# ALPHA CHROMIUM FORMATION IN ALLOY 718 AND ITS EFFECT ON CREEP CRACK PROPAGATION

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#### Abstract

Structure stability study on Alloy 718 disks with four different forging/heat treatment process combinations, i.e. Standard718, Super I 718,Super II 718 and DA718, has been conducted with long time thermal exposure at 593 °C(1100°F),649 °C(1200°F) and 677 °C(1250°F) till 2000hrs. A b.c.c chromium enriched phase  $\alpha$ -Cr has been identified by XRD,SEM,TEM and SAD methods. The existence of  $\alpha$ -Cr in Alloy 718 is also confirmed by Thermo-Calc method. The weight fraction of  $\alpha$ -Cr has been determined by special designed electrolytic isolation and micro-chemical analyses.  $\alpha$ -Cr precipitates are often in small globular particles ( $\sim$ 0.3  $\mu$  m) and form at the vicinity of  $\delta$ -Ni<sub>3</sub>Nb phase. More  $\alpha$ -Cr particles will precipitate in Alloy 718 at higher exposure temperatures and longer times. Retained stress can promote  $\alpha$ -Cr precipitation . The fractions of  $\alpha$ -Cr in 4 different processed Alloy 718 disks are not very high, only less than 1%wt. The highest fraction of  $\alpha$ -Cr in DA718 after 677°C/2000hrs is about 0.62%wt and the  $\alpha$ -Cr fraction in Standard 718 after 677°C/2000hrs is only 0.19%wt. Small amount of  $\alpha$ -Cr in Alloy 718 does not has serious deleterious effect on 650°C creep crack propagation rate. However, the impact energy does decrease with the thermal exposure times at 649°C and 677°C.

#### Introduction

Our previous studies on structure stability <sup>[1]</sup> and the effect of processing on stability of Alloy  $718^{[2]}$  show that when Alloy 718 was exposed for long time at temperature of  $593^{\circ}$ C,  $649^{\circ}$ C and  $677^{\circ}$ C,  $\alpha$ -Cr can precipitate from  $\gamma$ -matrix in addition to  $\delta$ -Ni<sub>3</sub>Nb phase.  $\alpha$ -Cr formation is not only dependent on chemical composition, especially chromium content in Alloy  $718^{[3]}$ , but also dependent on residual strain produced by cold work<sup>[4]</sup>.

Superalloy producers and engine manufacturers are very interested in  $\alpha$ -Cr behavior and its effect on mechanical properties, especially on high temperature creep crack propagation rate, which is critical for disk application in aircraft and land-base gas turbines.

A research project has been undertaken to study the stability of 4 different processed Alloy 718 disks at  $593\,^{\circ}\text{C}-677\,^{\circ}\text{C}$  thermal aging till 2000hrs. This is a continuing investigation followed our previous work<sup>[2]</sup> and the purpose of this paper concentrates on understanding the formation of  $\alpha$ -Cr and its effect on mechanical properties as follows:

- 1.  $\alpha$ -Cr behavior study including  $\alpha$ -Cr identification by XRD, SEM, TEM and SAD method, its morphology, size, distribution and precipitation behavior. The quantitative determination of  $\alpha$ -Cr in four different processed Alloy 718 disks with long time thermal aging has been conducted.
- 2. The effect of long time aging on  $\alpha$  -Cr formation and on mechanical properties, especially on 650 °C creep crack propagation rate has been studied in detail.

## **Experimental Procedures**

### Processes and Thermal Exposure

Triple melt (VIM+ESR+VAR) Alloy 718 with the chemical composition as shown in Tab. 1, was forged into a simple turbine disk configuration (230mm in diameter) using 4 different forging/heat treatment process combinations as shown in Tab. 2. These disks were then cut into sections for thermal exposure at 593°C, 650°C and 677°C till 2000hrs. The disks and samples ID are shown in Tab. 3.

