

## MELTING PROCESSES AND SOLIDIFICATION IN ALLOYS 718-625

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### Abstract

The present state of melting techniques for IN718 and IN625 has well-defined limitations both in terms of the ingot structure and maximum ingot size which is fabricable by the various processes. Although there are methods already in industrial use which would extend both of these limits, since the alloys must be produced at a relatively low cost here are further limitations on the practical manufacturing methods. The underlying scientific problems of solidification, inclusion removal and structure control are discussed with the conclusion that the present ingot structures probably cannot be substantially improved, but the inclusion population could be modified without much additional effort. Suggestions are made as to the possibilities for making larger pieceweights of the alloys should these become necessary for the marketplace.

## Introduction

The general principles of structure control in ingot have been known for many years, and essentially all casting processes contain some element of this control. The objectives are often not metallurgical but are connected with the need for the highest possible useable ingot yield, but nevertheless derived from the same basic principles of heat transfer and alloy solidification which govern even the most exotic of our present techniques. In the manufacture of superalloys the over-riding consideration is that of segregation control, for the following set of reasons. First, the alloy structure is such that there is extensive precipitation of primary phases, for example MC carbides, at temperatures at or above the solidus. In consequence, at least this component of the final product structure has its particle size and distribution fixed by the primary ingot-making process, since the carbides are not significantly altered by diffusional processes at temperatures below the solidus. Second, the nature of the alloy elements is such that their diffusion rates in the alloy solid structure are quite slow, even at temperatures approaching the solidus. This fact, when combined with the extreme sensitivity of the alloy properties to the distribution of the strengthening precipitate phases, means that we must attempt to minimise the segregation which takes place during solidification in order to simplify the problem of homogenisation of the ingot or forging at a later stage in the manufacturing process. The wavelength of the segregation is set by the dendrite spacing during solidification which in turn is directly related to the freezing conditions which operate during casting. Finally, we must control the solidification irregularities which play an important role in quality in the final ingot. Random process-related defects such as white-spots, freckles and tree-rings are not directly part of the solidification scheme but are connected to it in so far as they can be reduced to an acceptable degree if the stability of the solidification process is maximised.

Probably one of the least well-defined fields in the manufacture of these alloys is directly connected with the problem of the segregation taking place during solidification. Much of our basic design philosophy takes as a working premise that the alloy structure is a uniform isotropic medium for the purposes of fracture mechanics analysis. Even the more sophisticated methods of, for example, life-prediction improve on this only to the extent of imposing a given distribution of defects on the same isotropic background. It is clear from the examination of any real-life billet or bar that the alloy is far from being isotropic or uniform, and that much of the non-uniformity has its origin in the segregation processes of casting. The evaluation of this segregation at present rests on the comparison of etched samples, or worse still of photographs of etched samples, when more accurate and reproducible techniques are readily available. One such technique is that of computerised micro-analysis (CMA) which is capable of providing quantitative information on the exact degree of segregation in a given sample. An example of the technique as applied to a cast superalloy structure is shown in Fig. 1, where the effects of homogenisation can be seen. A wider application of CMA would eliminate much of the present uncertainty concerning the relative merits of the various manufacturing routes, and would probably also enable us to use the powerful tools of fracture analysis to a greater effect. Notwithstanding the above caveat, it is possible to use our present data collection on the alloys to come to valuable conclusions about the existing process for melting and casting and also to theorise effectively on the future possibilities for their manufacture.

### Melting Process and Inclusion Control.

In recent years we have begun to see the introduction of melting strategies which are leading to a situation in which the alloy produced has contents of non-metallic inclusions that approach zero. It is reasonable to ask in these circumstances what "zero" really means, and also to examine the relation to this apparent limiting condition to the true limits of precipitation of the inclusions as defined by their solution thermodynamics in the alloy. In the case of IN718 data is now available on the solutions of O, S and N in the liquid which permit us to predict the precipitation system. We will illustrate this scheme in the following manner.

