Effect of Long-Term Thermal Exposures on Tensile Behaviors of K416B Nickel-Based Superalloy

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Abstract

The effect of long-term thermal exposure on the tensile behavior of a high W content nickel-based superalloy K416B was investigated. The microstructure and the deformation characteristics were observed by scanning electron microscopy and transmission electron microscopy, and the phase transformation of the alloy during long-term thermal exposure was analyzed by X-ray diffraction patterns and differential thermal analysis. Results showed that after thermal exposure at 1000 °C, the MC carbides in the K416B alloy decomposed into M_6C . During tensile deformation, dislocations slipping in γ matrix crossed over the M_6C by Orowan bowing mechanism. With the increase of thermal exposure time, the secondary M_6C reduced greatly the yield strength of the alloy at room temperature. Meanwhile, the continuous distribution of the secondary M_6C with great brittleness in the grain boundary could become the main source of crack, which might change the fracture characteristic of the alloy from trans-granular to intergranular.

Keywords K416B superalloy · Thermal exposure · Deformation mechanism · Fracture characteristics

1 Introduction

Nickel-based superalloy with high tungsten (W) content has excellent oxidation resistance and mechanical properties [1–5]. Tungsten has prominent solid solution strengthening effect on nickel-based superalloys and the price of tungsten is much lower than other precious metals, such as Re and Ru. Therefore, nickel-based superalloy with high W content has gradually received the attention of material scientist.

Yang et al. [6] studied the precipitation behavior of γ' phase in K465 superalloy during thermal exposure, and reported that the decrease of γ/γ' lattice mismatch induced the degeneration of the cubic degree of γ' phase [7]. In addition, the formation of M_6C carbides in the nickel-based superalloy with high tungsten content might affect the

mechanical properties of the alloy [8, 9]. The research results of Xie et al. [10] and Qin et al. [11] showed that the secondary carbides precipitated with granular shape was caused by the decomposition of MC carbides at the grain boundary and followed the reactions as $MC + \gamma \rightarrow \gamma' + M_6C$ ($M_{23}C_6$), which could hinder the sliding of grain boundary and improved the performance of the alloy at high temperature.

The tensile fracture mechanism of cast nickel-based superalloys included mainly trans-granular, intergranular and microvoid coalescence fractures [12–14]. Aghaie-Khafri et al. [15] studied the relationship between aging time and tensile properties of IN-738LC alloy at 850 °C, and reported that the long-term aging caused rheological stress distribution of the alloy to be more uniform during the tensile tests and the resistance of dislocation movement to be reduced. Du et al. [16] reported that the tensile and yield strength of K417G alloy was basically same between room temperature and 700 °C. However, when the temperature increased to 900 °C, the tensile and yield strength of the alloy decreased rapidly, where the MC carbides and the eutectic structure were thought to be the crack sources and caused the crack to propagate along the grain boundary.

Although the tensile behaviors under different conditions and the microstructure evolution behavior of nickel-based

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superalloys during thermal exposure have been widely studied [17–23], but the relevant research on the superalloys with high tungsten content are still less. On the other hand, the tensile behavior of the high W content superalloy after long-term thermal exposure is still not clear. In this paper, the tensile behavior of K416B alloy which is a typical high W content nickel-based superalloy have been deeply investigated after thermal exposure for different hours. According to the results by phase analysis and microstructure observations, the effect mechanism has been well discussed.

2 Experimental

The K416B nickel-based superalloy in the form of an ingot was remelted by a 10 kg capacity vacuum induction melting furnace ZG-001 and cast into equiaxed crystal test bar. The chemical composition of the alloy is listed in Table 1. The alloys after thermal exposure for 100 h, 500 h, 1000 h and 1500 h at 1000 °C were processed into tensile specimens having dimensions of 35 mm × ϕ 5 mm, and the tensile test was carried out by AG-25KNE electronic tensile testing machine at room temperature, respectively.

To investigate the deformation and the characteristics of the alloy, samples were cut using wire cutting along the longitudinal axis direction of the fractured specimens. The samples were polished and etched in 20 g CuSO₄ + 100 ml HCl + 100 g H₂O, then put into the S-3400 N scanning

electron microscope (SEM) to observe the microstructure of the alloy. To investigate the deformation mechanism of the alloy, transmission electron microscope (TEM) has been used to observe the micro-morphologies of the alloy. The TEM samples were prepared by cutting into ϕ 3 mm and 0.5 mm, and milled into 50 μ m thickness. After electrolytic corroded by the ethanol solution with 10% perchloric acid at −25 °C, the TEM samples were put into the TECNAI 20 TEM to observe the micro-morphologies. To obtain the volume fractions of the MC and M₆C carbides, the regions of these two kinds of carbides were marked by the software of Photoshop and then calculated by Image-Pro Plus 6.0. To ensure the accuracy of the results, 15 photographs were selected for each condition.

