Effect of fine-grained structure on the mechanical properties of superalloys K3 and K4169

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Abstract

The fine-grained structures of superalloys K3 and K4169 were achieved by the addition of refiners. Test bars for the determination of mechanical properties were cast under the chosen conditions to study the tensile properties at room and intermediate temperatures, and the stress rupture properties at intermediate temperatures. Results show that for alloy K3, the yield and tensile strengths of the fine-grained samples are superior to those of the conventional ones at room and intermediate temperatures, but there is little difference in tensile plasticity. The stress rupture life of the fine-grained sample is much longer than that of the conventional one at 750°C, whereas it has no remarkable change at 800°C. For alloy K4169, the yield and tensile strengths of fine-grained samples are still superior to those of the conventional ones at room temperature and 760°C. In addition, the stress rupture life of the fine-grained sample is 1.1 times longer than that of the conventional one at 760°C. However, the permanent plasticity almost remains the same. The fracture of the samples was examined by scanning electron microscopy (SEM) and the fracture mechanisms were investigated. © 2001 Published by Elsevier Science Ltd.

Keywords: Superalloy K3; Superalloy K4169; Grain refinement; Mechanical properties; Fractography

1. Introduction

The major advantage of fine grain structures over conventional cast structures is the improvement in low cycle fatigue life, uniformity of properties, and comprehensive properties. Since the 1980s, integral fine-grained cast techniques have been extensively progressed [1,2]. Grain size can be controlled in three basic ways: thermally, chemically, or mechanically [3].

Much attention has been paid to the improvement in mechanical properties of castings refined in thermal or mechanical ways [4,5]. Contrarily, investigation into the mechanical properties of castings after grain refinement by the addition of refiners is still lacking intensive research. In a previous study [6], the present authors obtained fine-grained structures for superalloys K3 and K4169 by the addition of refiners to the alloy melt. This work is aimed to investigate the effect of fine-grained structure on the mechanical properties within different temperature ranges of both alloys and the fracture mechanism.

2. Experimental procedures

Mechanical-property test bars of superalloys K3 and K4169 were firstly cast in a VSG type vacuum melting and casting unit under conventional and fine-grained cast conditions (see Table 1). Then the test bars were processed into property samples according to Chinese National Standard GB 6397-860. Tensile properties at room and intermediate temperatures, and the stress rupture properties at intermediate temperatures were measured.

As for alloy K3, the tensile properties at room and intermediate temperatures (750 and 800°C) were tested with INSTRON 1195 and INSTRON 1196 universal strength tester, respectively. Stress rupture properties at intermediate temperatures were tested with MII tester. As for alloy K4169, tensile properties at room temperature and 760°C were tested with WJ-10B mechanical universal strength tester and ZDM-5 tensile tester, respectively, and the stress rupture properties at 760°C with SATE-JE tester. Fracture surfaces of the samples were examined by scanning electron microscope (SEM).

3. Results and discussion

3.1. Alloy K3

3.1.1. The tensile properties of alloy K3 at room and intermediate temperatures

Table 2 shows the data of the tensile properties. It can be...
Table 1
Process parameters for the test bars of alloys K3 and K4169 (\(T_p\), melt superheating temperature; \(T_p\), melt pouring temperature; \(T_m\), mould preheating temperature)

| Superalloy | Sample | Refiner | Content (wt%) | \(T_s\) (°C) | \(T_p\) (°C) | \(T_m\) (°C) | Grain size (min) |
|------------|--------|---------|---------------|-------------|-------------|-------------|-----------------|
| K3         | a      | –       | –             | 1550        | 1400        | 900         | 4.9             |
|            | b      | A + B   | 0.6           | 1550        | 1400        | 900         | 1.5             |
|            | g      | –       | –             | 1550        | 1420        | 850         | 4.3             |
| K4169      | h      | C       | 0.4           | –           | 1420        | 850         | 1.2             |

