High temperature tensile properties and fracture behavior of cast nickel-base K445 superalloy

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Abstract: The tensile properties and fracture behavior of a cast nickel-base superalloy K445 in the temperature range of 25−1 000 °C were investigated. The microstructure and fracture surfaces of the alloy were investigated by OM, SEM and TEM. The results revealed that an anomalous yield strength phenomenon exists in the alloy at medium high temperature. The yield strength decreases gradually with the increase of temperature, reaches the minimum value at 650 °C, and then increases again to obtain 940 MPa, which is almost the same as that at 400 °C. While the ultimate fracture strength attains the maximum value at 750 °C, the yield strength and fracture strength decrease entirely. The elongation and fracture shortage almost keep increasing trend with the increase of testing temperature, except the little drop at 400 °C and 900 °C. The fracture surface of K445 alloy below 400 °C exhibits most cleavage characteristic with some dimples. With the increase of temperature, the ductile feature increases, but the cleavage still dominates the fracture till 650 °C. Therefore, it can be concluded that the secondary hardening effect results in the high yield strength and fracture strength with better ductility, which makes it more appropriate to be used at medium high temperature.

Key words: nickel-based K445 superalloy; anomalous yield strength; tensile properties; fracture surface

1 Introduction

Cast nickel-base superalloys are widely used in manufacturing of modern gas turbine components, especially in the first stage turbine blades and vane, which are required to have high temperature strength, excellent oxidation and corrosion resistance, excellent fatigue and creep resistance and service reliability to fit for application at high temperature. Therefore, the cast nickel-base superalloys are continually developed to increase their thrust, operating efficiency and durability [1]. Recently, with the development of the land-base and aerospace gas turbine engines, the turbine inlet temperature increases year by year to satisfy the requirement of the thermal efficiency, so it is necessary to develop new materials to meet the requirements [2–4].

The nickel-based K445 superalloy is a kind of material that was designed and employed as high pressure turbine blades working in high performance industrial gas turbines. Due to its high Cr and Co content, the K445 superalloy has excellent hot corrosion resistance and high temperature oxidation resistance. Benefiting from the good combination of refractory elements and γ′ forming elements, it possesses superior high temperature properties, compared with other Ni-base superalloys [5]. However, the previous study [6] reported that low ductility at 800 °C called intermediate temperature brittleness (ITB) was observed, because of the relative high contents of W, Mo, Ta and Nb. In order to increase the strength of grain boundary, B and Zr elements are added to the superalloy [7], and the role of those alloying elements in Ni-base superalloys have been discussed previously [8].

Generally, in order to improve the hot resistance, the amount of γ′ in the γ'-strengthened superalloys almost reached about 70% [2]. According to the former investigations [9, 10], the mechanical properties at high temperature of the superalloy are related closely to the γ′ strengthening precipitates. The total mass fraction of γ′ in K445 superalloy is higher than 60%, so the high temperature behavior of the alloy depends strongly on the γ′ precipitates. In addition, the K445 superalloy is composed of γ matrix, bimodal γ′ precipitates, γ-γ′...
eutectics, carbides and a small amount of deleterious intermixture. The ′ phase is a superlattice with a nominal composition of Ni$_3$(AlTi), which may be considered derivatives of the best known L1$_2$ long range ordered intermetallic phase Ni$_3$Al [11, 12]. The volume percent and morphology of ′ precipitates depend on the alloys composition and lattice mismatch parameters [13]. The higher the volume percent of ′, the more effective the shear mechanism operating in ′ particles to alloy deformation.

To the superalloy, the high temperature creep and fatigue behavior are very important, due to its extreme operating conditions in gas turbine. Many studies have been carried out to understand the relationship between the creep strength and the amount of ′ phase. The tensile properties are also very important to the superalloy, but till now, few research has been done to study the relation between high temperature tensile strength or elongation and amount of ′ phase. The recent study [14] on the superalloy revealed that its tensile properties, elastic modulus and shear modulus relate with the ′ precipitates in the ′ strengthened superalloy. Therefore, the high temperature tensile properties and fracture morphology of the K445 superalloy are studied to further understand the deformation and fracture mechanism of the ′/′ dual phase alloy in this work.

2 Experimental

The alloy was remelted in a VIM25F vacuum induction furnace and cast into round bars with diameter of 16 mm and length of 130 mm. The casting mold preheating temperature and pouring temperature were 950 °C and 1 430 °C, respectively. The composition of the K445 alloy is shown in Table 1. The investigated samples were cut from the as-cast bars, and heat-treated to obtain the desired ′ precipitation microstructure. The heat treatments consisted of a solution treatment of (1120 °C, 2 h, air cooling) and an aging treatment of (850 °C, 24 h, air cooling).

