THE METALLURGICAL ASPECTS OF HOT ISOSTATICALLY PRESSED SUPERALLOY CASTINGS

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The effects of HIP and post-HIP heat treatment on the microstructures and mechanical properties of IN-713C and MAR-M-246 were investigated. IN-713C was found to be totally amenable to HIP, i.e., fully dense castings with controlled γ' size and distribution were developed through HIP and heat treatment without deleterious grain boundary carbide reactions or TCP phase precipitation. MAR-M-246, on the other hand, was found to be somewhat less amenable to HIP. Fully dense MAR-M-246 castings were achieved; but deleterious HIP-induced carbide reactions, which could not be ameliorated through heat treatment, were experienced. These results and observations were generalized to classify the HIP response of cast nickel-base superalloys depending on their Mo + W contents.

INTRODUCTION

The microstructures, hence properties, of cast alloys are determined by composition and solidification kinetics. Solidification kinetics are best described in terms of cooling curves such as illustrated in Figure 1, which simply describe metal temperature at a fixed position in the casting as a continuous function of time. In the case of nickel-base superalloys, the pour temperature determines primary carbide morphology and grain size; whereas, the freezing time determines the size and distribution of primary γ' and dendrite arm spacing. Secondary carbide and γ' morphologies are determined by the post-solidification cooling rate but may be modified by subsequent heat treatment.

Microporosity, a microstructural defect that invariably degrades mechanical properties, is inevitable in nickel-base superalloy castings. Microporosity can be minimized by

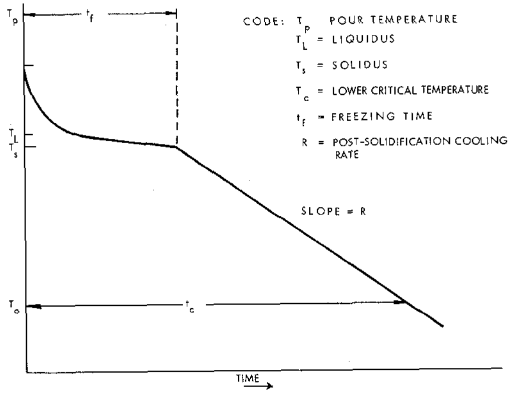

Fig. 1. Cooling Curve

adjusting composition and/or casting practice so as to increase the local liquid pressure during solidification (1). However, hot isostatic pressing (HIP) at temperatures approximating 95% of the absolute melting temperature is the only effective means of totally eliminating microporosity.

Microporosity elimination via HIP does not a'priori improve mechanical properties inasmuch as microstructure may be significantly altered and new phases may form during the HIP process. Homogenization occurs; secondary phases are solutioned and reprecipitate in a manner peculiar to the cooling rate of the HIP unit; and primary phases may, depending on composition, also be altered. Secondary-phase morphologies can be optimized through post-HIP heat treatment; but MC and γ' primary phases, once modified during HIP, cannot be returned to original morphologies.

This paper describes the effects of HIP on the microstructures and properties of IN-713C and discusses the effects of HIP on the microstructure of MAR-M-246.

IN-713C MICROSTRUCTURE AND PROPERTIES

IN-713C (Ni-13Cr-6Al-4.4Mo-2.3Cb-.75Ti-.15C), a "typical" cast nickel-base superalloy consisting of 50% γ' ($Ni_{2.95}$-$Cr_{.09}$-$Al_{.78}$-$Ti_{.06}$-$Cb_{.08}$-$Mo_{.06}$) in a γ matrix (Ni-22.6Cr-6.6Mo-3.9Al-.1Ti), is used extensively in gas turbine blade, vane, and integral wheel applications (2). Cast IN-713C also contains primary MC carbide phases which decompose during cooling or heat treatment into $M_{23}C_6$ depending on temperature and time.

The effects of heat treatment on the microstructure of IN-713C have been studied extensively (3,4,5). The size and distribution of the γ' as well as the carbide and boride phases are affected by heat treatment.

The mechanical properties of cast IN-713C vary depending on solidification kinetics. Rapid solidification (freezing time < 1 minute) generally produces the highest tensile and stress-rupture properties. Slow post-solidification cooling rates (< 10°C/min.) significantly reduce the high-temperature, stress-rupture properties of as-cast IN-713C and slightly degrade room-temperature tensile properties. Low cycle fatigue strength, and fracture toughness are thought to be influenced by microporosity more than by solidification kinetics.

