Materials Transactions, Vol. 49, No. 1 (2008) pp. 193 to 201

EXPRESS REGULAR ARTICLE

Effect of Hot Isostatic Pressing on Microstructure and Mechanical Properties of CM-681LC Nickel-Base Superalloy Using Microcast

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This study investigates how hot isostatic pressing (HIP) affects the microstructure and fracture modes of CM-681LC superalloy. As-cast test bars with grain size of 80 µm were prepared using the fine-grain process followed by HIP. Experimental results indicate that micropores formed during solidification and contraction degrade the tensile strengths and elongations of the fine-grain CM-681LC superalloy before HIP. The area fraction of micropores was reduced from 0.2% to 0.06% following HIP. Scriptlike MC carbides decompose into particlelike $M_{23}C_6$ carbides during HIP, revealing that HIP refines and spheroidizes the carbides. Eliminating the micropores and refining the carbides increases the mechanical strength by up to about 9% and the elongation by over 10% in room- and high-temperature tensile tests. The fracture analyses after tensile tests of the fine-grain test bars reveal that the microporosity and the scriptlike carbides at the grain boundaries are the main causes of the fracture of the test bars before HIP. The fracture mode of the fine-grain test bars following HIP, according to the tensile test, is typically intergranular because the micropores are eliminated and the carbides are refined. Since the elimination of the micropores and refinement of the carbides by HIP effectively improves the tensile strength and elongation, the fine-grain casting yields favorable mechanical properties.
[doi:10.2320/matertrans.MER2007088]

(Received April 20, 2007; Accepted October 29, 2007; Published December 12, 2007)

Keywords: *hot isostatic pressing, fine-grain, superalloy, carbide, tensile test*

1. Introduction

CM-681LC superalloy is a nickel-base superalloy developed in recent years. This superalloy exhibits superior mechanical properties, and is particularly useful in high-temperature, high-strain applications, such as aircraft gas turbine engines. This superalloy exhibits increased grain boundary strength and ductility when its microstructures are stable. The enhanced grain boundary strength and ductility allow both directionally solidified columnar grain casting and equiaxed casting of an integrally blade cast turbine wheel with superior capabilities at a substantially lower cost than conventional turbine wheels.[1)]

The fine-grain process (FGP) was developed to increase the strength, the elongation and the fatigue durability of conventional castings. Fine-grain castings have such advantages as refined carbides and precipitates, enhanced strength and elongation, improved low cycle fatigue life, and an isotropic microstructure and properties.[2)] Thus, fine-grain processes are beneficial for the strength and fatigue life of the turbine disk and integral wheels at moderate temperatures (427–760°C). Microcast developed by Howmet Corporation is one of the FGPs with controlled low superheat and a high heat extraction rate.[3)]

However, the fine-grain process brings about poor strength, low elongation, short creep life and short fatigue life, when micropores are present in the castings following the solidification and contraction of liquid metal. The micropores in castings are crack initiation sites and govern the propagation paths. Hot isostatic pressing (HIP) has been established to eliminate micropores and other defects from castings, but improving the microstructures and mechanical properties of superalloys remains an important area of study.[2–8)]

Although carbide morphology critically governs the performance of a superalloy, the effect of carbides in superalloys is unclear since the microstructures of superalloy are complex. Therefore, the main aim of this investigation is to study the effect of HIP on the micropores and carbide morphologies of the CM-681LC superalloy formed by the fine-grain process. This work also discusses the effects of micropores and carbides on the mechanical performance and fracture mode.

2. Experimental

CM-681LC superalloys were remelted in a vacuum furnace and then cast into test bars using the Microcast process, which is a fine-grain process with a low pouring temperature. The temperature gradient in the Microcast process was reduced to limit the growth of the grains. The pouring and mold temperatures were 1380°C and 1100°C, respectively. X-ray fluorescence analysis (XRF) was used to determine the compositions of the test bars. Some of the test bars were treated with HIP for comparison. The grain sizes of the test bars were observed by optical microscopy and measured by the intercepting method. HIP was performed at 1185°C under gaseous Ar at a pressure of 172.25 MPa for 5 h. A solution treatment of the test bars was performed in a vacuum at 1185°C for 4 h and cooled in an atmosphere of gaseous Ar to room temperature; then, an aging treatment was performed at 1038°C for 2 h in a vacuum, before cooling to room temperature in an atmosphere of gaseous Ar. The test bars were secondarily aged at 871°C for 20 h in a vacuum and then cooled to room temperature in a furnace. The tensile tests were performed using an Instron 1125 universal test machine at 21, 427, 760 and 982°C. The gauge size of all test

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Table 1 Major elements in chemical composition (weight percent) of CM-681LC superalloy analyzed by XRF.

(mass%)

Elements	Non-HIP test bar	HIPed test bar
Cr	5.5	5.3
Co	9.3	9.3
Mo	0.51	0.52
W	8.4	8.4
Re	2.9	2.8
Ta	6.1	6.21
Al	5.71	5.52
Ti	0.16	0.15
Hf	1.49	1.62
C	0.11	0.11
B	0.19	0.20
Zr	0.12	0.12
Ni	bal.	bal.

bars was 6.3 mm in diameter and 26 mm in length. The microstructures were characterized by scanning electron microscopy (SEM) and transmission election microscopy (TEM). The fracture morphologies were observed using SEM. Microanalysis was performed by energy dispersive spectroscopy (EDS) in SEM and TEM.

3. Results

3.1 Analysis of composition and observation of microstructure

Table 1 presents the composition of the fine-grain test bars, that were prepared by the Microcast technique, determined by XRF.

Figure 1 shows the typical microstructure of a fine-grain CM-681LC superalloy following heat treatment. The main phases are the matrix γ, the reinforced phase γ', the eutectic structure γ-γ' and the carbides. In CM-681LC superalloys, the γ' phase include Ni and other solid solution strengthening elements such as Ta, Mo and W among others. The carbides of the CM-681LC superalloy precipitated within the grains and at the grain boundaries. EDS analysis reveals that these are MC type carbides, which are enriched with Ta and Hf (Figs. 1(b) and 1(c)).