### Table 1 Composition of K445 alloy (mass fraction, %)

<table>
<thead>
<tr>
<th>C</th>
<th>Cr</th>
<th>Co</th>
<th>W</th>
<th>Mo</th>
<th>Ta</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.094</td>
<td>14.15</td>
<td>10.07</td>
<td>4.24</td>
<td>1.55</td>
<td>4.86</td>
</tr>
<tr>
<td>Ti</td>
<td>Al</td>
<td>B</td>
<td>Zr</td>
<td>Ni</td>
<td></td>
</tr>
<tr>
<td>2.62</td>
<td>4.02</td>
<td>0.029</td>
<td>0.026</td>
<td>Bal.</td>
<td></td>
</tr>
</tbody>
</table>

The standard specimens for tensile test with a diameter of 5 mm and a gage length of 25 mm were machined longitudinally from the heat-treated samples. The tensile tests were performed on a universal AG-250KNE test machine in air with an initial strain rate of $5 \times 10^{-5}$ to $1.04 \times 10^{-4}$ s$^{-1}$ at 25−1 000 °C. The tensile specimens were induction heated and the temperature was measured using the thermocouples placed in the gauge length. The temperature gradient over the gage length did not exceed ±2 °C. The inductive transducers could detect strain variations quickly as small as $5 \times 10^{-5}$ and load continuously which were recorded directly on an X-Y recorder. The tensile tests were run to rupture. Two identical specimens were tested at the same condition to obtain a tensile property.

Samples for microstructure observation were cut from the as-cast and heat-treated and fractured specimens. The samples were fabricated by conventional method and electrochemically etched with an electrolyte consisting of 25% H$_3$PO$_4$. The microstructure and fracture surface after the tensile tests were examined with optical microscopy and scanning electron microscopy (SEM) with energy-dispersive X-ray spectroscopy (EDS). The slices for transmission electron microscopy (TEM) observation were cut from the gauge part of the specimens deformed at different temperatures normal to the loading axis. The thickness of the slices was about 0.5 mm and polished to 50 μm. The polished slices were shaped into 3 mm in diameter followed by ion milling to perforation. The TEM observation was carried out in a JEM-200CX operated at 120 kV.

3 Results and discussion

3.1 Microstructure of heat treated K445 superalloy

The typical microstructure of the heat treated K445 superalloy is shown in Fig. 1(a). It can be seen that the alloy has a typical dendritic morphology, which consists of γ matrix, ′ particles, chrysanthemum-like ′/′ eutectic and carbides. All of the heat-treated specimens still maintain the columnar matrix grain morphology. All of the microstructures shown here were analyzed by X-ray diffraction to identify the preferred orientation or annealing texture. The serrated grain boundaries prevent the grain boundary gliding and enhance creep strength [15, 16]. TEM observation on the ′ precipitates is shown in Fig. 1(b). The primary ′ cuboids with average edge length of 600 nm are produced during solution under 1 120 °C. The secondary sphere ′ with average 60 nm in diameter is produced during aging. The eutectic distributed at dendrite boundaries, is often near micropores, which indicates they form at the end of solidification. The eutectic size varies from 5 to 15 μm. Micropores with various sizes are observed in the alloy, as shown in Fig. 1(c). They are solidification defects, which affect the ductility obviously [16]. Carbides
mainly distribute along the grain boundaries, as shown in Fig. 1(d). The EDS analysis reveals that the carbides uniformly distributed within the grains are MC type carbides, in which M is substituted for W, Ta and Ti. While the carbides found at grain boundaries are M$_2$C$_6$ type carbides, in which M refers to Co and Mo. Moreover, some small precipitates are found in the carbides, and EDS analysis shows that they are oxides, nitrides or carbonitride particles, which can act as the nuclei [16]. In fact, the effect of carbide on properties is contradictory. They can strengthen the grain and interdendritic boundaries, retarding boundary migration, hardening the matrix [17], but they also can become the origin of fracture [18].

### 3.2 Tensile properties

The tensile properties of the heat-treated K445 alloy at different temperatures are shown in Figs. 2(a) and (b), which are really different from the former research [19]. Generally, with the increase of testing temperature, the yield strength and tensile strength decrease monotonously [20, 21]. However, in the present alloy, the yield strength and tensile strength of the K445 alloy decrease with the increase of temperature and reaches the minimum at 650 °C, but they increase obviously again at 750 °C and reach the value of 940 MPa and 1040 MPa, respectively. When the testing temperature increases further, the yield strength and tensile strength drop gradually. The temperature dependence of ductility is different from the strength, as shown in Figs. 2(c) and 2(d). The changes of elongation and fracture shortage between 600–750 °C exhibit the same trend as the strength, while in the other temperature the changes are almost inverse. What is interesting is that the elongation and fracture shortage decrease slightly after 900 °C and then increase a little again.