Table 1. Chemical Composition of Investigated 718 Alloy(wt.%)

С	Mn	Si	S	Cr	Ni	Mo	Nb	Ti	Al	В	Fe
0.021	0.08	0.07	0.0003	17.89	53.88	2.99	5.36	0.94	0.49	0.0029	bal

### Mechanical Tests

Hardness, Charpy impact,  $650^{\circ}$ C tensile and notched stress rupture tests (notch factor  $k_t$ =3.6) were ran on all conditions as listed in Tab.3.  $650^{\circ}$ C creep crack propagation rate determination tests were ran by using compact tension specimens.

Table 2. Alloy 718 Disks Processed in Four Different Conditions

Disk	Classification	Processes	Grain Size
No.			
1,2	DA718	Fine grain forging with direct age heat treatment	ASTM 11-12
3,4	Super I 718	Fine grain forging with solution+age heat treatment	ASTM 11-12
5,6	Super II 718	Ladish proprietary process(special fine grain forging+DA)	ASTM10-11
7,8	Standard 718	Medium grain forging with solution +age heat treatment	ASTM7-9

Table 3. Thermal Exposure Tests for Four Different Processed 718 Disks

	0hr	500hrs	1000hrs	2000hrs
593℃(1100°F)			Disk 1,3,5,7(B)	Disk 1,3,5,7(C)
650°C(1200°F)	Disk 1,3,5,7(A)		Disk 1,3,5,7(D)	Disk 1,3,5,7(F)
677°C(1250°F)		Disk 2,4,6,8(G)	Disk 2,4,6,8(H)	Disk 2,4,6,8(I)

### **Structural Evaluation**

Modified electrolytic techniques for SEM observation were developed by J. Radavich<sup>[4]</sup> and were used to reveal the following: 1)  $\alpha$ -Cr phase in relief, 2)preferential etching of  $\alpha$ -Cr, or 3)both the  $\alpha$ -Cr and  $\delta$ -Ni<sub>3</sub>Nb as separate particles. X-ray diffraction of extracted residues was used to confirm the presence of the  $\alpha$ -Cr phase. TEM thin foil observation has been conducted with selected area diffraction (SAD) to identify  $\alpha$ -Cr phase directly. A special designed electrolytic isolation and micro-chemical analyses have been used for weight fraction determination of  $\alpha$ -Cr phase<sup>[5]</sup>. Thermo-Calc software was used to evaluate the  $\alpha$ -Cr formation and to compare with micro-structural evaluation.

#### Results and Discussions

## **Mechanical Properties**

Hardness. There was no significant difference in hardness among the groups of samples (as shown in Fig.1) after exposure at  $593^{\circ}$ C,  $650^{\circ}$ C and  $677^{\circ}$ C till 2000hrs. The mean hardness of the four group samples before the exposure was HRc45. After exposure at  $593^{\circ}$ C for 2000hrs the mean hardness of four groups increased to 48HRc, because of the complementary precipitation of Y'' and Y' for age hardening. The mean hardness was 42 after exposure at  $677^{\circ}$ C for 2000hrs. The largest decrease in mean hardness (from HRc45 to HRc40) appeared in samples exposed at  $677^{\circ}$ C for 2000hrs, because of the softening effect developed by the strengthening phases (Y'' + Y') coagulation and  $Y'' \rightarrow \delta$  transformation.

650°C Tensile Strength. These tensile strengths, YS and UTS, both have almost no noticeable reduction after 2000hrs exposures at 593°C and 650°C for 4 different processed disks as shown in Fig.2a. However, there is about 20% decrease in tensile strength after 677°C exposure for 2000hrs as shown in Fig.2b. These results are in accordance with hardness tests

(as shown in Fig.1) because of the softening effect developed at high temperature (677°C) long time exposure (2000hrs).

650°C Stress Rupture Lives. DA718 and Super II 718 (special fine grain processing +DA) characterize with highest hardness, 650°C tensile strengths and stress rupture lives (as shown In fig.1,2 and 3) among 4 different processed disks. However, a noticeable rupture time decrease was found after 649°C exposure for 2000hrs (see Fig.3a). A dramatic decrease of more than 80~90% in rupture times was found after the exposure at 677°C for 2000hrs (see Fig. 3b). All the times to rupture for DA718, Super II 718 Standard 718 and Super I 718 all decreased to very low levels, which were 34,22,15 and 8 hrs respectively.

