Effect of Zirconium on Microstructure and Mechanical Properties of Cast Fine-Grain CM 247 LC Superalloy

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This study investigates the effect of Zr minor additions, with Zr content from 0.015 to 0.15 mass%, on microstructure and mechanical properties of fine-grain (65 μm) CM 247 LC superalloy. Tensile test results indicate that minor addition of Zr to fine-grain CM 247 LC can dramatically improve the yield strength at 300 K as well as both the yield strength and the elongation at 1033 K. Under creep conditions of 1033 K/725 MPa or 1200 K/345 MPa, the Zr additions drastically increased the rupture life, creep rate and elongation. The AES observation reveal that Zr may enrich at grain-boundary and boride/matrix interface and dissolve in matrix, carbide or γ' phase. Thus, Zr may change the primary MC carbide characteristics and inhibit the script-like MC carbide formation. Moreover, Zr is apparently to increase the effects of the cohesive energy of both the precipitated phase/matrix interface and the grain boundaries, also it is beneficial to stress accommodation and retards the crack initiation and propagation.

(Received October 24, 2003; Accepted December 25, 2003)

Keywords: Cannon-Muskegon 247 Low-Carbon Superalloy, Microcast-X casting process, zirconium, tensile strength, creep resistance

1. Introduction

The turbine disk and blades of many small gas turbines, such as small aircraft engines, turbo-fan missile engines and vehicular engines, are usually cast into a single piece, generally known as “integral wheels”. The operating temperature of the turbine disk typically does not exceed 1033 K. The operating stresses, associated with the centrifugal loads of these rotating components are high at the rim and toward the center, and usually exceed 550 MPa. However, turbine blades are subject to relatively low stresses, generally below 345 MPa, but high operating temperatures. Materials used to construct integral wheel must possess the combined properties of blade and disk. These materials, therefore, must have a very high tensile strength at operating temperature of the hub to protect the parts from bursting and high creep strength under blade operating condition.¹⁾

Controlling the grain size is an important means of developing and maintaining both physical and mechanical properties. Microcast-X casting, developed by the Howmet Turbine Components Corporation, has been successfully demonstrated to generate fine-grain components that range from turbine disks to blades and vanes. Many fine-grain turbine wheels have been extensively cast with IN 713 LC, IN 718 and Mar-M247 nickel-based superalloys.^{1–3)} The rigid requirements of increased operating temperatures of integral turbine wheels, increased rotational speeds and extended lifetimes of components have forced designers of turbine engines to tighten their criteria for selecting alloys.

CM 247 LC is a cast nickel-based superalloy with a low carbon content. The alloy is a modified superalloy based on the chemical composition of Mar-M247, specifically designed for making directionally solidified (DS) turbine blades.^{4–6)} The previous study⁷⁾ indicates that when the casting parameters are well controlled, the CM 247 LC superalloy has an excellent castability to form an equiaxed

grain structure, with high strength and superior creep resistance. However, the elements (B, Zr) for grain boundary strengthening may be insufficient for generating a conventionally polycrystalline superalloy under fine-grain casting conditions. Consequently, studying the relationship between grain boundary chemistry and the mechanical characteristics of cast fine-grain superalloy is important.

The Microcast-X casting process, producing test specimens of CM 247 LC superalloy with controlled fine-grain structures, was performed herein. The aim of this study is to elucidate the microstructural characteristics and investigate the mechanical performance of the fine-grain CM 247 LC superalloy with various Zr additions. Furthermore, some mechanisms associated with microstructure and mechanical properties are discussed.

2. Experimental Procedure

The alloys used herein were originally melted by vacuum induction melting (VIM) at the Cannon-Muskegon Corporation in USA. The alloys were remelted in an alumina crucible in a VIM furnace and poured at 1643 K into a ceramic shell mold with a CoO facecoat at Chung Shan Institute of Science and Technology in Taiwan. The temperature of the ceramic shell mold was maintained at 1373 K. Four heats of fine-grain CM 247 LC superalloys with Zr contents from 0.015 mass% to 0.15 mass% were prepared during fine-grained casting. After casting, the major elemental constituents were detected by X-ray fluorescence (XRF). Table 1 presents the nominal compositions of the parent alloys used herein. After casting all test bars underwent hot isostatic pressing (HIP) at 1458 K/173 MPa/4 h in an atmosphere of argon. The specimens produced were then heat-treated in a vacuum furnace. The heat treatment processes consisted of solution treatments at 1494 K/2 h, followed by argon gas fan quenching (Ar-GFQ), subsequently aging treatments at 1144 K/20 h, and finally furnace cooling. The grain size was measured using the linear intercept method. The microstructure and fracture surface

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Table 1 Nominal composition (mass%) of fine-grain CM 247 LC with various Zr contents.