Table 2
Tensile properties of alloy K3 at room and intermediate temperatures

| Temperature (°C) | Sample | \(\sigma_{0.2}\) (MPa) | \(\sigma_t\) (MPa) | \(\delta\) (%) | \(\psi\) (%) |
|-----------------|--------|------------------------|-------------------|---------------|-------------|
| Room temperature| a      | 805                    | 970               | 9             | 10.5        |
|                 | b      | 885                    | 1070              | 14            | 6.5         |
| 750             | a      | 785                    | 950               | 3.5           | 8.0         |
|                 | b      | 885                    | 1020              | 4.0           | 4.5         |
| 800             | a      | 765                    | 915               | 7.0           | 7.0         |
|                 | b      | 880                    | 995               | 3.5           | 6.5         |

seen that the fine-grained sample has a higher yield strength (improved by 9.9% at room temperature, 12.7% at 750°C, and 15% at 800°C, respectively) and tensile strength (improved by 10.3% at room temperature, 7.3% at 750°C, and 8.7% at 800°C, respectively) than the conventional one. At room temperature and 750°C, there is no obvious difference in tensile plasticity between the fine and conventional grain structures. In contrast, the reduction in area decreases after grain refinement. It is believed that although well-distributed MC carbides are advantageous to improve plasticity, the addition of trace Zr — one component of refiner B — causes the incipient temperature of alloy K3 to decrease [7]. Therefore, the amount of eutectic increases, aggravating local segregation in front of the eutectic. Moreover, segregation areas might be the source of a crack. As a result the reduction in area reduces.

At 800°C, the plasticity level reduces after grain refinement, which may result from oxidation at grain boundaries. That is, the precipitates at grain boundaries have a lower ability than the \(\gamma\) matrix to resist oxidation. Furthermore, the crack propagation rate is faster at grain boundaries. Therefore, oxidation causes a crack at the surface of grain boundaries.

3.1.2. The stress rupture properties at intermediate temperatures

The test results are shown in Table 3. The fine grain has a much longer stress rupture life than the conventional one at 750°C, which is ascribed to the grain boundary strength being greater than the strength within the grains at 750°C. Thus, fine-grained structures are helpful to prevent crack propagation. However, there is no evident change at 800°C. That is the grain boundary strength is almost equivalent to the strength within the grains, indicating that 800°C is close to the equal strength temperature for alloy K3.

3.1.3. The mechanism of the fracture

The typical morphologies of brittle and mixed fractures are shown in Fig. 1. After examination with SEM, it can be concluded that the fracture of the fine grain tensile samples is mixed cracking at room and intermediate temperatures and that the fracture of the conventional tensile samples is brittle at room temperature and mixed fracture at intermediate temperatures. The fracture of the stress rupture properties of conventional and fine-grained samples at 760 and 800°C is mixed.

3.2. Alloy K4169

3.2.1. The tensile properties at room temperature and 760°C

The data of tensile properties at room temperature and 760°C are listed in Table 4. Apparently, at room temperature,
the fine-grained sample has higher yield and tensile strengths and tensile plasticity, which can be attributed to grain refinement and uniformity of structures. The plastic deformity is limited to comparatively small areas and distributed homogeneously. In addition, it is difficult for a crack to nucleate because Laves phase and MC carbides are reduced in size and distribute well. It can be seen that at 760°C, the strength of the fine-grained structure is still superior to that of the conventional one. Although the temperature rise results in the decrease of grain boundary strength and strength within grains, it can be concluded from the experiment results that the former is still higher than the latter at 760°C. Meanwhile, elongation improves 73% and reduction in area decreases slightly at 760°C after grain refinement.

3.2.2. The stress rupture properties at intermediate temperature of 760°C

The stress rupture properties at a temperature of 760°C and a stress of 480 MPa are listed in Table 5. It can be seen that the stress rupture life of the fine-grained sample is 1.1 times longer than that of the conventional one. This should be ascribed to grain refinement and uniform-distribution of grains. Because the grain boundaries can curve the interface, the nucleation speed and crack propagation for fine grain is much slower than that for the conventional one. However, there is no obvious difference in permanent plasticity between the two samples.

3.2.3. The mechanisms of the fracture

Fig. 2 shows the typical morphologies of tenacious dense and cleavage steps appearing in alloy K4169.

3.2.3.1. The tensile fracture at room temperature. For the fracture of fine-grained structures, there is a large amount of tenacious dense steps and there is no obvious cleavage step. As a result of grain refinement, the size of MC carbides and Laves phase decreases. Moreover, they are distributed uniformly. Therefore, crack forms in these limited local areas and tenacious fracture occurs. For conventional grain structures, there are few tenacious dense structures in fracture and the stress is easily assembled at grain boundaries because of large sized MC carbides and Laves phase. Under the enormous stress, because the deformity of coarse grain is not easily adjusted, cracks propagate along the cleavage facets at grain boundaries. When the cracks meet spiral dislocation or grain boundaries, the cleavage steps form.

