On incipient melting during high temperature heat treatment of cast Inconel 738 superalloy

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Cast Inconel 738 superalloy is one of the most widely used alloys used to manufacture hot section components of aircraft engines and gas power generator turbines, because of its high creep rupture strength and remarkable hot corrosion resistance. This heat resistant alloy derives its good elevated temperature strength primarily from precipitation hardening by ordered $L1_2$ intermetallic $Ni_3(Al,Ti)$ γ' phase. In a complex casting alloy like IN 738, the character of γ' precipitate particles is likely to be significantly variable due to dendritic microsegregation that occurs during ingot solidification. The dendritic core γ' particles in IN 738 are richer in negatively segregating Al atoms ($k > 1$) and exhibit solvus temperatures of between 1120–1130 °C, as compared to interdendritic particles, which are enriched with positively segregating Ti atoms, ($k < 1$) and have higher solvus temperatures between 1170–1180 °C [2]. In addition, γ-γ' eutectic constituent also forms along the interdendritic regions. Solution heat treatment at temperatures high enough to dissolve all the primary γ' particles and homogenize the alloy for subsequent γ' re-precipitation in the form of uniformly distributed fine γ' precipitates has been severely restricted in this alloy due to the occurrence of incipient melting [3]. Consequently, the standard solution heat treatment generally adopted for this alloy has been limited to 2 hrs at 1120 °C, which results only in a partial homogenization by exclusive dissolution of dendritic core γ' particles. The aim of the present work was to investigate the cause and nature of the limiting incipient melting, which should aid development of appropriate higher solution heat treatment schedule. This was performed by examining the microstructure of as-cast IN 738 superalloy for the presence of microconstituents that might be responsible for incipient melting at temperatures below what is needed to dissolve the interdendrtic γ' precipitate particles.

The microstructure of commercial investment cast IN 738 alloy with a chemical composition of (wt%) 0.11C, 15.84Cr, 8.5Co, 2.48W, 1.88Mo, 0.92Nb, 0.07Fe, 3.46Al, 3.47Ti, 1.69Ta, 0.04Zr, 0.012B balance nickel, was examined by optical and a scanning electron microscope (SEM) equipped with an ultrathin window energy dispersive X-ray spectrometer. Careful microstructural investigations of the alloy using backscatter and secondary electron imaging modes in SEM revealed the occurrence of different solidification products in front of some of the γ-γ' eutectic (Figs 1 and 2). The lamellar morphology of the solidification product suggests that it was formed by eutectic transformation involving at least ternary and quaternary eutectic reactions. The constituent phases of the eutectic-like products are delineated in the atomic number based backscatter electron SEM micrograph, shown in Fig. 2. Chemical composition of the two main phases, and that of another phase which was occasionally observed in association with gamma phase within the eutectic-like products, as determined by energy dispersive X-ray microanalysis, are shown in Table I. Significant boron concentration was detected in the Cr-Mo rich particle by the Oxford ultrathin window EDS detector. High Cr and Mo peaks observed in the EDS spectra in this work (Fig. 4) are also characteristic features of M_3B_2 boride phase in other Ni base superalloys [4]. M_3B_2 boride has been reported in heat-treated IN 738 superalloy by X-ray diffraction analysis [5]. The composition of Ni-Zr and Ni-Ti rich intermetallic phases suggests that they are based on Ni_5Zr and Ni_3Ti respectively. Ni_5Zr has been reported to be formed during directional solidification of Zr doped Ni-based MAR-M200 superalloy [6].

On the basis of differential thermal analysis (DTA), it has been suggested that ingot solidification of IN 738 is completed by the γ-γ' eutectic transformation at around 1198 °C [7] and 1230 °C [8]. Rosenthal *et al.* [2], however, indicated that the γ-γ' eutectic reaction in this alloy occurs over a range of temperatures, which could be as low as below 1180 °C. Zhu *et al.* [9] in their study of solidification behavior of some Ni-based superalloys reported that in an alloy, which is close to IN 738 in composition, 90% of the liquid had

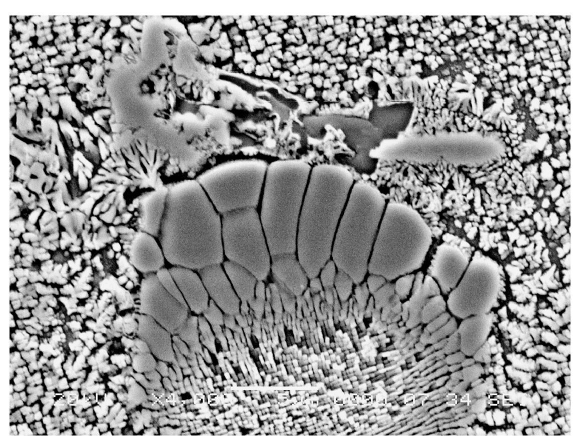

Figure 1 SEM secondary electron image showing eutecticlike constituent ahead of γ-γ' eutectic.

