A Microstructural Study of Alloy 718 Plus<sup>TM</sup>

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#### Abstract

A microstructural study using metallographic, x-ray diffraction, and Edax analyses techniques was carried out on alloy 718 Plus samples which were in various thermal conditions. Results of this study showed that the strengthening phase is gamma prime which grows larger with increasing time of exposure. A delta phase nucleates and grows as the gamma prime phase becomes larger. No gamma double prime is found in the standard heat treatment and no  $\alpha$ Cr, sigma or Cr carbides are found in samples exposed for 2500 hours at 732°C (1350°F) in Alloy 718 plus while  $\alpha$ Cr forms in as little as 1000 hours at 704°C in Alloy 718.

#### Introduction

Alloy 718 has been the work horse in the gas turbine field for the past 50 years. As disk temperatures keep rising, alloy 718 which is currently the alloy becomes temperature limited to  $650^{\circ}$ C as its strength drops due to the transitional changes in the gamma double prime and gamma prime to delta and  $\alpha$ Cr phases.

A new alloy has been developed by Allvac called alloy 718 Plus<sup>TM</sup> which is claimed to have a 55°C (100°F) higher temperature capability than alloy 718 (1). The major changes in its composition from alloy 718 are the substitution of 10% Co for 10% Fe and a three fold increase in the Al content. The resultant strengthening phase is reported to be gamma prime ( $\gamma$ ') rather than double gamme prime ( $\gamma$ '') (1)

Because the Cr content remains at 18% and the continual formation of  $\gamma'$  depletes Ni from the matrix, there exists the possibility of the formation of Cr rich phases such as  $\alpha$ Cr, sigma or Cr carbides and a loss of stability with long exposures. The objectives of this study are two-fold: (1) describe an optimum sample preparation for alloy 718 Plus, and (2) determine the phase behavior of alloy 718 Plus as it relates to the phase behavior in alloy 718.

## Samples Studied

A group of alloy 718 Plus<sup>TM</sup> samples were received from Allvac with thermal treatments ranging from 982°C/4 hours to standard heat treatment and exposures of 2500 hours at 732°C (1350°F). The samples selected for study and their thermal histories are given in Table 1.

	Table 1.
Sample	Thermal History
1.	982°C/4 hours
2. As heat treated	954°C/1 hr/+788°C/2 hr, FC to
	650°C + 650°C/8 hr.*
3. As heat treated plus	$760^{\circ}$ C/500 hr + Creep tested
	@ 706°C/70 KSI/256 hr
4. As heat treated plus	705°C/1000 hr.
5. As heat treated plus	732°C/2500 hr.

\*Standard Alloy 718 Plus heat treatment.

# Metallography of Alloy 718 Plus<sup>TM</sup>

# A. Electropolishing

The choice of either a mechanical or electrolytic polish for Alloy 718 Plus<sup>TM</sup> depends on the size and type of phases present. It is difficult to remove all of the flow layer present on a mechanically polished surface and still reveal fine  $\gamma'$  precipitation using a HCl containing etchant solution. This type of problem is overcome by electropolishing since no flow layer is formed by electropolishing.

All the samples were ground through 6 micron diamond paste and then electropolished for 10 seconds in a 20% H<sub>2</sub>SO<sub>4</sub>-methanol solution at 25 volts. During polishing, the samples were agitated to remove the surface residue.

The structures produced by electropolishing are first studied optically since there is a selective attack on various phases. Carbides, borides and oxides will be in relief while  $\gamma'$ ,  $\gamma''$ , and delta phases will be polished flat in the plane of the matrix. Although electropolished samples can be studied on the SEM, the structures in the samples need to be etched for greater contrast.

# B. Etching of Alloy 718 Plus<sup>TM</sup>

To reveal the  $\gamma''$ ,  $\gamma'$ , and/or delta phases, the electropolished samples are electro-etched in a solution composed of 170 cc  $H_3PO_4+10cc$   $H_2SO_4+15$  grams of  $CrO_3$  at 5 volts for 8 seconds. In addition to putting the  $\gamma'$ ,  $\gamma''$  and delta phases in relief, the  $CrO_3$  etch will preferentially attack Cr rich phases like  $\alpha Cr$  and sigma phase. The delta phase and grain boundaries can be accentuated without the presence of  $\gamma'$  by electro-etching samples in a 15% HCl-methanol solution. The delta phase of each sample in this study was prepared in this manner as well by the  $CrO_3$  technique.

