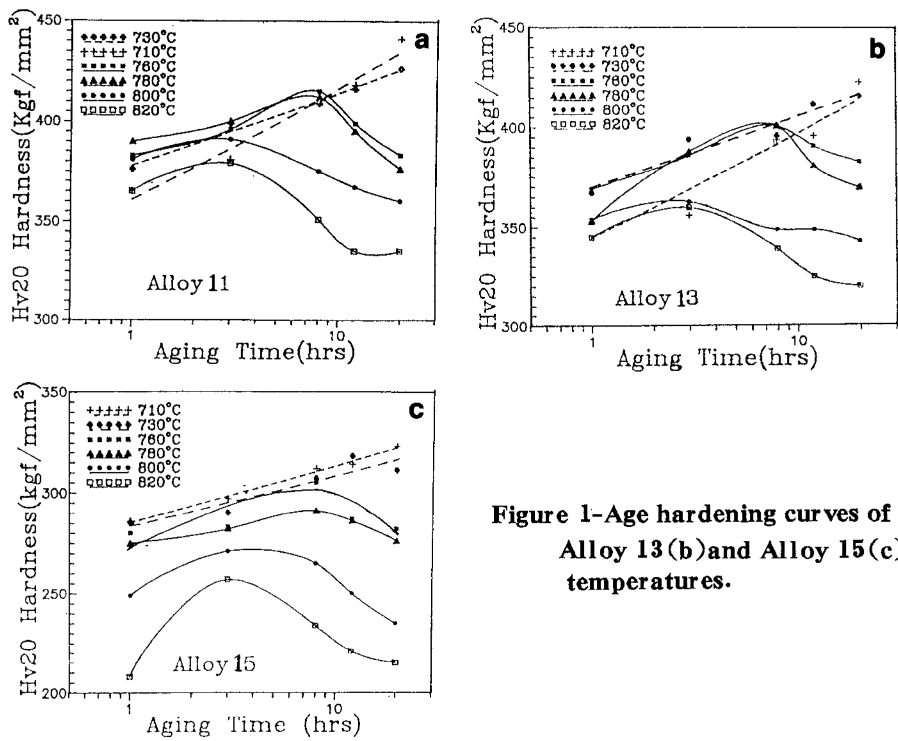

Figure 1-Age hardening curves of Alloy 11(a), Alloy 13(b)and Alloy 15(c)at different temperatures.

Table Ⅱ shows hardnesses of 3 Alloys for long time aging (730℃/100 and 200h) after reaching peak hardnesses at 760℃/8h. For Alloys 11 and 13 after 100 and 200h long time aging hardnesses keep same levels almost as the hardnesses of 760℃/8h aging. However, the hardnesses of Alloy 15 are increasing continiously from Hv 311 till 375 for 730℃/200h aging.

Table Ⅱ Hardnesses Hv 20(kgf / mm²) of Alloys 11, 13, 15 after 730℃ long time aging.

Alloy	760℃ / 8h	700℃ / 8h+730℃ / 100h	760℃ / 8h+730℃ / 200h
11	415	415	415
13	401	404	404
15	311	343	375