3.2 Relationship between pouring temperature and grain size

The grain sizes obtained by traditional investment casting (using a superheating temperature of about 150°C) are 3 to 5 mm, and the morphology tends to be coarse and columnar. The grain size obtained by the Microcast process herein is 80 μm and the structure is equiaxed (Fig. 2). This result indicates that reducing the temperature gradient between the liquid metal and the mould temperature by lowering the pouring temperature and controlling the mould temperature, shortens the solidification time and, thereby, limits the grain growth.

3.3 Microstructures before and after HIP

Some micropores were generated in the fine-grain CM-681LC superalloy by the Microcast technique because of a lack of melt supplement (Fig. 3(a)). The micropores were distributed mainly between the interdendrites and at the grain boundaries. Most of these micropores can be eliminated by HIP as presented in Fig. 3(b). The porosity of fine-grain test bars was measured before and after HIP (Fig. 4). The experimental results reveal that HIP can significantly reduce the porosity of CM-681LC superalloys. HIP reduces the area fraction from 0.2% to 0.06%.

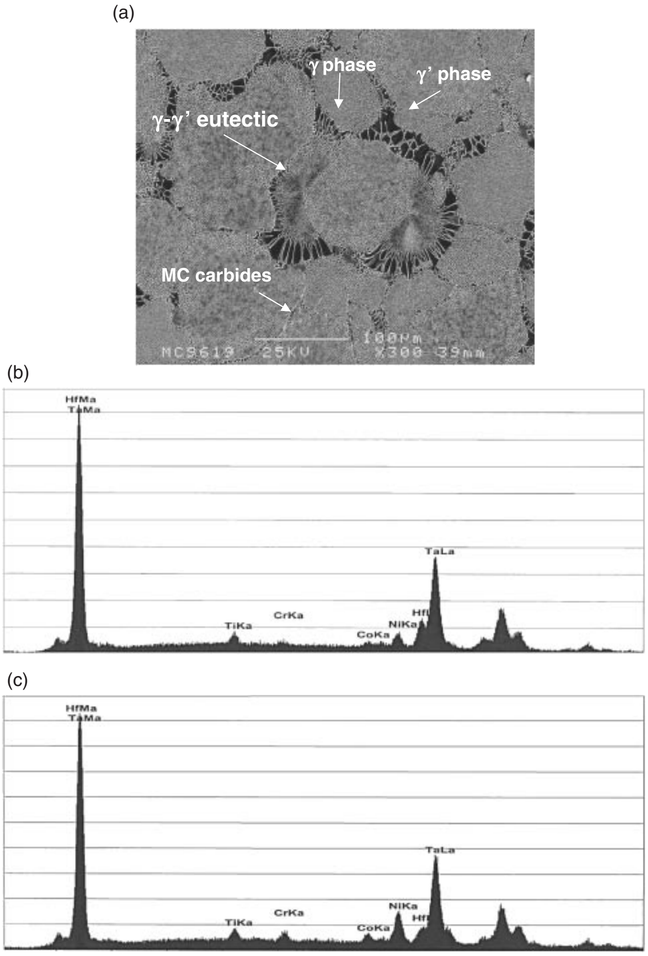

Fig. 1 (a) SEM micrograph of microstructure, (b) EDS analysis of carbides within grains, and(c) EDS analysis of carbides at a grain boundary of non-HIP CM-681LC superalloy following heat treatment.

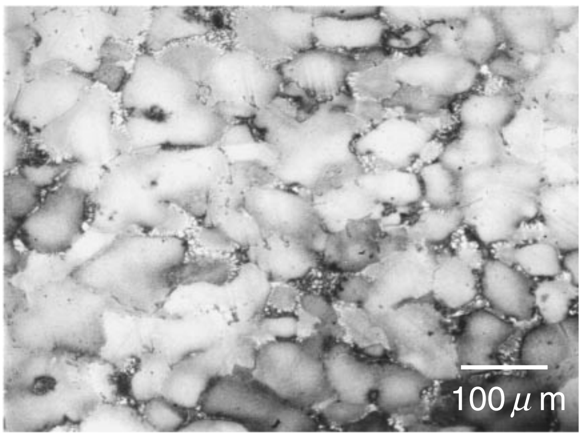

Fig. 2 Grain size of non-HIP CM-681LC superalloy obtained using Microcast process.

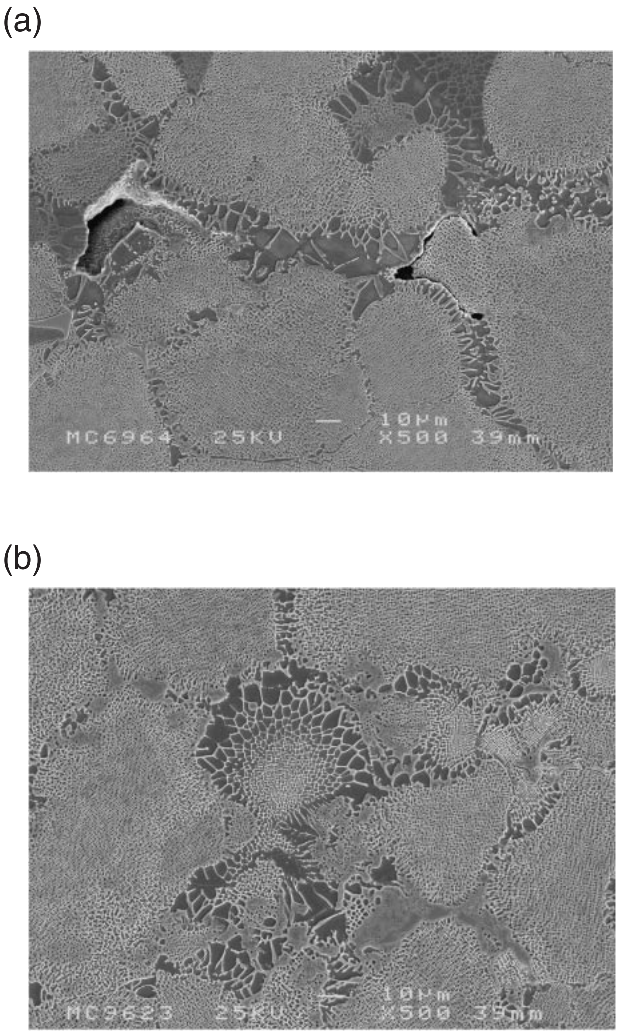

Fig. 3 Morphology of microporosity of CM-681LC superalloy with fine grains (a) before HIP and (b) after HIP.