Composition	CM 247 LC	CZ1	CZ2	CZ3	CZ4
Cr	8.0~8.5	8.1	8.2	8.1	8.1
Co	9.0~9.5	9.3	9.29	9.4	9.32
W	9.3~9.7	9.4	9.4	9.4	9.4
Mo	0.4~0.6	0.58	0.58	0.58	0.58
Ta	3.1~3.3	3.26	3.3	3.3	3.3
Al	5.4~5.7	5.5	5.51	5.6	5.6
Ti	0.6~0.9	0.68	0.72	0.72	0.7
Hf	1.0~1.6	1.35	1.42	1.34	1.35
C	0.07~0.08	0.08	0.08	0.08	0.08
B	0.01~0.02	0.015	0.015	0.015	0.015
Zr	0.007~0.02	0.015	0.04	0.08	0.15
Ni	BAL	BAL	BAL	BAL	BAL

were observed using optical microscopy (OM) and scanning electron microscopy (SEM). The segregation of Zr was analyzed by Auger electron spectrometry (AES). Specimens were impacted in the vacuum chamber of the Auger spectroscope at a liquid nitrogen temperature to obtain a fresh fracture surface, and thereby minimize contamination of the specimen and absorption of surface gas. Tensile tests were performed at 300 K and 1033 K, using an Instron 1125 mechanical testing machine. Creep tests were conducted at 1033 K/725 MPa or 1200 K/345 MPa using SATEC M3 creep testers. The gauge size of the test bars was 6.3 mm in diameter and 25.4 mm in length.

3. Results

3.1 Microstructure and carbide morphology

Figure 1 depicts the typical as-cast microstructure of the fine-grain CM 247 LC superalloy. The structure comprises a γ' intermetallic phase dispersed in a γ matrix. The rosette structure of the primary γ - γ' eutectic phase was observed mainly at the grain boundaries, with only a small portion within the grains. Additionally, the as-cast MC carbide has a predominantly irregular shape in the grains and at the grain boundaries.

Fig. 1 The microstructure of cast fine-grain CM 247 LC superalloy.

Fig. 2 The grain size of cast fine-grain CM 247 LC (65 μm) with (a) 0.015 mass% Zr, and (b) 0.15 mass% Zr.

Minor additions of Zr were observed not to affect the grain size of the fine-grain CM 247 LC superalloy. The average grain size of approximately 65 μm determined in the study as shown in Fig. 2 was obtained by maintaining the pouring temperature about 10 K above the liquidus. The fine-grain alloy produced by the Microcast-X casting process results in a substantially cellular and nondendritic microstructure. However, Zr additions greatly altered morphology of the carbides. Coarse and script-like carbides are present in the fine-grain CM 247 LC superalloy, and their sizes are in excess of 30 μm (Fig. 3(a)). As revealed in Figs. 3(b)–(d), minor Zr addition changed the carbide morphology from coarse and script-like to fine and spherical. The γ' size of fine-grain CM 247 LC superalloy with various Zr contents following heat-treatment at HIPing + 1494 K/2 h GFQ + 1144 K/20 h F.C. is about 0.15 μm , and the morphology of the γ' is fine and globular, as shown in Fig. 4.

