SOLIDIFICATION OF ALLOY 718, ATI 718PLUS[®] AND WASPALOY

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Abstract

Alloy 718, ATI 718Plus[®] and Waspaloy have been investigated in terms of what their respective solidification process reveals. Differential thermal analysis was used to approach the task together with secondary electron and back scattered electron detectors equipped with an energy dispersive X-ray spectroscopy detector. These experimental methods were used to construct pseudo binary phase diagrams that could aid in explaining solidification as well as liquation mechanisms in processes such as welding and casting. Furthermore, it was seen that Waspaloy has the smallest solidification range, followed by Alloy 718, and finally ATI 718Plus[®] possessing the largest solidification interval in comparison.

Introduction

Alloy 718 (FeNi based superalloy), and Waspaloy (Ni based superalloy) are two widely used superalloys that have been around for about half a decade. ATI 718Plus[®] (Ni-based superalloy), on the other hand, is a fairly newly developed superalloy which possesses a slight service temperature improvement compared to that of Alloy 718 (704 °C vs 650 °C). This is primarily because ATI 718Plus[®] is a so called γ' hardening alloy compared to a γ'' hardening alloy which is the case of Alloy 718. Waspaloy is also a γ' hardening alloy and possess an even higher temperature capability than that of ATI 718Plus[®] (750 °C vs 704 °C) due to its higher content of γ' forming elements (i.e. Al and/or Ti). However, the higher volume fraction of γ' , the harder it will be to process the alloy. This trade off between alloy performance and process ability is therefore the basic concern in alloy development and was also one of the major drivers for the development of Alloy 718 back in the old days, that is, the industry demanded an alloy that was less sensitive to cracking during various processing steps. Even though both Alloy 718 as well as Waspaloy have been around for quite some years, there are still fundamental aspects related to their respective behaviour during solidification and melting that are still not fully understood. The aim of this paper was therefore to investigate their respective solidification processes which later can be used to comprehend various processing methods such as welding and casting in a better way.

Experimental

The experimental work carried out in this study is based on differential thermal analysis (DTA) and scanning electron microscopy together with an energy dispersive X-ray spectroscopy detector (SEM-EDX).

Materials

The materials used in the present investigation were wrought Waspaloy, Alloy 718, and ATI 718Plus[®] with the composition (in wt. %) as tabulated in table 1.

Table 1. The chemical composition of Waspaloy, Alloy 718, and ATI 718Plus[®], in wt. %.

Alloy	Ni	Fe	Cr	Co	Mo	Nb	Al	Ti	C	Mn	W	B	P	Si	S	Cu
Alloy 718	Bal.	17.92	18.41	0.17	3.05	5.00	0.60	0.94	0.03	0.11	-	0.002	0.010	0.08	0.0004	0.13
ATI 718Plus [®]	Bal.	9.6	18.0	8.9	2.6	5.47	1.52	0.74	0.02	0.12	1.0	0.005	0.010	0.08	0.0003	-
Waspaloy	Bal.	1.13	19.13	13.34	4.22	-	1.36	3.03	0.08	0.02	-	0.006	0.004	0.09	0.002	0.02

DTA

DTA experiments have been carried out on Alloy 718, ATI 718Plus[®] and Waspaloy. Each thermal cycle had a heating, soak and cooling part in which the samples have been heated up to the liquidus temperature, soaked at that temperature and then cooled down to a temperature below the solidus temperature. The starting temperature was set to 25 °C and 1400 °C as the maximum soak temperature since all of the present alloys are fully liquefied at this temperature. The dwell time was set to ~300 s which is in accordance with a previous study on Alloy 718 [1].

Before testing, all samples were weighed. A ceramic tube was cut by a high speed steel cutter to fit the sample and then after the sample was placed in an alumina crucible which was covered by a ceramic lid. Another crucible and thermocouple was used for the graphite reference. The whole sample assembly were covered by a ceramic shielding tube to ensure protection from contamination.

At testing, the power was switched on and start mode initiated. The recording unit was set to heating mode. Argon and water was tapped on before starting experiments. When the temperature reached the maximum limit, after ~300 s of dwell time, cooling mode was initiated whereas the sample cooled to room temperature. Argon inert gas protection and water cooling were shut off and stop mode initiated by end of experiment. The recorded data was collected and exported to an excel data sheet. Two different graphs were derived, one graph revealing the reference and furnace temperature and a second one showing their respective differential curves for analyzing the experiments.

The samples used in the present study have the following dimension and an example can be visualized in Figure 1 below:

Outer diameter of the sample: 7.5 mm

Length of the sample: 13 mm

Depth of the cylindrical hole in the centre: 8 mm

Diameter of the hole: 3.5mm

Figure 1. Example of a sample used at DTA testing.