EFFECT OF HIP ON MECHANICAL PROPERTIES

The effects of HIP on the mechanical properties of IN-713C were determined by comparing selected mechanical properties of specimens machined from various castings. One-half of each casting was tested in the cast and heat-treated condition. The other half of each casting was HIP'ed at 1232°C, 10.9 kg/mm^2 for 4 hours and tested in both the as-HIP'ed and HIP'ed and heat-treated conditions. Heat treatment conditions (955°C, 1 hr./1150°C, 8 hrs./955°C, 1 hr.) were selected to simulate typical stabilization/coating/aging cycles as might be used by gas turbine engine manufacturers.

The maximum microporosity levels in the hub and disc sections of the IN-713C integral wheel castings used in this investigation were typically 1.5% and 0.4%, respectively, with void diameters ranging up to .06mm. Detailed metallographic examination indicated that these microporosity levels were totally eliminated under the HIP conditions used in this investigation.

Measured mechanical properties are compared in Table 1. Room-temperature tensile ductility and fracture toughness were somewhat improved by HIP, but intermediate-temperature low cycle fatigue was substantially increased by HIP and post-HIP heat treatment. High-temperature, stress-rupture properties, on the other hand, were virtually unaffected by HIP. Preceding the heat treatment sequence with a brief (1/2 hour) return to the HIP temperature followed by a rapid air cool (RAC) surprisingly improved "microporosity-sensitive" mechanical properties but did not improve high-temperature creep rupture properties.

TABLE 1: IN-713C MECHANICAL PROPERTIES

Property	Cast + H.T.	HIP	HIP + 2250F, 1/2 Hr., RAC + H.T.
R. T. Tensile	153	155	160
0.2% Yield Strength (Kg_2/mm^2)	166	176	169
Ultimate Tensile (kg/mm^2)	3	5	4
Elongation (%)	4	5	8
Reduction in Area (%)			
982C/15.5 kg/mm^2 Stress-Rupture			
Life (Hrs.)	--	43	38
Elongation (%)	--	9	10
649C/156 to 0 kg/mm^2 Low Cycle Fatigue N_f	2570	4410	6793
Fracture Toughness K_Q ($kg/mm^2 \sqrt{mm}$)	386	—	436

EFFECT OF HEAT TREATMENT ON MICROSTRUCTURE

The γ' in HIP'ed and heat-treated (1080°C, 10 min., RAC) IN-713C was coarse (0.5 to 2.5μ) and irregular in shape (see Figure 2). Conversely, the γ' in HIP'ed and heat-treated IN-713C in which the heat treatment step was preceded by a 1/2-hour return to the HIP temperature followed by a rapid air cool was uniformly fine (see Figure 3). These observations suggest that the γ' in IN-713C is totally solutioned at the HIP temperature. The coarse γ' in HIP'ed IN-713C associated with the slow cooling rates (∿ 30°C/min.) of the HIP unit can be modified, virtually at will, through post-HIP heat treatment.

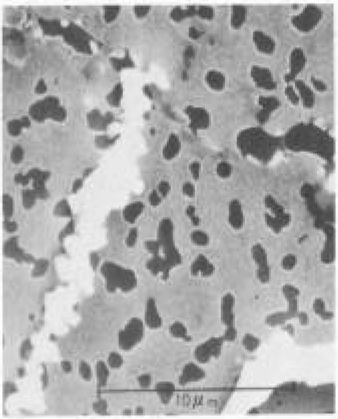

Fig. 2. HIP'ed IN713-C + 1080°C, 10 min.

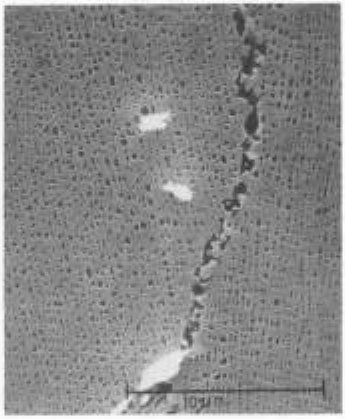

Fig. 3. HIP'ed IN-713C + 1232°C, 1/2 hr., RAC + 1080°C 10 min.

(Note: Surface preparation of all SEM specimens included in this study was such that carbides appear bright and γ' appears dark.)

High-temperature exposure (∿ 1050 to 1150°C), such as might occur during a coating operation, can develop γ' morphologies ranging from fine and uniform (see Figure 4) to duplex and irregular (see Figure 5). Grain boundary reactions and further decomposition of primary MC carbides also occur during high-temperature exposure. However, no evidence of continuous grain boundary precipitation, such as might be deleterious to mechanical properties, was observed in any of the heat treatment sequences included in this investigation.