### **Structural Results**

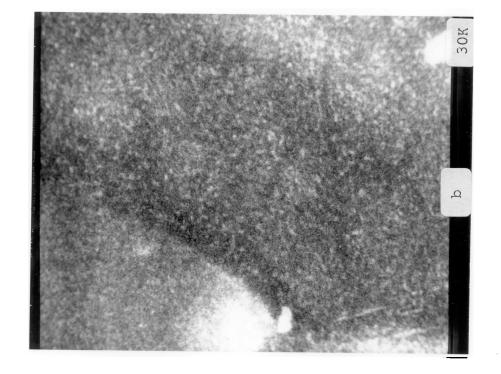
## y' Phase

The  $\gamma'$  solvus in alloy 718 Plus<sup>PM</sup> is 954°C as reported by CaO (1). The amount of Al+Ti in alloy 718 Plus<sup>PM</sup> should give a  $\gamma'$  solvus of 982°C. The decrease in  $\gamma'$  solvus temperature of alloy 718 Plus is probably due to the presence of the 10% Fe.

The size of the  $\gamma'$  in alloy 718 Plus<sup>PM</sup> which has been given a 760°C age is very small,

The size of the  $\gamma'$  in alloy 718 Plus<sup>PM</sup> which has been given a 760°C age is very small, probably less than 200°A, and a magnification of 30,000X is necessary to detect it. If the 718 Plus is aged at 760°C or lower for extended times, the  $\gamma'$  grows and is more easily seen. In order to follow the changes of the  $\gamma'$  in the different samples, all of the  $\gamma'$  pictures will be shown at 30,000X in this paper.

Figure 1a shows the  $\gamma'$  precipitation in the as heat treated condition (954\*C/1 hr + 788\*C/2 hr, FC to 650C and hold for 8 hrs at 650°C). If the samples is D.A. (direct aged) heat treated and exposed for 100 hours at 760°C the  $\gamma'$  also is very small. Figure 1b. If the alloy 718 Plus is heat treated below the 954°C  $\gamma'$  solvus temperature, a large  $\gamma'$  precipitate will form and a smaller  $\gamma'$  will precipitate during the 760°C age as seen in Figure 2a. When the alloy is exposed



DA + 760C/100hr

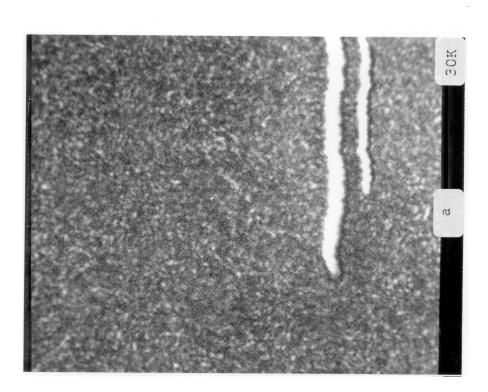
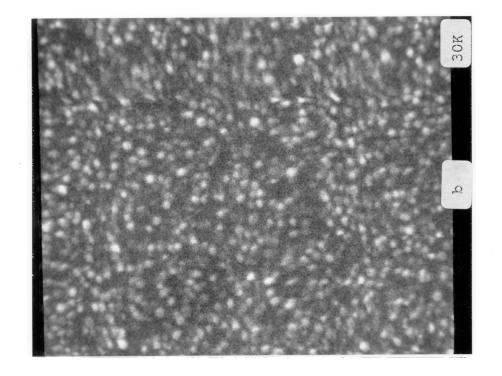