SEM-EDX

A high-resolution Leo 1550 Gemini-FEG SEM equipped with detectors were used to obtain high resolution images whereas an X-MAX energy dispersive X-ray spectroscopy (EDS) detector was used for qualitative chemical analysis at 20kV.

Results

Thermal analysis is generally carried out to investigate the melting, solidification and phase reactions for different alloys. Cooling curves can be used to determine the liquidus, solidus temperatures and also the total time of solidification. The cooling curves are generally presented in a temperature and time plot as the ones presented below, Figures 2 to 4. A change in slope as registered as a deviation from the reference curve indicates a phase reaction.

DTA

The cooling (Temperature °C versus Time, s) together with its differential curves at 10 °C/min for Waspaloy, Alloy 718, and ATI 718Plus[®] can be seen in Figures 2-4, respectively.

Figure 2. Cooling and differential curves for Waspaloy at a cooling rate of 10 °C/min.

Figure 3. Cooling and differential curves for Alloy 718 at a cooling rate of 10 °C/min.

Figure 4. Cooling and differential curves for ATI 718Plus[®] at a cooling rate of 10 °C/min.

In Figure 5, a comparison in between the three alloys is made by plotting each respective differential curve (Thermal effects versus Temperature) to comprehend the solidification events in a better way.

Figure 5. Differential curves for Alloy 718, ATI 718Plus[®], and Waspaloy at a cooling rate of 10 °C/min.

SEM-EDX

The SEM images of relevant areas in Waspaloy samples together with its elemental analyses (summarized in terms of average, standard deviation, minimum, and maximum composition) are disclosed below. The SEM-EDS spot analyses of the Waspaloy matrix, the TiC, and the eutectic phases are based on 10, 10, and 6 different EDS spectrums, respectively. These spectrums and microstructural appearance are summarized in tables 2 through 4 as well as in figures 6a and 6b.

Table 2. Chemical composition (10 spectrums) in wt. % of the matrix phase in Waspaloy.

Element	Al	Ti	Cr	Fe	Co	Ni	Mo	Phase
Average	1.3	3.2	19.0	1.1	13.1	58.1	4.3	Matrix
Standard deviation	0.2	0.9	1.1	0.2	1.0	1.0	0.3	
Minimum	1.1	2.0	16.2	0.8	11.1	57.4	3.9	
Maximum	1.9	5.2	20.0	1.4	14.4	60.5	4.6	

Table 3. Chemical composition (10 spectrums) in wt. % of the TiC phase in Waspaloy.

Element	Al	Ti	Cr	Fe	Co	Ni	Mo	Phase
Average	0.5	56.4	6.5	0.5	2.8	10.4	26.7	TiC
Standard deviation	0.1	16.9	3.6	0.1	2.0	8.9	5.0	
Minimum	0.4	38.4	1.0	0.4	0.6	2.1	17.0	
Maximum	0.5	96.4	11.2	0.6	5.6	25.2	31.6	

Table 4. Chemical composition (6 spectrums) in wt. % of the eutectic phase in Waspaloy.

Element	Al	Si	Ti	Cr	Fe	Co	Ni	Zr	Mo	Phase
Average	2.5	0.3	8.7	10.0	0.5	8.5	67.6	0.9	2.5	Eutectic
Standard deviation	0.2	0.0	1.4	2.6	0.1	1.1	2.7	0.5	0.7	
Minimum	2.2	0.3	7.7	4.9	0.3	6.4	64.5	0.6	1.1	
Maximum	2.6	0.3	11.4	12.3	0.6	9.3	71.5	1.5	3.3

a. **b.**
Figure 6a and 6b. SEM-EDS analysis of Waspaloy matrix phase in Fig. 6a and TiC (i.e spectrum 9) and eutectic phases (i.e. spectrum 13) in Fig. 6b.

The SEM-EDS analyses of Alloy 718 are tabulated in tables 5 through 7 together with its microstructure as presented in Figure 7. The SEM-EDS analyses of the Alloy 718 matrix, the Laves, and the eutectic phase constituents are based on 11, 41, and 8 different EDS spectrum analyses, respectively.

Table 5. Chemical composition (11 spectrums) in wt. % of the matrix phase in Alloy 718.

Element	Al	Si	Ti	Cr	Fe	Ni	Nb	Mo	Phase
Average	0.4	0.1	0.8	18.9	17.9	53.9	5.3	3.0	Matrix
Standard Deviation	0.0	0.0	0.2	0.6	1.0	0.5	1.4	0.3	
Minimum	0.4	0.1	0.5	18.0	16.6	52.8	2.7	2.6	
Maximum	0.5	0.2	1.1	20.2	19.6	54.6	7.2	3.7	

Table 6. Chemical composition (41 spectrums) in wt. % of the Laves phase in Alloy 718.

