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Development of Microstructure and Mechanical Properties of a Ni-Base Single-Crystal Superalloy by Hot-Isostatic Pressing

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Effects of the hot-isostatic pressing (HIP) process on microstructures and mechanical properties of Ni-base single crystal superalloy CMSX-4 were investigated. In the overall heat treatment process, the HIP treatment remarkably induced the healing behavior of micropores and decreased the pore size and porosity of superalloy CMSX-4 compared with normally treated specimens. The microstructure of γ' phase after the HIP process showed rather a coarsening tendency and could be developed by the partially solution and aging treatment. Consequently, the elimination of cast micropores using the HIP step resulted in the inhibition of crack initiation in microstructure and improved the stress-rupture lives of Ni-base single crystal superalloy by 185%.

Keywords CMSX-4, healing behavior, HIP treatment, micropores, single-crystal superalloy

1. Introduction

Turbine blades with complex cooling paths in a gas turbine engine are cast generally from nickel-base superalloys in single crystals.[1,2] High creep resistance and high temperature strength of Ni-base superalloys can be achieved from a strengthening by a coherent precipitation of the Ni_3Al type, γ' phase in the Ni-base γ matrix.[3-5] Under the operation condition, the polycrystalline gas turbine blades, fabricated by a vacuum investment casting, show some problems related to grain boundaries, which act as a weak point. Thus, there has been steady work for the application of Ni-base single crystal superalloys.

Superalloy CMSX-4 is one of the second-generation single crystal Ni-base superalloys.[6] In general, the introduction of the refractory elements to these superalloys enhances the strength and creep resistance by the solid solution strengthening in the γ matrix. Actually, the single-crystal superalloys, obtained by the investment casting, contain porosity of 0.2 ~ 0.5 vol.% due to the casting micropores around the γ/γ' eutectic phase. At elevated temperatures, the micropores of single-crystal superalloys are the main source of crack initiation to external stress and increase the scattering band of fatigue and creep strength.[7]

Micropores are formed inevitably in the inter-dendrite area, isolated by the growth of secondary dendrite during solidification.[8] This phenomenon is due to the insufficient supply of upper liquid phase; thus the micropores are created at the end of γ/γ' eutectic and are last to solidify. Generally, the micropores are connected in the early stage of rupture, and these micropores have a harmful effect on mechanical properties. Unfortunately, it is difficult to estimate the remaining lives and service lives of components due to the different porosities of superalloys.

Hot-isostatic pressing (HIP) treatment is able to release the scattering band of mechanical properties due to the concentration difference of micropores. In general, densification through the HIP process can be accomplished by plastic deformation and creep behavior, and then by the diffusion bonding of the surfaces of the collapsed regions under applied pressure and elevated temperature. By this HIP method, the casting blades can be optimized to a homogenous, dense microstructure; an additional benefit is the resultant increase in alloy homogeneity.[1,9]

In this research, the micropores in Ni-base single-crystal superalloy CMSX-4 were eliminated by the HIP process. After the HIP treatment and the partially solution and aging treatment, the microstructural morphologies of the γ' phase precipitated in the γ matrix was observed, and the stress-rupture behavior of the Ni-base single-crystal superalloy CMSX-4 was investigated.

2. Experimental Procedures

The single-crystal superalloy CMSX-4 (Ingot, Cannon-Muskegon Co.,) was directionally solidified by a Bridgeman

Table 1 Heat Treatments of Ni-Base Single-Crystal Superalloy CMSX-4

Specimen	Heat Treatment	Remark
1	As-cast + full solution + aging	Stress-rupture LCF test
2	As-cast + full solution + HIP	…
3	As-cast + full solution + HIP + partial solution	…
4	As-cast + full solution + HIP + partial solution + aging	Stress-rupture LCF test