Impact Energy. The decrease in Charpy impact energy is noticeable for all 4 different processed disks even the hardness and tensile strengths both have almost no reduction after 650°C exposure till 2000hrs (see Fig.4a and 2a). A dramatic charpy impact energy decrease happened for all 4 different processed disks at 677°C exposure till 2000hrs (see Fig.4b). Among them the strength level of DA718 and Super II 718 dropped to lowest levels. For example, after 677°C, 2000hrs exposure the impact energies of DA718 and Super II 718 were only 8.1J and 10.8J respectively and the impact energies of Standard and Super I 718 can keep 12.2J and 13.5J respectively.

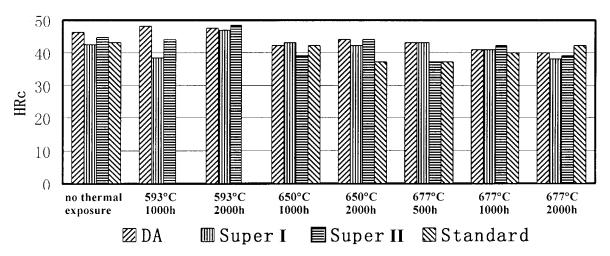


Figure 1: The hardness changes with thermal exposure time for 4 different processed 718 disks at temperature of 593 °C, 649 °C and 677 °C

### Structural and Phase analysis

SEM structure observation on 4 different processed 718 disks after  $649^{\circ}\text{C}$  exposure for 2000hrs reveals the existence of  $\alpha$ -Cr (etched out black holes as shown in Fig.5). It is very clear that DA718 contains highest fraction of  $\alpha$ -Cr among them (in comparison Fig.5b with 5a,5c and 5d). At higher temperature (677°C) exposure, more  $\alpha$ -Cr formed, as an example certain amount of  $\alpha$ -Cr can be found in Standard 718 (compare Fig.6a with 5a). Fig.6b also show the increase of  $\alpha$ -Cr fraction in Super I 718 after 677°C exposure for 2000hrs. The coagulation of main strengthening phase ( $\gamma$ ") can be also detected by SEM observation (in comparison with Fig.6 with Fig.5).

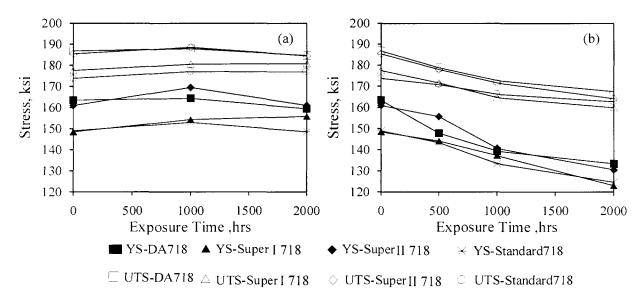


Figure 2The relationship of 650°C tensile strengths with thermal exposure time for 4 different processed 718 disks at temperatures (a) 649°C, (b) 677°C

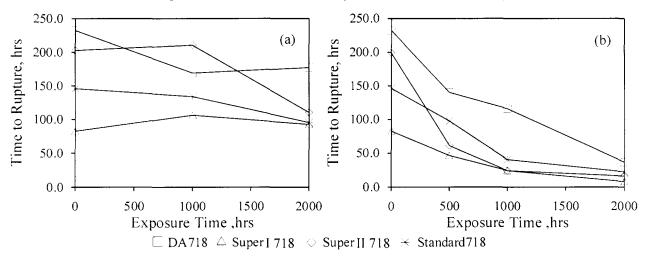


Figure.3 The dependence of notched stress rupture (650°C, 724MPa) lives on thermal exposure time for 4 different processed 718disks at temperatures (a) 649°C, (b) 677°C

