DIFFERENTIAL THERMAL ANALYSIS (DTA) STUDY OF

THE HOMOGENIZATION PROCESS IN ALLOY 718

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Abstract

The DTA thermograms of as-cast and homogenized Alloy 718 have been determined. In combination with the results of an interrupted melting experiment, it was found that the incipient melting temperature (solidus) of as-cast alloy was about $1165\,^{\circ}$ C and homogenization increased the incipient temperature of Alloy 718 to about $1230\,^{\circ}$ C for a fully homogenized alloy. An endothermic peak was found at about $1290\,^{\circ}$ C for homogenized alloys in contrast with a slight inflection in as-cast alloys at the same temperature. A special melting experiment below, at, and above the inflection temperature around $1290\,^{\circ}$ C showed that this peak was associated with the dissolution of NbC/ γ eutectic in cast alloy and carbide particles in homogenized alloys instead of the melting of bulk γ matrix. The monovariant NbC/ γ eutectic reaction occurred over the temperature range from $1260\,^{\circ}$ C to $1297\,^{\circ}$ C (as-cast) and carbide dissolution from $1267\,^{\circ}$ C to $1305\,^{\circ}$ C (homogenized).

Introduction

In recent years there have been several studies of the phase transformation behavior of superalloys using the DTA (differential thermal analysis) technique (1-11). Most of the work (1-9) concentrated on the behavior of Alloy 718, one of the most intensively utilized nickel-base superalloys. The objectives of these DTA studies, generally speaking, included identifying the characteristic temperature and nature of phase two aspects: transformations of the superalloys studied, and determining the effect of chemical composition and processing on phase transformation behavior in these alloys with the first aspect being the major concern in referenced studies, especially for Alloy 718. Because DTA is an indirect method of examining phase transformations, there is always a problem of explaining the observed DTA thermograms. Therefore, certain discrepancies concerning the characteristic temperatures and nature of phase transformations in Alloy 718 exist in the literature, although very similar curve profiles have been obtained by different authors. These discrepancies can be eliminated only by the combination of the DTA technique with other direct experimental methods. Furthermore, the alloys with different degrees of homogenization could have different profiles of DTA thermograms, and it is worth exploring the possibility of tracing the homogenization process by using the DTA technique. Little work has been done in this area.

In this paper, the liquid-solid phase transformations of Alloy 718 with different degrees of homogenization were studied using the DTA technique in combination with quantitative metallography, SEM (scanning electron microscopy) and EMP (electron microprobe) analyses with the aim of clarifying the cause of the discrepancies existing in the literature and exploring the effect of homogenization on DTA thermograms.

Experimental Procedure

<u>Material</u>. The material used in this study was a VIM (vacuum induction melted)/VAR (vacuum arc remelted) Alloy 718 having the following chemical composition:

C	Si	Fe	Cr	Mo	Nb	Ti	Al	Ni
0.029	0.08	19.18	17.86	2.93	5.31	0.90	0.46	Balance

All of the samples came from the central portion of a slice cut from the top of a 500 mm (20 inch) diameter ingot.

Heat Treatment. To create different degrees of homogenization, part of the as-cast samples were heat treated at 1190 °C for 1 hr, 10 hrs and 48 hrs. These four structures were designated as as-cast, 1 hr, 10 hr and 48 hr homogenized states. To further homogenize the alloy, a portion of the 48 hr homogenized alloy was forged at 1121 °C with a reduction ratio of 6:1. The forged alloy represents a fully homogenized one and was used to determine the incipient melting temperature of the homogenized alloy.