3 Results

3.1 Microstructure of the As-Cast Alloy

The microstructures of the as-cast K416B alloy are shown in Fig. 1. The light contrast area was the dendritic structure while the dark one was the inter-dendritic structure. On the other hand, large number of eutectic γ' structure could be observed in the inter-dendritic region, as shown in Fig. 1a. Large blocky carbides were mainly distributed in the eutectic structure with the size was about 15–20 μ m. Skeletal carbides precipitate discontinuously at grain

Table 1 Chemical composition of K416B alloy (mass fraction, wt%)

C	Cr	Co	Al	W	Ti	Nb	Hf	Ni
0.12	4.85	6.80	5.70	16.0	1.0	2.04	1.0	Bal.

Fig. 1 Microstructures of as-cast K416B alloy: **a** dendritic and inter-dendritic structures, **b** carbide morphologies and EDS analysis, **c** γ/γ' phase in dendrite and inter-dendrite regions

boundary and irregular carbides were observed in the inter-dendritic region, as demonstrated in Fig. 1b. Moreover, it was illustrated that the size of γ' phase had great inhomogeneity between dendrite and inter-dendritic region in the as-cast alloy. The size of the γ' phase in the inter-dendritic region was about 1–2 μm , while that in the dendritic region was about 0.4–0.6 μm , as shown in Fig. 1c.

The structure of the as-cast K416B alloy mainly consisted of MC carbide, M_6C carbide, γ' phase and γ matrix. The XRD pattern of the alloy after thermal exposure for different hours is shown in Fig. 2. According to the composition analysis by energy dispersive spectrometer (EDS), the large blocky carbide was M_6C enriched in W, while the smaller ones precipitated along the grain boundary and the inter-dendritic region were MC enriched in Nb and Ti, as shown in the illustration in Fig. 1b. However, the alloy had obvious intensity peaks on the (400), (511), and (440) planes after long-term thermal exposure, respectively. Meanwhile, the intensity peaks of MC carbides near 60° and 73° of the as-cast alloy disappeared after thermal exposure. It was illustrated that the content of MC carbides decreased and secondary M_6C precipitated in the alloy during long-term aging.

The phase transition temperature of as-cast K416B superalloy was measured by differential thermal analysis, and the cooling curve is shown in Fig. 3. The initial solidification temperature of the alloy was 1366 $^\circ\text{C}$ and the final solidification temperature was 1234 $^\circ\text{C}$. Obviously, the solidification sequence was successively as γ matrix, MC carbide, eutectic and γ' phase.

Fig. 2 XRD patterns of the K416B superalloy with thermal exposure for different hours

Fig. 3 DTA cooling curve of as-cast K416B alloy

3.2 Microstructure of K416B Alloy After Thermal Exposure

The morphologies of γ' phase in the inter-dendritic region after thermal exposure for different hours are shown in Fig. 4. It was indicated that the sizes of irregular γ' phase increased gradually and the volume fractions of γ' phase decreased in the inter-dendritic region with the increase of aging time. As a result, the coarsening extent of γ' phase would become larger with the addition of thermal exposure time.

The morphologies of carbides in K416B alloy after thermal exposure for different hours are shown in Fig. 5. After the alloy was exposed for 500 h, there were blocky M_6C carbides with large size existing in the eutectic region and a small amount of discontinuous secondary M_6C particles in the grain boundary. Although the alloy had been exposed for 500 h, there were still skeletal MC carbides existing in the inter-dendritic region, as shown in Fig. 5a.

On the other hand, the secondary M_6C particles were converted into a strip shape, where the morphology of the MC carbides was not changed significantly. The grain boundary of the alloy had coarsened obviously and the width was about 3–4 μm , as shown in Fig. 5b. Due to the large amounts of the strip and the blocky M_6C carbides precipitated and the size of the MC carbides was reduced obviously which were basically granulated at the grain boundary. The main reaction at the grain boundary during thermal exposure is as follows: $MC + \gamma \rightarrow \gamma' + M_6C$.

After the alloy was exposed for 1500 h, there were only a few MC carbides that could be observed in the inter-dendritic region. As the decomposed reaction proceeded, the MC carbides at the grain boundary had almost decomposed into the M_6C carbides, and the width of grain boundary increased to about 7–8 μm , as shown in Fig. 5c, d.