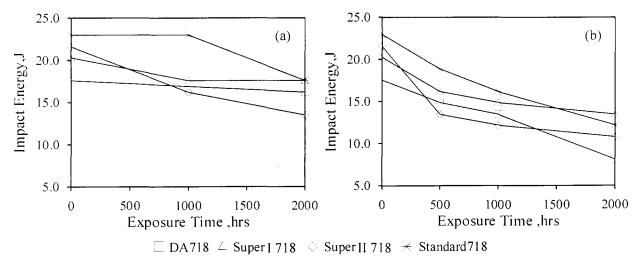


Figure.4 The dependence of impact energy on thermal exposure time for 4 different processed 718 disks at temperatures (a) 649°C, (b) 677°C

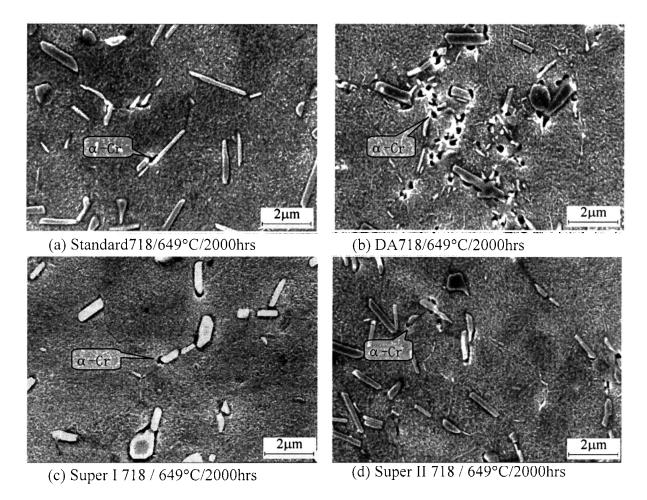


Figure 5: SEM observation on 4 different processed 718 disks thermally exposed at  $649^{\circ}C$  ( $1200^{\circ}F$ ) for 2000hrs

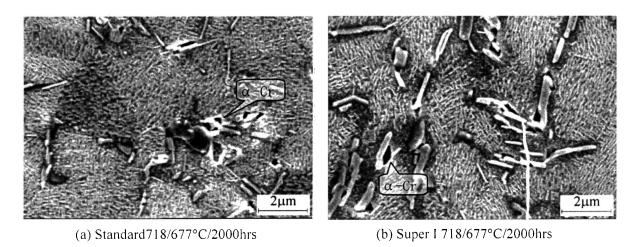


Figure 6: SEM structure observation on standard 718 (a) and Super I 718 (b) disks thermally exposed at 677°C(1250°F) for 2000hrs

X-ray diffraction on isolated residue confirms  $\alpha$ -Cr formation in 4 different processed 718, Fig.7 is an example of X-ray diffraction spectrum of Standard 718 after 677°C exposure for 2000hrs. Fig.8 shows the selective etching on  $\alpha$ -Cr (white particles) and EDS analysis directly on a "white particle", which characterizes with high content of Cr. These results reveal  $\alpha$ -Cr identification in agreement with XRD diffraction. Further detail TEM observation shows  $\alpha$ -Cr often forms with  $\delta$ -Ni<sub>3</sub>Nb and SAD analysis confirms the co-existence of  $\alpha$ -Cr with  $\delta$ -Ni<sub>3</sub>Nb phase as shown in Fig.9. More TEM observations confirm the co-existence of  $\alpha$ -Cr with  $\delta$ -Ni<sub>3</sub>Nb phase as shown in Fig.10. However, the detail mechanism of  $\alpha$ -Cr formation with  $\delta$ -Ni<sub>3</sub>Nb phase in Alloy 718 should be studied further.

Thermo-Calc results show that when the chromium content is higher than 16%,  $\alpha$ -Cr (and also  $\sigma$  phase) should form in Alloy 718 as shown in a Cr-IN718 pseudo-binary phase diagram (see Fig.11) and the  $\alpha$ -Cr fraction is linearly increased with the chromium content as shown in Fig.12.