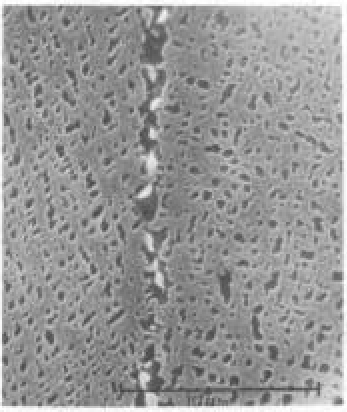

Fig. 4. HIP'ed IN-713C + 1232°C, 1/4 hr. RAC + 1080°C, 4 hrs. + 760°C, 8 hrs.

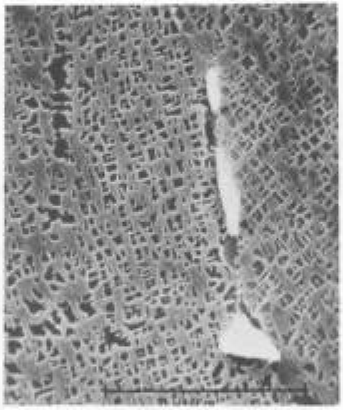

Fig. 5. HIP'ed IN-713C + 1232°C, 1/2 hr. RAC + 1150°C, 8 hrs. + 955°C, 1 hr.

No plates of sigma and/or M_6C were observed in either HIP'ed or HIP'ed and heat-treated IN-713C. The absence of detrimental sigma and/or M_6C phases is attributed to the homogenizing effects of HIP and the low molybdenum content of the alloy. It would appear that the noted changes in the mechanical properties of the HIP'ed and heat-treated IN-713C were related to micropore closure and changes in the amount, size, and distribution of the γ'.

EFFECT OF HIP ON THE MICROSTRUCTURE OF OTHER NICKEL-BASE SUPERALLOYS

Nickel-base superalloys containing large amounts of refractory elements Mo + W may behave in an erratic way after HIP and post-HIP heat treatments. This behavior is associated with possible formation of M_6C and sigma phases in the temperature range of 980 to 1200°C.

The effects of HIP (1205°C, 10.9 kg/mm^2, 4 hrs.) and heat treatment (1093°C, 2 hrs. + 843°C, 50 hrs.) on the 760°C 68.6 kg/mm^2 creep-rupture properties of MAR-M-246 (Ni-10Co-10W-9Cr-5.5Al-2.5Mo-1.5Ti-1.5Ta), a nickel-base superalloy typical of the "high refractory type", are summarized in Table 2. In some cases, the post-HIP heat treatment was preceded by a 2-hour exposure at 1218°C, a temperature higher than the HIP temperature, followed by a rapid air cool.

The data given in Table 2 represents an average of a number of heats, and it appears that the HIP treatment was very beneficial to the creep rupture properties of MAR-M-246. However, HIP did not reduce the scatter in rupture life and ductility of this alloy.

Structural studies were carried out on a number of samples representing the three thermal conditions described above. The as-cast plus heat-treated sample showed the normal MC plus some precipitation in the grain boundaries of M_6C formed at 1093°C and $M_{23}C_6$ phase formed at 863°C. Typical microstructures found in this condition are seen in Figure 6.

TABLE 2: MAR-M-246 760C/68.6 kg/mm^2 CREEP RUPTURE PROPERTIES

Condition	Life (Hrs.)	Elong. (%)	RA (%)	Prior Creep (%)
As-Cast + 1093C, 2 Hrs. + 843C, 50 Hrs.	81	2.4	4.3	2.0
HIP'ed + 1093C, 2 Hrs. + 843C, 50 Hrs.	68	2.7	3.4	2.4
HIP'ed + 1218C, 2 Hrs. RAC + 1093C, 2 Hrs. + 843C, 50 Hrs.	123	3.5	5.7	3.2

Compared to the as-cast plus heat treated condition, the HIP'ed plus heat treated samples showed that the precipitation in the grain boundaries was very much heavier. The MC showed evidence of breakdown, and a plate-like phase was present in the matrix. A number of the plates were growing out of the MC phase in the grain boundaries and matrix. X-ray diffraction studies showed the unmistakable presence of M_6C phase in addition to the MC phase. No $M_{23}C_6$ was detected. It appears that both the grain boundary and plate-like phases are M_6C. Figure 7 shows the typical structures found. SEM fractographic studies showed that no porosity was present. A part of the fracture path was through the plate M_6C phase which showed cracking.