Fig. 4 SEM images of γ' precipitates in K416B alloy in inter-dendritic region after thermal exposure for different hours at 1000°C: **a** 100 h, **b** 500 h, **c** 1000 h, **d** 1500 h

Fig. 5 SEM images of microstructures in K416B superalloy after thermal exposure for different hours at 1000 °C: **a, b** 500 h, **c, d** 1500 h

Fig. 6 Variation of the area fractions of two kinds of carbides in K416B alloy after thermal exposure for different hours at 1000 °C

Figure 6 shows the variation of the area fractions of *MC* and *M₆C* carbides in K416B alloy after thermal exposure for different hours. When the alloy was exposed for 500 h, the area fraction of *M₆C* carbides was larger than that of *MC* carbides. After thermal exposure for 1000 h, the decomposing rate of the *MC* into *M₆C* became slowly, which was thought to be caused by the almost completely decomposition of *MC* into *M₆C* at the grain boundary. After thermal exposure for 1500 h, the area fraction of *M₆C* carbides was more than 90%, as shown in Fig. 6.

Analysis showed that, with the increase of thermal exposure time, there were still a few *MC* carbides observed in the inter-dendritic region but none at the grain boundary, caused by the free energy of the grain boundary was much higher than that of the inter-dendritic region. On the other hand, as thermal exposure goes on, it might cause the increase of *M₆C* and the γ' phase coarsening in the grain boundary by *MC* carbides kept decomposing into *M₆C* plus γ' phase.

3.3 Tensile Behavior of K416B Alloy at Room Temperature After Thermal Exposure

The relationship between yield strength, tensile strength, elongation and aging time during tensile test at room temperature had been shown in Fig. 7. It was obvious that the yield strength and tensile strength of the K416B alloy decreased after thermal exposure.

The yield strength of the alloys was about 652 MPa, 628 MPa, 623 MPa and 614 MPa after thermal exposure for 100 h to 1500 h, respectively. However, the thermal exposure time almost had no effect on the tensile strength of the alloy, which was about 880 MPa. In addition, the elongation had decreased from 8% to about 5% after the alloy was exposed for longer than 100 h.

Fig. 7 Tensile properties of K416B alloy after thermal exposure for different hours at room temperature ($\sigma_{0.2}$ —yield strength, σ_b —tensile strength, δ —elongation)

After tensile fracture, the dislocation configurations of the alloys with different thermal exposure hours have been shown in Fig. 8. In the as-cast alloy, dislocation tangle at the interface of γ/γ' phase could be observed, the shearing dislocations might be decomposed into the configuration of partial dislocations plus stacking faults with a narrower width [24], as shown in Fig. 8a. On the other hand, some shearing dislocations could be observed in γ' phase. The great lattice mismatch between γ and γ' phase could cause the dislocation piling-up at the interface and hinder the dislocation shearing into the γ' phase [25]. As a result, less amount of the dislocation in the γ' phase could be observed, as shown in Fig. 8b. Dislocations slipping in γ matrix and shearing into γ' phase were thought to be the main deformation mechanism of the alloy during the tensile test.

When the alloy was exposed for 500 h, some fine *M₆C* carbides with the size were about 200–300 nm could be observed in the γ matrix and γ/γ' interface, whose diffraction pattern had been shown in the left bottom of Fig. 8c. Due to the incoherent crystallographic relationship between *M₆C* and γ matrix, the fine *M₆C* carbides at the γ/γ' interface could hinder the dislocation slipping and shearing into γ' phase, and caused great amount of dislocation tangles around the *M₆C*, as shown in the arrow of Fig. 8c. On the other hand, some dislocation loops and $(a/2)\langle 110 \rangle$ partial dislocation plus antiphase boundary (APB) could be observed in γ' phase. It was indicated that the deformation mechanism of the alloy was dislocations slipping in γ matrix and crossing over the fine *M₆C* carbides by Orowan bowing mechanism. However, some dislocations might decompose into $(a/2)\langle 110 \rangle$ partials to shear into γ' phase, as shown in Fig. 8d. The density of the shearing dislocations in the γ' phase was obviously greater than that in Fig. 8b, caused by

Fig. 8 Morphologies of the regions near the fracture after the alloy thermal exposure for different hours at 1000 °C: **a, b** as-cast, **c, d** 500 h, **e, f** 1500 h

the reduction of the lattice misfit of γ/γ' phase after thermal exposure [26].

