THE PRESENT STATUS OF MELTING

TECHNOLOGY FOR ALLOY 718

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Abstract

Alloy 718 has been made by air and vacuum melting for many years. In this presentation, the present status will be described for Alloy 718 melting with particular reference to present quality problems. It is clear that the present processes are all at a mature stage of development, with the exceptions of electron-beam and VADER melting, and that very significant problems still exist with quality aspects in respect to both structure and composition. The principles governing these problems are, however, well-understood and we can anticipate progress using the new processes over the next development period.

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INTRODUCTION

Alloy 718 has presented severe difficulties in melting behaviour since its inception. The problems encountered have been ones of both principle and practice compounded by the ever-increasing demands of the user community in application and in inspection. The alloy has a wide melting range with strong density differences developing between liquid and solid during solidification, which together constitute a good recipe for segregation problems in the solid irrespective of the melting and casting technique. Additionally, the user requirements for extreme limits of properties impose severe conditions on the chemical control of the alloy in major, minor, and trace elements. The combination of these difficulties with the over-riding need for an economically-viable product has been the environment in which the industry has created the present melting and casting technology for Alloy 718. The procedures vary very widely, reflecting the wide range of applications for this alloy. It is probably correct to state that the alloy is not now air-melted for any substantial market and that all liquid processing is carried out under atmospheric protection or vacuum, with the special exception of some ESR To a large extent, the nature of the melting and casting process used is dictated by chemical specifications, such as the necessary precision of analysis, the trace element contents (particularly in respect to sulphur) and the required freedom from non-metallic inclusions. following review addresses these problems separately as far as is possible, but it is clear that they interact in a very complex and variable pattern.

Chemical Aspects of Alloy 718 Melting Composition Control.

The chemical compositions developed for the various versions of the alloy largely centre around the niobium concentration. The highest strength versions of the alloy require the maximum Nb content possible without creating congruent melting Laves phase. The formation of such a structure cannot be corrected by homogenization in any realistic time/temperature framework. The composition of the Alloy 718 Laves phase is not constant and can be influenced by minor elements such as silicon. However, it is clear from structural examinations that particularly with high Nb alloys (Nb>5.3 wt%) very great care is necessary in accurate alloy formulation. This requirement alone imposes a melting process restriction in that the alloy batch must be fully liquid in its entirety at some time

in the processing to ensure not only a homogenous mixture, but also a representative chemical analysis. Since niobium forms a very stable carbide with a weight ratio of Nb:C, 93:12, small changes in the carbon activity of the alloy can greatly influence the phase distribution of Nb. In this regard, a complex interactive system of Si, Ti, C, N, and Nb all influence the Laves phase stability and must be controlled during alloy formulation.

Sulphur Content.

The effect of suphur in Alloy 718 is well-established and it is clear that low (<10 ppm) levels are required for optimum properties, particularly in dynamic properties depending on ductility. Since the alloy contains very strong deoxidizers at high chemical activities we cannot remove sulphur by oxidative reactions, but must instead rely on the general reactions:

$$[S] + M \stackrel{\Rightarrow}{\leftarrow} MS(s, l) \tag{1}$$

or

$$\{s\} + M0 \stackrel{\Rightarrow}{\leftarrow} Ms + \{0\}$$
 (2)

Reaction (1), utilizing the solubility product of MS, requires the addition of an element with a very stable sulphide and with a slight solubility in Alloy 718. The practical conclusion is that only Mg, Ca, or the rare-earth metals will fit these requirements. In a vacuum process, there are obvious problems in using Mg or Ca, which will be discussed below, but the major disadvantage of all three possibilities is that their extreme reactivity towards refractory oxides makes their controlled addition to a melt a very difficult process. Reaction (2), on the other hand 2 is not prone to this difficulty because the product, an oxygen activity, is part of the alloy deoxidation scheme. Provided that the required degree of desulphurization is not too large the resulting oxygen content will be an insignificant contributor to the overall deoxidation The reaction has been primarily applied to alloy treatment with CaO, as solid addition or as refractory container, but it is also the ruling reaction for desulphurization by ESR. Partly by the use of reactions (1) & (2), and partly by a judicious choice of raw materials, levels of sulphur may be obtained which are essentially impossible to check by conventional chemical analysis (<1 ppm). However, because it is economically advantageous to recycle scrap alloy, the basic problem of sulphur removal remains active in the melting scheme.