The cast structure of IN718 normally shows four types of inclusions:  $\text{Al}_2\text{O}_3$ , TiN, MgO, and MgS. The form of these types is that TiN particles nucleate and grow on MgO precipitates, and are subsequently agglomerated by  $\text{Al}_2\text{O}_3$  particles, whilst MgS particles are to be found only in the interdendritic regions in the final liquid to solidify. The solubility products of TiN and  $\text{Al}_2\text{O}_3$  have been determined and those of MgO and MgS may be calculated from literature data.<sup>1</sup> MgO particles exist in the liquid at all process temperatures whilst TiN and alumina precipitate progressively as the liquid cools during the casting process. The solubility product of TiN may be used as an example<sup>2</sup> where we see that for a typical composition of 60ppm N, all of the nitrogen is in solution at temperatures in excess of 1500°C, but as the liquid cools to the liquidus temperature slightly more than 20ppm N is precipitated as TiN crystals which nucleate isomorphously on MgO. At the same time, alumina is being precipitated as separate particles, which (presumably since alumina is not isomorphous with TiN) do not surround the TiN crystals but instead act as agglomerating bridges. At the liquidus temperature, approximately 5ppm O and 40ppm N remain in solution to be precipitated further during the liquidus/solidus interval. No sulphides precipitate at temperatures above the liquidus.

During the solidification process, the segregation of both sulphur and magnesium leads to a rapid rise in their concentration in the final liquid to solidify. Following the Pfann equation and using partition coefficients derived from the binary phase diagrams of Ni-S and Mg-Ni we may make an estimate<sup>3</sup> of their concentration profiles during solidification, with the results shown in Fig. 2. The concentration exceeds the solubility product of MgS (as calculated from the data of Samuelsson et al)<sup>1</sup> only in the very last stages of the solidification, leading to a situation where MgS will be precipitated at the sole site of primary grain boundaries, which is in fact observed in practice.

From the point-of view of melting and refining practices, the implications of the above sequence are quite clear. A "clean" metal will be one in which any non-metallic inclusions are precipitated only at temperatures less than the liquidus, and hence do not have the opportunity to grow or coalesce. We should, therefore, aim to produce liquid metal which has a content of O, N and S which satisfies this condition with respect to the solubility relationships given above. For IN718 with an Mg content of approximately 10-20ppm, the limiting contents are  $\text{S} < 2\text{ppm}$ ,  $\text{O} < 3\text{ppm}$  and  $\text{N} < 38\text{ppm}$ . It is necessary to review various combinations of refining techniques in this context as well as their implications in ingot structure control.

### Surface Quality Considerations.

One of the characteristics of remelted ingots is that they have a surface structure which is controlled by the form of the initial contact of the liquid metal with the chill of the water-cooled crucible. In static crucible processes the surface is composed of a layer of fine-grained material which is a major contributor to the success of the initial forging breakdown steps. For processes in which there is a relative movement between the mold and the ingot, the surface layers are complex and involve some degree of "leakage" of interdendritic liquid during the micro-tearing which accompanies the movement, giving a surface in which we find high local concentrations of eutectic. This effect causes embrittlement of the surface layer and can give rise to cracking in the breakdown steps.

The above situation is part of the overall quality requirements of the process and is further compounded by the influence of the surface contact on the mold in determining the heat transfer coefficient at the ingot/mold interface. The value of this contact is an extremely important parameter in the solidification process as will be seen below. It is often neglected in the analysis of the remelting processes but is in fact probably one of the most important areas of focus in any quality control scheme for ingot structure.

### Present Processing Techniques.

The need for a very controlled composition dictates a primary process in which the entire bulk of the alloy is liquid, followed by further processes in which we carry out the refining and solidification steps. The bulk of high quality IN718 is made by the route of VIM followed by VAR, in order to fit this requirement. The VIM process can accomplish a degree of refining by removing oxides and nitrides as a slag or causing them to stick on the sidewalls of the crucible. Obviously this mechanism will only operate if the particles are present as a second phase, which implies that O & N must be present at concentrations above the solubility limit at the melt temperature in the VIM liquid. As we have pointed out above, if this is the case then it follows that we will produce a liquid, after the separation of the particles, which has O & N at a concentration well in excess of that at the solidus and therefore which will produce particles of TiN and  $\text{Al}_2\text{O}_3$  during the cooling and solidification processes. It is this point which provides the major challenge in making clean IN718 in VIM, namely, that of controlling the temperature during the particle separation phase at one which is as close as possible to the liquidus. At present this requirement is not well-accepted by the industry, which as a result follows practices which largely defeat the purpose of tundish and filtration separation by adding superheat to compensate for the effects of tundish systems which are too cold or which cause too much heat loss during teeming.<sup>4-6</sup>