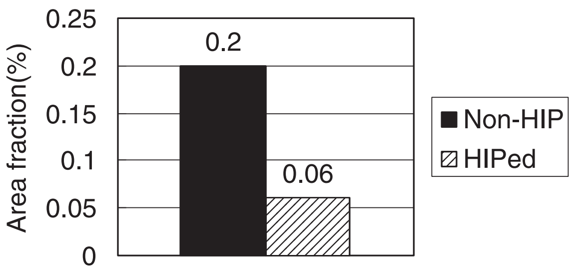

Fig. 4 Effect of HIP on area fraction of microporosity of CM-681LC superalloy with fine grains.

Figure 5 shows the grain size of fine-grain CM-681LC superalloys following HIP. The grain size after HIP, about 85 μm, is slightly larger than that before HIP; the structure is also equiaxed.

Figure 6 presents the γ' phase morphology of CM-681LC superalloys before and after HIP. The size of the γ' phase grew from 0.5 μm to 1.0 μm after the alloys were heat-treated at a high temperature for a long period and then cooled slowly from the high temperature in HIP.

Figure 7 presents the carbide morphologies of CM-681LC superalloys before and after HIP. Figure 7(a) shows the morphology of carbides before HIP, most of which are script-like and distributed within grains and at grain boundaries. Some particle-like carbides precipitated inside the grains.

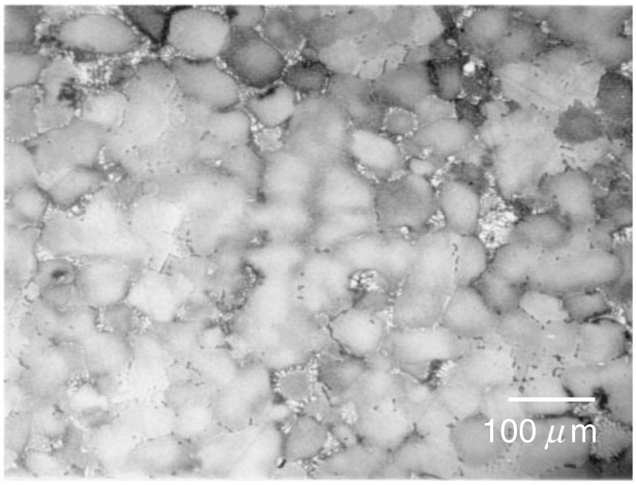

Fig. 5 Grain size, 85 μm, of HIPed CM-681LC superalloy obtained using Microcast process.

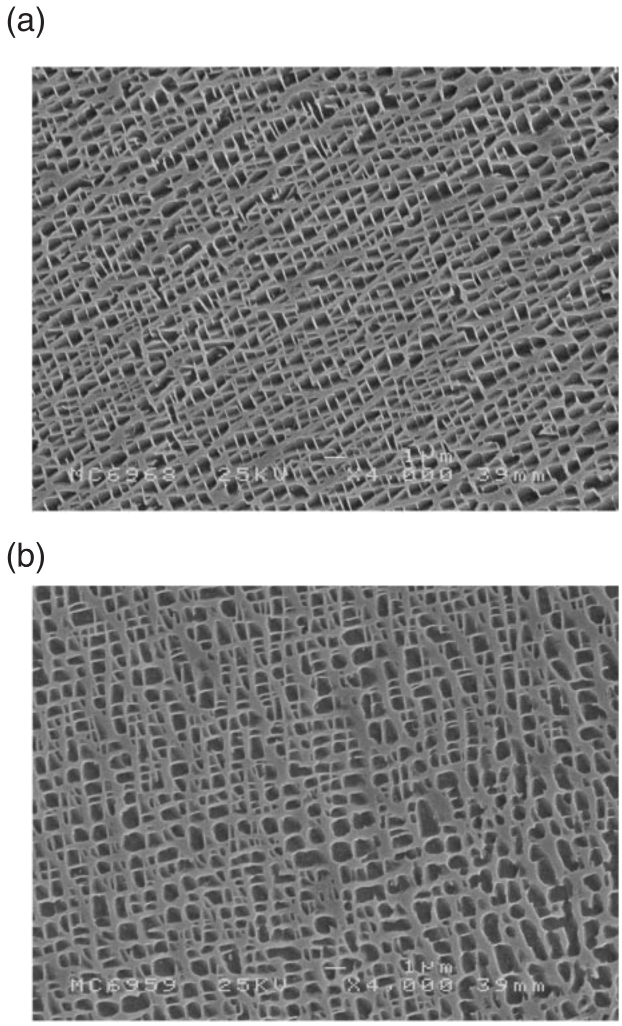

Fig. 6 γ' phase morphology of CM-681LC superalloy with fine grains (a) before HIP and (b) after HIP.