Trace Elements.

Control of trace element contamination in Alloy 718 is a well-established field. Since most of the trace elements are highly volatile, their concentrations are influenced by vacuum treatment in spite of their low chemical activity. The problem is one of analytical technique rather than of melting variables. However, the need to control the wide range of trace elements impacts are the melting techniques through the consequent need to control all potential sources such as slags, cements, refractories, etc. as well as the metallic components of the alloy.

Non-Metallic Inclusions.

The thermochemistry of this alloy leads to the conclusion that there are essentially only two true inclusion precipitation reactions:⁴

$$\{Ti\} + \{N\} \stackrel{?}{\leftarrow} TiN(s) \tag{3}$$

$$2\{A1\} + 3\{0\} \stackrel{?}{\leftarrow} A1_2 0_3(s)$$
 (4)

The primary carbide, NbC is precipitated during freezing also, but is onsidered to be part of the alloy structure. The other oxide which is requently found, MgO, is formed as a solid in the liquid alloy processing at the time of addition of Mg to the melt. The solid particles of TiN, $\mathrm{Al}_2\mathrm{O}_3$ and MgO are present in the liquid only when their respective solubility limits are exceeded. In the three cases above, at the liquidus temperature these correspond to a nitrogen content of 35 ppm and an oxygen content of 5 ppm. In most industrial melts, therefore, we can expect to see solid particles. This finding partially accounts for the success of refining procedures such as filtering or EBCHM refining in removal of It also leads to the conclusion that to reduce 0 & N to levels lower than the above would require techniques other than physical removal of particles. The particles themselves not only influence mechanical properties but also influence solidification structure. Fig. 1 demonstrates the effect of nitrogen in changing the dendritic growth of the alloy, whilst Figs. 2 & 3 show the influence of prior-existing particles on the nucleation and growth of carbides during solidification. Obviously, it is necessary to use a melting route in which we can control this variable.

MELTING PROCESSES

Raw Materials

The various uses of Alloy 718 all require that the alloy is formulated at the least cost, with the melting and refining steps accomplishing as much chemical improvement as is possible. The use of alloy scrap is problematical because of trace element contamination and also because it necessarily entails the addition of reactive elements at an early process point, thus precluding any oxidative refining. Scrap alloy is therefore used only in lower-grade applications, or in small quantities as a late addition, or after prior processing, for example by AOD to recover the primary element value.

The elemental additions are selected to minimize the chemical problems described above. One advantage of Alloy 718 being its iron content which permits the use of some ferro-alloys, for example ferro-chromium, The alloy cost is directly related to the charge quantity and considerable effort is made to use those materials which can be refined suitably during melting. example, carbon contents in the primary Ni + Fe + Cr charge are lowered by vacuum deoxidation, but carbon cannot be oxidized after the addition of Ti Al, Nb, etc. Conversely, although nitrogen contents can be reduced by vac exposure, the rate is slow and it is usually more economical to control nitrogen by other measures such as raw material selection.

Vacuum Induction Melting

The VIM process has been very adequately described elsewhere, but it pertinent to discuss the specific procedures which are applicable to Alloy 718. The most fundamental question is the extent to which VIM may be used a refining, as opposed to a melting, process. In this regard, the modern process has three major problems:

- -de-sulphurization
- -control of volatile elements
- -removal of slag and inclusions

Evaporation principles are well covered in the literature 5 and the conclusions may be summarized as follows. At the ppm levels of most volatile impurities in Alloy 718, the evaporation rates are finite (e.g.

for Zn at 1 ppm, the rate is 0.5 ppm/hr in a typical 5 tonne VIM geometry) and are controlled almost completely by liquid-phase mass transfer. There is hence an appreciable decrease in volatile trace elements during VIM processing which, written typical vacuum ranges, is not dependent on furnace pressure but is a function of surface/volume ratio and Gas removal is similarly limited by liquid-phase mass temperature. transfer once the initial carbon boil has been accomplished. The description rates depends on the precise alloy composition, but are approximately the same as for volatile elements. It is to be recalled that the nitrogen removal is partly through the physical flotation of TiN(s) and partly by the latter's decomposition into solution prior to a gas description reaction. This combination of processes leads to the apparent contradiction that lower nitrogen contents are obtained by long processing times at high temperature. The reactions involving TiN(s) are exploited to remove nitrogen by filtration in the tundish. filters have been used for some time in the casting of Alloy 718, their use is relatively new in the production of forging material. The filter is placed either in the tundish or directly above the mold and offer a The term "filter" is a misnomer significant resistance to metal flow. since the mechanism of removing particles is largely one of adsorption on the interior pore surfaces. The average filter pore size is much larger than the inclusion particle size. It is to be noted that since filtering is the physical removal of solid inclusion particles, the efficiency of oxygen or nitrogen removal by this mechanism is increased at temperatures This conclusion leads to the problem of close to the liquidus. maintaining metal flow through the filter at low temperatures and has initiated developments in heating the tundish area.