Figure 1

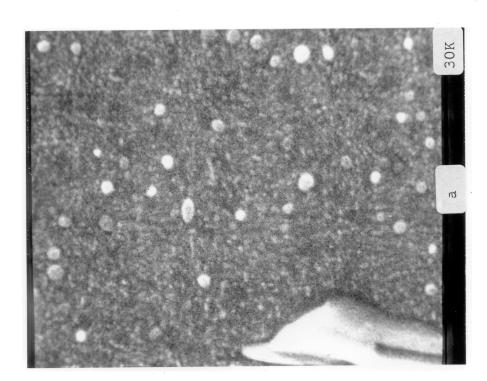
As heat treated



DA + 872C/1.5hr

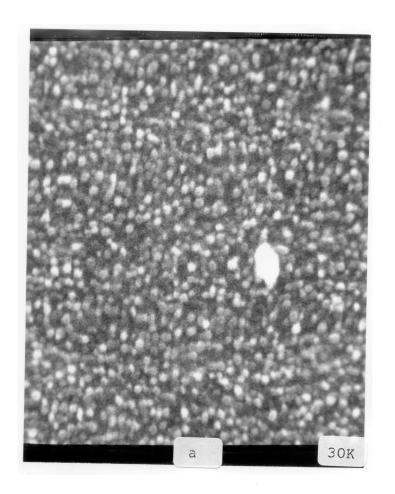


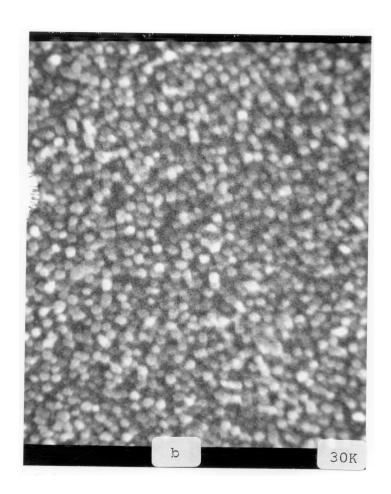




F1:

Subsolvus HT





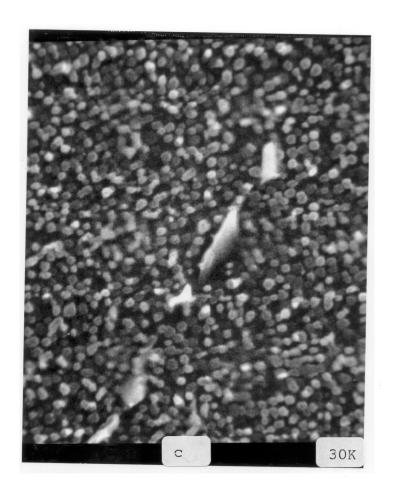
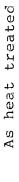


Figure 3

a-HT + 705C/1000 hr b-HT + 760C/350 hr c-HT + 760C/500hr + creep tested at706C/256hr





982C/4hr

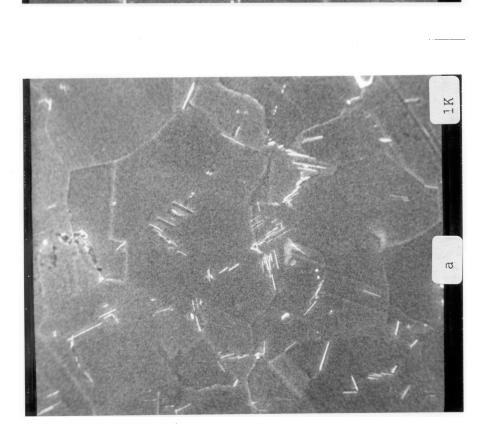
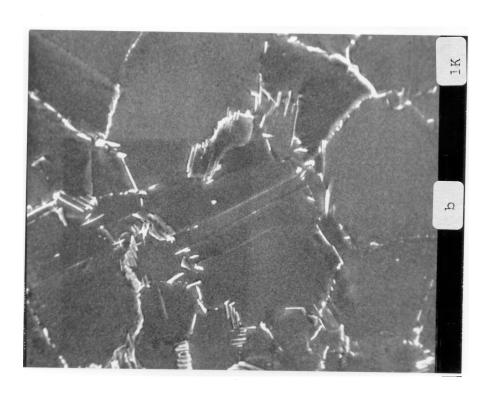
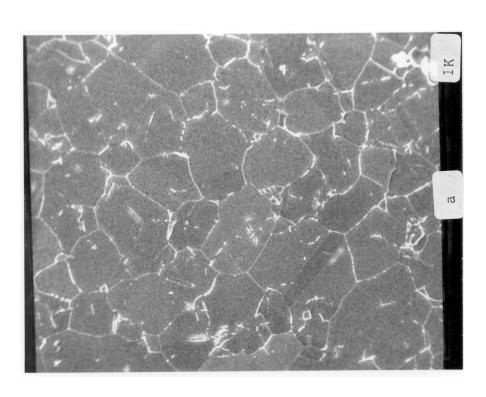


Figure 4



760C/500hr+ Creer tested@704C/256hr



704C/1000hr

at some intermediate temperature such as  $872^{\circ}$ C, an intermediate  $\gamma'$  size will form at the temperature as seen in Figure 2b. Figure 3 shows the resultant  $\gamma'$  in samples exposed for an extended time at  $705^{\circ}$ C and shorter times at  $760^{\circ}$ C. There is no evidence of  $\alpha$ Cr or sigma phases in the samples.