3.2 Tensile properties at 300 K and 1033 K

Table 2 presents the tensile properties at 300 K and 1033 K of fine-grain CM 247 LC superalloy with various Zr contents. The yield strength at 300 K increases from 1020 MPa for test bars with 0.015 mass% Zr to 1095 MPa for test bars with 0.15 mass% Zr. Furthermore, the ultimate tensile strength and elongation at room temperature change only slightly with the Zr content. At 1033 K, the fine grain structure retains its

Fig. 3 The morphology of carbide in fine-grain CM 247 LC superalloy with the content of (a) 0.015 mass% Zr (b) 0.04 mass% Zr (c) 0.08 mass% Zr (d) 0.15 mass% Zr.

strength and elongation. As shown in Table 2, the yield strength increases from 1020 MPa, for test bars with 0.015 mass% Zr, to 1053 MPa for test bars with 0.15 mass% Zr; the elongation increases from 8.3 to 9.7% as the Zr content is increased from 0.015 to 0.15 mass%. Similar results are also obtained for ultimate strength, which increases from 1190 MPa up to 1220 MPa as the Zr content is increased as stated. Consequently, the tensile test results indicate that the minor addition of Zr increases yield strength at both room temperature and 1033 K.

3.3 Creep tests at 1033 K/725 MPa and 1200 K/345 MPa

Table 3 shows that the creep life and ductility of polycrystalline CM 247 LC at 1033 K/725 MPa at any specified Zr content; increasing the Zr content can remarkably enhance the creep life and ductility at 1033 K/725 MPa. When the Zr content is 0.15 mass%, the rupture elongation and creep life are 4.8–5.0% and 154–162 h, respectively. In other words, the rupture life of fine grain CM 247 LC with 0.15 mass% Zr is approximately 2.5 times greater than that of the 0.015 mass% Zr. In particular, the 1% creep life increases from 37 h, for test bars with 0.015 mass% Zr, to 67 h for test bars with 0.15 mass% Zr.

According to Table 4, under creep conditions of high temperature and moderate stress (1200 K/345 MPa), increasing the Zr content can substantially increase the creep life and ductility. When the Zr content exceeds 0.08 mass%, the rupture elongation and life can reach 10.3–11.0% and 35–38 h, respectively. Restated, the rupture life of fine grain CM 247 LC with 0.08 mass% Zr is approximately double that with 0.015 mass% Zr. In particular, the 1% creep life

increases from 9 h for test bars with 0.015 mass% Zr to 14 h for test bars of 0.08 mass% Zr. Apparently, optimal minor Zr additions enhance both the rupture elongation and the life of the fine-grain CM 247 LC superalloy.

3.4 AES analysis

AES was employed for interfacial segregation analysis to investigate the effect of Zr on microstructural characteristics. Standard AES samples with 0.015 mass% and with 0.15 mass% Zr were prepared and treated with liquid nitrogen for 4 h before being impact-tested. Figure 5(a) displays a secondary image of the fractured surface of a 0.015 mass% Zr specimen, wherein five typically distinct areas are marked as area 1 to area 5. In this analysis, no or a limited amount of Zr is detected in areas 1 to 5. Figures 5(b)–(c) are the corresponding Auger spectra, revealing areas that correspond to the matrix (area 3) and the MC carbide (area 5), respectively. Apparently, a limited amount of Zr is detected in the matrix and in the interior of the carbide. Moreover, Fig. 6 presents a secondary image of the fractured surface of a 0.15 mass% Zr specimen, wherein three typically distinct areas are selected and marked, including areas 1 to 3. Figures 7(a)–(c) are the corresponding Auger spectra, revealing boride (area 1), carbide (area 2) and matrix (area 3). Clearly, the Zr peaks in the spectra of the areas are detected, indicate Zr enriches at the boride/matrix interface, at the carbide/matrix interface, and at the grain boundaries. Furthermore, the depth of Zr segregation is determined in three distinct areas (areas 1 to 3) selected for ion sputtering. The speed of ion sputtering is 0.8 nm/min. Figures 7(d)–(f) show the test spectra of areas 1 to 3 following ion sputtering. Firstly, no or limited Zr is detected after 10 mins of sputtering in area 1. Zr is thus shown to segregate at the boride/matrix interface. Secondly, a large quantity of Zr is detected after 25 mins of sputtering in area 2. Zr is thus proven to be in solid solution in MC carbide when Zr is added. Finally, no or limited Zr is detected after 20 mins of sputtering in area 3, implying that Zr segregates at the grain boundaries. Those results indicate that Zr segregates at the boride/matrix interface or at the grain boundaries, and in solid solution in MC carbides.