It is interesting to consider the case of a VIM heat in which we have arranged for the O & N contents to be below the required solubility limits by virtue of choosing low-interstitial charge materials. In this case there is only a removal of O in compositions before the addition of Al or Ti and removal of N only before the addition of Ti. Both of these reactions involve a very slow desorption step and it has been observed that the reduction rate of N under these circumstances is about 4 - 5ppm/hr, a rate which agrees well with theoretical calculations based on Machlin's model of fluid flow in VIM.<sup>7</sup>

As a practical conclusion, then, it follows that if we wish to make IN718 which does not precipitate inclusions at temperatures above the liquidus we must start with a charge material which has contents of O & N less than the target values of 3ppm and 38ppm, respectively. This condition has been met in the melting of IN718 for aerospace castings

for some time, probably because the presence of even a small fraction of inclusions during solidification leads to the formation of easily-detectable micro-porosity. However, in the forging grades the present levels are far from this target as the result of the use of low-quality raw materials and also the various practices of reverting scrap materials directly into VIM.

The high-strength nickel-base alloys are all very susceptible to small contents of sulphur a condition which is ameliorated by compensating additions of magnesium. It is clear that in DA IN718 we must have a maximum of 5-10ppm S for adequate intermediate temperature ductility and a parallel addition of between 5 and 20ppm Mg. Indeed it appears that even at S levels which approach the limits of analysis (1-2 ppm) the ductility is still improving, and that the additions of Mg are still necessary. These conditions pose a significant problem in VIM since the desulphurisation processes used introduce considerable operational difficulties and also the predictability of Mg additions in the process is low.

The subsequent remelting steps are strongly influenced by the problems of the VIM practice, VAR more so than ESR. The VAR process was originally conceived as a technique for removing inclusions, but is now operated as a process for controlled solidification. There are numerous literature descriptions of the VAR heat balance and its influence on ingot structure,<sup>8-10</sup> but it is germane to this stage of its development to consider the process' limits in regard to IN718.

The relation between the solidification structure obtained in the ingot and the VAR process variables has been described<sup>11</sup> with the result that we may place a limit on the possible structure refinement available to us. In practical terms, the limits are set by two processes; the precipitation of the primary MC carbides and the formation of eutectic Laves phase. *Interdendritic segregation leads to carbide growth giving rise to carbide clusters which although they can be manipulated to a certain extent by prolonged high temperature homogenisation, remain a limiting source for crack initiation.* The formation of eutectic Laves phase, also due to segregation, prevents effective homogenisation by giving rise to grain - boundary liquation. It also can be manipulated to a degree by two-stage homogenisations since it may be decomposed slowly by diffusional processes below the solidus temperature, but the cost is usually prohibitive. These limits combine to tell us that the effective limit on the production of ingots by VAR is set at a diameter of approximately 500mm in the case aeroengine quality, and probably of not beyond 600mm for any mechanical purpose which involves using the whole ingot cross-section.

All of the above presupposes that the VAR process is being carried out in a controlled and stable fashion. As has been highlighted in the literature control of the VAR process involves precise arc-gap control by techniques whose scientific foundation is still obscure. It also presupposes that the electrode received from the VIM contains no flaws such as cavities or cracks -- a situation which is often not the case. The principal defect which arises from a failure of control in both of the above areas is that of white-spot formation,<sup>12,14</sup> for which we normally compensate by increasing the melting rate so as to enhance the probability of dissolving the white-spot-forming particles. As our control strategies have improved over the years, it is noteworthy that the industry average melting rates at the maximum ingot diameters have gradually increased so that they are now approximately 20% higher than they were 10 years ago. It is evident, however, that the white-spot problem interacts with the limitations on segregation in that to minimise the former we increase the melting rate whereas to minimise the latter we must decrease it. The combined result is that the process window becomes progressively smaller as the

ingot diameter increases, to the point that at the limits described above it is at an industrially-acceptable minimum.