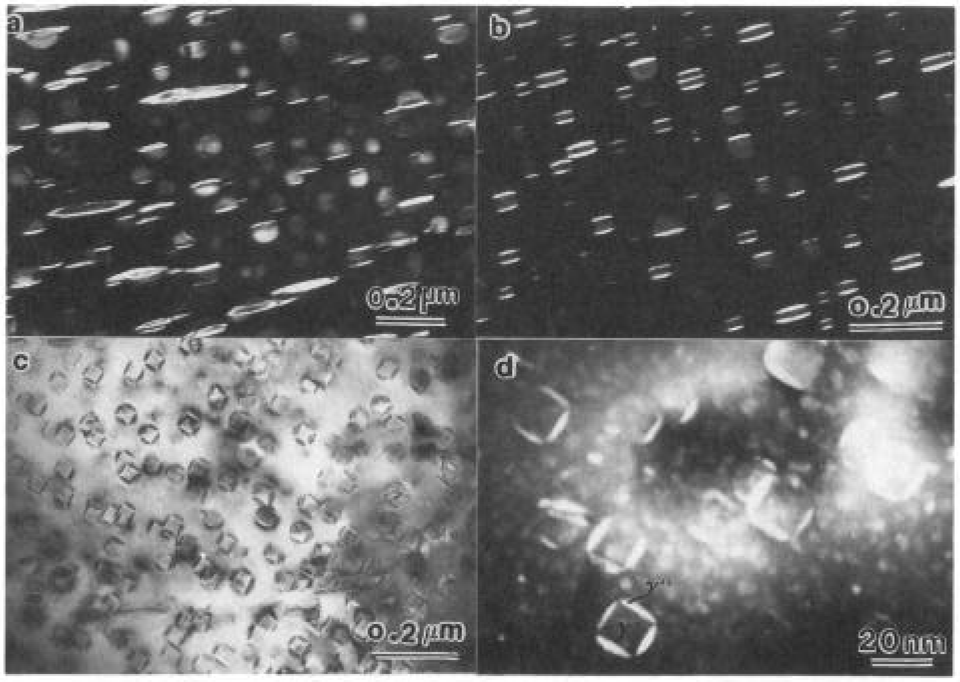

Figure 2-Associated precipitation and compact morphology of γ'' and γ' in Alloy 13 (a-dark field) and Alloy 15 (b, d-dark field, c-bright field) after long time aging at 730℃ for 200hrs.

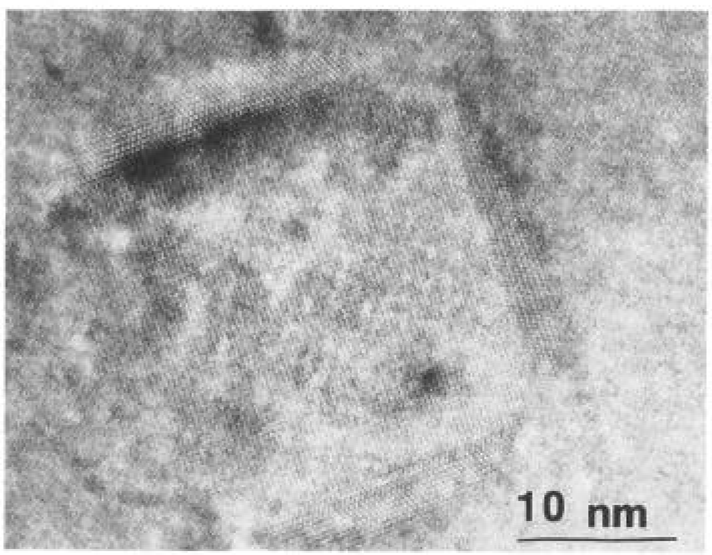

Figure 3-HREM image of compact morphology of γ'' and γ' in Alloy 15 after long-time aging at 730℃ for 200hrs.

TEM and HREM observation

Chemical composition of Alloy 11 is close to INCONEL 718 but with a little high contents of Al and Ti and higher ratios of Al+Ti/Nb and Al/Ti. Most of γ'' and γ' are precipitated separately from γ-matrix during the aging and intensively grow especially for main strengthening phase γ''. Two types of combined precipitation of γ'' and γ' are found in modified 718 Alloys 13 and 15, especially.

(A) Associated precipitation of γ'' and γ'. γ'' can be bound up with almost hemispherical γ' or can be alternatively precipitated as a " sandwich" morphology as shown in Fig. 2 (a) and (b).

(B) Compact morphology of γ'' and γ'. γ'' can directly precipitate at cuboid-shaped γ' particles coated with its shell as shown in Fig. 2 (c) and (d).

These two types combined precipitation of γ'' with γ' are different to separate precipitation of γ'' and γ' as the mixture of disk-shaped γ'' and round γ' particles in conventional INCONEL 718.