4. Discussion

4.1 Mechanisms of Zr segregation strengthening

One of the most remarkable alloying effects in a superalloy is the enhancement of creep properties when minute amounts of boron and zirconium are added.⁸⁾ Boron and zirconium clearly retard the grain boundary cracking. The case of early U-500 alloy is a good example.⁸⁾ In U-500 without boron and zirconium, microcracks developed at the end of first-stage creep at 23 h; but with boron and zirconium, microcracks did not develop until third-stage creep at 214 h. A general mechanism of the above effects is that odd-size atoms (21–29% oversize or undersize) segregate at grain boundaries, filling vacancies and reducing grain boundary diffusion.⁹⁾

A later work¹⁰⁾ has revealed a new mechanism, by which zirconium has been found to form $Zr_4C_2S_2$, significantly reducing the amount of elemental sulfur at the grain boundaries and retarding the generation of grain boundary cracking. In this work, however, the contents of S, P and

Fig. 4 SEM micrographs showing the distribution and size of γ' phase of fine-grain CM 247 LC superalloy with 0.015 mass% Zr after heat treatment (HIPing + 1494 K/2 h + 1144 K/20 h) and the observation at (a) grain boundary, and (b) the center of grain as well as fine-grain CM 247 LC superalloy with 0.15 mass% Zr after heat treatment (HIPing + 1494 K/2 h + 1144 K/20 h) and the observation at (c) grain boundary, and (d) the center of grain.

Table 2 Tensile test results of fine-grain CM 247 LC with various Zr contents. (Material Condition: HIPing + 1494 K/2 h + 1144 K/20 h)

Condition	Test Temp. (K)	U.T.S. (MPa)	Y.S. (MPa)	Elongation (%)
CZ1(0.015 mass% Zr)	300	1242	1020	8.2
CZ2(0.040 mass% Zr)	300	1238	1026	7.7
CZ3(0.080 mass% Zr)	300	1273	1062	8.0
CZ4(0.150 mass% Zr)	300	1230	1095	7.6
CZ1(0.015 mass% Zr)	1033	1190	1020	8.3
CZ2(0.040 mass% Zr)	1033	1215	1040	8.7
CZ3(0.080 mass% Zr)	1033	1215	1049	8.8
CZ4(0.150 mass% Zr)	1033	1220	1053	9.7

Table 3 The creep test results of fine-grain CM 247 LC with various Zr contents at 1033 K/725 MPa. (Material Condition: HIPing + 1494 K/2 h + 1144 K/20 h)

Condition	Creep life (h)	Elongation (%)	1% Creep life (h)
CZ1(0.015 mass% Zr)	40~61	3.0~3.3	37
CZ2(0.040 mass% Zr)	110~113	3.3~3.8	51
CZ3(0.080 mass% Zr)	124~140	3.8~4.7	53
CZ4(0.150 mass% Zr)	154~162	4.8~5.0	67

Table 4 The creep test results of fine-grain CM 247 LC with various Zr contents at 1200 K/345 MPa. (Material Condition: HIPing + 1494 K/2 h + 1144 K/20 h)

Condition	Creep life (h)	Elongation (%)	1% Creep life (h)
CZ1(0.015 mass% Zr)	15–23	7.8–9.4	9
CZ2(0.040 mass% Zr)	28–32	9.8–10.5	12
CZ3(0.080 mass% Zr)	35–38	10.3–11.0	14
CZ4(0.150 mass% Zr)	36–38	10.2–10.5	15

Fig. 5 Fine-grained CM 247 LC superalloy with 0.015 mass% Zr (a) the area of the AES analysis, (b) the spectrum at area 3, and (c) the spectrum at area 5.

other trace elements were far below 5 ppm. Therefore, the $Zr_4C_2S_2$ phase was observed neither at the matrix/carbides interfaces nor in the GBs, according to the analysis of AES in fine-grain CM 247 LC with various Zr contents. Thus, an improvement of the high-temperature mechanical properties of fine-grain CM 247 LC may not be directly related to the

Fig. 6 The area of the AES analysis for fine-grain CM 247 LC superalloy with 0.15 mass%Zr.

getting mechanism.