Although the freckle defect was a significant problem in the early stages of the melting development of IN718, the VAR control systems have progressed to the point that it is very seldom seen in the normal production of the alloy. However, due to the rather open dendritic network which is characteristic of this alloy, interdendritic flow of highly-segregated liquid can readily occur and is easily initiated by instabilities in melting, by high melting rates and by intermittent variations in the helium flow commonly used to accelerate cooling of the ingot surface during melting. The result is a heavy dendritic pattern in the ingot or billet centre, but more importantly, the dendrite structure itself can be denuded locally of interdendritic liquid producing, after forging, local areas of low Nb concentration which appear on etching as white spots. The characteristic of this type of white spot is that the Nb concentration is seldom lower than 4.5wt% in contrast to the white spots originating in the electrode which typically have lower concentrations. Unfortunately, since the electrode white spots also have concentration gradients with areas having Nb concentrations in the range of 4.5wt%, it is impossible to distinguish between the two types of defect without an extensive destructive examination.

The use of the ESR process for manufacturing IN718 has been a point of development for some considerable time.<sup>15</sup> The process has been used for the manufacture of IN718 in non-rotating part quality and also for the manufacture of small engine parts for many years. The discussion concerning its applicability to large engine forgings centres around two points: the ability to control composition and the problem of centre-segregation. The first of these is a positive advantage whilst the first provides a very distinct limitation to the technology.

It has been shown that the choice of a slag with the correct chemical activities of titania and alumina is compatible with the rest of the physical property requirements of the ESR process and so we can make an ingot of IN718 with Ti and Al composition variations which are well within the most exacting specifications, without incurring any penalties in, for example, the ingot surface quality due to the other properties of the slag. At the same time, the ESR technique can reduce the sulphur content by up to 50% even when the incoming electrode has contents as low as 5ppm.

Extensive studies using both LCF testing and EB button assessment<sup>16</sup> have shown that the ESR process is capable of a high degree of refining with respect to oxide inclusions as would be expected from the nature of the process. It is unusual for any inclusions present in the electrode to penetrate to the ingot and the ingot's inclusion population is a characteristic of the slag/metal reactions in the system rather than the electrode's composition. ESR ingots tested in this way have been shown consistently to be cleaner than VAR material with respect to oxide inclusions. ESR, on the other hand, does not remove nitride inclusions except to the extent that at high nitrogen contents some of the nitrides are rejected to the ingot/slag/mold interface by surface forces and are found to be concentrated on the final ingot surface. The nitrides are modified in that the oxide inclusions which play an important role in the agglomeration mechanism of the nitrides, are no longer the simple alumina inclusions present in VIM or VAR. The result is that the nitrides are less likely to agglomerate in ESR even when the nitrogen content is well above the saturation solubility at the liquidus. It has been shown that when IN718 is melted in air by ESR the nitrogen content equilibrates at around 60ppm. Hence, an electrode which has been made from high-purity raw materials and has a nitrogen content of, say, 20ppm (as might be typical of the casting industry) will add nitrogen on

processing by ESR. For ESR processing at very low nitrogen contents we are faced with the need to develop protective atmospheres and the added complexity which that involves.

These factors, as can be seen from the discussion above, are very important in producing the requisite ductility, particularly when working with revert or low-cost raw materials. By choosing a slag which has approximately 5% MgO content we can retain an Mg content in the ingot of 50ppm, which is a level appropriate for the composition balance either of a final product material or for the electrode composition in a subsequent remelting operation. The formation of white spots in the ESR process is very rare and requires a combination of circumstances which it is not possible to attain without there being a clear indication of the problem on the melt record. With the appropriate adjustment of the melting parameters it is possible to obtain a micro- and macro-structure in the ESR ingot which is identical to a high quality VAR ingot up to an ingot diameter of 400-450mm.