γ'' is the most inportant strengthening phase in Alloy 718. However, separately precipitated disk-shaped γ'' grows rapidly in conventional INCONEL 718 beyond 650℃. Same tendency occurs in modified 718 Alloys, that separately precipitated γ'' grows rapidly and the average length of γ'' disks can be increased from ~200Å (760℃/8h aging) to almost 1700Å (after 730℃/200h long time aging) as shown in Fig. 2 (a) for Alloy 13. The γ'' coarsening rate is as high as 7Å/h. However, the associated precipitated γ'' bound with hemispherical shaped γ' grows slowly as shown in the same picture (Fig. 2a, b) for good comparison. Especially the γ'' in compact morphology of $\gamma'' + \gamma'$ grows more slowly as shown in Fig. 2 (d). The γ'' coarsening rate is much lower (~3Å/h) for Alloy 15 in comparison with Alloy 11 and 13. These results clearly show higher thermal stability of main strengthenign phase of γ'' in modified 718 Alloys with associated precipitation or compact morphology of γ'' and γ'.

Compact morphology of γ''andγ' is shown in detail on Fig. 3 by high resolution electron microscopy image. The interfaces of γ' /γ'' and γ'' /γ will be studied further.

Associated precipitation and compact morphology of γ'' and γ' can be formed in modified 718 Alloys with higher contents of Al (≥1. 0%) and Ti (≥1. 0%) and higher ratios of Al+Ti/Nb and Al/Ti at different levels of Nb (~5. 5%Nb for Alloy 1, 2; ~5. 1% Nb for Alloy 4, 6 and ~4. 75% Nb for Alloy 7, 9, see Fig4a, b, c). However, γ'' and γ' separately precipitate from γ-matrix in conventional Alloy 718 with lower contents of Al (~0. 5%) and Ti (~1. 0%) and lower ratios of Al+Ti/Nb=0. 70 and Al/Ti=0. 79 (see Fig 4d).

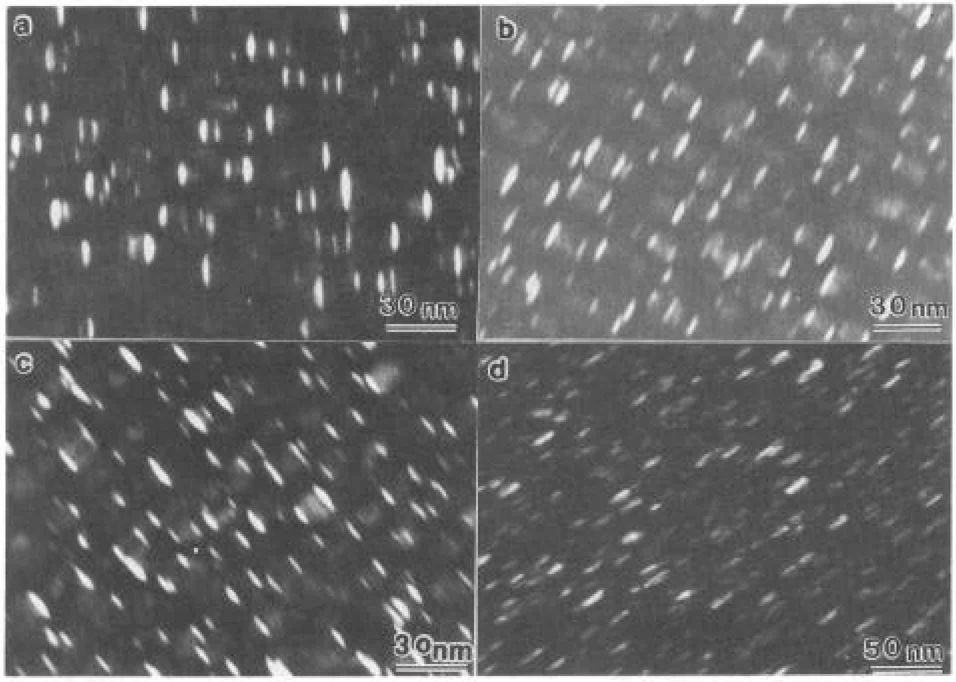

Figure 4-Associated precipitation and compact morphology of γ'' and γ' in modified 718 Alloy 1 (a), Alloy 6 (b), Alloy 9 (c) and separate precipitation of γ'' and γ' in conventional INCONEL 718 (Alloy 10-d).