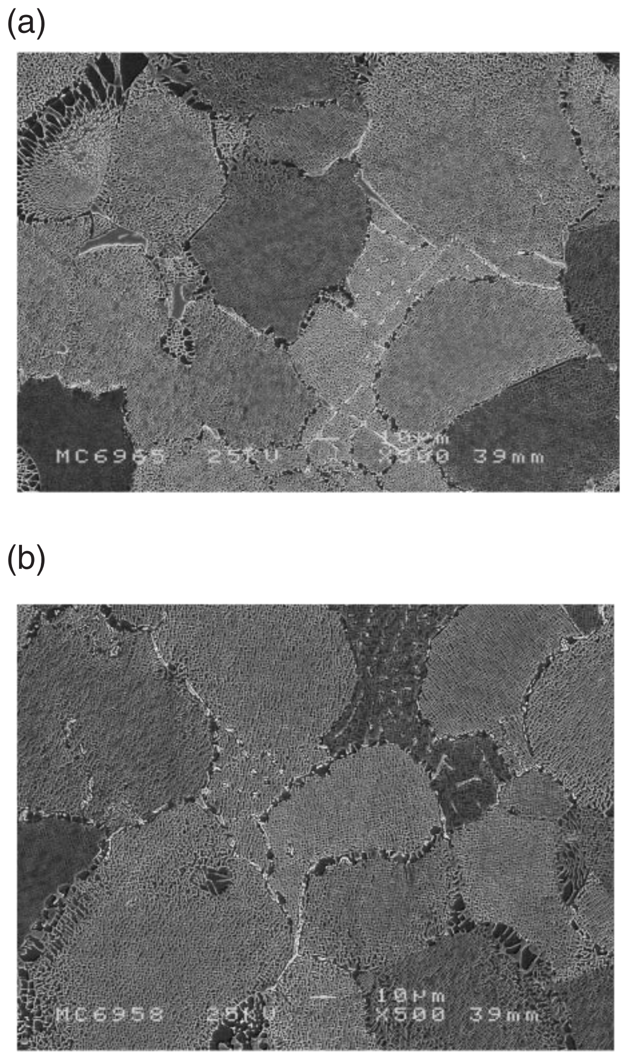

Fig. 7 Carbide morphology of CM-681LC superalloy with fine grains (a) before HIP and (b) after HIP.

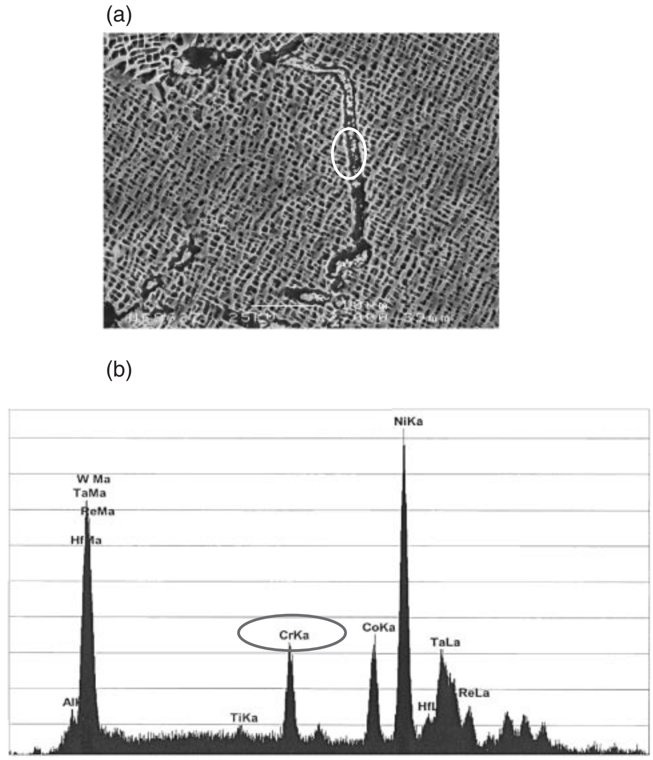

Fig. 8 (a) SEM micrograph of carbide morphology after HIP. (b) EDS spectrum of carbides at a grain boundary.

The EDS analyses in Figs. 1(b) and 1(c) indicated that these carbides that were rich in Ta and Hf were MC carbides. Figure 7(b) presents the carbide morphology of the fine-grain test bars after HIP, in which the carbides were finer than in the fine-grain test bars before HIP. Some carbides were observed inside the grains of the fine-grain test bars after HIP.

High-magnification SEM in Fig. 8(a) reveals that some fine particles precipitated discontinuously at the grain boundary following HIP. The EDS analysis (Fig. 8(b)) demonstrates that these precipitates are Cr-rich, indicating that they are $M_{23}C_6$ carbides. These carbides were also identified and determined by TEM/EDS. Figure 9 presents the bright-field image of a $M_{23}C_6$ carbide, the dark-field image of the carbide, the selected-area diffraction pattern of the carbide and the EDS spectrum of the carbide.

Figure 10 plots the statistical results concerning the carbide characteristics of CM-681LC superalloys before and after HIP. Figures 10(a) and (b) demonstrate that carbides in the fine-grain castings tend to be refined and spheroidized after HIP. The size of carbide particles was reduced from 16.8 μm to 12.8 μm, and the aspect ratio was reduced from 1.96 to 1.85. Moreover, the total carbide area fraction in grains and at GBs following HIP was reduced from 1.12% to 0.91% (Fig. 10(c)), suggesting that carbides were dissolved in the matrix during HIP. The following discussion will show the causes of the change in carbide morphology.

3.4 Tensile Properties for Various Temperatures

Tensile tests of test bars that had undergone HIP were performed at 21, 427, 760 and 982°C. Table 2 and Fig. 11 show the results.

Figure 11(a) plots the tensile strengths of the test bars before and after HIP for the various test temperatures. The tensile strength of the test bar at room temperature was 990 MPa and 1085 MPa before and after HIP, respectively. The increase was 9.6%. At a test temperature of 427°C, the tensile strength before HIP was 1060 MPa, while that after HIP was higher at 1156 MPa. The strengths of both samples without and with HIP decreased rapidly as the temperature increased over 982°C, but the tensile strengths following HIP always exceeded those before HIP.

Figure 11(b) reveals that the variation of yield strength with temperature is similar to the variation of tensile strengths. At all of the test temperatures, the yield strengths after HIP exceeded those before HIP. At room temperature, the yield strength before HIP was 900 MPa, which increased to 967 MPa following HIP. In the 760°C test, the yield strength after HIP was 945 MPa, which was 9.9% higher than that before HIP. At 982°C, HIP increased the yield strength, but by markedly less than at 760°C.