The morphologies of the alloy at 1500 h exposure time and tensile fracture are shown in Fig. 8e, f. Compared to the alloy which was exposed for 500 h, the width of γ matrix and the amount of the fine M_6C carbides were increased. Furthermore, some dislocation networks existed at the γ/γ' interface. It was meant that the crystallographic relationship between γ/γ' phase had changed from coherent to semi-coherent [4], as shown in Fig. 8e. On the other hand, the great amount of the fine M_6C carbides at the interface of γ/γ' could cause increasing of the amount of the bowing dislocation loops, as shown in Fig. 8f. Analysis showed that dislocations slipping in γ phase might be hindered by the

fine M_6C carbides and the dislocation networks at the γ/γ' interface. The piling dislocations might slip across the M_6C by Orowan bowing mechanism or climb over the γ' phase by the dislocation reactions with the dislocation networks [10].

The morphologies of crack initiation and propagation of the K416B superalloy under different conditions after tensile fracture at room temperature are shown in Fig. 9. The morphologies of crack initiation and propagation in the as-cast alloy are shown in Fig. 9a, b. It could be observed that the fracture mechanism of the as-cast alloy was mainly trans-granular, where the crack initiation mainly occurred in the M_6C carbide in the eutectic and the MC carbides at the grain boundary, and the crack might propagate across the grain. Analysis showed that since the crystallographic

Fig. 9 SEM images of tensile fracture of K416B alloy at room temperature after thermal exposure for different hours at 1000 °C: **a, b** as-cast, **c, d** 500 h, **e, f** 1500 h

relationship between M_6C and γ matrix was incoherent, which might cause the dislocation piling up and the stress concentration at the interface of the brittle M_6C and γ matrix [27]. It was thought to be the main reason that the M_6C carbides were the main crack source of the alloy.

After the alloy was exposed for 500 h, the cracks mainly initiated in the blocky M_6C carbides in the eutectic, as shown in Fig. 9c, d. It was indicated that M_6C carbides were easily to become the crack source of the alloy during room temperature tensile. It was thought to be one of the main reasons that the yield strength of the alloys decreased, caused by the increase of the volume fraction of M_6C carbides in the thermally exposed alloys, as shown in Fig. 7.

When the alloy was exposed for 1500 h, the tensile crack mainly initiated on the M_6C in the eutectic and the grain boundary, due to the fact that there were few MC carbides in the alloy and the crack might mainly propagate along the grain boundary, as shown in Fig. 9e, f. However, it was also observed that the micro-cracks around the M_6C particles existed at the grain boundary, as illustrated in Fig. 9e, f. The morphologies of the crack initiation and propagation in the alloy after thermal exposure for 1000 h were similar as those in Fig. 9e, f, the pictures were omitted.

The morphologies of the tensile fractures of K416B alloy with different thermal exposure hours are shown in Fig. 10. The fracture morphologies of the as-cast alloy are shown in Fig. 10a–c. It could be observed that the fracture was smooth

Fig. 10 SEM images of tensile fracture morphologies of K416B superalloy at room temperature after thermal exposure for different hours at 1000 °C: **a, d, g** the macroscopic morphologies of the as-cast alloy, the alloy was exposed for 500 h and 1000 h, respectively; **b, e, h** high magnified morphologies of different alloys; **c, f, i** back scattering images of **b, e, h**

and there were large numbers of cleavage steps and planes, which conformed with the typical cleavage fracture characteristic, as shown in Fig. 10a, b. The broken MC and M_6C carbides could be observed on edge of the cleavage step, as shown in Fig. 10c. Figure 9 shows that the carbide is the main crack source of the alloy during tensile test.

After the alloy was exposed for 500 h, the fracture morphologies of the alloy as shown in Fig. 10e–g could be observed that the amount of the cleavage planes was less than that in the as-cast alloy. However, because of much MC transformed into M_6C carbides, more broken M_6C carbides could be observed on the edge of the cleavage planes, as shown in Fig. 10g.

Conversely, when the alloy was exposed for 1500 h, there appeared to be few cleavage planes but more crack could be observed on the fracture, as shown in Fig. 10h–j. It was indicated that the fracture mechanism of the alloy had been changed. According to Figs. 5 and 6, the MC precipitated

on the grain boundary had almost completely transformed into M_6C , the fracture characteristic of the alloy was closer to intergranular.