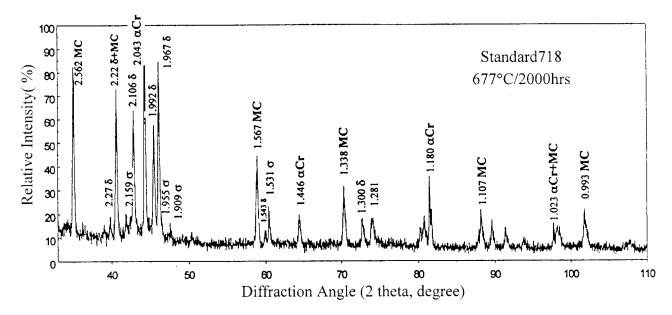


Figure 7: The X-ray diffraction spectrum of Standard 718 alloy thermally exposed at 677°C(1250°F) for 2000hrs

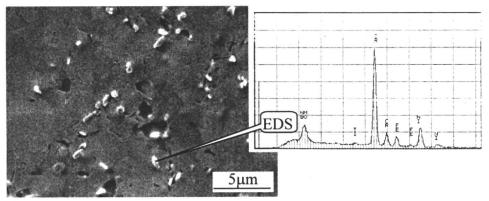


Figure 8: SEM morphology and EDS analysis of  $\alpha$ -Cr for Standard 718 alloy thermally exposed at 677°C(1250°F) for 2000hrs

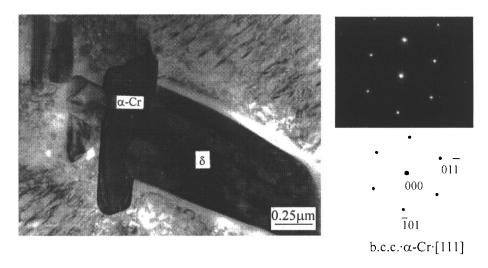


Figure 9: The morphology and selected area diffraction of  $\alpha$  -Cr phase in Standard 718 alloy thermally exposed at 677 °C (1250 °F) for 2000hrs

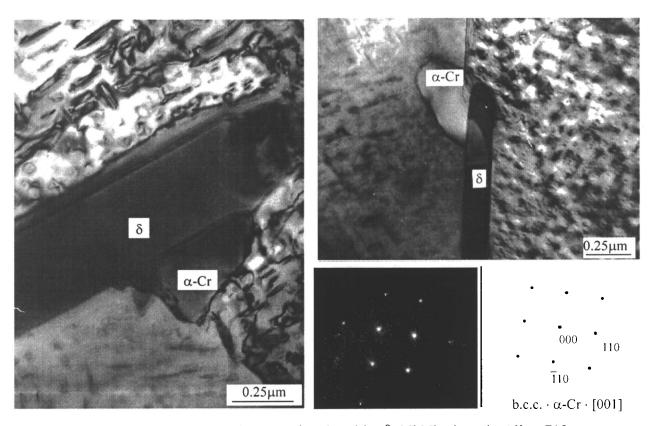


Figure 10: The co-existence of  $\alpha$  -Cr with  $\delta$  -Ni $_3$ Nb phase in Alloy 718 after 677°C(1250°F),2000hrs exposure in (a) super I 718 disk and (b) Super II 718 disk

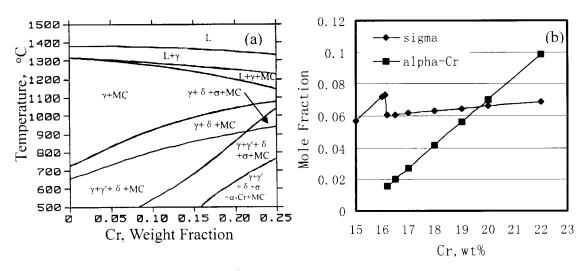


Figure 11: The Pseudo-Binary phase diagram of Cr-IN718

Figure 12: The mole fraction of  $\alpha$  -Cr and  $\sigma$  phase vs Cr content in IN718