3.2.3.2. The tensile fracture at 760°C. The fracture of the conventional grain structures at 760°C is somewhat smooth, and there is obvious macro-contraction. Consequently, it can be concluded that plasticity deformity has occurred before rupture. Observed by SEM, almost the same amount of cleavage steps and tenacious dense structure can be found, indicating that the crack is mixed. At 760°C, although the grain boundary strength decreases, the coarse grains can mitigate stress aggregation at grain boundaries by deformity among grains. Therefore, it is difficult for cracks along cleavage facets to propagate compared with crack propagation at room temperature. At

Table 4
The data of tensile properties of alloy K4169 at room and intermediate temperatures

| Temperature (°C) | Sample | $\sigma_{0.2}$ (MPa) | $\sigma_{b}$ (MPa) | $\delta$ (%) | $\varphi$ (%) |
|------------------|--------|---------------------|-------------------|--------------|--------------|
| Room temperature | g      | 590                 | 805               | 7            | 9            |
|                  | h      | 6                   | 850               | 17           | 14           |
| 760              | g      | 505                 | 610               | 7.5          | 20           |
|                  | h      | 515                 | 660               | 13           | 18           |

Table 5
The data of stress rupture properties

| Sample | Stress rupture life (min) | $\delta$ (%) | $\varphi$ (%) |
|--------|--------------------------|--------------|--------------|
| g      | 15.45                    | 8.80         | 11.94        |
| h      | 33.15                    | 9.20         | 7.84         |
the same time, tenacious dense structure form at the precipitates.

The fracture of the fine-grained structures seems to be uneven and there are more tenacious dense structures than cleavage steps. At 760°C, the strengths within the grains and between grain boundaries both decrease compared with those at room temperature, which leads to more serious local stress concentration. When the direction of stress is favourable for crack to propagate along certain cleavage facets, local cleavage crack appears. Despite that, the amount of tenacious dense structure is much more than that of cleavage steps at 760°C.

3.2.3.3. The fracture of the stress rupture. Since there is no significant difference in permanent plasticity between fine and conventional grains, the fractures of both are similar. Their fractures have nearly the same amount of tenacious dense structure and cleavage steps, suggesting that the fracture is mixed.

Although there is little difference in permanent plasticity, great difference exists in rupture life between conventional and fine-grained structures. The reason is that the size of grains influences the ability of structures to resist stress. For conventional grain structures, no matter what kind of crack, tenacious or cleavage crack, the propagation of a crack takes place in one or several grains, thus cracks propagates fast. However, for fine-grained structures, both tenacious and cleavage cracks appear in small areas because of fine grain size. Therefore, the resistance against the propagation of cracks is great. As a result, the stress rupture life of fine-grained structures has been improved significantly.

4. Conclusions

From the above experimental results, some important conclusions can be drawn:

1. For alloy K3, the yield and tensile strengths of the fine-grained samples are superior to those of the conventionally cast ones at room temperature, 750 and 800°C. However, tensile plasticity has no obvious change at room temperature and 750°C, and it decreases at 800°C after grain refinement. The stress rupture life of the fine-grained sample is much longer than that of the conventional one at 750°C, but it has no obvious difference at 800°C.

2. For alloy K3, the fracture of the fine-grained tensile samples is mixed at room and intermediate temperatures, and the fracture of the conventional tensile samples is brittle at room temperature and mixed at intermediate temperature. The fracture of conventional and fine-grained stress rupture samples at 760 and 800°C is mixed.

3. For alloy K4169, the yield and tensile strengths of the fine-grained test bars are superior to those of the conventional ones at room temperature and 760°C, and the tensile plasticity is also higher for fine grains. The stress rupture life of the fine-grained sample improves 110% compared to that of the conventional one at 760°C, whereas the permanent plasticity remains almost the same.

4. For alloy K4169, the fracture of the fine-grained tensile samples is tenacious at room and intermediate temperatures. The fracture of the conventional tensile samples is brittle at room temperature and mixed at intermediate temperature, and the fracture of conventional and fine-grained stress rupture samples is mixed.

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