The Precipitation of Primary Carbides in IN718 and its Relation to Solidification Conditions

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Abstract

The solidification structure of remelt ingots of IN718 has been modeled by many authors using a number of different computation techniques. The models have generally been validated by comparing the computed liquidus line with the "pool profile" as outlined by a variety of techniques in industrial ingots. Recent models have also been validated by comparing the dendrite spacings predicted by the computed solidification parameters to those derived from laboratory directional solidification experiments. Both these methods have strong drawbacks. The "pool profile" is almost certainly not the liquidus line in a practical ingot due to supercooling and to momentum transfer from the bulk liquid to the interdendritic liquid in the region where the solid fraction is small. In the case of the dendrite spacings, the nature of the spacings' dependence on the solidification parameters means that the variations expected in practical ingots are close to the precision with which the spacings can be determined. We therefore lack sufficient means to properly test the computed ingot structures against actual practice.

A further test of computed ingot thermal conditions has been used. In this test, the average size of primary NbC precipitates is used as the solidification monitor. Laboratory directional solidification experiments have shown that this parameter is sensitive to the local solidification time and can thus be used to check the computed position of the solidus relative to the computed liquidus. The purpose of the present work is to determine the extent to which this method is valid over the range of compositions which are use for alloy IN718.

We find that the method is valid provided that the correct relationship is used for the alloy composition in question. The method, however, is difficult to implement in practice due to the problem of determining exactly which carbides in the microstructure are the appropriate primary particles. This difficulty reduces the method to one which is of the same order of precision as the one which uses dendrite spacings. Nonetheless, the method is a useful additional determination of the solidification parameters.

Introduction

Several authors have studied the solidification structure of IN718 and produced pseudo phase diagrams for the alloy. Floreen et al (1), Cieslak (2,3), Bouse (4), Mitchell (5) and Frederiksson (6) all comment on the precipitation of NbC in the early stages of solidification of IN718. The majority of phase diagram studies have, however, concentrated on the nature and form of Laves phase precipitation since it is this feature which presents the most problematical aspect of subsequent property development. The Laves phase eutectic represents the final solidification of the alloy, in a sequence which commences with a liquidus at 1320 – 1340C, a carbide start temperature of 1240 – 1280C with NbC precipitation and growth until the eutectic is reached. The temperature ranges cover the composition variation found in formulations of the alloy. The primary carbides are nucleated on TiN precipitates which are formed at or above the liquidus temperature and the "NbC" precipitates contain some Ti by both diffusion from the nitrides and from the cooling liquid. They have consequently been identified as "carbonitrides, and have a variable composition in the range (Ti,Nb)(C,N) but the primary carbide precipitating close to the initial temperature of 1280/1260C contains very little Ti or N outside of the TiN nucleus.

The carbide precipitates have two different morphologies; blocky and script, described by Fredericksson (6) and Mitchell (5). The blocky carbides are formed in the early stages of solidification when there is little or no growth restriction by diffusive supply of Nb or C from the interdendritic liquid. The script carbides form later in the sequence when growth rates are restricted by the available liquid volume. The as-cast morphology is shown in Fig 1 (a) (6), carbide stringers (Fig1 (b) and the growth sequence is shown in Fig 2 (6). Both carbides have a size distribution which is a function of solidification parameters, but the function is different in the two cases and in practice it has been found that only the size distribution of the blocky carbides can be measured with reasonable accuracy.

In the report below we describe the precipitation reaction in terms of local solidification time and the Nb/C ratio of the alloy, comparing the former with results on cooling rate obtained from industrial ingots.

Experimental

The technique used to produce the required samples was that of directional solidification plus quenching. The equipment used employed an induction-heated graphite susceptor of sufficient thickness to prevent any of the 450kHz induction field penetrating to the sample. The sample size was 5mm dia x 100mm long and was melted/cast under argon. The high-speed quenching (into water) was made possible in an automatic mode through the use of a computer-driven variable-speed motor as the motion system. The temperature gradients were calibrated using an Mo dummy bar with a thermocouple inserted on the centerline, as a function of the induction power input and a control thermocouple located in the susceptor. The detailed operation of the equipment has been described (7).