### Quantitative Determination of $\alpha$ -Cr fraction

The weight fraction of  $\alpha$ -Cr phase in 4 different processed 718 disks at 649°C and 677°C thermal exposure till 2000hrs was carefully determined by special designed electrolytic isolation and micro-chemical analyses<sup>[5]</sup>. Fig.13 show the dependence of  $\alpha$ -Cr fraction on thermal exposure times at 649°C(a) and 677°C(b). It clearly shows that  $\alpha$ -Cr fraction keeps at very low levels for 649°C thermal exposure till 2000hrs and its fraction of Standard and Super I 718 are less than 0.018 and 0.042%wt. Super II 718 (fine grain forging+DA) contains 0.135%wt  $\alpha$ -Cr. However, DA718 contains highest level of  $\alpha$ -Cr fraction, which almost reaches 0.331%wt because of the retained stress effect on promotion of  $\alpha$ -Cr formation due to direct aging after forging. Higher temperature (677°C) exposure promotes  $\alpha$ -Cr formation because of high diffusion process in  $\gamma$ -matrix of Alloy 718. Fig.13b shows that  $\alpha$ -Cr fraction at 677°C exposure for 2000hrs can reach 0.190%, 0.364%, 0.419% and 0.618% for

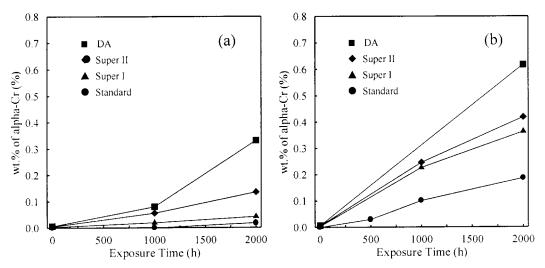


Fig.13: The dependence of  $\alpha$  -Cr on thermal exposure time for 4 different processed 718 disks

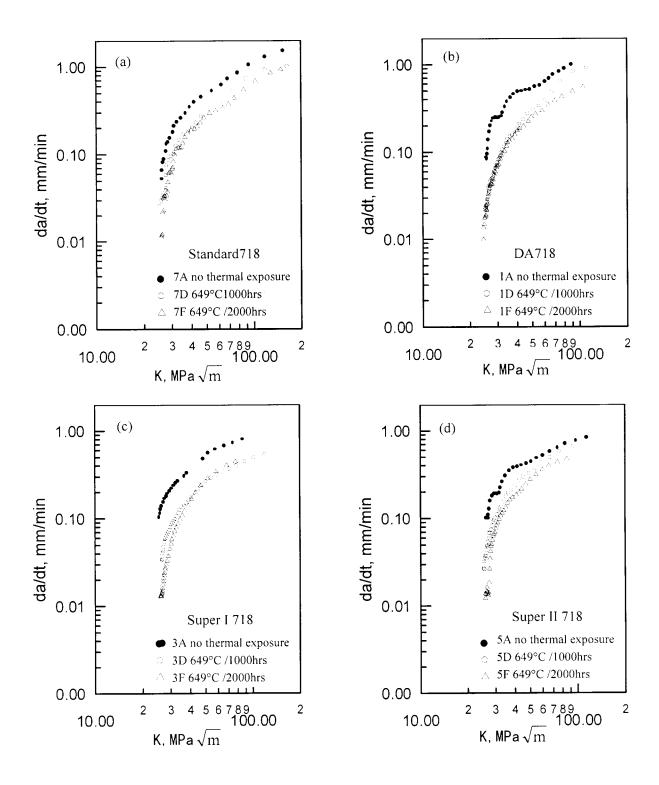


Figure 14: 650°C creep crack growth rate curves of 4 different processed 718 disks thermally exposed at 649°C(1200°F) till 2000hrs,