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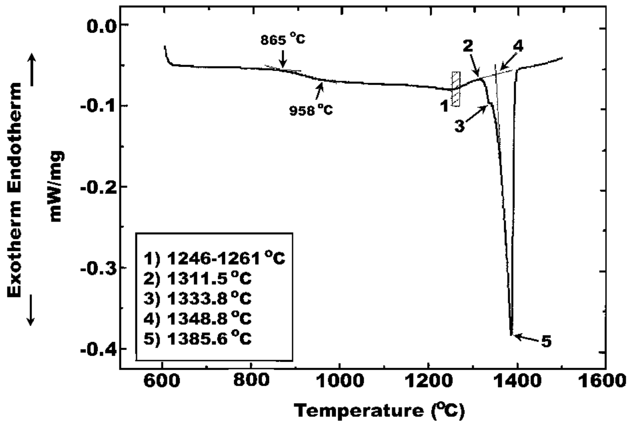

Fig. 1 DTA curve of Ni-based single-crystal superalloy CMSX-4

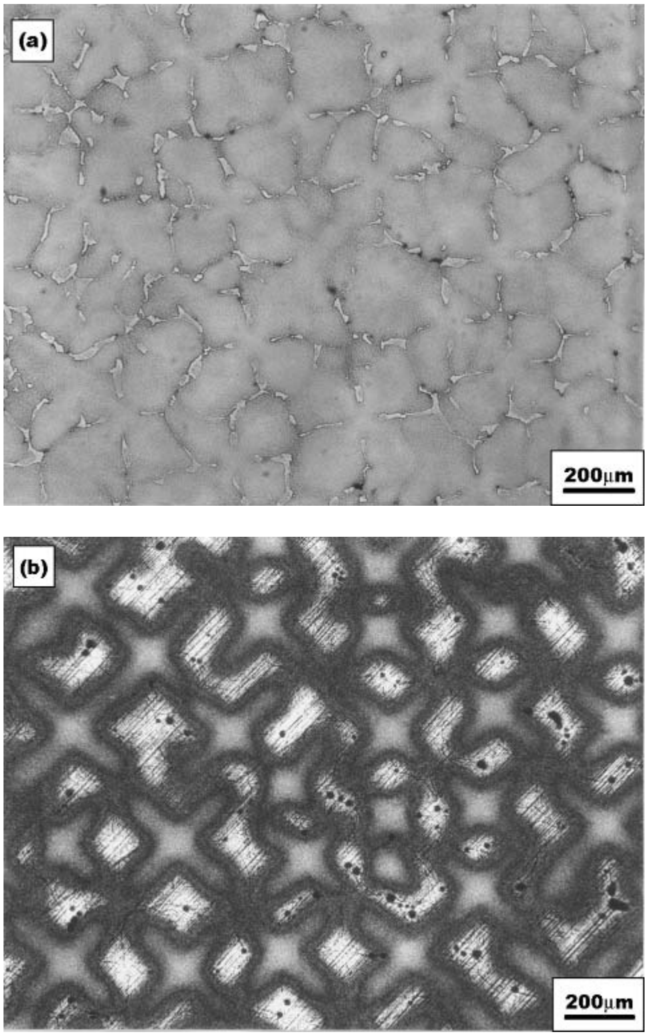

Fig. 2 Microstructure of **(a)** as-cast and **(b)** after full solution treatment of Ni-base single-crystal superalloy CMSX-4