In sizes larger than 450mm, however, it appears that we start to encounter the limits set by the process heat balance. It has been commonly observed that ESR ingots of diameters larger than 450mm have extensive centre segregation,<sup>17</sup> but present irregularly along the ingot axis. The effect is connected to the control of the electrode position in the slag and its effect on the partition of current flowing to the mold and to the ingot. Very small changes in the electrode position when it is "floating" on the slag surface, can play a disproportionately large role in this partition, to the extent that a variation of less than 1mm in this position is able to produce the ingot pool-shape change which in turn brings about the fluid flow leading to centre segregation. It is evidently possible to make ESR ingots for rotating part quality in diameters as large as 500mm, but the reproducibility of the method is still in doubt. This finding has led to the development of the so-called triple-melt route in which we use a VIM electrode to feed an ESR furnace followed by melting in VAR. The purpose of the VIM stage is to prepare the alloy with a uniform composition and N and S contents acceptable to the ESR process. The ESR is then used partly to accomplish refining with respect to the inclusion content and partly to produce an ingot which has a very uniform structure (compared with the VIM product) and which then is much easier to use in the VAR process. We may then use the VAR technique solely as a means to obtain the desired solidification structure, having substantially reduced the incidence of white-spot defects and also reduced the probability of those white spots being associated with inclusion agglomerations.

This combination of techniques has proved to be a cost-effective way to obtain high-quality material, but is almost certainly the limit of the technologies concerned. The VAR ingots from triple melting still contain a finite number of white spot defects and a finite number of inclusion agglomerations. LCF testing demonstrates that we still have not achieved the goal of clean metal -- a situation in which all LCF failures are seen to originate either at surface features or at inherent microstructural features such as carbides, which are assessable by random metallographic testing. The inevitable conclusion from this examination of the present melting practice is that we have reached the limit on ingot diameter (and therefore on pieceweight), and we have also reached the limit on metal cleanliness and structural refinement for this alloy.

#### Possibilities for Future Processing of IN718.

For the full utilisation of IN718 in high temperature machinery we need to accomplish several further objectives:

- structure refinement with respect to the carbide size and distribution.
- elimination of inclusions greater than the critical defect size
- develop a fabrication route to give bigger pieceweights in the alloy.

The first of these objectives can only be attained by increasing the rate of solidification since the carbides are primary and their size is fixed by the precipitation from solution either during freezing or at the eutectic temperature. Powder routes will of course have this effect, but carry all of the concomitant disadvantages, and an ingot route is to be preferred if possible because of the added security of quality control. Several alternatives have been suggested for such a route; the casting of forging preforms in a pancake,<sup>18</sup> VADER solidification<sup>19</sup> and the welding together of smaller ingots prior to forging.<sup>20</sup>

The casting of preforms into a pancake shape achieves the required structural refinement by utilising the heat transfer at the base and top surfaces of the ingot in a situation where the ruling section for heat transfer is less than that of the ingot radius. Obviously, this technique will work for pancake heights which are less than the ingot diameter, but ultimately the limits on the pancake diameter will be the same as those for the conventional ingot. If the pancake method could be made to work perfectly, therefore, its theoretical limit would be a pieceweight which would be double that obtainable from a VAR ingot containing the same structure. However, given the practical problems of metal feeding and superheat it is probably unrealistic to suggest that this is an attainable goal, and the actual limits of this type of process are probably contained in height-to-diameter ratios of around 0.4 as computer modelling would indicate. Several methods have been proposed for the casting of such pancakes; casting from an EB hearth (either as a single cast from a tilting hearth followed by EB hot-topping or progressive casting from a hearth using careful beam scanning on the cast surface to control the heat loss), rheo- or squeeze-casting from a VIM melt, or electroslog casting in a manner similar to the preparation of a slab ingot. All of these methods have been shown to work on the laboratory or pilot scale, but as yet none of them has faced the test of full-scale melting. It is worthwhile noting that the shape requirements for heat transfer would pose definite problems in the need for minimum amounts of mechanical work and also in the needs for minimum strain-rates, on the majority of the industry's presses.