<u>DTA Experiments</u>. DTA was performed for all the four structures on a Dupont Model 2100 instrument. Specimens weighing 0.25 g were thermally cycled from 400°C to 1450°C under a flowing helium atmosphere at a heating rate of 10°C/minute. A 99.999% pure platinum was used as the reference material. Reaction temperatures were taken as the temperature at which the DTA curve deviated from the local baseline and were determined from three repeated measurements. To facilitate the interpretation of

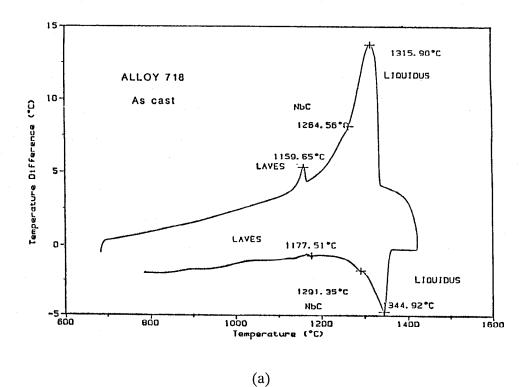
DTA thermograms, determine the incipient melting temperature, and trace the melting process, a special interrupted melting experiment was performed which involved heating samples to different temperatures, holding for 2 hrs, and then cooling down to ambient temperature.

Study of Phase Transformation at 1294°C on Heating. As shown in many studies (2-9), an event occurred at about 1290°-1300°C on DTA heating curves. The same phenomenon has been found in this study (Figures 1 and 2). The nature of this event is not clear yet. Some investigators regarded it as the beginning of melting of bulk γ matrix (solidus), but no detailed work has been conducted to justify this assumption. In this study, a series of as-cast and 48 hr homogenized + forged samples (5 x 20 x 20 mm) were heated to three temperatures (1270°C, 1290°C, and 1310°C) at a heating rate of 10°C/min. to simulate DTA heating. After reaching the specified temperature, the samples were water quenched to an ambient temperature. The resulting structures represent those of as-cast and 48 hr homogenized materials at corresponding temperatures in the DTA test. Metallography, SEM and EMP were performed on these samples to clarify the nature of phase transformation at about 1290°-1300°C.

Results and Discussion

<u>DTA Thermograms</u>. Figure 1 shows the typical DTA thermograms of as-cast and homogenized materials. The general profiles of these thermograms are in agreement with those reported in the literature. A comparison between thermograms of as-cast and of homogenized alloys revealed several differences in curve profile on heating:

- A. There is an endothermic peak within the temperature range of 1162°-1190°C in as-cast material, but no such peak was observed in the 48 hr homogenized alloy. Obviously this peak is associated with the dissolution of Laves phase as described in many studies. In less homogenized alloys, e.g. 1 hr, or especially 10 hr homogenized, it is not always possible to observe this peak on the thermograms although metallographic examination revealed the existence of Laves phase in small amounts. This may be explained by two factors: one is the low sensitivity of the DTA method in detecting small amounts of Laves phase, and the other is the sampling effect, i.e. a variation in DTA thermograms could be observed when the sample was small and the bulk material was severely non-uniform. Therefore, to trace the homogenization process, a larger sample and several runs are necessary in the DTA test.
- В. Immediately after the dissolution of Laves phase, there is a continuous endothermic process occurring in the as-cast alloy, but the DTA curve deviated from the baseline at much higher temperatures in homogenized alloys. This deviation from the baseline, i.e. the endothermic process, was explained to be the Nb carbides beginning to solution, i.e. carbide liquation (4, 6-9). However, as shown below, the nature and volume fraction of the carbides in as-cast alloy by EMP and quantitative metallography show no noticeable differences up to 1230°C (NbC carbide with a volume fraction of about 1.5%) and up to 1260°C in homogenized alloys, except the carbide size in homogenized alloys is slightly larger. An accelerated dissolution of carbide particles was found in as-cast alloys only at temperatures higher than 1230°C. Therefore, it is very unlikely that the different behavior of as-cast and homogenized alloys totally came from the different dissolution behavior of Nb carbides. Moreover, no experimental evidence has been given to show an unquestionable correlation between the initial deviation from the baseline and the dissolution of Nb carbides. All these problems will be clarified in the incipient melting experiment (see below).



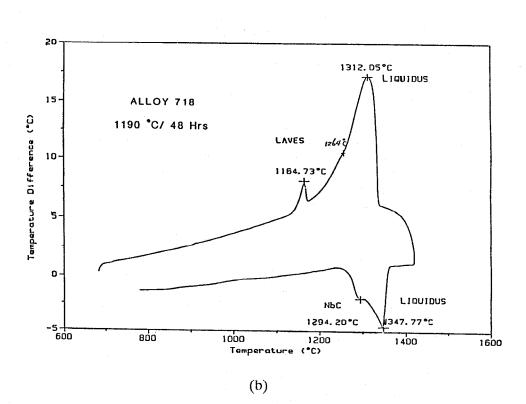


Figure 1 - Typical DTA Thermograms of Alloy 718 (a) As-Cast and (b) 48 Hr Homogenized Materials.