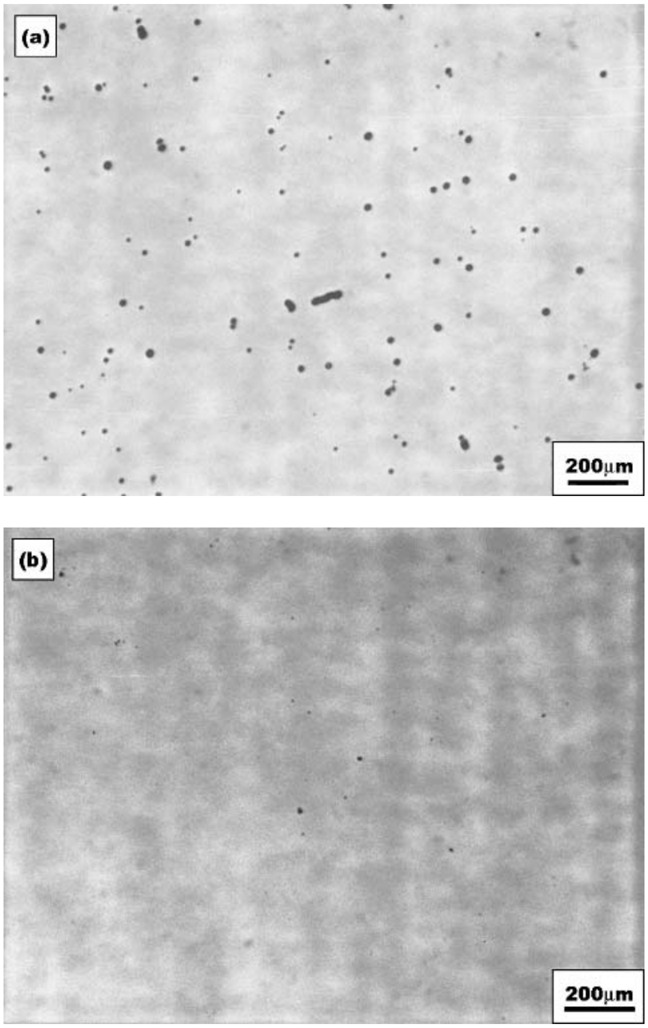

Fig. 3 Microstructural morphologies of **(a)** specimen 1 and **(b)** specimen 4

Table 2 Chemical Composition, in wt.%, of Ni-Base Single Crystal Superalloy CMSX-4 Used in This Work

	Cr	Mo	W	Al	Ti	Ta	Co	Hf	Re	Ni
Core of primary dendrite	5.9	0.6	6.1	5.1	0.9	5.3	9.7	…	3.6	Bal
Interdendritic region	5.8	0.5	4.6	5.8	1.0	6.5	9.6	…	2.0	Bal

method (Korea Institute of Machinery and Materials, Changwon, Korea). Each specimen had the <001> direction of crystallographic lattice within a few degrees of its long axis. Differential thermal analysis (DTA) testing was performed at a heating rate of 10 °C/min in Ar gas.

To examine the effects of the HIP process, the heat-treatment steps were classified (Table 1). For the uniform distribution of alloying elements in matrix, the fully solution treatment of superalloys was carried out in the sequential steps of 1277 °C/2h, 1288 °C/3h, 1296 °C/3h, 1304 °C/2h, 1313 °C/2h, 1316 °C/2h, 1318 °C/2h, and 1312 °C/2h, and then were cooled by Ar gas fan quenching (AGF). The HIP processing of the single-crystal superalloys was carried out by the schedule of