### 3.3 Fracture surface morphology

The morphology of fracture surface of the tensile specimens tested at room temperature, 400 °C and 650 °C is shown in Fig. 3. Clearly, the quasi-cleavage and decohesion combined with some dimples are the main characteristics in the specimen tested at room temperature, as shown in Figs. 3(a)–(c). In general, the alloy exhibits semi-brittle features at room temperature, so it can be seen that the elongation and fracture shortage are so low at this condition. Though there are some dimples, the tear-off dimples are not elongated, so the deformation taken place by the shear mode is limited [14]. Facets develop in conjunction with casting pores, which appear as irregular holes with a rounded surface morphology.
At 400 °C, the fracture morphology shows the cracked MC carbide and decohesion along the carbide/matrix interface, as shown in Fig. 3(d). Most of the carbides are present at interdendritic regions. The EDS analysis also confirms that a number of carbides appear on the fracture surface. Most of the carbides are determined to be MC, which can lead to initiate microvoids at grain boundaries. These carbides along the interface of matrix would lead to dimples and provide strong strengthening effect, which is beneficial to the mechanical properties of the alloy. However, cracks between coarse carbide particles and matrix propagate easily, which makes fracture a cleavage characteristic. When the tested temperature increases to 650 °C, the cleavage decreases and the dimple increases obviously, as shown in Fig. 3(e). Therefore, it can be found that the elongation increase a little.

The morphology of fracture surface of the alloy tested at 750 °C is shown in Fig. 4. It can be seen that the flowery cleavage feature appears in some adjacent regions, as shown in Fig. 4(a). Further observations find some small dimples and amount of carbides distributed on the fracture surface, as shown in Fig. 4(b). Regardless of the tensile test variation (microstructure, temperature and strain rate), several of the fractured specimens show intergranular-type secondary cracks. Particularly, the microstructures are coarse. In addition, long intergranular cracks form along the columnar matrix grain boundaries, as shown in Fig. 4(c).

The observation on the specimen tested at 800 °C reveals that many cleaved carbides decorate in the ductile fracture area randomly, as shown in Fig. 5(a). Transcrystalline crystallographic facets are more frequently observed, suggesting that slide plane decohesion plays an important role in the fracture process [14]. Fracture surfaces of the specimens tested at 850 °C show some unique features, as shown in Fig. 5(b). The dimples are large, 5–15 μm in size. Among the dimples, distinct cleavage facets can be observed, as shown in Fig. 5(c). It can be postulated that the γ′ begins to soften, when the temperature exceeds 850 °C, and more dislocations are activated. Therefore, the deformation is more homogeneous, and plenty of dimples are observed on the fracture surface. All the
Fig. 3 Fractographs of specimens after tensile test to fracture:
(a) At room temperature, a faceted-cleavage type fracture surface; (b) Cleavage fracture with steps in some regions in (a) at higher magnification; (c) At room temperature, irregular holes with rounded surface morphology; (d) At 400 °C, cracked MC carbides and decohesion at carbide/matrix interface; (e) At 650 °C, quasi-cleavage and dimple fracture pattern.

Fig. 4 Fractographs showing mixed quasi-cleavage fracture in specimens tensile tested at 750 °C: (a) Mixed quasi-cleavage fracture morphology; (b) Small dimples and amount of carbides on fracture surface; (c) Intergranular fracture morphology.