TABLE I Chemical composition of terminal solidification constituents

Elements	Ni-Zr intermetallic (at.%)	Cr-Mo boride phase (at.%)	Ni-Ti intermetallic (at.%)
Al	0.7	–	4.7
Ti	2.5	2	11.7
Cr	3.2	67.9	5.1
Co	7.4	1.73	6.6
Ni	67	4.6	66.3
Zr	16	0.9	2.18
Nb	2.5	2.4	1.8
Mo	–	16	0.28
Ta	0.8	–	0.95
W	–	4.45	0.34

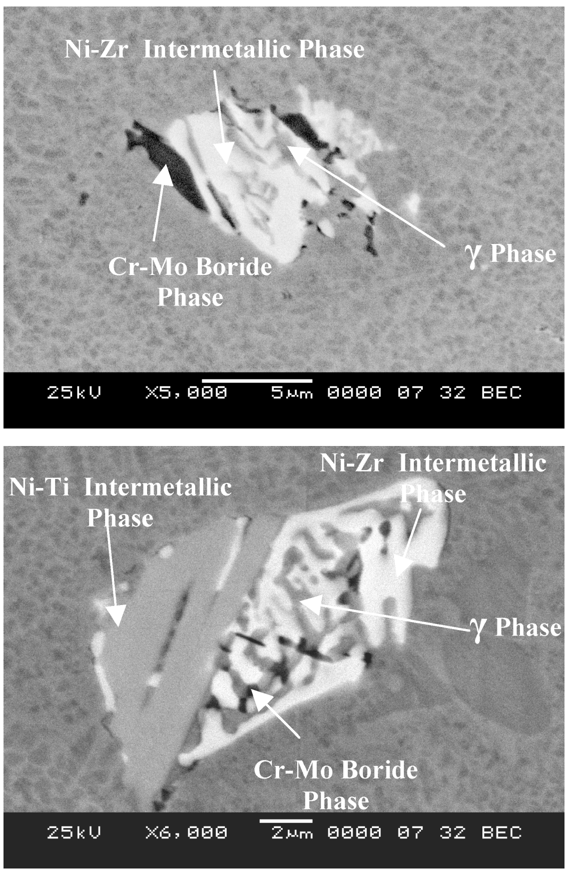

Figure 2 (a) SEM backscatter electron image of ternary eutectic-like constituent. (b) SEM backscatter electron image of quaternary eutectic-like constituent.

solidified before 1230 °C but the remaining liquid did not solidify until the temperature was about 1120 °C. Zou *et al*. [10] in a separate work, to study the solidification behavior of another derivative of IN 738 using a slow heating rate of 0.28 K/s in DTA, reported that the terminal eutectic reaction temperature in the alloy was 1094 °C. The nature of such terminal eutectic was, however, not discussed. The results of these investigations [9, 10] thus suggest that, contrary to the generally accepted view that solidification of IN 738 ingot is completed by the γ-γ' eutectic reaction at around 1180–1198 °C, a different eutectic transformation could be occurring in this alloy at a significantly lower temperature.

TABLE II Element partition coefficients in IN 738.

Element	Experimental *k* [10]
Al	1.2
Co	1.1
Cr	1.05
Ni	1.05
Zr	0.06
Nb	0.4
Ti	0.6
Ta	0.7
Mo	0.85
W	1.4
Element	**Theoretical *k* [11]**
B	0.0082
S	0.01
C	0.3

Solidification behavior and the accompanying products that formed in IN 738 alloy can be reasonably understood by the knowledge of solute partition coefficients of the alloying elements during solidification. Taha *et al*. [11] have determined solidification partition coefficients (k) of metallic elements in IN 738. Their results and those of Nakao *et al*. [12] on theoretical k values of B, S, and C, as estimated from their Ni-X binary phase diagrams, are listed in Table II. During solidification, the first solid to form from the liquid was in the form of gamma dendrites. As these dendrites grow during cooling, dendritic microsegregation occurs with interdendritic liquid getting enriched with positively segregating elements ($k < 1$). Supersaturation of the liquid with these solutes due to continual enrichment on cooling, would invariably result in the formation of secondary solidification constituents such as MC carbides, M_2SC sulphocarbide, and γ-γ' eutectic in the interdendritic regions. During the formation of γ-γ' eutectic in the later stages of solidification, which has been reported to be occurring over a range of temperatures [2], solute partitioning occurs with elements having low solubility in γ and γ' phases being rejected into the residual liquid pool ahead of the γ-γ' eutectic. Zr and B are known to have low solubility in γ and γ' phases and as such, would partition into the residual liquid pool. Increase in concentrations of the these elements, which are also known to be melting point depressants in Ni, would result in further lowering of the freezing temperature of the residual liquid.

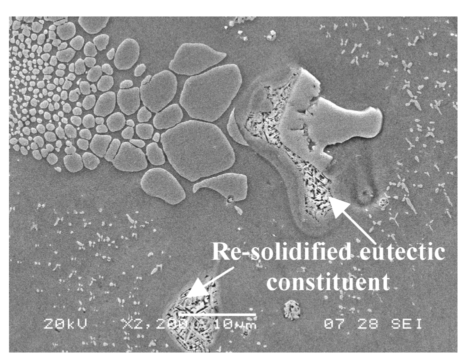

Figure 3 Melting of terminal solidification constituents at 1155 °C.