An event occurred at about 1294°C in both the as-cast and homogenized C. alloys but with different DTA profiles. Figure 2 illustrates the details of DTA curves in the as-cast and homogenized alloys. An inflection occurred at about 1292°C in the as-cast alloys, but it gradually transformed to a more distinct endothermic peak at almost the same temperature in those alloys with the longer homogenization times. Such an inflection has been observed by many investigators (2-9). Some authors (4, 6-9) regarded it as the beginning of the bulk y matrix melting, and the temperature at which the inflection occurred was taken to be the solidus of Alloy 718. However, the observation in this study sheds doubt on this assumption because the DTA curves showed a distinct endothermic peak at 1294°C, particularly in homogenized alloys, and this type of profile cannot be explained by the melting of γ matrix. The nature of this reaction cannot be clarified solely by DTA thermograms and will be explored by the above stated interrupted melting experiment (see below). The DTA thermograms on cooling are the same for both as-cast and homogenized alloys; there are two events at about 1264°C and 1162°C, corresponding to carbide and Laves phase formation, respectively, as proven in numerous studies (2-9).

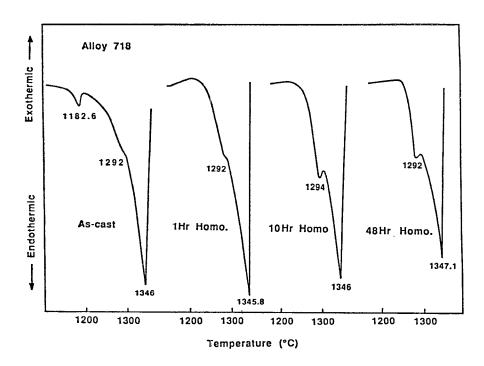


Figure 2 - Comparison of DTA Thermograms on Heating of Alloys with Different Degrees of Homogenization. Note the Feature of Endothermic Reaction at 1294 °C in Homogenized Materials.

Incipient Melting in As-Cast and Homogenized Alloys. The critical (i.e. incipient melting) temperatures of as-cast and homogenized alloys were determined to be 1165°C and 1230°C from the interrupted melting experiment. As shown in Figure 3, incipient melting started at existing Laves phase particles for the as-cast alloy and at grain boundaries and around carbide particles for the homogenized alloy. The melting phenomenon in the ascast alloy is obviously associated with the dissolution of Laves phase and the increasing

amount of melting of γ matrix and carbides at temperatures higher than 1230 °C explains the endothermic process after the Laves peak observed on the DTA thermogram.

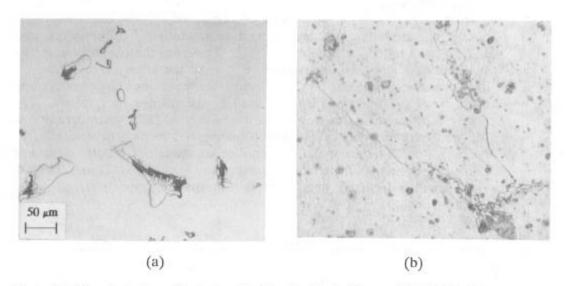


Figure 3 - Morphologies of Incipient Melting in (a) As-Cast and (b) 48 Hr Homogenized + Forged Alloy 718.

Therefore, the incipient melting temperature measured in the as-cast alloy represents the Laves/γ eutectic temperature. The melting phenomenon in fully homogenized material is most likely associated with the melting of γ matrix instead of Nb carbide liquation. This is because, as proved by quantitative metallography, the area fraction of melted regions quickly increased to a level much higher than what can be formed from carbides by increasing the temperature above 1230 °C, and no significant dissolution of carbide particles has been observed below the temperature of 1260 °C even though there is much local melting around carbide particles. Therefore, the measured incipient melting temperature of the fully homogenized alloy more nearly represents the equilibrium solidus of Alloy 718.