De-sulphurization is accomplished in two ways; reaction with the refractory and reaction with additions of slag or metal components. The refractory/metal contact is particularly good in VIM and enchances the removal of refractory sulphides formed by de-sulphurizing reactions. For example, rare-earth additions to the melt produce oxy-sulphides which are readily absorbed into the VIM refractory walls. Unfortunately, these compounds are leached back into successive melts in the same crucible and the technique leads to very undesirable fluctuations in trace rare-earth contents. The same reactions also hold for Mg and Ca additions although in this case the residual trace variation is more acceptable in the Alloy 718 composition. Very effective desulphurization is produced by the use

of lime or dolomite as the refractory lining, but as yet it appears that the problems of using such linings in practice have not been resolved.

VAR Processing

The VAR process has altered considerably in detail over the years, but in principle remains a problematic process. The aim of the procedure is to produce a controlled, segregation-free ingot structure, but the precise mechanisms which govern this are still obscure. The development of the liquid ingot pool by a dynamic heat balance; the relation of pool dimensions and ingot isotherms to that heat balance; 8 and the influence of melting rate to structure have all been intensively studied and the theoretical postulates largely verified. However, production problems are seldom associated with the above, but lie more in the aspect of the inherent instability of VAR. After paying due attention to process control, defects associated with the intrinsic solidification process, e.g. carbide distributions or laves phase, are seldom reported. However, the pressure of instability-related defects such as white spots or agglomerated inclusions is a recurring problem. White spots have been broadly classified into four categories: clean, dirty, large, small (<~1mm). The combination clean, small is obviously the least detrimental to properties, with the combination dirty, large being the most harmful. The majority of the large white spots appear to originate as electrode fall-in and their incidence, can be greatly reduced by either prior ESR melting, or using forged electrodes. In addition, since the defect survival in the mother pool is related to the pool superheat and volume, the incidence can also be reduced by increasing the melting rate. solution has obvious limits imposed by the segregation requirements. The small, clean white spots and the diffuse type are related to liquid flow in the liquid + solid region. Their origin is not yet precisely understood but is connected with random liquid pool stirring due to either thermal or electromagnetic instabilities.

Inclusion removal by VAR is a complex process. As indicated above, nitride and oxide inclusions are present in the liquid when their respective saturation solubilities are exceeded and could therefore be removed by liquid process. However, although some inclusion removal by surface forces does take place, the resulting films of agglomerated inclusions collect at the rim of the liquid pool and random stirring can

readily entrap them in the ingot. A random micro-examination of the alloy hence shows an apparent decrease in average inclusion content, but at the expense of a finite probability of an incidence of large inclusion agglomerations. Although the existing standards for NDT should adequately remove such material, the existence of these inclusion collections remains an in-principle problem of VAR and underlines the need to manufacture the cleanest possible VIM electrode material.

ESR Processing

Although one of the first commercial applications of the ESR process was to superalloys, it has not been widely specified for the highest quantity in this application. Alloy 718 is extensively processed by ESR for non-rotating part use and much has been established concerning the behaviour of this reaction system. It is clear that the alloy composition can be accurately controlled by utilizing the reaction:

$$3\{Ti\} + 2A1_2O_3 \stackrel{?}{\leftarrow} 4\{A1\} + 3TiO_2$$

through slag compositions which are compatible with the low melting rates required for segregation control. These slag compositions are also ones in which a large degree of de-sulphurization can be attained and in which the trace element content of Mg can be controlled.