(a) Standard 718 (b) DA718 (c) Super I 718 (d) Super II 718

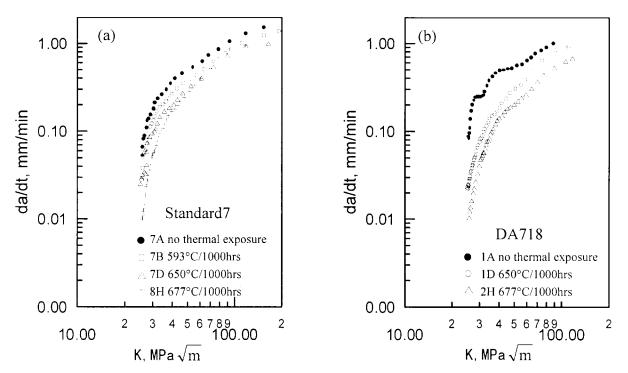


Fig.15: 650°C creep crack growth rate curves of Standard 718 (a) and DA718 (b) disks thermally exposed at different temperatures for 1000hrs

Standard 718, Super I 718, Super II 718 and DA718 respectively. These quantitative results of  $\alpha$  -Cr fraction are in accordance with micro-structural analysis.

### 650°C Creep Crack Propagation

Fig.14 shows 650°C creep crack growth rate curves of 4 different processed 718 disks thermally exposed at 649°C till 2000hrs. There are two main structure changes, which will effect the mechanical properties including creep crack propagation rate. Firstly, the coagulation of strengthening phases ( $\chi'' + \chi'$ ), which is a softening process and may reduce creep crack propagation rate. Secondly,  $\alpha$ -Cr formation which may cause embrittling effect to increase creep crack propagation rate. However, all creep crack growth rate curves of 4 different processed 718 disks decrease to lower levels with the thermal exposure times till 2000hrs at 677°C. It seems to us a small amount of  $\alpha$ -Cr (less than 0.6%wt) will be not harmful for 650°C creep crack propagation resistance. Fig.15 shows the creep crack growth rate curves of Standard 718 and DA 718 disks thermally exposed at 593°C,649°C and 677°C for 1000hrs. All creep crack growth rate curves drop to lower levels because of the intensive softening effect at higher temperatures. These experimental results indicate us that a small amount (less than 0.6%wt) of  $\alpha$ -Cr formation in 4 different processed 718 disks will not be harmful for 650°C creep crack propagation behavior, which is critical for disk application in aircraft and land-base gas turbines.

#### **Conclusions**

Structure stability study on Alloy 718 with 4 different forging/heat treatment process combinations, i.e., Standard 718, Super I 718, Super II 718 and DA718, has been conducted

with long time thermal exposure at 593°C, 649°C and 677°C till 2000hrs.

- 1. Alpha chromium ( $\alpha$ -Cr), a b.c.c. chromium enriched phase has been identified by XRD, SEM, TEM and SAD analyses. The existence of  $\alpha$ -Cr in Alloy 718 is also confirmed by Thermo-Calc method.
- 2. Alpha chromium (  $\alpha$  -Cr) precipitates are often in small globular particles ( $\sim$ 0.3  $\mu$  m) and form at the vicinity of  $\delta$  -Ni<sub>3</sub>Nb phase.
- 3. More  $\alpha$ -Cr particles will precipitate in Alloy 718 at higher exposure temperatures and longer times.
- 4. DA718 contains  $\alpha$ -Cr phase even without high temperature exposure. The fraction of  $\alpha$ -Cr grows as the exposure temperature and time increase.DA718 contains highest fraction of  $\alpha$ -Cr (0.62%wt) and Standard 718 contains lowest fraction of  $\alpha$ -Cr (0.19%wt) after 677°C, 2000hrs exposure among the 4 processed 718 disks, because retained stress promotes  $\alpha$ -Cr precipitation.
- 5. The fraction of  $\alpha$ -Cr in 4 different processed Alloy 718 disks after high temperature long time exposure are not very high, only less than 1%wt. The small amount of  $\alpha$ -Cr in Alloy 718 does not has serious deleterious effect on 650°C creep crack propagation rate. However, the impact energy does decrease with thermal exposure time at 649°C and 677°C.
- 6. A noticeable  $650^{\circ}$ C strengths drop, especially stress rupture time decrease was found after  $649^{\circ}$ C exposure for 2000hrs. A dramatic decrease of more than  $80 \sim 90\%$  in rupture times of 4 different processed 718 disks was found after  $677^{\circ}$ C exposure for 2000hrs.

### References

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