According to the present AES results, Zr enriches at austenitic boundaries, carbide or the boride/matrix interfaces. Computer simulations¹¹⁾ have suggested that micro-cavities at grain boundaries are on the atomic scale and extremely dense. Hence, the grain boundary can be considered effectively as a very thin layer of porous material, which has a very low effective elastic modulus and, therefore, reduced shear stress transmission. In order to minimize the energy, Zr thus tends to fill lattice discontinuities at interface boundaries or grain boundaries, increasing the structural regularity and the strength of bonds between the angulated lattices.

4.2 Mechanisms of Zr solid-solution strengthening

Nickel-based superalloy is strengthened primarily by the γ' phase in the matrix. The Ni-Al phase diagram indicates that the Ni_3Al phase has a broad range of chemical compositions. A region with an Al content from 23 to 27.5 at% is single-phase, and the Al content increases as the temperature increases.¹²⁾ The broad range of chemical compositions implies that the stable secondary phase can be formed easily. The atoms of the Ni_3Al are replaced by three types of the elements, that depending on the positions of the replacements.¹³⁾ Replacement at the position of the Al atom is by such atoms as Si, Ge, Ti, V, Hf, Zr, Mo, W, or Nb, whereas replacement at the position of the Ni atom is by such atoms as Cu, Co, or Pt, and replacement at positions of both Ni and Al atoms are by such atoms as Fe, Mn, or Cr. Hf and Zr are the most effectively strengthening elements in Ni_3Al , because of the size of the atoms, difference between the

Fig. 7 The spectrum of the AES analysis before ion sputtering at (a) area 1, (b) area 5, (c) area 3 for fine-grain CM 247 LC superalloy with 0.15 mass% Zr and after ion sputtering at (d) area 1, (e) area 2, (f) area 3.

valence of these atoms, the APB energy, and the energy associated with cross-slip on the (100) face.

Liu¹⁴⁾ and Hsu¹⁵⁾ found two reasons for strengthening by Hf and Zr. Firstly, both Hf and Zr increase the CRSS (critical resolved shear stress) on the (100) face and only weakly affect the (111) face. Thus, it increases the temperature of transfer from the $\{111\}\langle 110 \rangle$ slip system to the $\{100\}\langle 110 \rangle$ slip system. However, some elements, such as Mo, Ta, Nb, Ti and W, decrease the CRSS on the $\{100\}\langle 110 \rangle$ slip system. Secondly, both Hf and Zr reduce the APB energy, increasing the rate of the cross-slip from $\{111\}$ to $\{100\}$ associated with super-dislocation. This study found little evidence of Zr segregation in 0.015 mass% Zr specimens. Clearly, Zr prefers to dissolve in solid-solution in matrix (γ or γ') of 0.015 mass% Zr specimens, because the Zr content is relatively low.

4.3 Effect of Zr on carbide morphology

In the late 1970s, alloy designers added Hf to nickel-based superalloys to produce the γ - γ' eutectic phase and enhance the GB's toughness.¹⁶⁻¹⁸⁾ However, nickel-based cast super-

alloy remained brittle at elevated temperatures.¹⁹⁾ Fractographic observations revealed that cracks are preferentially initiated at the MC carbide/matrix interface in Mar-M247 superalloys at elevated temperature.²⁰⁾ The same conclusion was reached by Kaufman,²¹⁾ who proved that script-like MC carbides are extremely brittle and may act as crack initiation sites or crack propagation paths. Baldan²²⁾ also noted that the size of the carbide particles dominates the creep rupture ductility, and that fine dispersion increases creep ductility.

Bhambri *et al.*²³⁾ proposed that MC carbide is faceted and octahedral with $\{111\}$ faces and rapid growth in the $\langle 100 \rangle$ direction during solidification. Accordingly, elongated or script-like morphology of MC carbide is formed. As mentioned previously, Zr addition has a strong tendency to dissolve in carbide, possibly affecting the transport of carbon and carbide-forming elements, such as Ta, Hf, or Ti. Therefore, the addition of Zr to superalloy possibly results in the isotropic growth of carbides during solidification, and eventually leads to the refinement and spheroidization of MC carbide in fine-grain CM 247 LC superalloy.