Figure 11(c) presents the relationship between the tensile elongation and the test temperature. The tensile elongation of the test bar at room temperature before HIP was 4.3%, which

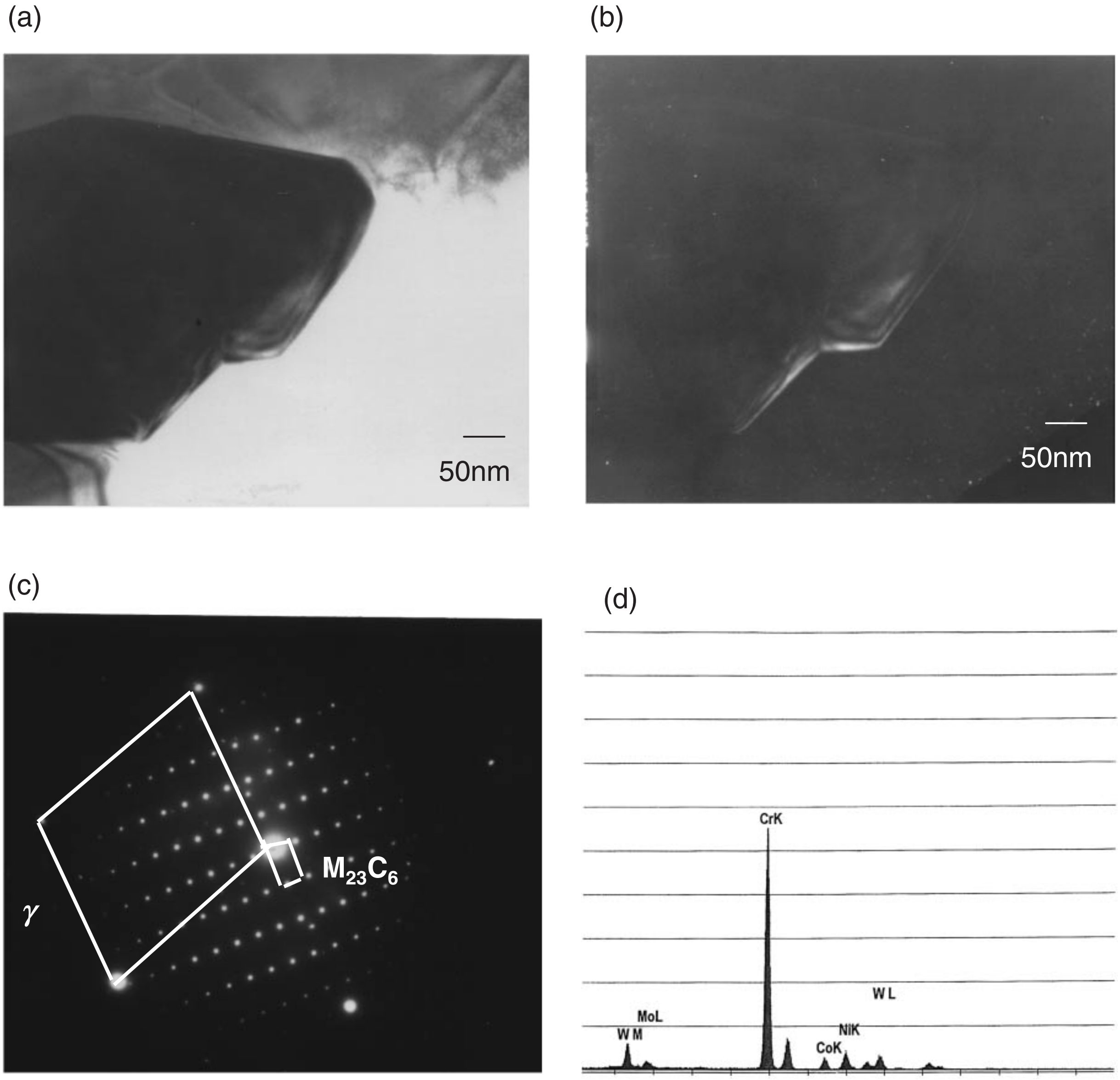

Fig. 9 (a) Bright-field image of a $Cr_{23}C_6$ ($M_{23}C_6$) carbide, (b) dark-field image of the carbide, (c) selected-area diffraction pattern corresponding (a), and (d) EDS spectrum of the carbide.

increased to 4.7% after HIP. HIP improved the tensile elongation from 5.5% to 6.9% at 427°C. The tensile elongations at 760°C and 982°C before HIP were always less than the specified value of 4% for practical applications, and all tensile elongations were larger after HIP.

3.5 Fracture morphology

The fracture surface and the longitudinal section were observed using SEM to study the effect of HIP on the tensile fracture mode. Figure 12(a) displays the room-temperature tensile fracture surface of the CM-681LC superalloy before HIP. The main cracks were distributed along MC carbides and the micropores. The longitudinal section of the fracture sample (Fig. 12(b)) indicates that scriptlike carbides at grain boundaries were also the main crack-production sites, in addition to the micropores. Therefore, micropores and scriptlike MC carbides are the main determinants the fracture of the fine-grain test bars before HIP.

Following HIP, no cracks was observed from the micropores in Fig. 13(a). The main cracks were observed at GBs. The longitudinal section of a fracture sample (Fig. 13(b)) also indicates that the cracks are distributed along GBs after HIP, and not along the micropores of the fine-grain test bars. Additionally, Fig. 13(c) shows that some cracks are distributed near the carbides at the grain boundaries. However, significantly fewer cracks are present near the scriptlike carbides at the GBs than before HIP. The fracture mode of the fine-grain test bars following HIP was a typical intergranular fracture mode.

The fracture surface and longitudinal section were observed in tensile tests at 427, 760 and 982°C. The fracture modes were the same as those at room temperature.

4. Discussion

HIP effectively improve the mechanical properties of CM-681LC superalloy by favorably affecting the microstructure and fracture mode. This section discusses the effects of HIP