VADER solidification has been discussed extensively in the literature and clearly has a potential to manufacture ingot diameters which are beyond those limits outlined above. The mechanisms which govern the formation of the fine structure are not yet understood and it is not possible to predict the exact extent to which the process might be used to achieve the aim of a large section in IN718, although it appears that there is certainly considerable potential. The method of welding together large ingots to form a larger preform before initial breakdown forging has been used successfully on a variety of steel grades to make turbine and rotor shafts with weights of up to 250 tonnes, and also in the manufacture of heavy steel plates. The welds are made using plate electrodes and the electroslog method, both of which are adaptable to IN718 in the requisite quality. The technique has not been used on any high alloy nickel-base materials, but since the heat transfer situation is a highly predictable one, it should be possible to simulate the process on the required scale and so to predict the metallurgical structure of the heat-affected zone. Once that structure is known, we would be able to assess whether or not the desired final structure could be obtained by mechanical working.

It appears from the above discussion that there are still possibilities for the production of larger pieceweights in IN718 containing structures which have at least an acceptable as-cast structure. Whether or not these structures can be converted to final product



requirements, given the necessary restrictions on strain-rates, heating and cooling rates during processing remains a serious question. From the viewpoint of conventional technology, however, the present maximum sizes made by VAR represent not only the practical but also the theoretical limits of the process.

The aim of making IN718 with a much lower inclusion content is, in contrast, relatively straightforward. The judicious choice of a raw material, leak rates, degassing and heat-up rates (at the penalty of a cost increase) can reduce the nitrogen level to a point where a VIM with a heated filtering system could produce liquid IN718 containing nitrogen at less than the liquidus solubility limit. Equivalent contents could also be produced with known technology probably at closely similar cost using EB hearth refining techniques. The agglomeration of nitrides would thus be avoided. The sulphur level can similarly be reduced by the application of established technology, either by using de-sulphurisation and calcia or dolomite refractories in the VIM and/or by using ESR. Reduction of the oxide inclusion content poses a more difficult problem, since the maximum oxygen level permitted by the solubility limits is very low, and the initial VIM melt necessarily involves the use of oxide crucibles. There are some indications that the use of calcia or dolomite refractories can produce low oxygen levels, but even these levels are not below the oxide solubility limits. The very low oxygen contents concerned make the efficiency of filtration systems equally low, and even by using a heated system with low superheat is doubtful that the oxide inclusion content could be removed with adequate reliability. The ESR process reliably removes oxide inclusions from the electrode, but in turn introduces a slag/metal equilibrium at a significant superheat temperature, which gives an oxygen content to the ingot pool that is significantly in excess of the solubility limit. As indicated above the result is a lower content of inclusions with a better distribution than in either VAR or VIM, but still not fulfilling the objective of clean melting. It would appear that the only way in which we can remove oxide inclusions whilst maintaining the requirement of little or no superheat is to use EB hearth refining. The technology for this procedure has been developed and is regularly applied<sup>21</sup> to superalloys with a higher intrinsic value than IN718. It is clearly possible to make IN718 with the oxide content qualifying it as clean alloy, but it remains in question whether the added cost of the EB process, small though it is, could be justified by the applications.

### Conclusions

We may conclude from the above discussion that:

- The present use of melting and casting techniques shows little potential for dramatic improvements in the intrinsic quality of IN718, although the detailed operations are likely to benefit substantially from the application of more sophisticated QA and QC systems in conjunction with up-to-date equipment.
- Improvements in the structure and inclusion content of IN718 can certainly be made by the application of technology which is feasible within the present state of development. However, it remains to be seen whether or not this improvement carries a cost which can be justified by the projected uses of IN718.
- It appears that should the need be found for larger pieceweights of IN718 than are presently available, that there are routes available which would make this possible without substantially adding to the cost or detracting from the quality.