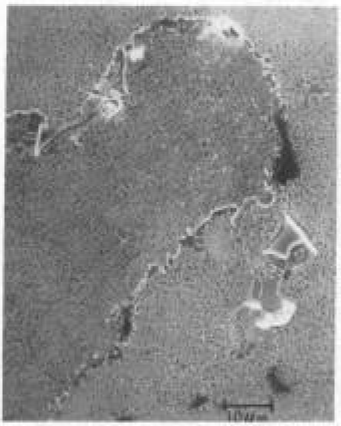

Fig. 6. MAR-M-246 cast and heat treated

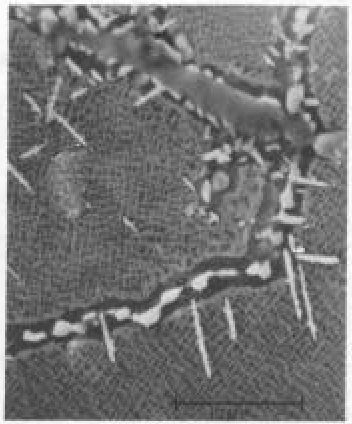

Fig. 7. MAR-M-246, HIP'ed and heat treated

The HIP'ed samples which were treated at 1218°C, 2 hrs., RAC prior to heat treatment were microstructurally similar to the HIP plus heat treated samples except there were more variations in the number of plates formed. Those samples which showed the least number of plates gave the longer rupture life and the highest ductility. Thus, from the structural studies and mechanical properties of the various MAR-M-246 samples, it appears that the M_6C plates account for the scatter in mechanical properties. Originally, it was thought that the variations in the amount of plates were mainly attributed to subtle differences in the temperature control during the post-HIP heat treatment, especially the 1093°C coating step. However, recent results of other high-temperature studies of nickel-base alloys with similar refractory contents as in the MAR-M-246 showed that the M_6C phase is stable and can form at temperatures of 1205°C or higher.

In order to confirm that M_6C plates are stable in MAR-M-246 in the HIP and post-HIP heat treatment temperature range, samples of MAR-M-246 in various thermal conditions were exposed for 4 hours at 1218°C in vacuum and examined. Microstructural and x-ray diffraction analyses showed that M_6C was present after the 1218°C exposure and was not solutioned. The amount of M_6C plates was greater in those samples which previously had been HIP'ed and/or post-HIP heat treated. The authors suspect that the increase in the amount of M_6C plates occurred as a result of breakdown of Ti-rich primary MC carbides.

DISCUSSION

Microporosity in typical nickel-base superalloys such as IN-713C and MAR-M-246 is totally eliminated by HIP at sufficiently high temperatures and pressures. As a rule, "microporosity-sensitive" properties improve as a result of HIP. However, mechanical property scatter remains a problem with HIP'ed MAR-M-246.

In IN-713C the γ' size and distribution can be controlled virtually at will through post-HIP heat treatment. MAR-M-246, on the other hand, with its high refractory content is subject to HIP-induced M_6C reactions. Extreme care and control of the temperatures of HIP and post-HIP heat treatments is necessary to minimize the size and amount of the M_6C plates formed in the matrix and grain boundaries. Long and repetitive exposures in a temperature range of 1205 to 1218°C produces increased amounts of M_6C plates.

CONCLUSIONS

The results of this investigation suggest that nickel-base superalloys with reasonably low refractory (Mo + W) solute contents are fully amenable to HIP and post-HIP heat treatment; whereas, nickel-base superalloys with high refractory solute controls are marginally responsive and require extreme care in HIP and post-HIP heat treatment. The authors suggest that alloying additions which improve the high temperature stability of primary MC phases might relax the stringent heat treatment requirements associated with nickel-base superalloys with high refractory solute contents. Primary MC stabilization would, in effect, preclude or minimize deleterious plate-like M_6C precipitation in high refractory solute content alloys. Tantalum, columbium, and particularly hafnium, are preferred alloying additions in this regard. Titanium, perhaps the most common "MC former" utilized to date, is less desirable because titanium-rich MC carbides are less stable and introduce deleterious M_6C reactions. Future nickel-base superalloy development studies should consider HIP response and utilize titanium and/or high refractory solute contents only in the presence of strong MC carbide formers.

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