The characteristic temperatures of liquid-solid transformation of Alloy 718 have been determined by a number of investigators. The available data are listed in Table 1, together with those in this study. It can be seen that there is a remarkable discrepancy in reported solidus temperatures. This results from the different methods used to define the solidus; all the work which defined the inflection temperature as the solidus (4, 6-9) gave a quite high value from 1280° - 1300° C, and the works which defined the solidus according to hot bend testing (12), melting experiments (6), or the deviation from the base line of DTA thermographs at the lowest temperature (1,5) reported much lower values. The different definitions, therefore, led to different pseudo-binary representation, (compare Figure 9 in Ref 8 and Figure 4 in Ref 6). It seems that the former definition ignored the liquation events that occurred at the lower temperatures and the latter took the temperatures at which alloy liquation occurred as the solidus. As shown in this study, the initial liquation involved mainly the melting of γ matrix and, to a lesser extent, carbide liquation, and the latter definition is much closer to the true solidus.

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Table I - Characteristic Temperatures of Phase Transformations in Alloy 718*

Authors	Eiselstein	Knorovsky et. al.	Cieslak et.al.	Fayman	Bouse	Ballantyne & Mitchell	Poole	This Study
Ref.	(12)	(8)	(7,9)	(4)	(6)	(1)	(5)	
Liquidus	1260	(1364)	(1351)	1316 (1345)	1330** (1355)	1340	(1341)	1315 (13 45)
Solidus	1227	(1320)	(1297)	(1281)	1240** (1280)	1245	(1243)	1230** (1235)
γ/NbC Eutectic		1250	1257 (1230)	1225-1245 (1225-1290)		1270	(1271-1321)	1262 (1267-1305)
γ/Laves Eutectic	1177	1198	1185	1120-1155	1165	1140-1173	(1149-1177)	1165** 1162(1167)

^{*}The values in brackets are those obtained from DTA thermographs on heating, others on cooling.

^{**}Measured by quantitative metallography on the specimens quenched from different temperatures.

The solidus given in this study is even lower than those obtained by similar methods (3,6). The reason for this may be that the alloy used in this study has a slightly higher Nb level than other studies, and the holding time (2 hrs) in this study is much longer than others (15 min.), i.e. the value reported in this study more nearly approaches equilibrium. The solidus determined from the first deviation from the baseline on heating in the homogenized alloy is quite close to the true solidus (Table 1).

Nature of the Reaction at 1294 °C on Heating. An interrupted melting experiment was performed to assist in clarifying the nature of transformation at 1294°C on heating. The microstructures that resulted from heating at 1270 °C, 1290 °C and 1310 °C are illustrated in Figure 4, together with those of the initial as-cast and 48 hr homogenized, wrought alloys. These microstructures represent those just below, at, and above inflection temperature. The initial as-cast structure contains blocky Laves phase (or Laves/y divorced eutectic-like constituents) and script-like carbides (or MC/γ divorced eutectic-like constituents) (Figure 4a). Upon heating to 1270°C, the existing Laves phase started to melt and some newly liquated regions formed. The new liquated region which transformed into a mixture of γ and Laves/ γ eutectic having very fine structure upon quenching from heating temperature was surrounded by a transitional region which can be identified from etching effects (Figure 4b). Upon raising the temperature to 1290 °C, melted regions continuously extended, the amount of existing MC/y divorced eutectic was reduced, and more importantly, a new, typical eutectic-like constituent formed. Figure 4c illustrates a typical example. There is an old MC/ γ divorced eutectic-like constituent on the left side of the photo and a typical eutectic-like constituent, which was not observed in the initial as-cast structure, on its right side. The new eutectic-like constituent can be seen more clearly in the microstructure resulting from heating at 1310°C (Figure 4d). A similar situation occurred with the homogenized alloys. The initial structure of the 48 hr homogenized alloy was y matrix plus certain coarse equiaxed carbide particles (Figure 4e). Raising the temperature to 1270 °C resulted in many locally melted regions which have a morphology similar to that of newly formed melted regions in the as-cast alloys at 1270°C (Figure 4f). A significant change in the morphology of the melted region occurred when the temperature increased to 1290 °C. Some liquated regions changed into y plus carbide-like particles with surrounding transitional areas, as shown in Figure 4g. These regions still can be seen in the microstructure of the homogenized alloys at 1310°C (Figure 4h). The details of the newly formed constituents in the as-cast and homogenized alloys are illustrated in Figure 5 (a) and (b). Electron microprobe analysis was performed on these constituents to identify their nature, and the results are shown in Figure 6 for the homogenized alloys. The liquated regions formed at 1270°C contained y and fine Laves/y eutectic with quite high levels of Nb and were enveloped by the transitional regions with a Nb content less than that of γ matrix (Figure 6a). The liquated regions may not always be associated with carbide particles. Upon increasing the temperature to 1290 °C and 1310 °C, the area fraction of liquated regions increased and some of them transformed to Nb carbides or Laves phase and y-phase with Nb levels higher than that of y matrix. Figure 6b illustrated an example of this transformation; the liquated region on the left side of the picture might have partly changed into Laves and γ -phase. Figure 6c showed an example of the transformation into carbide plus γ phase, i.e. NbC/y eutectics.