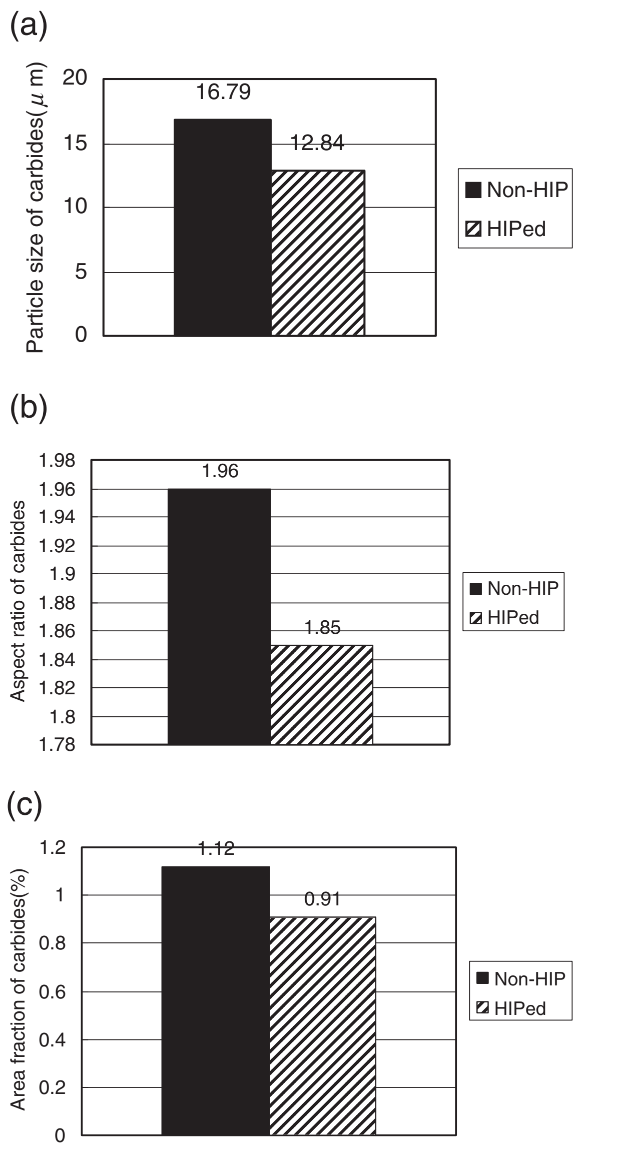

Fig. 10 Effect of HIP on the characteristics of carbides: (a) particle size, (b) aspect ratio and (c) area fraction.

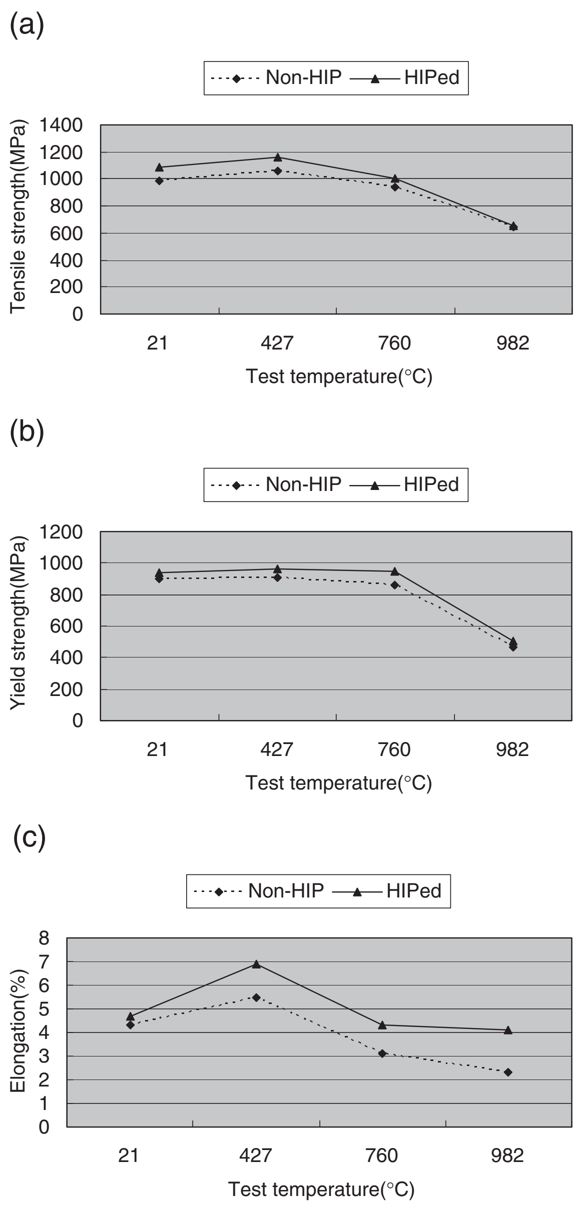

Fig. 11 Comparison of the mechanical properties at various testing temperatures before and after HIP.

Table 2 Tensile test results of non-HIP and HIPed CM-681LC superalloys with fine grains.

Test Temp. (°C)	U.T.S. (MPa)		Y.S. (MPa)		Elongation (E %)	
	Non-HIP	HIPed	Non-HIP	HIPed	Non-HIP	HIPed
21	990	1085	900	936	4.3	4.7
427	1060	1156	910	967	5.5	6.9
760	940	1003	860	945	3.1	4.3
982	642	657	468	505	2.3	4.1

on micropores, carbides, tensile properties and fracture modes.

4.1 Micropores

The most common defects in the fine-grain superalloy castings are micorpores that areformed during solidification. Two mechanisms contribute to the formation of pores. The shrinkage micropores are caused by the shrinkage of the melt. The gas micropores are due to the gas trapped in the melt. In this work, the shrinkage forms irregularly shaped micropores, rather than spherical gas micropores. The micropores are formed because of the insufficiency of melt supplement.

According to Pascal's law, the fluid exhibits an isotropic pressure in a closed tank. HIP provides high temperature and hydrostatic pressure, which closes the pores in casting. Accordingly, creep and diffusion soften the alloy at high temperature and pressure.[9] Therefore, HIP eliminates the microporosity in the interdendrite zones and at the grain boundaries.

HIP coarsens the γ' phase because it involves holding the fine-grain test bars at high temperatures, and then slow cooling. Coarsening of the γ' phase typically reduces its coherency with the matrix, its stability and its strength.[10,11] Therefore, it is essential that solid solution and aging treatment must be performed just after HIP to compensate for the degradation of mechanical properties.[2,5,7,8]