Creep tests.

Creep fracture lives (tr) at 700℃ and different stresses (500-600MPa) increase with the atomic sum of Al, Ti, and Nb and reach the peaks at Al+Ti+Nb=7, then mildly decrease again when Al+Ti+Nb increases furthur. (see Fig 5a). In consideration of the complex effect of Al, Ti, and Nb a coefficient of $K=\sqrt{(Al+Ti/Nb)^2+(Al/Ti)^2}\cdot Nb$ is suggested for creep lives evaluation. Creep rupture lives are almost proportional to the coefficient K at the stress levels of 500, 550 and 600MPa for 700℃ (see Fig. 5b). Creep rupture lives of almost all modified 718 Alloys in Group B are longer than conventional Alloy 718 (Alloy 10 in Fig 5a and b). It indicates that associated precipitation and compact morphology of γ'' and γ' in modified Alloy 718 with higher Al+Ti+Nb and higher ratios of Al+Ti/Nb and Al/Ti possess with not only higher thermal stability but also longer creep rupture life.

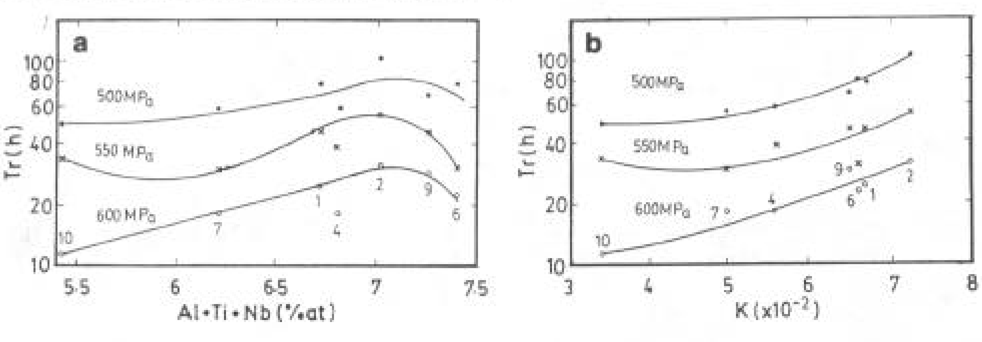

Figure 5-The dependence of 700℃ creep fracture time (tr) on atomic sum of Al+Ti+Nb- (a) and on coefficient K at 500, 550, 600MPa- (b).