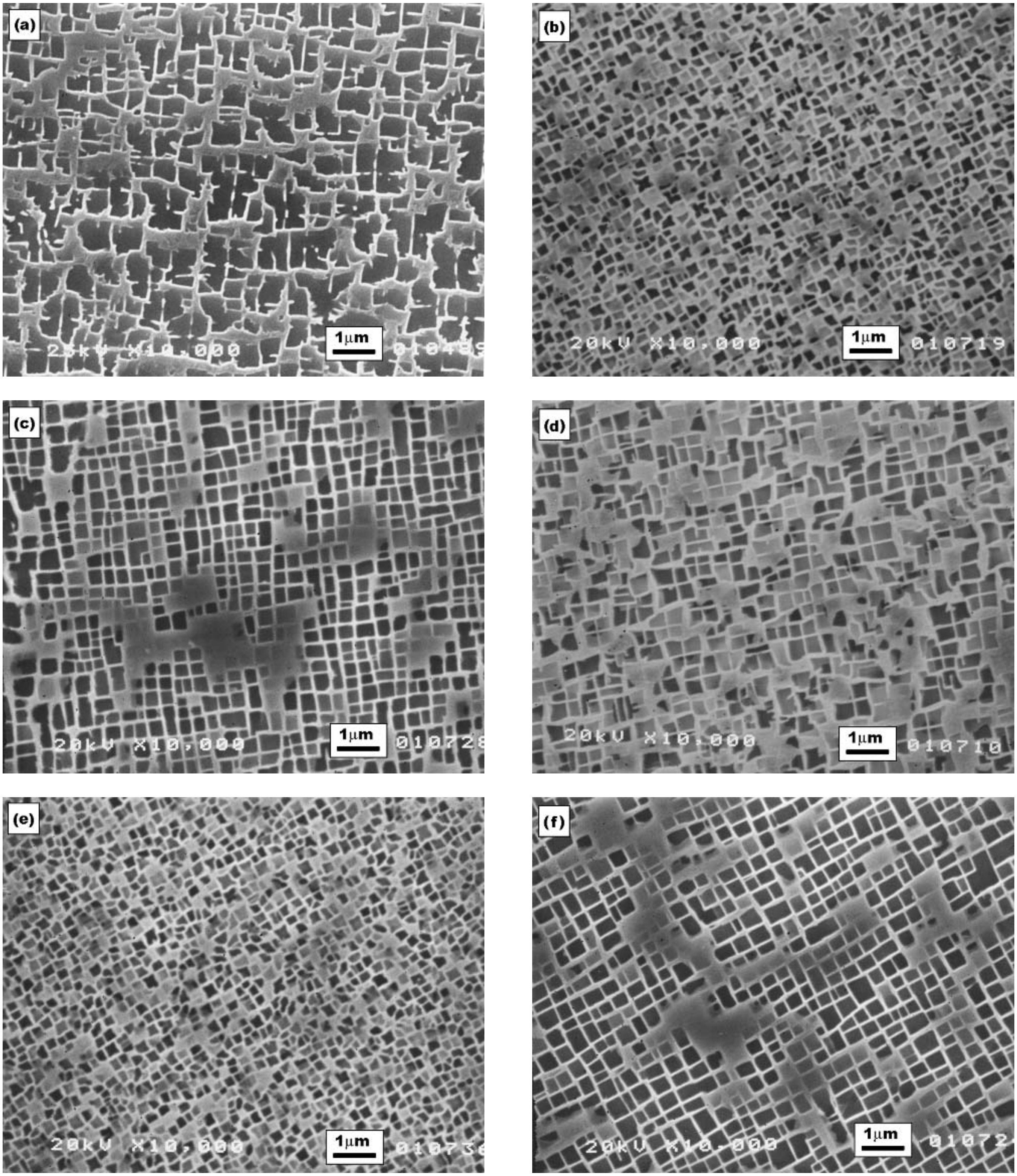

Fig. 4 Microstructural morphologies of the γ′ phase in Ni-base single-crystal superalloy CMSX-4: **(a)** as-cast, **(b)** after full solution treatment, **(c)** specimen 1, **(d)** specimen 2, **(e)** specimen 3, **(f)** specimen 4

1288 °C, 35 MPa/1h, at 1288 °C, 100 MPa/4h, and then the AGF step. During HIP processing, the treatment temperature is maintained carefully for γ′ dissolution not to exceed the incipient melting temperature. Then the partially solution treatment (1140 °C /2h and AGF) and aging heat treatment (870 °C/20h) and air cooling (AC) followed.