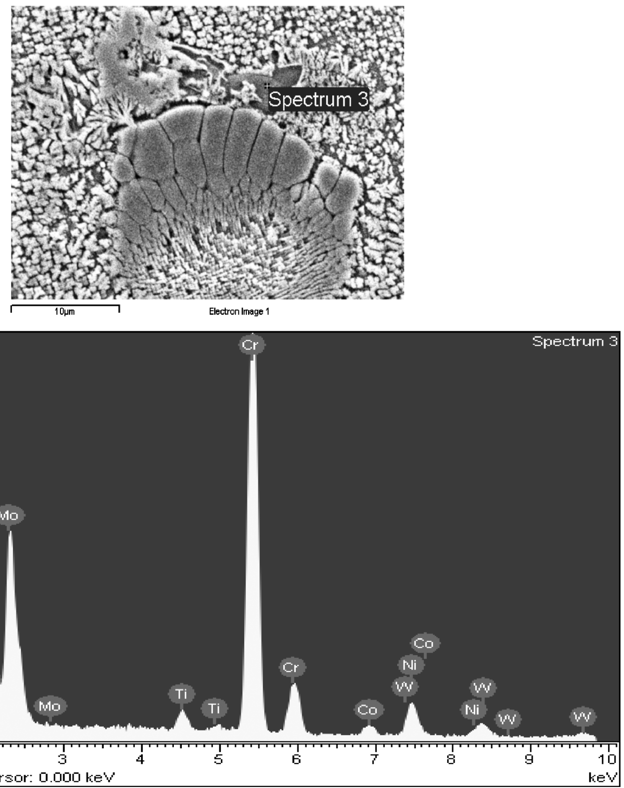

Figure 4 EDX spectrum from Cr-Mo boride.

Cr and Mo, which have high affinity for boron, have also been found to exhibit positive segregation ($k < 1$) into the liquid during γ-γ' eutectic formation [9]. On continuous cooling to the invariant freezing temperature for a given alloy composition, below which liquid phase could no longer exist, the remaining liquid would most likely complete solidification by terminal eutectic transformation which may involve formation of multiple constituent phases. This could then explain the formation of ternary eutectic-like products containing γ phase, Ni-Zr intermetallic, and Cr-Mo rich boride observed in the present work. In addition, it is known that once the ratio of Ti/Al exceeds 3:1, Ni_3Ti would form preferentially, which could also account for the formation of what appears to be Ni_3Ti based intermetallic in the quaternary terminal eutectic-like product, considering the positive segregation behavior of Ti ($k = 0.6$) versus negative segregation of Al ($k = 1.2$). Confirmation of the exact nature of these constituent phases is, however, being pursued through analytical transmission electron microscopy involving a detailed electron diffraction analysis. However, what is clear at this time is the occurrence of a solidification product, which is believed to form in this alloy by terminal eutectic transformation and as such, could be responsible for the limiting incipient melting.

The low melting temperature of the terminal solidification product was confirmed by heating a cylindrical as-cast sample of diameter 6 mm and length 115 mm in a Gleeble thermomechanical simulator system to 1155 °C (15 °C below 1170 °C the quoted solvus temperature for interdendritic γ' precipitates) at a rate of 38 °C/min, held for 1 s and then water quenched. Evidence of melting of the terminal solidification constituents was observed, as shown in Fig. 3. Incipient melting of the terminal solidification product, as observed in this work at 1155 °C, could thus be the major factor limiting higher solution heat treatment temperature (>1170 °C) needed to dissolve the interdendritic γ' precipitates, as well as eutectic γ' particles in cast IN 738 superalloy. It has been reported in DS MAR-M200, that annealing at temperatures below Ni-Ni_5Zr eutectic temperature eliminates the Ni_5Zr phase through a solid state reaction. In the present work, the Ni-Zr intermetallic phase was not observed after solution heat treatment at 1120 °C for 2 hrs, while the Cr-Mo rich boride particles were observed to persist after this heat treatment.

This suggests that the constituent phases of the terminal solidification product in this alloy could respond differently to heat treatment. Therefore, an adequate understanding of the response of the terminal solidification microconstituents to thermal treatment as observed in this work is crucial for developing appropriate higher homogenization solution heat treatment for IN 738 superalloy. This will be beneficial towards achieving uniform dispersion of fine γ' precipitates for optimum creep rupture strength of cast components during service. In addition, it is also pertinent for producing suitable microstructure that will be resistant to cracking during welding of IN 738 superalloy, which is known to be highly susceptible to heat affected zone (HAZ) cracking. Eutectic melting of the terminal solidification constituents during fabrication welding will contribute to sub-solidus intergranular liquation, which is known to reduce resistance to HAZ cracking.

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