Element	Al	Si	Ti	Cr	Fe	Ni	Nb	Mo	Phase
Average	0.2	0.7	1.3	9.1	8.9	46.7	28.9	5.8	Laves
Standard Deviation	0.0	0.2	0.8	4.8	4.1	12.4	1.6	2.3	
Minimum	0.1	0.2	0.3	2.6	3.4	33.2	23.9	1.0	
Maximum	0.2	0.9	3.1	15.3	13.5	63.4	31.2	9.0	

Table 7. Chemical composition (8 spectrums) in wt. % of the eutectic phase in Alloy 718.

Element	Ti	Cr	Fe	Ni	Nb	Mo	Phase
Average	1.6	7.5	7.3	61.0	21.7	1.9	Eutectic
Standard Deviation	0.3	2.2	1.6	1.5	3.3	0.4	
Minimum	1.2	5.4	5.8	59.2	17.5	1.5	
Maximum	2.0	10.3	9.4	62.7	25.5	2.3

Figure 7. SEM-EDS analysis of Alloy 718 matrix (i.e. spectrum 5), Laves (i.e. spectrum 3) and eutectic phases (spectrum 4).

The SEM-EDS analyses of ATI 718Plus[®] and its microstructure is presented in tables 8 through 10 as well as Figures 8a and 8b below. The SEM-EDS analyses of the matrix, the Laves, and the eutectic phase constituents are based on 12, 38, and 4 different EDS spectrum analyses, respectively.

Table 8. Chemical composition (12 spectrums) in wt. % of the matrix phase in ATI 718Plus[®].

Element	Al	Ti	Cr	Fe	Co	Ni	Nb	Mo	W	Phase
Average	1.0	0.5	18.0	9.5	8.9	52.3	6.4	2.5	1.2	Matrix
Standard Deviation	0.1	0.2	1.3	0.9	0.4	0.7	2.4	0.2	0.2	
Minimum	0.9	0.3	16.3	8.3	8.4	51.5	3.0	2.2	0.8	
Maximum	1.1	0.9	19.7	10.7	9.6	53.9	9.3	2.8	1.5	

Table 9. Chemical composition (38 spectrums) in wt. % of the Laves phase in ATI 718Plus[®].

Element	Al	Si	Ti	Cr	Fe	Co	Ni	Nb	Mo	Phase
Average	0.2	0.9	0.6	10.0	5.1	9.1	40.0	30.1	5.2	Laves
Standard Deviation	0.1	0.3	0.3	3.8	1.6	1.5	9.6	0.9	1.2	
Minimum	0.2	0.2	0.2	2.9	2.2	6.3	32.0	28.0	2.2	
Maximum	0.3	1.2	1.5	13.9	6.7	10.4	57.8	31.6	6.8	

Table 10. Chemical composition (4 spectrums) in wt. % of the eutectic phase in ATI 718Plus[®].

Element	Ti	Cr	Fe	Co	Ni	Nb	Mo	W	Phase
Average	0.7	8.7	4.4	6.7	56.9	20.2	1.5	0.7	Eutectic
Standard Deviation	0.3	2.0	1.1	0.5	1.7	2.4	0.2	0.1	
Minimum	0.5	5.9	2.8	6.2	55.3	17.5	1.3	0.5	
Maximum	1.2	10.4	5.3	7.3	59.3	23.2	1.7	0.8

a. **b.**
Figure 8a and 8b. SEM-EDS analysis of ATI 718Plus[®] matrix (spectrum 1), Laves phases (i.e. spectrum 3) in Fig. 8a and Laves (i.e. spectrum 4) and eutectic phases (spectrum 1) in Fig. 8b.

Discussion

The DTA and the SEM-EDS analyses reveal that Waspaloy has the narrowest solidification interval when compared to Alloy 718 and ATI 718Plus[®]. This observation is a consequence of the Nb content in Alloy 718 and ATI 718Plus[®] alloys. The partition coefficient of Nb is significantly lower than 1 and Nb is therefore highly enriched into the last solidifying melt. However as can be seen in Figure 5 for Waspaloy, there is a small deviation from the exothermic matrix base line curve at around 1280 °C which can be associated with the TiC carbide precipitation. The solidification interval for Waspaloy is therefore found to be approximately ~70 °C (T_L : 1350 °C - T_{EUT} : 1280 °C). By carefully investigating the samples as exposed to the DTA cycle it was revealed that both the matrix, TiC and eutectic phase constituents co-exists, Figure 6b, which further on supports the conclusion regarding eutectic matrix – TiC precipitation upon solidification. It can also be seen that small spherical precipitates exist. These precipitates may be γ' phase, however, no phase reaction was disclosed in the DTA thermograph (at lower temperature than what is shown in the thermograph) even though these precipitates were present to a very large extent. Pseudo binary phase diagrams are sometimes helpful in explaining main effects associated with melting and solidification. Based on the SEM-EDS and DTA measurements a pseudo binary phase diagram was constructed based on Ni and Ti, Figure 9. As the investigated alloys are constituted by a multi component system, it is very difficult to make very accurate estimates. However, based on the EDS it seems like the maximum solubility of Ti in Ni is approximately 5 wt. % where above the matrix phase is tied to the TiC phase through the eutectic component with a Ti content of approximately 9 wt. %. This information may be useful to explain liquation mechanisms which in turn may explain any ductility drop linked to hot cracking in i.e. welding [2].