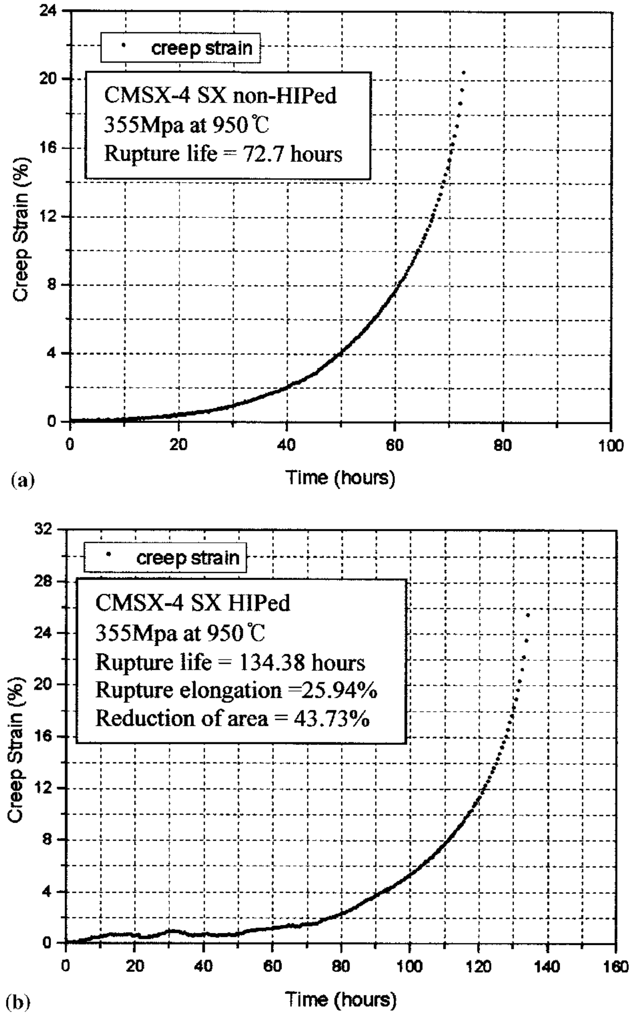

Fig. 5 Stress-rupture curves of **(a)** specimen 1 (non-HIP treated) and **(b)** specimen 4 (HIP treated)

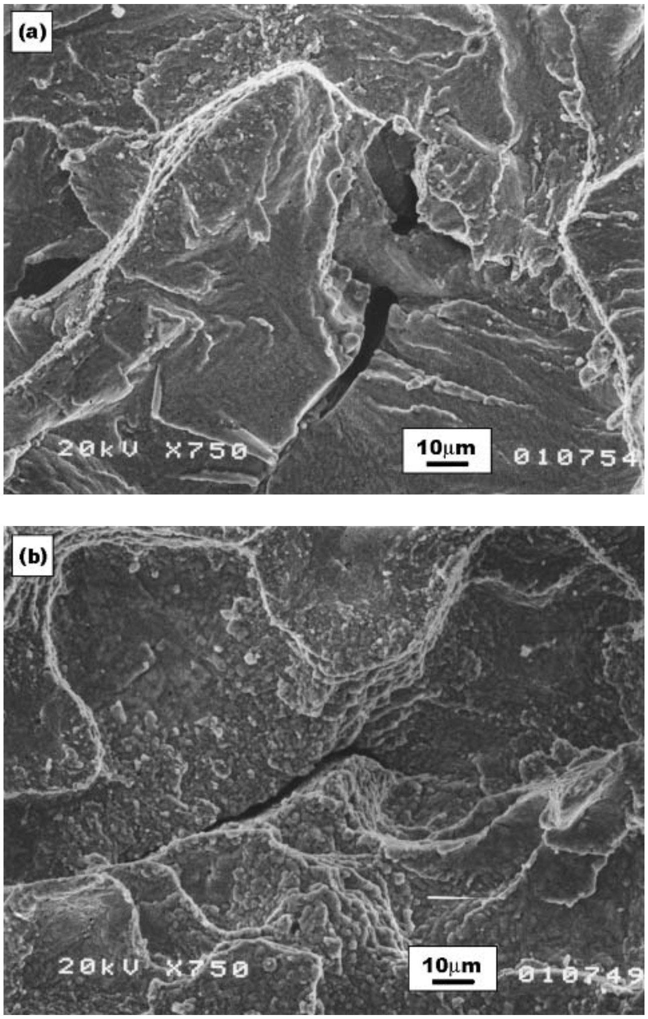

Fig. 6 Microstructure of Ni-base single-crystal superalloy CMSX-4 after stress-rupture test: **(a)** specimen 1 (non-HIP treated) **(b)** specimen 4 (HIP treated)

The distribution of micropores in specimens was observed with an optical microscope and an image analyzer after polishing. The specimens were etched with Kalling solution (1g $CuCl_2$, 15 Mℓ HCl, 40 Mℓ ethanol) to observe the microstructural morphologies of γ' phase by the scanning electron microscope (SEM; JSM5410, JEOL, Tokyo, Japan). The stress-rupture and low cycle fatigue (LCF) tests of HIP-treated specimens were evaluated. The stress-rupture test was performed at 355 MPa and 950 °C with a constant load type creep tester (ATS, Toshin RT-50T, Japan), which has a thermocouple attached on the center of the specimens to measure the real test temperature. The load control LCF test was performed at 580 MPa, 950 °C and cycle of 0.35Hz, ratio (R) $= 0$. The fractured surface of the single-crystal superalloys was observed by SEM.