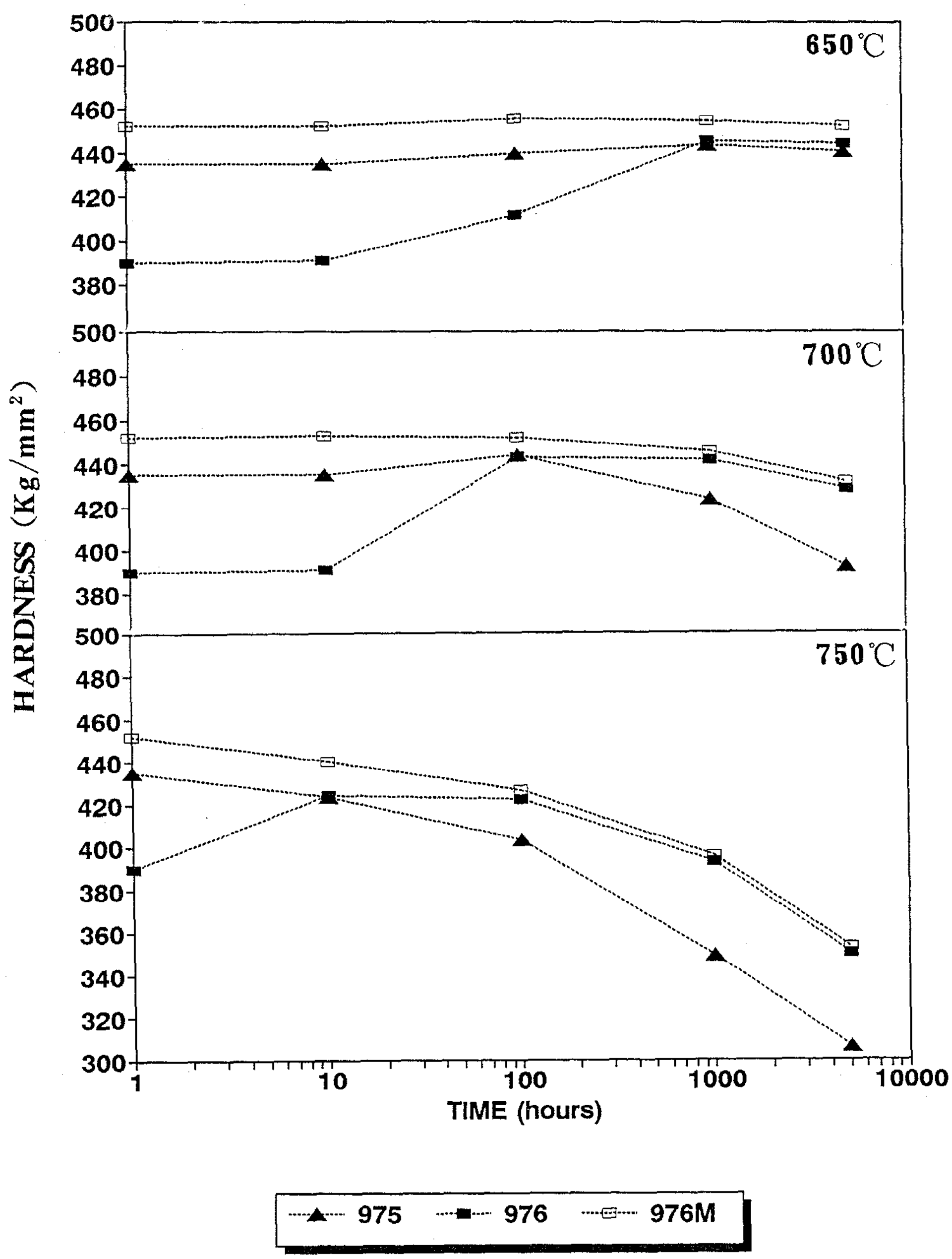

Figure 6-The dependence of hardness (Hv20) on long time aging (5000hrs) at 650, 700 and 750℃ for alloy specimens 975, 976 and 976M.

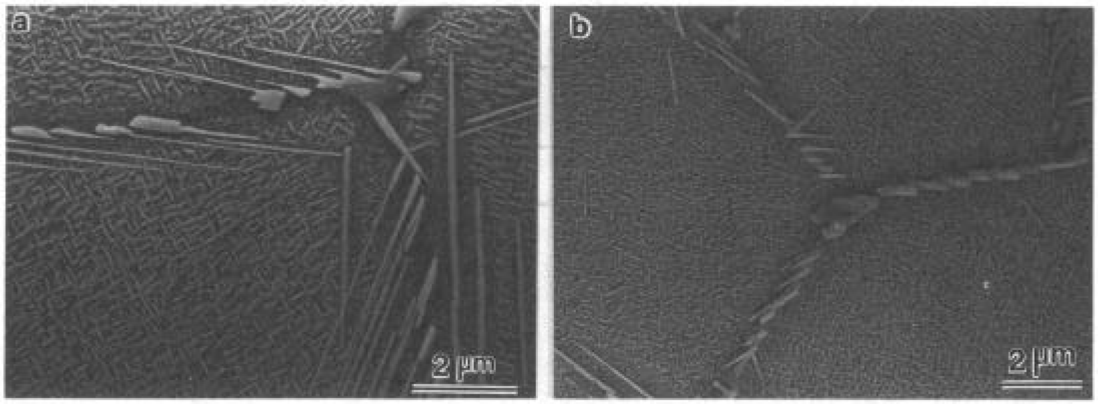

Figure 7-SEM structure comparison of 975 (a) 976 (b) after long time exposure at 750℃ for 1000hrs.