Figure 9. Pseudo-binary phase diagram for Waspaloy.

As previously mentioned, Alloy 718 contains Nb which segregates heavily into the interdendritic regime in a melt upon solidification causing Laves phase to precipitate [3]. The solidification interval of Alloy 718 was found to be in the range of ~ 170 °C (T_L : 1320 °C - T_{EUT} : 1150 °C), more than 100 °C larger than for Waspaloy. In Figure 3 and 5 it can clearly be seen that an exothermic reaction takes place at ~ 1150 °C which is associated with the Laves phase. Most probably, NbC reaction takes place upon solidification. However, this reaction as well as the TiC reaction in Waspaloy, is slightly hidden within the matrix phase reaction. It is believed to the author's best judgment that the NbC reaction takes place at approximately the same temperature as for TiC in Waspaloy, which is at ~ 1280 °C. There is a small deviation at this temperature in the thermographs and it corresponds well with other investigations as well [4]. The maximum solubility of Nb in Ni within the matrix phase of Alloy 718 is around ~ 7 wt. % wherein the eutectic phase contain ~ 22 wt. % Nb.

Figure 10. Pseudo-binary phase diagram for Alloy 718.

ATI 718Plus[®] possesses the largest solidification interval as compared to that of Waspaloy and Alloy 718. It has a liquidus temperature of ~1340 °C and does as well as Alloy 718 reveal a final eutectic reaction at around 1150 °C which give a solidification interval of ~190 °C. The final eutectic reaction is once again as based on the SEM-EDS analyses believed to be associated with the matrix and Laves phase. The EDS spectrums reveal that the maximum solubility of the matrix phase is around 9 wt. % Nb as compared to 7 wt. % Nb in Alloy 718. The eutectic constituent in ATI 718Plus[®] contain ~ 20 wt. % Nb as compared to ~ 22 wt. % Nb in Alloy 718. As pointed out earlier, these phase diagrams may aid in explaining various liquation mechanisms during welding. Assume the alloys (ATI 718Plus[®] or Alloy 718) contain other phases that contain large amounts of Nb such as δ and NbC, and that they are exposed to rapid heating which is encountered during welding. Then, it may in fact be that these phases do not have enough time to dissolve through solid state diffusion before the temperature hits the eutectic temperature of the matrix – Laves phases. It means that dissolution process is now in a local equilibrium with the matrix – Laves – liquid phases and will consequently liquate. This is what happens to the NbC during welding and is known as constitutional liquation [5, 6]. Therefore, it is possible that this same type of liquation may occur also to the δ phase [7]. Since it seems like the matrix phase in ATI 718Plus[®] does have a slightly higher solubility for Nb in the matrix, it would be harder for this liquation to take place as compared to Alloy 718 which in turn may promote a better resistance towards hot cracking.

Figure 11. Pseudo-binary phase diagram for ATI 718Plus®.

Conclusions

A thorough investigation of the solidification process of Waspaloy, Alloy 718, and ATI 718Plus® have been carried out using DTA coupled with SEM-EDS analyses. It was concluded that:

- Pseudo binary phase diagrams have been developed for Waspaloy, Alloy 718, and ATI 718Plus®.
 - These diagrams may aid in explaining liquation mechanisms in i.e. welding.
- Waspaloy has a solidification interval of ~70 °C.
 - It solidifies through a final eutectic reaction between the matrix and TiC at ~1280 °C.
 - The matrix phase has a maximum solubility of around 5 wt. % Ti.
- Alloy 718 has a solidification interval of ~170 °C
 - It solidifies through a final eutectic reaction between the matrix and Laves at ~1150 °C.
 - The matrix phase has a maximum solubility maximum solubility of around 7 wt. % Nb.
- ATI 718Plus® has a solidification interval of ~190 °C.
 - It solidifies through a final eutectic reaction between the matrix and Laves at ~1150 °C.

- The matrix phase has a maximum solubility maximum solubility of around 9 wt. % Nb.

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