In summary, the experimental observation of this study proved two points: 1) the only reactions observed in the tested temperature range ($1270 \,^{\circ}\text{C}$ - $1310 \,^{\circ}\text{C}$) are the dissolution of initial NbC/ γ eutectic or eutectic carbides and formation of new NbC/ γ eutectics, and 2) the increase in area fraction of liquated regions with increasing temperature causes

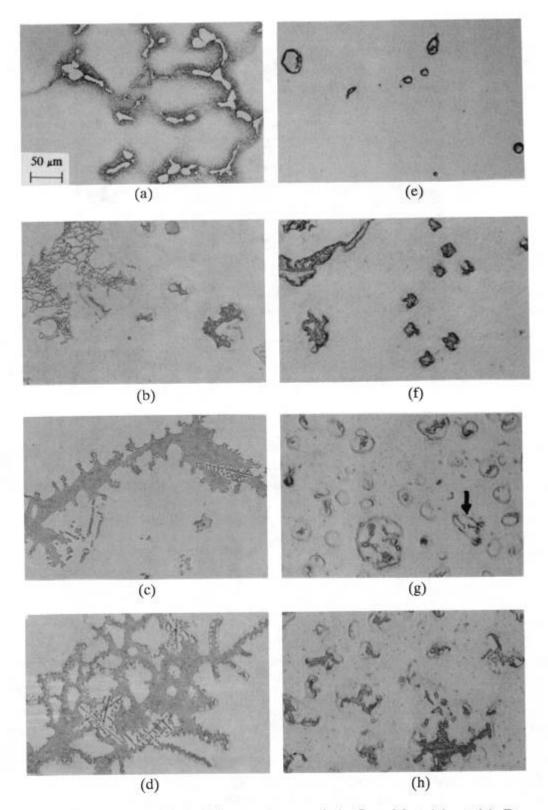
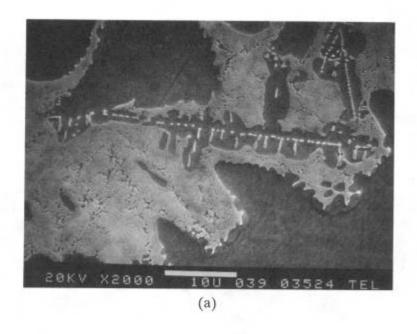


Figure 4 - Comparison of the Microstructures of As-Cast Material at (a) Room Temperature, (b) 1270°C, (c) 1290°C, (d) 1310°C, and 48 Hr Homogenized, Wrought Material at (e) Room Temperature, (f) 1270°C, (g) 1290°C, and (h) 1310°C.



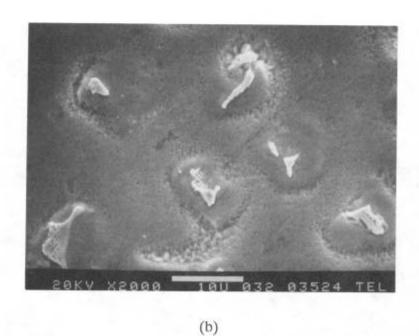


Figure 5 - SEM Photographs of the Eutectics Formed by Eutectic Reaction (at 1290°C) in (a) As-Cast and (b) 48 Hr Homogenized, Wrought Materials.