4.4 Effect of Zr on mechanical properties

Tensile test results can be explained by the Hall-Petch equation; $\sigma = \sigma_0 + Kd^{-1/2}$, where σ is the yield stress; σ_0 is the “fraction stress,” representing the overall resistance of the crystal lattice to dislocation movement; K is the “locking parameter,” which measures the relative contribution of the grain boundaries to hardening, and d is the grain size. In this study, d is constant, such that σ_0 and K strongly affect the strength of fine-grain CM 247 LC alloy. Zr prefers to be in solid solution in matrix (γ , γ' or carbide), so σ_0 increases as Zr is added. Zr additions also appear to annihilate grain-boundary vacancies, result in increasing the value of K thus increasing the structural perfection and increasing bond strength between the GBs. Hence, the tensile strength increases with the amount of minor Zr addition at 300 K. The strength of the grain boundary determines the high-temperature strength of polycrystalline alloys, because the grain boundary is weaker than the inside of the grains at elevated temperature. The results concerning the fine-grain CM 247 LC superalloys show that minor Zr addition may alter the carbide morphology which promotes the retardation of the dislocation gliding and crack propagation. Therefore the yield strength and elongation of fine-grain CM 247 LC alloy increase with the Zr content at 1033 K.

Based on the creep tests results, the initiation and propagation of cracks during creep are clearly retarded in

fine-grain CM 247 LC with 0.15 mass% Zr, substantially prolonging the rupture life. According to the creep deformation map of a CM 247 LC-related superalloy,²⁴⁾ the creep mechanism at 1033 K/725 MPa involves the gliding of dislocations and the creep mechanism at 1200 K/345 MPa involves the sliding of grain boundaries and the climbing of dislocations. Thus, creep behavior is dominated by the interaction between dislocations and obstacles such as grain boundaries, precipitates or carbides. Figure 3 indicates that fine-grain CM 247 LC castings with more than 0.04 mass% Zr exhibit sparse script-like carbides with a tendency to have a blocky or discrete carbide morphology, which promotes the retardation of the dislocation gliding and the grain boundary sliding, and to form barriers to the crack propagation. Additionally, Zr was detected to enrich at the grain boundary, which leads to spread the deformations that reduce stress concentrations at grain boundaries, substantially retarding crack initiation and propagation, and increasing the rupture life and elongation. Moreover, a mixed crack propagation mode is observed (Figs. 8(a)–(d)) in fine-grain CM 247 LC at 1033 K/725 MPa and 1200 K/345 MPa. The crack propagation mode associated with a fine-grain structure is determined to be principally intergranular and transgranular, cracking along the grain boundary is less in CZ4 (0.15 mass% Zr) specimens.

Fig. 8 Fractography of fine-grain CM 247 LC superalloy with (a) 0.015 mass% Zr, and (b) 0.15 mass%Zr tested at 1033 K/725 MPa and fine-grain CM 247 LC superalloy with (c) 0.015 mass% Zr, and (d) 0.15 mass% Zr tested at 1200 K/345 MPa.

5. Conclusion

- (1) Minor Zr additions inhibit the formation of script-like MC carbides and cause the refinement and spheroidization of MC carbides, while not affecting the sizes of grains.
- (2) Minor Zr additions promote the yield strength at 300 K as well as both the yield strength and elongation at 1033 K, reducing the possibility of bursting of the hub in small gas turbine engines.
- (3) Minor Zr additions effectively increase creep life and rupture elongation. In particular, the creep life of fine-grain CM 247 LC superalloy with 0.15 mass% Zr is double that of the alloy with 0.015 mass% Zr at either 1033 K/725 MPa or 1200 K/345 MPa.
- (4) AES observations reveal that Zr may enrich at grain boundaries and boride/matrix interfaces, and become solid solution in the carbide and matrix, possibly changing the primary MC carbide morphology and inhibiting the formation of script-like carbide. These tendencies promote the accommodation of stress and retard the initiation and propagation of cracks, increasing the tensile strength and creep resistance of the alloy.
- (5) The experimental results demonstrate that optimal Zr additions promote the performance of fine-grain CM 247 LC superalloys in small gas turbine engines, by increasing yield strength at operating temperature of the hub, protecting the parts from bursting, and increasing creep strength under operating condition of the blade.

Acknowledgements

The authors would like to appreciate the National Science Council of the Republic of China for financially supporting this research under Contract No. NSC 91-2623-7-002-008.

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