The samples were sectioned, polished and etched for determination of the dendrite structure and the position of the dendrite tips thus located. A typical interface is shown in

Fig 3. The primary dendrite arms were marked using a micro-hardness indenter and the sample polished to remove the etched surface. The carbides were then measured, using the dendrite arm locations together with the equivalent micrographs of the etched surface to ensure that only the blocky primary carbides were included in the measurements. The "carbide size" is expressed as the average maximum linear dimension of the five largest carbides measured in 40 separate fields. This technique was adopted due to the difficulty of devising an automated method for the identification of the primary carbides. Samples from all of the compositions shown in Table 1 were examined by DTA to determine the carbide start temperature with the results shown in Fig 4

In the case of the industrial ingot samples a macrosection was cut axially from an ESR ingot top, using ingots in which melting had been terminated abruptly during steady-state melting with the application of any hot-top procedure. This technique enabled the accurate determination of a "pool profile" for the conditions used. Micro samples were extracted from the ingot sections by EDM, polished and then treated as above for carbide examination.

Results of the determinations are tabulated (Tables 2 & 3) and also shown graphically together with previous results from the literature (8) in Figs 3, 4 and 5.

Discussion

It can be seen from the results that the size of the blocky carbides is a function of the solidification conditions, as has been previously indicated. As might be anticipated, the size distribution is related to the available volume from which precipitation can take place (represented by the dendrite arm spacing) and the time available for growth (represented by the solidification growth rate). The two conditions are conveniently combined as the local solidification time for the purposes of comparison with ingot thermal regime computations. The form of the size dependence on composition follows the expected path in that as the total Nb and C available for precipitation is decreased, the range of carbide size is also decreased. It is found that the precipitation start temperature is not strongly dependent on the Nb/C ratio, but carbide precipitation (in the form of blocky carbides) becomes absent abruptly when the solubility limit is not exceeded. In the relationships shown in Figs 4 & 5, the precipitation start temperature falls from 1280C to 1260C (accompanied by a small decrease in the size range) as the composition approaches the solubility limit, but becomes abruptly undetectable by DTA when the composition falls below the solubility limit.

The relation of the present results to the manufacture of remelt ingots of IN718 lies in the practicality of two items; the extent to which we may reduce the content of C and Nb, and the extent to which Nb and C segregation in the final remelting solidification can be controlled. In respect of the former aspect, work on the mechanical properties of the alloy indicates that the role of the Nb content is very complex and lies not only in the formation of the delta phase, but also in the formation of Laves phases. If we attempt to reduce the carbon content without the parallel balance in Nb content, the alloy will not only be deficient in delta but will also be prone to the formation of eutectic Laves. In summary, given our present knowledge of the effect of composition variation on the alloy behaviour it does not appear practical to reduce the Nb/C contents to the point where primary carbide does not form. Also, even given the absence of primary carbide, the

problem of carbide stringers originating in the eutectic areas would still remain. The conclusion to be drawn is that the control of the solidification parameters is the critical point leading to alloy structure refinement rather than composition manipulation.

The control of the solidification process requires definition of the process thermal regime. To this end, a number of modeling exercises have been carried out on both the VAR and ESR processes. The model structures now available have usually been checked for accuracy against the "pool profile" obtained by ingot sectioning, with the assumption that this line represents the liquidus line during melting. By suitably adjusting the model input conditions, an excellent fit has been found for many cases. However, the form of the heat release during solidification, and also the temperature gradients close to the liquidus line have a large influence on both the rate, "R", and the temperature gradient, "G", in the liquid + solid zone of the ingot. Fitting a model to give a good result for the liquidus line does not uniquely define these parameters and it is highly desirable to have at least one other experiment verification of the model output. The most commonly-used second method is to measure the primary and secondary dendrite arm spacings, which are controlled by a combination of G and R. A typical relationship for IN718 would be:

Secondary spacing (micron) = 19.5 x (Cooling rate,
$$C/s$$
)^{-0.63}(1)

The method has the potential to provide the required verification, but suffers from two drawbacks: a lack of precision in the experimental determination of the average spacings and the rather low dependence of the spacings on G and R. An alternative method lies in the use of the primary precipitate size to define G and R; in the case of IN718 the technique would use the NbC precipitation described above.

In this work, we have used ESR modeling as the test vehicle for the method, using the modeling method of Kelkar et al (9) to produce an output describing the G and R values of the solidifying region. A schematic of the output is shown in Fig 6 and the equivalent ingot section in Fig 7.which was used as described above to obtain the carbide size distribution profile. The values obtained experimentally for the carbide size variation with cooling rate agree well with the cooling rates predicted for the model ingot. Although the method is evidently valid for the ingot model chosen, there remains the experimental difficulty of determining the primary carbide size, since the primary carbides themselves are difficult to identify in any automated assessment technique. In addition, the method has limited industrial applicability since the property-determining carbide stringers are not isolated primary carbides but instead are the carbide + gamma + Laves-forming pools of segregated liquid remaining at the end of solidification (2,3). The size distribution of the latter has a relationship to both composition and dendrite spacing (hence to cooling rate) which has not yet been determined.