3. Results and Discussion

3.1 Determination of the Homogenization and HIP Conditions From DTA Diagram

The temperature range of γ' phase dissolution was determined by the DTA diagram (Fig. 1). The dissolution of γ' phase was initiated at 865 °C and completed in the temperature range of 1246-1261 °C (γ' solvus). The γ/γ' eutectic was formed at 1334 °C; the incipient melting occurred in the segregated regions over this eutectic temperature. Thus the homogenization temperature was determined in the range of 1277-1321 °C. The liquidus line and the solidus line of this sample were 1349 and 1386 °C, respectively.

Dissolution temperature is very important in determining a heating procedure of superalloys. Actually, an unsuitable temperature in HIP process—that is, over the eutectic temperature or below the dissolution temperature—may cause the internal recrystallization or deter the incorporation of micropores and the deformation in superalloy. HIP treatment is usually done over the γ' dissolution temperature for the effective elimination of micropores through plastic deformation or creep behavior. Thus, in this work, the HIP condition was determined at 1288 °C between the γ' dissolution temperature and the γ/γ' eutectic temperature of CMSX-4 alloy. In some cases, the local dissolution of the secondary phase in matrix could be initiated; thus the HIP process was preceded by the partially solution treatment.

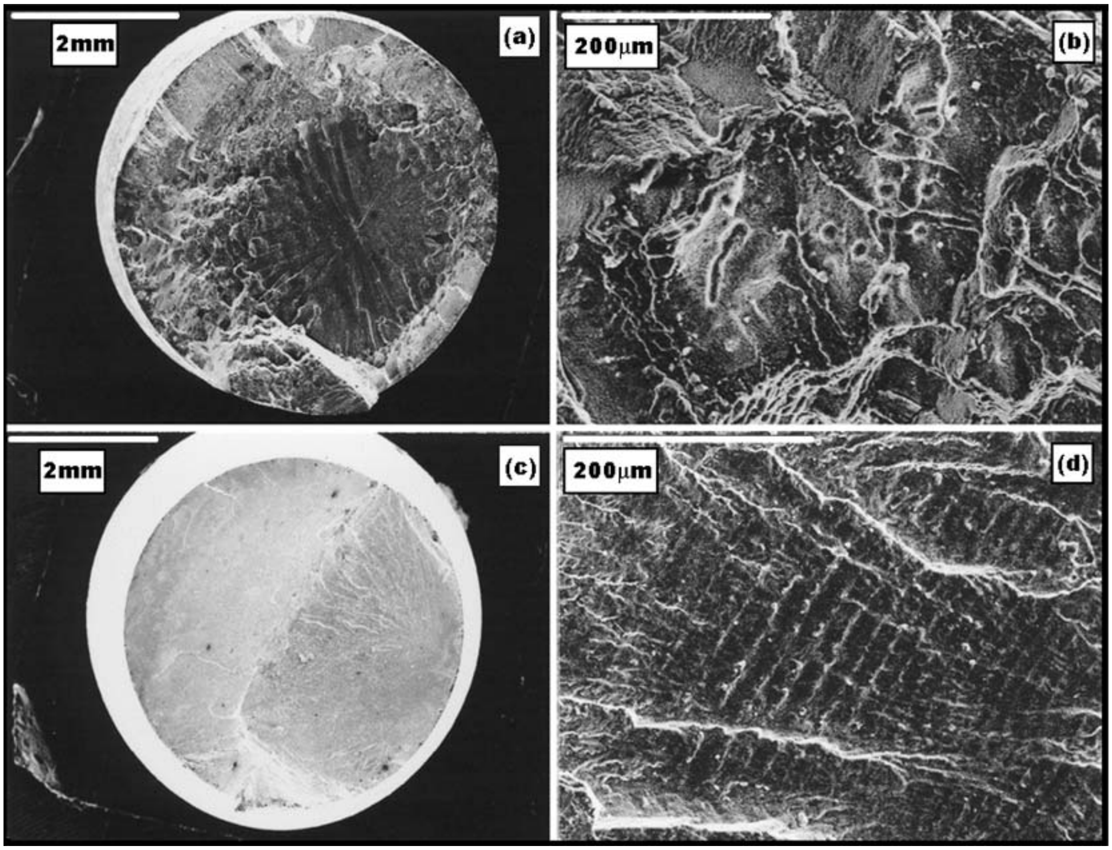

Fig. 7 Micrographs of Ni-base single-crystal superalloy CMSX-4 after LCF test: **(a,b)** specimen 1 (non HIP treated), **(c,d)** specimen 4 (HIP treated); (a,c) optical micrographs, (b,d) SEM micrographs