4 Discussion

4.1 Influence of Thermal Exposure on Tensile Yield Strength

It could be seen in Fig. 7 that the yield strength of the alloy obviously decreases after thermal exposure. While as the exposure hours extend, the yield strength of the alloys displays almost the same. It is well known that the primary deformation mechanism of nickel-based superalloy during tensile process is dislocations slipping in the matrix and shearing the γ' phase [28–30]. While in Fig. 9e, due to the precipitation of the fine M_6C carbides in γ matrix and

Fig. 11 Schematic diagram of crack initiation and propagation near grain boundary at room temperature after thermal exposure for different hours at 1000 °C

the formation of dislocation network at the γ/γ' interface when the alloy was exposed for 1500 h, the deformation mechanism of the alloy during tensile process changed into Orowan bowing mechanism and dislocation climbing over γ' phase. On the one hand, according to the theory of dislocation slipping, dislocations slipping in the matrix channel must overcome the Orowan resistance of the local area, the latter could be expressed as follows [31]:

$$\tau_{or} = \sqrt{\frac{2}{3}} \frac{Gb}{h},$$

where τ_{or} is the critical shear stress for dislocation slipping, G is the shear modulus, b is the Burgers vector, and h is the width of γ matrix channel. The critical shear stress of the dislocations decreased when the matrix channel width increased. It could be seen from Figs. 4 and 8 that the width of γ matrix channel increased as the thermal exposure hours extends, which may cause the increase of the slipping dislocation amount in γ matrix channel. It is thought to be one of the main reasons why the yield strength of the alloy decreases after thermal exposure.

On the other hand, the K416B alloy is mainly strengthened by W element. Simultaneously, W atoms can cause significant lattice expansion of the γ matrix in the superalloy and forming a large long-range stress field for increasing yield strength [32]. The precipitation of the M_6C carbides with high W content consumes a large amount of W atoms in the matrix [33]. Therefore, the solid solution strengthening effect of the W element is greatly reduced with the increase of the M_6C carbides in the K416B alloy. It is another reason why the yield strength of the alloy decrease after thermal exposure.

4.2 Tensile Fracture Characteristics Transformation of K416B Alloy

The schematic diagram of crack initiation and propagation in the alloys after thermal exposure for different hours at 1000 °C are shown in Fig. 10, where micro-crack initiation could occur on MC and M_6C carbide, and M_6C is the mainly crack source of the alloy [34]. In the as-cast alloy, the blocky M_6C carbides mainly distribute in the grain, while the distribution of MC is in the grain and grain boundary, as shown in Fig. 11a. As a result, when micro-crack is initiated in the blocky M_6C and MC, the micro-crack in M_6C could be propagated rapidly to crack because of its larger size. During tensile test, the crack may propagate perpendicular to the stress axis, which may result in the smooth fracture and the trans-granular fracture mechanism of the alloy.

When the alloy was exposed for 500 h, some of the MC carbides in the grain boundary transformed into M_6C by $MC + \gamma \rightarrow \gamma' + M_6C$ [2, 35, 36]. During tensile test, the micro-crack on blocky M_6C in the grain and the fine M_6C with great brittleness [8, 37] in the grain boundary could simultaneously propagate to crack and connect, as shown in Fig. 11b.

At 1500 h exposure time, the MC carbides in the grain boundary was observed to almost completely transform into the secondary M_6C and γ' , which may cause the distribution of M_6C in the boundary to be continuous and the width of grain boundary to be increased, as shown in Fig. 11c. During tensile test, due to the brittleness of the secondary precipitated M_6C particles, it was easy to form micro-voids along the grain boundary in the alloy. With the increase of the tension stress, micro-voids could connect to each other

to form the crack along the grain boundary, which is thought to be the main reason that the fracture of the alloy displays an intergranular fracture mechanism, as shown in Fig. 10h–j.

5 Conclusions

- (1) After thermal exposure for 1500 h at 1000 °C, the MC in the grain boundary would transform into fine continuous M_6C , and the width of the grain boundary is much wider than that in the as-cast alloy.
- (2) The deformation mechanism of the as-cast K416B alloy during tensile test at room temperature is dislocations slipping in γ matrix and shearing γ' phase. The secondary M_6C precipitations in the matrix could hinder the dislocations to shear γ' phase, and the slipping dislocations may cross over the secondary M_6C particle by Orowan bowing mechanism and form the dislocation loops in the thermally exposed alloy.
- (3) During tensile test, crack mainly initiates on M_6C and propagates perpendicular to the stress axis direction. With the increase of the thermal exposure hours, the increase of the M_6C carbides in the grain boundary could change the fracture characteristic from transgranular to intergranular. The increase of the M_6C volume fraction and the γ matrix width would reduce the yield strength of the alloy.

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