Many investigations have been carried out to study the unusual variation of tensile properties at high temperature in superalloy. Most of all, the effect of dislocations in $\gamma'$ precipitates is quite important to the anomalous yield behavior. Dislocations dissociate in partials, because the Burgers vectors of these dislocations in $\gamma'$ precipitates are bigger than those in other FCC structures such as $\gamma$. Dissociations in L1$_2$ $\gamma'$ structure are more complex [6]. Secondly, the former research showed that the strength of $\gamma'$ increases with the rise of temperature at relative low temperature. Therefore, the decrease of yield strength by the $\gamma$ matrix during temperature rising could be compensated by the $\gamma'$, because of the high volume fraction of $\gamma'$ in the K445 alloy. Thirdly, the diffusion-controlled process is appreciated at high temperature, so more dislocations and slip system can be activated, which would decrease...
the strain-hardening rate. But if the dislocations and slip system are handicapped, the strain hardening exponent \( n \) will increase and then the strength increases [22]. Therefore, at low temperature (<400 °C), the decrease of \( \sigma_s \) is governed by the disordered or short range ordered solid solution strengthened \( \gamma \)-matrix. Because most dislocations lie in the \( \gamma \)-matrix, and two identical \( 1/2 \langle 011 \rangle \) dislocations form a pair and glide together [19, 23]. Once the dislocations start to move, they will accumulate before the \( \gamma' \) and then harden the alloy further. So it can be seen that the fracture strength of the alloy almost does not drop before 750 °C. The previous research [19] also found that the high density of superlattice-intrinsic stacking faults (S-ISFs) forms in the \( \gamma' \). The density of the S-ISFs decreases with the increase of deformation temperature. When the temperature is higher than 400 °C, no S-ISFs are left. Based on the recent research [24], the formation of S-ISFs is beneficial to the strength but detrimental to the ductility. Then one can find that the ductility drops obviously at 400 °C, but the yield and fracture strength almost have no change. Above 400 °C, the tangled and piled dislocations begin to act as barriers to the glide, so the yield strength increases again from 600 °C. At this stage the dislocations prefer to bypass the \( \gamma' \) precipitates and stop at the grain boundary [25]. The carbides along the grain boundary become the obstacle to suppress the dislocations. With the accumulation of dislocations, the strength increases and the ductility increases as well, because the softening of \( \gamma \) contributes much. When the stress along the grain boundary exceeds its bearability, the grain boundary will migrate [26]. Simultaneously, the dislocations begin to shear the \( \gamma' \), and the stress will decrease gradually with the increase of temperature.

More carbide particles, which are larger than the \( \gamma' \) precipitates, are found in the fractograph, and the microcracks at grain boundary are almost observed at all test temperatures, which indicates the brittleness of the grain boundary. Particularly, the prevalence of dominant intergranular cracks could be attributed to the distributed

**Fig. 5** Fractographs of specimens after tensile testing at different temperatures: (a) Cleaved carbides linked by areas of ductile fracture in 800 °C tested specimen; (b) Large dimples; (c) Cleavage facets among dimples in 850 °C tested specimen; (d)–(f) Morphologies of dimple in specimens tested at 900, 950 and 1000 °C, respectively
carbides along the grain boundaries. If the dislocations are mainly trapped in slip bands within γ' channel, the deformation will be inhomogeneous. From fracture surface observations, it can be seen that the microstructures consist of ductile regions with lots of dimples and cleavage facet with many carbides and γ', and the facet cleavage increases with decreasing the size of γ' particles. The above-mentioned morphology characteristics are consistent with the decrease of elongation, with increasing solution treated temperature. The observation on the cross section finds that initial microvoids form along the interface of coarse precipitate and the matrix, as shown in Fig. 6(a). Further observation reveals that intergranular fracture with crack along the columnar matrix grain boundary, as shown in Fig. 6(b). Therefore, it can be deduced that if the microvoids develop further, the dimples will generate here. Moreover, the fracture microstructure is composed of facet of carbides and γ'. That is because the cracks propagate along the interface of MC carbide and γ' easily. Since the microvoid prefers to nucleate at the γ/γ' interface, the specimen with relative big γ' possesses more dimple features.

According to the former research [14], cleavage prefers to start on the weak {100} planes at room temperature, which leads to the faceted-cleavage fracture commonly observed in fine and duplex microstructure. When the crack meets the γ', it can still propagate along the {100} planes in γ', since these are also the cleavage planes of the precipitates and there is usually similar preferred orientation in both the matrix and precipitate [15]. It can be noted also that the cleavage feature observed at 750 °C is quite different from the faceted {100} cleavage generally found at room temperature, compared Fig. 4(a) with Fig. 3(a). A different cleavage mechanism or set of cleavage planes may be associated with this fracture morphology. This cleavage feature may, however, relate to the ‘localized deformations in single slip bands’ contributing to {111} slip plane decohesion [19]. The brittle {100} faceted fracture is dominant at room temperature and {111} faceted cleavage at 750 °C where lower ductility prevails. The low toughness of the microstructures having fine precipitates might be associated with the easy occurrence of cleavage type fracture on the {100} planes at low temperature and on the {111} slip planes at higher temperatures [14].

4 Conclusions

1) The K445 alloy exhibits anomalous yield strength at medium high temperature, which obtains the maximum fracture strength and almost the secondary high yield strength at 750 °C. With the increase of temperature, the ductility decreases firstly, reaches the minimum value at 400 °C, and then increases gradually, but exhibits a little drop at 900 °C.

2) The fracture surface of K445 alloy below 400 °C exhibits most cleavage characteristic with some dimples. With the increase of temperature, the ductile feature increases, but the cleavage still dominates the fracture till 650 °C.

3) The secondary hardening effect results in the high yield strength and fracture strength with better ductility, which makes it appropriate to be used at 750 °C.

References