In general, alloys melted by ESR have no "memory" for the inclusion content of the electrode, and the ingot inclusion content is established entirely by alloy composition, ESR slag and ESR deoxidation practice. Il A very strong positive aspect of ESR is the process' tolerance for poor quality electrodes, in contrast to VAR. The above axiom can be rationalized for oxide inclusions in terms of reactions with the slag, but since TiN is not soluble in ESR slags, reactions involving TiN removal are predominantly physical, surface mechanisms. TiN is rejected to the electrode/slag interface upon melting and subsequently to the rim of the ingot surface by the forces operating on the ingot pool meniscus. processed Alloy 718 has a high surface concentration of TiN particles, which represents a nitrogen removal from the bulk metal. However, since there exists a finite nitrogen solubility in the slags used. 12 and the remelting is carried out under air, a low nitrogen electrode will pick-up nitrogen during ESR melting. For example, an electrode content of 80 ppm may be decreased to an ingot content of 60 ppm, but in the same conditions a 40 ppm electrode will rise to a 60 ppm ingot. This effect is relatively unimportant in the context of present specifications, but would constitute a reason for argon-ESR processing in the future.

The tolerance of ESR for electrode defects is exploited in triple melting, where the ESR step reduces sulphur content, removes large inclusion agglomerations and entrapped slag, and produces a fully-dense structure with no shrinkage cavities. This ingot is then used as the VAR electrode; the object of VAR processing being the sole one of controlling solidification structure. The procedure has been shown to have a lower incidence of defects in the final ingot than has single VAR melting, but its greatest significance is probably in balancing the Mg/S ratio. Magnesium is added at a late stage in the VIM process and is generally present at levels between 80 and 150 ppm in the VAR electrode, the recovery depending on VIM practice and on the pouring time. After VAR, the magnesium content of the alloy is in the region 5-10 ppm, determined by evaporation reactions during VAR. This Mg content is sufficient to give adequate ductility with sulphur levels of 5-10 ppm, but it is inadequate for higher values. Attempts to add more Mg in the electrode, so as to compensate for higher sulphur levels lead to excessive Mg vapour in the arc region of VAR with consequent instability and solidification defects. The interposition of ESR reduces the total sulphur content and by a correct choice of slag composition having an MgO content, will also produce an ingot of the desired Mg/S ratio. 13 The low contents of both Mg and S are not changed by the subsequent VAR step.

The heat balance of the ESR process is superficially very similar to that in VAR, and at similar melting rates, the ingot isotherms are of the same form in the two processes. The ESR process is inherently more stable due both to the presence of the liquid slag and to the more stable current paths. For this reason, it appears that the structure of the VAR liquid + solid zone differs from that in ESR, permitting a greater interdendritic fluid flow in the latter process. The result is that ESR ingots exhibit a greater degree of centre segregation than do VAR ingots processed under the same conditions. It is possible that the inherent instability of VAR may have the advantage of preventing long term, stable interdendritic liquid movement, hence avoiding the centre segregation so characteristic of Alloy 718.

Cold-Hearth Processes

The cost/benefit structure of Alloy 718's use does not justify high-cost processing to obtain increased properties, but since it is a well-characterized alloy, it has been used as a model system to test the newer refining processes. The principal objectives in these processes are to obtain a less-segregated solidification structure and also to reduce the total inclusion content. Secondary benefits are also to be obtained from better process control and monitoring, leading to a reduced incidence of defects such as white spots.

The principal process which has been applied is electron beam melting utilizing melting into a cold hearth followed by ingot casting by a withdrawal technique. The prime objective of the process is the removal of non-metallic inclusions in master alloys for casting and powder as well as in the forging grades. The removal takes place during the hearth processing and the slag film formed is separated from the liquid metal either by a physical barrier, or dam, or by using the surface forces generated by the electron beam. The relative utility of these techniques has been discussed in previous reports, but it is clear that in both cases, even with ESR or VAR feedstock to the hearth furnace, there is a remarkable separation of inclusion material.

An examination of the melting process 15 shows that inclusions are not separated by flotation, but instead are rejected to the liquid metal surface by interfacial tension during the melting of the feedstock. subsequently remain on the surface during the hearth processing. Although Al_20_3 are decomposed if they are heated to a very high both TiN and tempeature by direct beam impingement, most of the separated inclusion material remains on the metal surface during processing. It is to be concluded, therefore, that the metal immediately below the slag layer is in chemical equilibrium with it and is saturated with respect to both TiN and Al, 0, . Upon cooling and solidification, therefore, it will precipitate these particles and form secondary inclusion, defeating to a large extent the purpose of the hearth refining process. In order to prevent these reactions we must design the process so that the slag film separation takes place in the initial part of the hearth, followed by a significant metal exposure to the vacuum environment. This latter step then reduces the oxygen and nitrogen contents to less than the respective saturation solubilities of ${\rm Al}_2{\rm O}_3$ and TiN at the solidus and no inclusions agglomerations are formed in the ingot product.