Conclusions

We may conclude from the above study that the primary carbide size distribution can be used as a monitor of cooling rate in IN718 remelted ingots, provided that the correct composition relationships are taken into account. The results also demonstrate that there is good agreement with computed cooling rates. The relationship of the findings to the mechanical properties of IN718 is not direct due to the fact that the

carbide stringer formation in the final product is mainly the result of the solidification of the final liquid remaining at the eutectic rather than of the isolated primary carbides.

Acknowledgements

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References

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Table I Alloy compositions examined, base IN718; 18%Fe, 0.0 5%Si, 18.25%Cr, 0.57%Al, 1.02%Ti, 2.8%Mo bal Ni

Wt%Nb	Wt%C
5.63	0.05, 0.03, 0.015, 0.01, 0.007,
	0.005
5.40	ibid
5.25	ibid
5.10	ibid
4.90	ibid

Table 2 ESR Secondary Dendrite Arm Spacings, (500mm dia ingot)

Ingot radial position (mm)	Dendrite spacing (microns)
centreline	130
50	105
100	104
150	94
200	81
edge	68

Table 3 Carbide size distribution in the ESR ingot

Radial position (mm)	Average maximum carbide size (microns)
centreline	24.9
50	23.6
100	22.4
150	15.7
200	13.4
edge	12.4

<u>Figures</u>

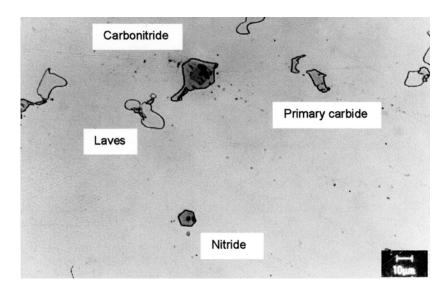


Figure 1 (a) Carbide morphology in IN718 (6)

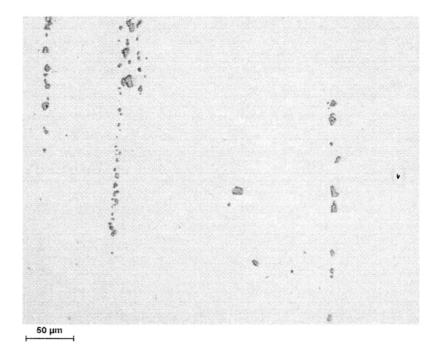


Figure 1 (b) Carbide "stringers" in wrought product IN718

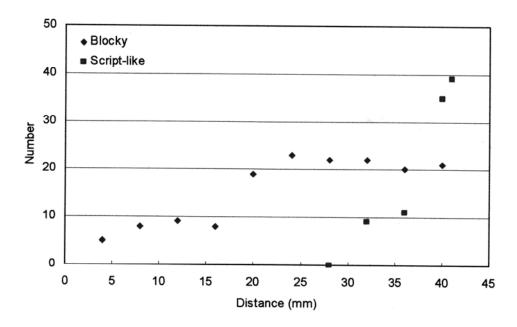


Figure 2 Carbide precipitation in IN718 showing precipitation start for "blocky" or primary, carbides as a function of distance from liquidus line in a directionally solidified sample of IN718 (6).

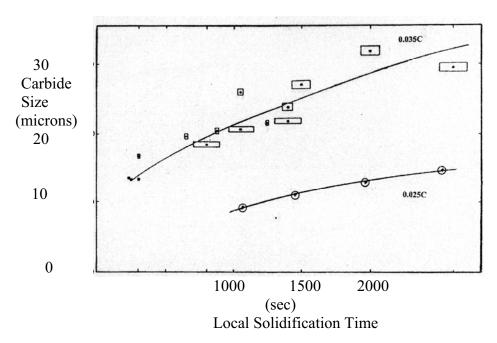


Figure 3 Primary carbide size as a function of alloy composition and local solidification time