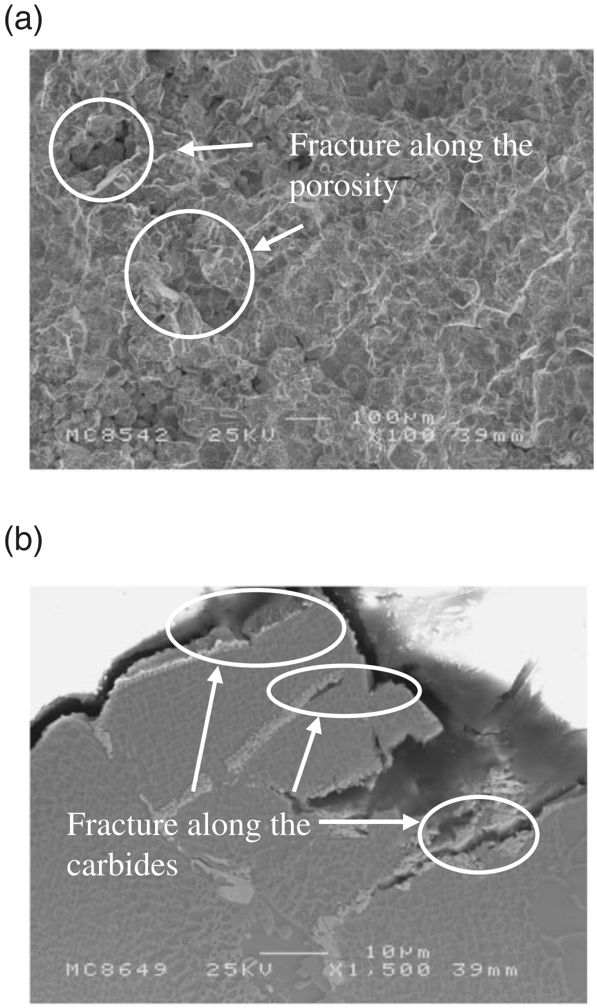

Fig. 12 SEM micrographs of non-HIP CM-681LC superalloy after room-temperature tensile test: (a) fracture along porosity and (b) cracks along carbides.

4.2 Carbides

SEM observations of microstructures and EDS analyses of CM-681LC superalloys before HIP demonstrate that the scriptlike and blocky carbides are MC carbides, which are rich in Ta and Hf. CM-681LC superalloys have a relatively low titanium content and a relatively high tantalum content. Sims[12] pointed out that the stabilities of MC carbides in the nickel-base superalloys follow the order HfC, TaC, NbC and TiC. The relatively high hafnium and tantalum contents cause the grain boundaries (GBs) to comprise discrete (Hf, Ta)-rich carbides, whose stability at high temperatures is better than that of titanium carbide, increasing the grain boundary strength and ductility.[1]

However, the carbide morphology importantly affects the characteristics of the alloy. Scriptlike MC carbides generally have a negative effect on alloy performance because they can pile up many dislocations, causing stresses to concentrate around the MC carbides. The transformation from scriptlike MC carbides to $M_{23}C_6$ carbide improves the mechanical properties. The results in this study reveal that scriptlike carbides at the GBs were transformed to particlelike carbides after HIP. According to TEM analysis, these carbides are Cr-rich $M_{23}C_6$ carbides.

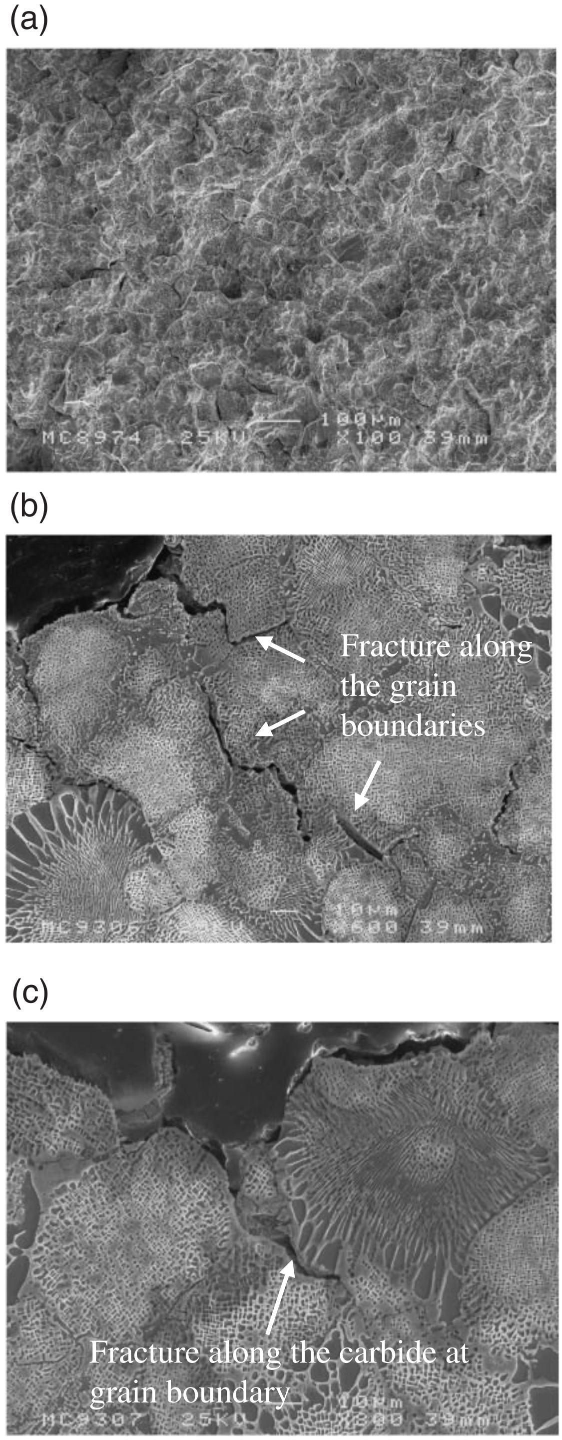

Fig. 13 SEM micrographs of HIPed CM-681LC superalloy with fine grains following room-temperature tensile test, showing (a) fracture surface, (b) cracks along grain boundaries and (c) cracks along carbides at grain boundaries.

Sims[12] also stated that the MC carbides in nickel-base superalloys can decompose into $M_{23}C_6$ carbides by heat treatment at a temperature of over 980°C:[12]

$$MC + \gamma \rightarrow M_{23}C_6 + \gamma'$$

However, the MC carbides of the CM-681LC superalloy are (Hf, Ta)C. They are more stable at high temperature, and so solution treatment at 1185°C alone cannot cause them to decompose. However, the HIP process used herein was performed at high temperature and high pressure. Since the free energy varies with pressure, the equilibrium temperature varies with pressures.[13] Some of the scriptlike MC carbides that precipitate at the GBs in CM-681LC superalloys decompose into the matrix at high temperature and high pressure during HIP. Carbon atoms combine with chromium atoms and reprecipitate as discontinuous particle-like $M_{23}C_6$ carbides at GBs during aging. Therefore, the carbides were refined and spheroidized, as indicated by the observation of the microstructure. These inferences agree with the results of EDS analyses. HIP decomposes the MC carbides and dissolves them in the matrix. The total amount of carbides in CM-681LC superalloys is slightly lower following HIP because there are few sites of $M_{23}C_6$ precipitate at GBs and chromium atoms.