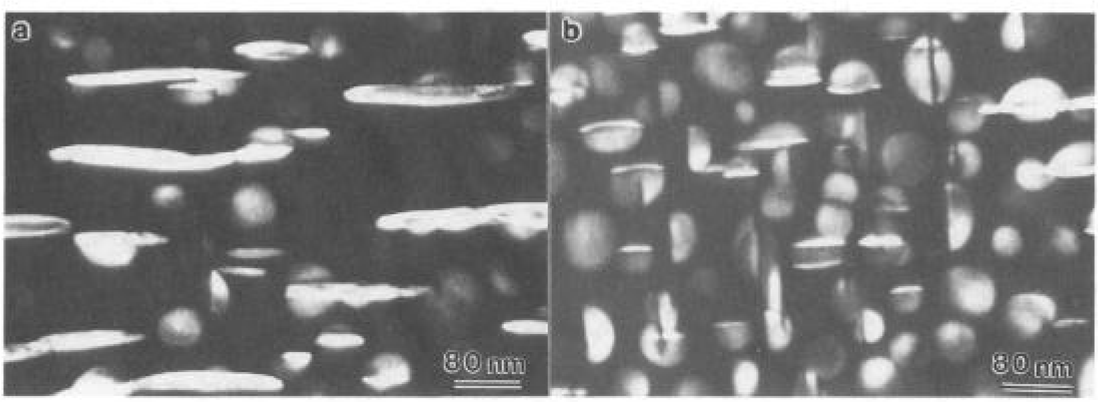

Figure 8-TEM dark field images of γ'' and γ' precipitates after long time exposure at 750℃ for 1000hrs in alloy specimens of 975 (a) and 976M (b).

Long time structure stability.

A modified 718 (Alloy 976) was specially designed for long time structure stability study in comparison with Super 718 Alloy (Alloy 975). Both alloys were heat treated as 1025℃/30 min/AC. +720℃/8h→FC. 50℃/h+620℃/8h/AC. For obtaining compact morphology of γ'' and γ' a special heat treatment 1025℃/30 min/AC+850℃/30 min→300℃/h→750℃→50℃/h+650℃/16h/AC was conducted for Alloy 976 (designated as 976M). Fig. 6 shows the dependence of hardness on long time aging (till 5000hrs) at 650, 700 and 750℃ for alloy specimens 975, 976 and 976M. It clearly shows that 976M possesses with the highest hardness level at all aging temperatures. SEM observation gives a clear comparison that microstructure of 976M is more stable than 975 after 750℃ aging for 1000hrs (see Fig. 7). TEM observation shows the rapid growth of disk-shaped γ'' in Alloy 975 (see Fig. 8a). However, γ'' bound with γ' as associated precipitation and compact morphology in Alloy 976M possesses with excellent thermal stability even at 750℃ for 1000hrs (see Fig 8b).

Conclusions

1. Thermal stability and creep resistance can be improved in modified 718 Alloys by increasing the ratios of Al+Ti/Nb and Al/Ti and the Al+Ti+Nb content above that of the conventional INCONEL 718.
2. The main structural factors for thermal stability improvement above 650℃ are contributed by associated precipitation and compact morphology of γ'' and γ' because of the slow growth rate of γ'' bound with γ' in modified 718 Alloys at high temperature.
3. A modified 718 Alloy with appropriate heat treatment (976M) possesses with excellent thermal stability and strengthening effect even at 750℃ long time aging for 5000hrs.

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