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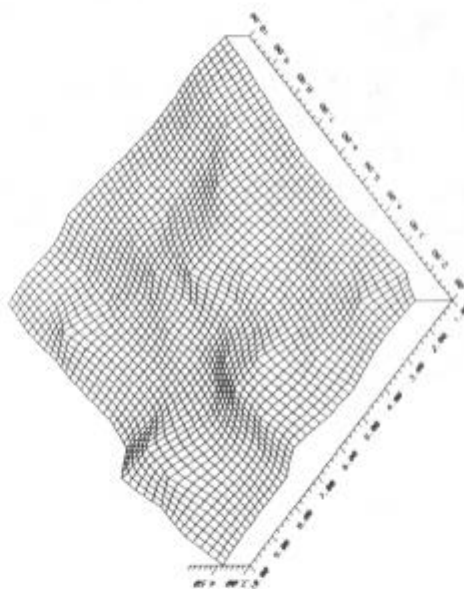
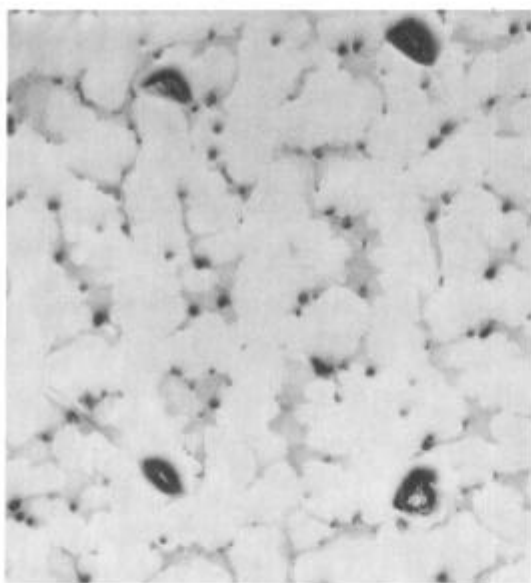
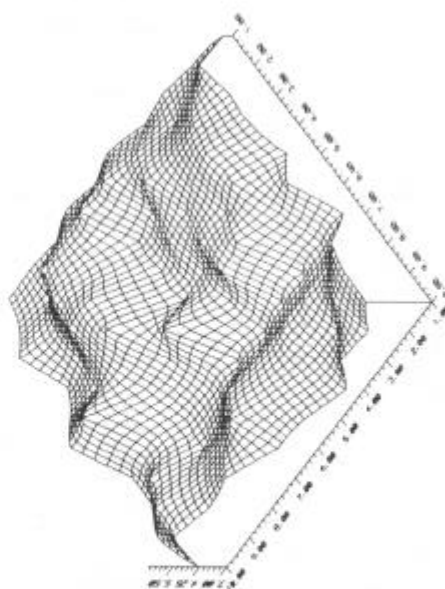
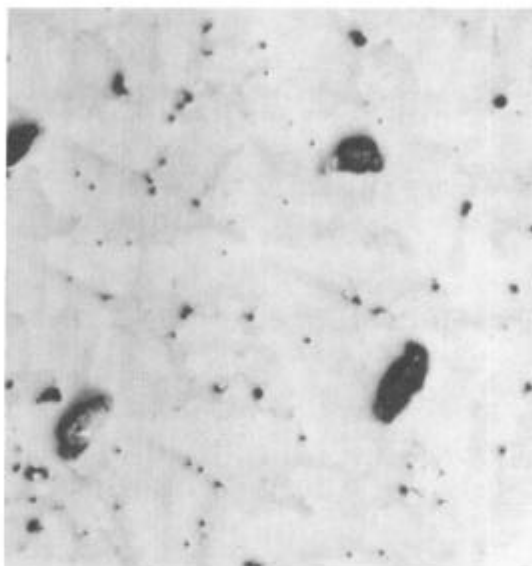


Fig 1. CMA analysis of Udimet 720. Upper micrograph, as cast x125, Kalling's etch. Graph shows Ti concentrations. Lower micrograph, same conditions, but the sample has been homogenised for 12h at 1160°C. Data reproduced courtesy of Mr J Fernihough.