The superalloy compositions processed in this way are all similar to Alloy 718 in that they have one principal volatile component, chromium. Although in principle it is possible to make a corrective addition directly to the process, it has proven to be difficult to control the composition in this way to within the limits required by specification. The solution adopted is to provide a feedstock which has been melted and cast by VIM with a corrective addition of chromium.

Whilst the technique described above appears to have opened up a new era of super-clean alloys, the problem of solidification control still remains. Various methods have been used to refine the structure of Alloy 718, from the obvious ones of powder and RST to the VADER 16 process and variations of the same technique in both EB and plasma melting. The methods have all produced a refined grain size, but no comprehensived study is yet available on their comparative effectiveness in influencing the distribution of LCF crack nucleation sites which must be taken as the critical test of their success. Possibly the most intriguing, but as yet unexploited, feature of the solidification control processes based on melting is their potential to make large diameter pieces of Alloy 718 available containing an acceptable structure throughout the section. The availability of such larger pieceweights in Alloy 718 could quite possibly open up new uses for this material.

CONCLUSION

Over the long development of melting techniques for Alloy 718, it has become clear that the primary melting and remelting techniques of VIM, VAR and ESR can produce adequate qualities of the alloy. They all, however, have well-understood limitations which have now limited the ultimate applications of the alloy in quality, quality assurance and pieceweight. The newer techniques of hearth melting show promise in solving all of these questions and will certainly develop the use of this and similar alloys in the future.

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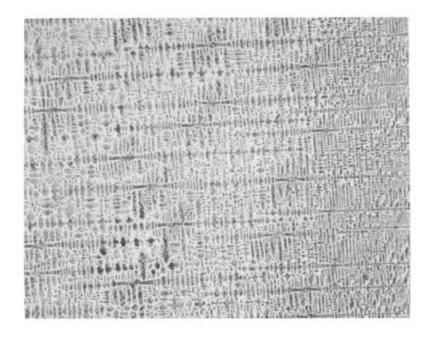


Fig. 1(a) Directionally solidified Alloy 718, 20 ppm N, x 40

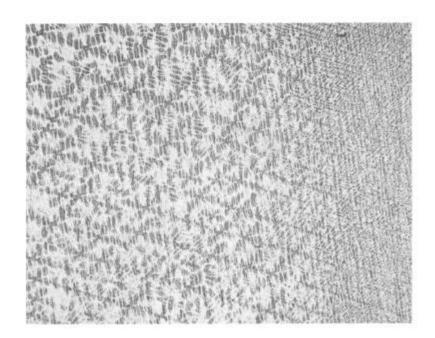


Fig. 1(b) Same conditions as Fig. 1(a) but with 120 ppm N, \times 40

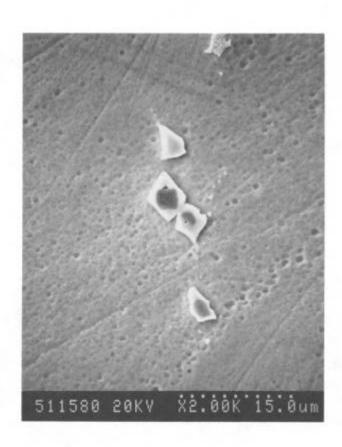


Fig. 2 Carbides nucleated by prior TiN particles.

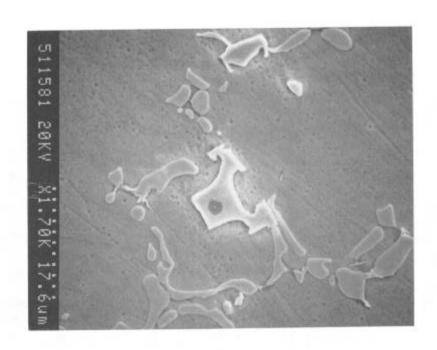


Fig. 3 Core of TiN particle showing MgO prior particle.