4.3 Tensile properties

The causes of the improvement of the tensile strength, the yield strength and the tensile elongation of CM-681LC superalloys by HIP can be discussed in four ways.

Grain refinement is well known to improve tensile performance. The Microcast technique adopted in this investigation yielded a grain size of 80 µm in the test bars. However, the grains grow slightly during HIP, because a high temperature is maintained for a long time before the slow cooling during HIP. Hence, the grain size is not the main cause of the improvement of the tensile properties by HIP.

The formation of the micropores originates from the lack of the supplement of melt in the dendrites during rapid solidification. The micropores reduce the tensile strength, the yield strength and the tensile elongation, and cause the fracture to occur earlier since most of the crack initiation sites are close to the micropores. As HIP dramatically reduces the number of micropores, the formation of cracks along the micropores is inhibited, improving the tensile properties.

The carbides have an important and complex role in superalloys. The effects of carbides are still debated. Finer particlelike carbides precipitated at the GBs improve the mechanical properties.[14] In this study, the carbide morphology before HIP is scriptlike, causing cracks to be formed near the carbides, as revealed by observations of the fracture surface, mainly because the interface between the carbide and the matrix is incoherent. The MC carbides that are precipitated at the GBs make the GBs brittle. The cracks are easily formed at the brittle interface of the carbides. The early rupture is caused by the very poor capacity of the brittle carbides at GBs to tolerate strain, reducing rapidly the strength and elongation.[15] The scriptlike MC carbides decompose into particlelike $M_{23}C_6$ carbides during the HIP at high temperature for a long time. The ability of the interface between the carbides and the matrix to release stress can be improved by the refinement and spheroidization of the scriptlike carbides. Opportunities for producing cracks and causing fracture along scriptlike MC carbides can be reduced. Hence, the strength and elongation can be improved.[16–21]

The (Hf, Ta)C decomposed into the matrix can increase the Hf and Ta contents in the matrix. Hf can provide good grain boundary ductility and Ta can increase the strength of both the γ and the γ' phases by solid solution strengthening in nickel-base superalloy.[1]

Sims[22] has emphasized that the amount, size and distribution of the γ' phase in superalloys importantly affect the tensile properties. The high-temperature strength increases with the amount of γ' phase and declines as the size of the γ' phase increases. However, the γ' phase grows when it is treated with HIP at a high temperature for a long time and then slowly cooled. Some investigations[2,5,7,8] have reported that HIP slightly coarsens the γ' phase. Accordingly, the mechanical properties decrease as a result of the coarsening of the γ' phase. Hence, solid solution and aging treatments must be applied following HIP to improve the properties of fine-grain castings.

In summary, HIP improves the tensile properties of the fine-grain CM-681LC superalloys mainly by eliminating micropores and refining and spheroidizing MC carbides.

Sims[22] has showed that the strength increases with temperature, but the strength of the γ' phase declines as the temperature increases above a critical temperature. The presence of the γ' phase inhibits the motion of dislocations and improves the properties at high temperature. Therefore, the tensile strength and yield strength of the CM-681LC superalloy increase with temperature, and high strength is retained at 760°C. The strength of the CM-681LC superalloy begins to decline as the temperature increases above 760°C, indicating that the CM-681LC superalloys following HIP exhibit excellent high-temperature mechanical properties up to 760°C.

4.4 Fracture modes

The observations of the fracture surface in tensile tests at room temperature and elevated temperature show that the cracks of the fine-grain CM-681LC superalloys before HIP were along the micropores and the scriptlike carbides. The presence of the micropores in fine-grain test bars reduces tensile strength, yield strength and tensile elongation, and causes fracture to occur an earlier time. The scriptlike carbides easily pile up dislocations and cause stresses to be locally concentrated, since the carbides are brittle and a sharp interface is present between the carbides and the matrixes. Finally, the formation and propagation of cracks around the carbides cause the test bars to fracture.

HIP eliminates most of the micropores in fine-grain castings, by closing them at high temperature and hydrostatic high pressure. The opportunities to generate cracks along the carbides are reduced because of the decomposition of scriptlike MC carbides into particlelike $M_{23}C_6$ carbides following the HIP process. Hence, the grain boundary of the fine-grain test bars following HIP inhibits the sliding of dislocations at high stress during the tensile test. The fracture mode changes to intergraular fracture, since most of the micropores are eliminated and the scriptlike carbides are refined and spheroidized, indicating that the tensile strength, the yield strength and the tensile elongation can be effectively improved.

5. Conclusions

This study elucidated the effects of hot isostatic pressing on the microstructure and the tensile fracture modes of CM-681LC superalloy by the Microcast process. Based on the results of this study, we conclude the following.

(1) A fine-grain microstructure of the CM-681LC superalloy test bar with a grain size of 80 μm can be obtained using the Microcast process.
(2) HIP can reduce the microporosity of fine-grain casting from 0.2% to 0.06%.
(3) HIP decomposes the MC carbides at grain boundaries to $M_{23}C_6$ carbides at high temperature and high pressure. The morphology of the carbides is transformed from script-type to discontinuous particles, indicating that HIP tends to refine and spheroidize the carbides in fine-grain castings.
(4) Eliminating the mircopores and refining carbides improve the mechanical strength by about 9% and the elongation by over 10% in room- and high-temperature tensile tests. The tensile strength at 760°C is 1003 MPa, and the yield strength is 945 MPa.
(5) Fracture analyses of the tensile test specimens suggest that the microporosity and the scriptlike carbides at grain boundaries are the main factors that determine the early fracture of the fine-grain test bars before HIP. However, the tensile test fracture of the fine-grain test bars following HIP is typically intergranular because the micropores are eliminated and the carbides are refined.

Acknowledgments

The authors would like to thank Mr. Y. P. Huang for his help in mechanical property evaluation. The equipment support of processing from Mr. J.S. Chen is also thankfully acknowledged.

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