### B. Delta Phase

The high Nb contents in both alloys 718 and alloy 718 Plus forms a delta phase at the grain boundaries which is used to control grain size during processing into the desired shapes. It is well known that delta phase in the grain boundaries is beneficial for notch ductility.

To more readly follow the delta phase in the alloy 718 Plus, the samples were re-prepared using an electro-etch in 10% HCl-methanol which dissolves away the  $\gamma'$  phase and leaves the delta in relief. Thus, the delta phase can be readily examined at magnification of 1000X to 3000X.

Figure 4a shows the residual delta left from processing after a 4 hour solution at  $982^{\circ}$ C. Figure 4b shows the delta phase after the standard heat treatment. Because only a small amount of fine  $\gamma'$  forms during the standard heat treatment, only a small amount of delta phase forms.

Figure 5a shows more delta phase at the boundaries as the material is exposed for 1000 hours at 704°C. However, if the alloy 718 Plus is exposed at 760° for 500 hours and then creep tested at 704°C for 256 hours, a greater amount of delta has grown as seen in Figure 5b.

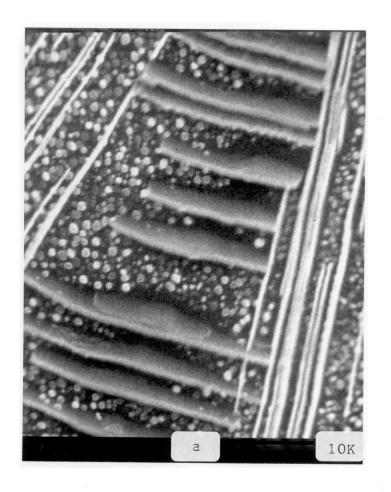
# Longtime Exposure at 732°C

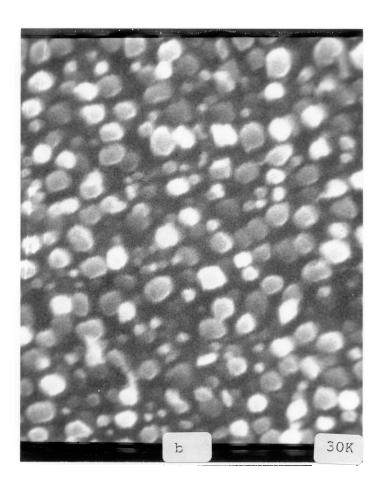
A sample of alloy 718 Plus was exposed at 732°C for 2500 hours to follow the growth of  $\gamma'$  and delta phases and to note the possible formation of any other secondary phases. Figure 6a shows that the  $\gamma'$  and delta phases have grown such that they can be seen at 10,000X. When the  $\gamma'$  is magnified 30,000X, it appears that the main  $\gamma'$  is about 2000Å and there are a number of  $\gamma'$  sizes as seen in Figure 6b. Figure 6c shows that the extracted  $\gamma'$  residue also contains various size  $\gamma'$ s.

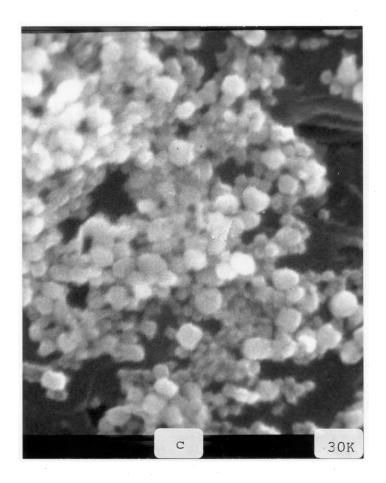
Figure 7a shows that a great amount of delta phase has formed after 2500 hours at 732°C. When the phases in the sample are extracted in ammonium sulphate and citric acid in water, both the delta phase left in the matrix and the extracted delta show  $\gamma'$  clinging to the delta plates, Figures 7b and 7c.