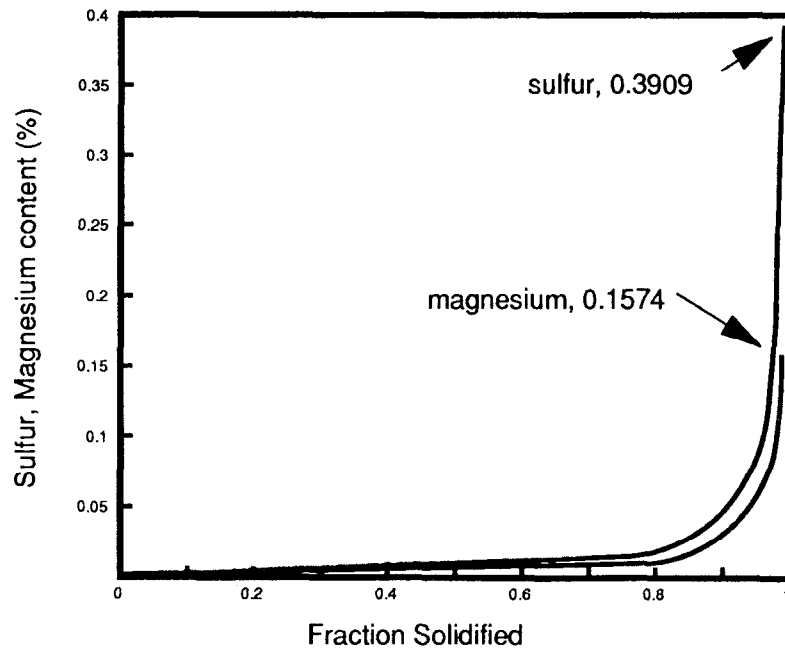


Fig 2a. Sulphur and Magnesium contents as a function of fraction solidified for IN718 at a starting concentration of 5ppm S and 20ppm Mg assuming the same segregation coefficients as apply to the binary systems Ni-Mg and Ni-S. Data from Ref.3.

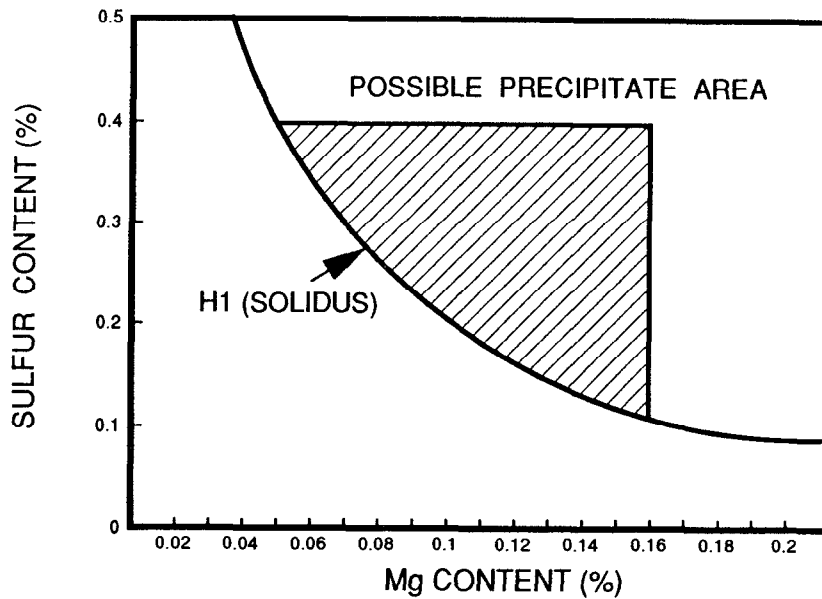


Fig 2b. Solubility product in the reaction;  
 $[Mg] + [S] = MgS(s)$   
 Calculated for the solidus temperature of IN718, indicating the composition range where segregation should lead to the precipitation of MgS, from Fig 2a. Data from Ref 1.