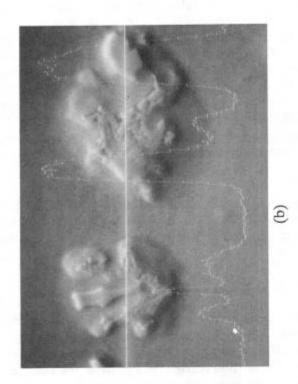
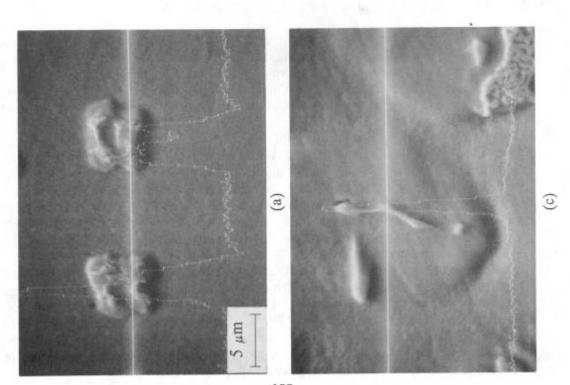


Figure 6 - Nb Distributions Across (a) Locally Liquated Region at 1270°C, (b) Partially Transformed Liquated Region at 1290°C, (c) NbC/ γ Eutectic Formed on Quenching 1310°C in 48 Hr Homogenized, Wrought Material.



no abrupt change in the DTA thermogram within this temperature range. The above facts imply that the inflection or peak profile at about 1290 °C is associated with carbide dissolution reaction instead of the melting of γ matrix. The initial carbides dissolved on heating, inducing the inflection or peak profile in DTA thermograms, and the observed new NbC/ γ eutectic most likely formed during water quenching if the liquid temperature was higher than the eutectic temperature. From this, the NbC/ γ eutectic temperature should be higher than 1270 °F because there is almost no newly formed eutectic in 1270 °C structures. It can be seen from the DTA thermograms that the curves deviated from the baseline at about 1260 °C (as-cast) or 1267 °C (homogenized) and resumed the path of the baseline at 1297 °C (as-cast) or 1305 °C (homogenized) with an endothermic peak at about 1290 °C, implying that the dissolution of eutectic carbides occurred within the temperature range from 1260 °C to 1287 °C for as-cast and from 1267 °C to 1305 °C for homogenized materials, which is in agreement with the experimental observation in Figures 5 and 6 and implies that NbC/ γ eutectic formation is a monovariant reaction occurred over a range of temperatures.

The endothermic peak was much clearer in the homogenized alloys, and probably this phenomenon is associated with the accelerated dissolution of carbide particles in cast alloys at a rate higher than that in homogenized alloys which will lead to a less endothermic peak at about 1290 °C.

Summary and Conclusions

The Differential Thermal Analysis (DTA) thermograms of Alloy 718 in the as-cast and homogenized states have been determined. A special melting experiment has been performed to accurately determine the solidus of the as-cast and homogenized alloys. Optical metallography, SEM and Electron Microprobe (EMP) studies have been carried out on the specimens with microstructures corresponding to those below, at, and above 1290 °C to clarify the nature of phase transformation occurring at 1294 °C during the DTA test on heating. The following results have been obtained:

- 1) The Laves eutectic peak existing on the DTA thermograms of as-cast alloys was eliminated by increasing homogenization time at 1190°C, and the homogenization also pushed the deviation of the curve from the local baseline to a much higher temperature. A distinct endothermic peak profile occurred at 1294°C in the thermograms of homogenized alloys, but only an inflection was found in the as-cast alloys.
- 2) The incipient melting temperature of the as-cast alloys was about 1165°C, as determined by the melting experiment. Immediately following the initial melting, the liquation of the existing Laves eutectic and γ matrix rapidly increased with increasing temperature. This was responsible for the endothermic process after the Laves peak on the DTA thermogram. The incipient melting temperature of a fully homogenized alloy was about 1230°C which is the solidus temperature of 718 and roughly corresponded to the temperature of the first deviation of the DTA curve from the local baseline on heating.
- The inflection or peak profile on the DTA thermograms at about 1294°C in the ascast and homogenized alloys came from the dissolution of the eutectic carbides instead of the melting of γ matrix. The NbC/γ eutectic or carbide dissolution reaction occurred from 1260°C to 1297°C for the as-cast and from 1267°C to 1305°C for the homogenized, wrought alloys. The sharper peak profile at 1294°C in homogenized alloys probably is due to the different dissolution behaviors of carbide particles in cast and homogenized alloys.

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References

- 1. A. S. Ballantyne and A. Mitchell, "The Prediction of Ingot Structure in VAR/ESR 718", in <u>Sixth International Vacuum Metallurgy Conference Proceedings</u>, ed. G. K. Bhat and R. Schattler, (San Diego, California, 1979), pp. 599-627.
- 2. G. K. Bouse and P. W. Schilke, "Process Optimization of Cast Alloy 718 for Water Cooled Gas Turbine Applications", in <u>Superalloy 1980</u>, <u>Proceedings of the Fourth International Symposium on Superalloys</u>, (ASM, Ohio, 1980), pp. 303-310.
- 3. B. Prinz and G. Rudolph, "Microprobe Measurements to Determine the Melt Equilibria of High-Alloy Nickel Materials", <u>Mikrochimica Acta</u>, (Wien), Suppl. 11 (1985), pp. 275-287.
- 4. Yvonne C. Fayman, "Microstructural Characterization and Elemental Partitioning in a Direct-Aged Superalloy (DA718)", <u>Materials Science & Engineering</u>, 92 (1987), pp. 159-171.
- 5. J. M. Poole, "Homogenization of VIM-VAR Inconel Alloy 718", in <u>Conference Proceedings of the Special Melting and Processing Technologies</u>, (San Diego, California, Moyes Publishers, 1988), pp. 508-539.
- 6. G. K. Bouse, "Application of a Modified Phase Diagram to the Production of Cast Alloy 718 Components", in <u>Superalloy 718, Metallurgy and Applications</u>, ed. E. A. Loria, (TMS, 1989), pp. 69-77.
- 7. M. J. Cieslak, G. A. Knorovsky, T. J. Headley and A. D. Romig, Jr., "The Solidification Metallurgy of Alloy 718 and Other Nb-Containing Superalloys", Ibid pp. 59-68.
- 8. G. A. Knorovsky, M. J. Cieslak, T. J. Headley, A. D. Romig, Jr. and W. F. Hammetter, "Inconel 718. A Solidification Diagram", <u>Metallurgical Transactions</u>, 20A (1989), pp. 2149-2158.
- 9. M. J. Cieslak, T. J. Headley, G. A. Knorovsky, A. D. Romig, Jr. and T. Kollie, "A Comparison of the Solidification Behavior of INCOLOY 909 and INCONEL 718", Ibid, 21A (1990), pp. 479-488.
- 10. C. J. Burton, "Differential Thermal Analysis and the Mechanisms of Minor Additions in Superalloys", in <u>Superalloys Metallurgy and Manufacture</u>, ed. R. H. Kear, D. R. Muzyka, J. K. Tien and S. T. Wlodek, (Claitor's Publishing, 1976), pp. 147-158.
- 11. C. L. Jeanfils, "Solidification Behavior of Some Nickel- and Cobalt-Base Alloys", in <u>Proceedings of Vacuum Metallurgy Conference 1984</u>, ed. G. K. Bhat and L. W. Lherbien, (ISI, 1984), pp. 93-98.

References (Con't)

- 12. H. L. Eiselstein, in Advances in the Technology of Stainless Steels, (ASTM STP 369, 1965), pp. 62-79.
- 13. B. Radhakrishnan and R. G. Thompson, "Solidification of the Ni-Base Superalloy 718. A Phase Diagram Approach", <u>Metallurgical Transactions</u>, 20A (1989), pp. 2866-2868.