## X-ray Diffraction Results

A limited amount of phase extractions were carried out to confirm the nature of phases in Alloy 718 Plus. The  $\gamma'$  was extracted using a solution of 1 gram ammonium sulphate + 1 gram of citric acid in water while the inert phases were extracted in a 10% HCl-methanol solution. Delta phase was present in the inert phase extraction as well as a primary MC with a lattice parameter of 4.43°A. Gamma prime was found to be present in the as-heat treated condition as well as in the 2500 hour exposure at 705°C. No  $\gamma''$  was present in the as heat treated condition nor was any  $\alpha$ Cr found after long time exposure.







a-732C/2500hr-delta + Y' b-732C/2500hr-insitu Y' c- 732C/2500hr-extracted Y'

Figure 6





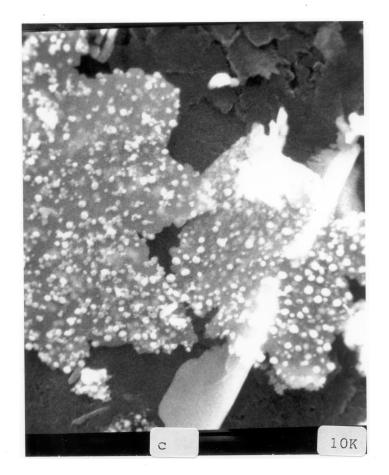
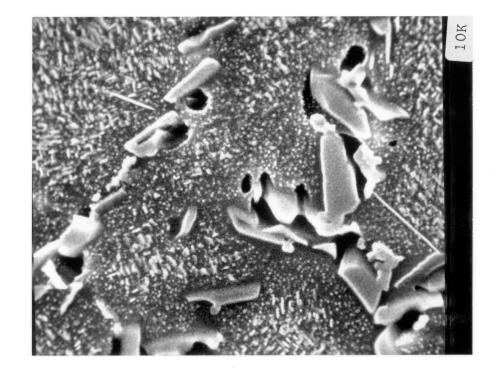


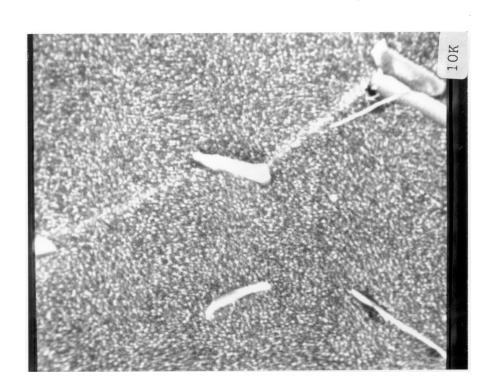
Figure 7

a-732C/2500hr-delta b-732C/2500hr-delta plates c-732C/2500hr-extracted delta



Alloy 718 704C/1000hr

Figure 8



Alloy 718Plus 704C/1000hr

## **EDAX Study**

Some of the residue used for x-ray analysis was placed on a carbon stub and probed in the SEM. The  $\gamma'$  and delta particles showed high Nb contents. A typical analysis would be 53.8 Ni, 4 Fe, 4.6 Co, 3.6 Cr, 8.1 Ti, 17.4 Nb and 8.9 Al. Similar compositions for  $\gamma''$  and delta were found in alloy 625 by Cozer (2). No evidence was found of particles containing high Cr contents to signify the presence of  $\alpha$ Cr or sigma phases.

### **Conclusions**

The metallographic study shows the  $\gamma'$  and delta phases to be the main phases in alloy 718 Plus. The  $\gamma'$  and delta phases continue to grow with increased time of exposure. The  $\gamma'$  size in the as heat treated condition is less than 200 Å while in longtime exposures at 732°C, the  $\gamma'$  size may be 2000 Å or larger.

There is no evidence of  $\alpha$ Cr formation by metallographic, X-ray analysis, or EDAX study in samples exposed for 2500 hours at 732°C.

While alloy 718 undergoes changes in the  $\gamma$ " and the formation of  $\alpha$ Cr when exposed at 704°C for 1000 hours, alloy 718 Plus continues to exhibit a stable microstructure as seen in Figure 8.

## Acknowledgements

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