3.2 Microstructure of Superalloys

Figure 2 shows the morphologies of the typical dendrite structure on transversal section as reported by some researches.[10,11] The composition of the core area is different from that of the interdendritic area due to micro-segregation during the cast process, which should be homogenized by heat treatment. The arm spacing *L*p of the primary dendrite lies in the range of 150-100 μm. Energy dispersive spectroscopy (EDS) analysis of cast specimens indicates the residual dendritic micro-segregation as shown in Table 2. This result was consistent with the work of Karunaratne[12] and Fuchs.[13]

The microstructure morphologies of superalloy CMSX-4, after HIP treatment and heat treatments, are shown in Fig. 3 and 4. The HIP-treated specimens (Fig. 3b) have small pore size, and porosity decreased markedly as compared with the non-HIP treated specimens (Fig. 3a). The as-cast specimen shows an irregular cubic shape of about 1 μm (Fig. 4a); the coarsening of the γ' phase was depressed to the size of 0.3 μm by the full solution treatment (Fig. 4b). After aging, the cuboidal γ' phase was irregularly precipitated in the γ matrix (Fig. 4c). In the γ' morphology of the superalloy, the irregular morphology caused by the HIP step (Fig. 4d) disappeared through the partially solution treatment (Fig. 4e) and aging treatment (Fig. 4f). Consequently, the HIP process with the follow-up heating procedure resulted in the uniform morphology of γ' morphology.

3.3 Stress-Rupture Behavior of Superalloys

In the stress-rupture behavior (Fig. 5), the lives of specimen 1 and 4 were 72.72 and 134.38 h, respectively. This indicates that HIP treatment improved the stress-rupture life of superalloys up to 185%. Figure 6 shows the fractured surfaces of the superalloy after the stress-rupture test. The fracture behavior, initiated by flaws, was shown more prominently in the non-HIP treated specimen of Fig. 6(a) than in the HIP-treated specimen of Fig. 6(b). It was inferred in the fractured plane of the HIP-treated superalloy that the fatigue crack initiation would be restrained throughout the entire specimen by the densification and healing behavior in the microstructure (Fig. 6b). Consequently, the flaws and micropores in specimens were removed markedly by HIP treatment, and the cracks, propagated from the surface, were not easily connected with each other in the form of a three-dimensional network under the stress.

3.4 Microstructural Morphology After LCF Test

The results of the LCF test of specimens 1 and 4 showed little difference, but the fractured surface for non-HIP treated (Fig. 7a and b) and HIP treated specimens (Fig. 7c and d) showed a conspicuous morphology change after the LCF test. The non-HIP-treated specimen had the failure pattern by micropores in the creep and fatigue fractured specimens. The HIP-treated specimen did not show the fracture pattern related with micropores but showed the striation pattern in the fractured surface (Fig. 7d). It is supposed that the striation appearance is related to the residual micropores with small size after healing behavior by HIP treatment.

4. Conclusion

HIP treatment (100 MPa, 1288 °C) could densify the microstructure of Ni-base single-crystal superalloy CMSX-4 by the removal of micropores. The control of the coarsening and uniform distribution of the cuboidal γ' phase in γ matrix, after HIP processing, was accomplished through partially solution treatment and aging treatment. Development in the microstructure of the single-crystal superalloy enhanced the stress-rupture properties of the turbine blade material up to 158%. The stress-rupture lives of specimens, before and after HIP treatment, were 134.38 and 72.72 h, respectively. The single-crystal superalloy CMSX-4 after healing exhibited no noticeable increase in LCF life but showed peculiar striations in the fractured surface without the fracture patterns due to the micropores.

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