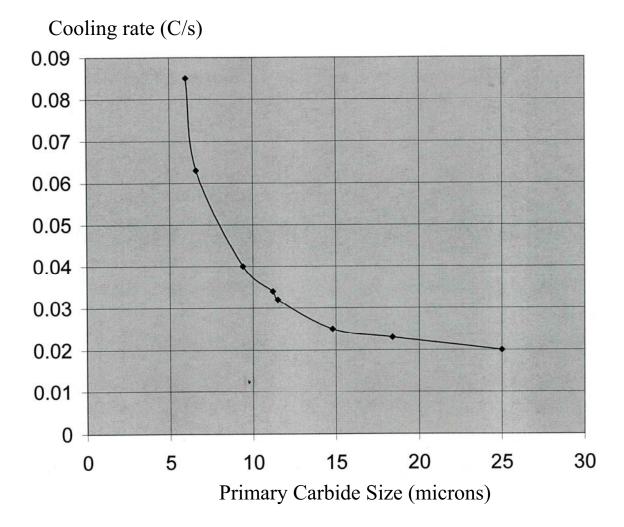


Figure 4 (a) Primary carbide size as a function of cooling rate

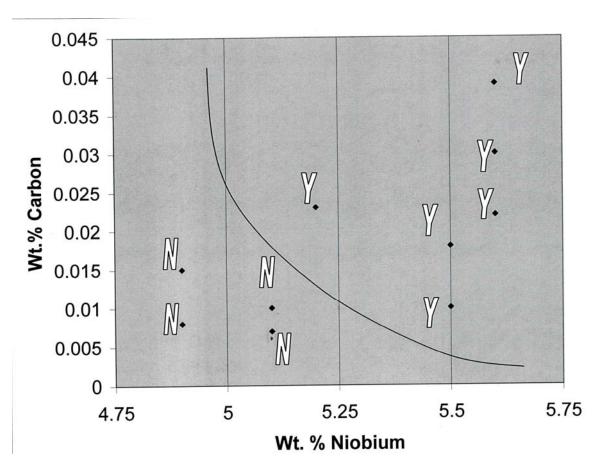


Figure 4 (b) Primary carbide precipitation as a function of composition

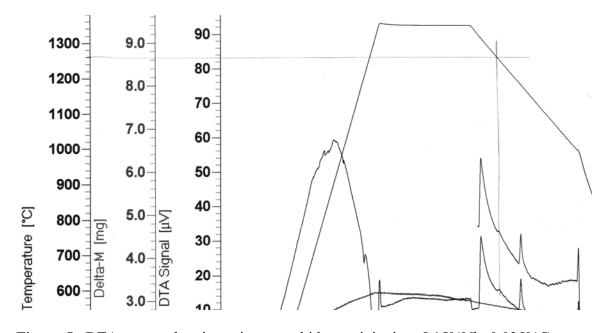


Figure 5 DTA curves showing primary carbide precipitation, 5.15%Nb, 0.035%C

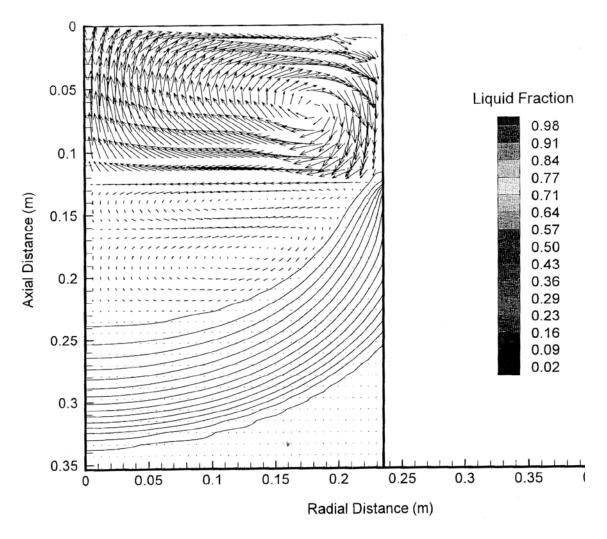


Figure 6 Model result for an ESR ingot of IN718 melted at a high rate (5 Kg/min) (9)

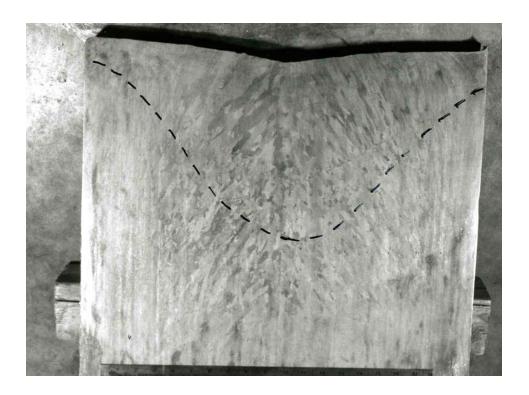


Figure 7 Ingot section corresponding to the conditions of Fig 6 above.