Deformation and fracture behaviors of K403 Ni-based superalloy at elevated temperatures

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ABSTRACT

The effects of the deformation temperatures (850 °C–1000 °C), the strain rates (0.01 s⁻¹ to 10 s⁻¹) and the stress states on the deformation and fracture behaviors of K403 Ni-based superalloy were investigated by thermal compression and tensile simulating tests using Gleeble-1500 thermo-simulation machine. The microstructure evolution and fracture surface was also investigated using scanning electron microscope and transmission electron microscope, with the aim to elucidate the deformation and fracture mechanism. It was found that increasing the strain rates increases the peak stress and the yield stress, but decreases the elongation and the yield strain. In contrast, increasing the deformation temperatures decreases the peak stress and the yield stress, but increases the elongation and the yield strain. Various dislocation cells were observed after the deformation at 850 °C. Increasing the deformation temperatures decreases the dislocation density. Brittle quasi-cleavage fracture morphology was observed at the temperatures below 900 °C. Increasing the deformation temperatures changes the fracture from quasi-cleavage fracture to inter-granular fracture. The fracture mechanism of K403 alloy at high temperatures was revealed, and the high temperature fracture model of K403 alloy was established.

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1. Introduction

The K403 alloy is a Ni–Cr based cast superalloy: alloying with Co, W, Mo, Al, and Ti [1]. It has an excellent castability and can be cast using different casting technologies, i.e. investment casting under vacuum. Therefore, it has been widely used in various fields, including gas turbine guide vanes with working temperatures below 1000 °C, turbine rotor blades with working temperatures below 900 °C and isothermal forging forming dies to deform the hardly deformable materials such as Ti alloys [1,2]. Under these conditions, the K403 alloy is exposed to the high temperature and subjected to different strain states. A better understanding of the deformation and fracture at elevated temperatures is of great necessity to predict the life time and thereby ensure the safety.

In terms of the thermal exposure, it is generally accepted that there may be significant structural changes, including the aggregation and the growth of strengthening phases (γ′ precipitates) [3–14], precipitation and transformation of carbides [15,16], and precipitation of topologically close-packed (TCP) phases [16,17]. This microstructure evolution will definitely affect the deformation properties. For example, the coarsening of γ′ precipitates has been reported to be responsible for the degradation of the creep properties after thermal exposure in CMSX-10 single crystal Ni-base superalloy [3], the decrease of the hardness and the creep lifetime during long-term thermal exposures in IN738C [4–7], a large reduction in strength and sharp reduction in stress rupture life of GH783 alloy at 650 °C [8], the decrease of yield stress at 900 °C in a directionally solidified Ni-base superalloy DZ951 [9,10]. Furthermore, the precipitation of carbides and TCP phases will definitely deplete these elements within the matrix and thereby reduce their solid strengthening effect. More importantly, the precipitation of carbides and TCP phases is very often related to the formation of voids, which may potentially act as initiation sites for fracture. Therefore, the precipitation of carbides and TCP phases causes the deterioration of mechanical properties and thereby reduce their service life and reliability [16,17]. For example, the intergranular fracture in a Ni–Co–Cr–Si alloy (HAYNES HR 160 alloy) during the thermal exposures or ageing was attributed to the formation of M23C6 carbides and Ni16Ti6Si7-G phase at the grain boundaries [11]. A significant decrease in creep resistance in a nickel base...
superalloy EI698 VD was attributed to the volumetric $\gamma'$ changes and additional precipitation of carbides [13]. However, it should be noted here that the temperature (thermal exposure) is only one of the most important factors affecting the microstructure evolution and thereby the mechanical properties. At least two other factors (strain rate and strain state) should be taken into consideration when discussing the deformation and fracture behavior at elevated temperatures.

It has been reported that the flow stress is sensitive to the forming temperature and strain rate [18–23]. In Ni-base superalloy GTD-111 [18], the temperature dependence of the tensile behavior has been studied by tensile tests in the temperature range of 25–900 °C with a constant strain rate of 10−4 s−1. An abnormal tensile property variation was observed with increasing temperatures. The yield strength decreased slightly with temperatures up to about 650 °C and then increased between 650 °C and 750 °C. Above 750 °C, a rapid decrease in the yield strength was found. The tensile strength showed a similar behavior except for its maximum that occurred at 650 °C. The elongation fluctuated more with temperatures. In a single crystal nickel-base superalloy [19], the effect of various strain rates on the tensile behavior has been studied. The yield strength increased with the increase of strain rate, whereas the configuration of the stress-strain curves was independent of strain rates. In a nickel-based alloy (Inconel 617) [20], tensile tests at 25 °C, 600 °C, 800 °C, and 950 °C were conducted in air at different strain rates. In a commercially Ni-base superalloy (Ni-19Cr-5Nb-3Mo-1Ti-0.6Al-0.03C-0.03Co. wt.%), it was found that the flow stress significantly decreases with increasing forming temperatures or decreasing strain rates. Under relatively low deformation temperatures (920 °C, 950 °C and 980 °C), the stress-strain curves are composed of three distinct stages, including work hardening, steady stress and flow softening stages. However, under relatively high deformation temperatures (1010 °C and 1040 °C), the flow curves show the typical dynamic recrystallization characteristics [22]. The formation of the $\delta$ phase was also found to cause the obvious work hardening at the beginning of hot deformation, and then accelerates the flow softening by promoting the dynamic recrystallization with further straining [23]. The formation of $\delta$ phase (Ni$_3$(Nb) and carbides was believed to be responsible for the formation of microvoids [22].

In terms of the deformation mechanisms, it has been reported that two different deformation mechanisms occur in Ni-based alloy Haynes230 depending on temperatures and strain rates [24]. The saw - tooth type serrations at intermediate temperatures and higher strain rates were associated with dynamic strain aging whereas the oscillations with a sinusoidal shape were associated with dynamic recrystallization at temperatures higher than 800 °C and slower strain rates. In M951 alloy [25] and directionally solidified Ni-base superalloy DZ951 alloy [26], at low temperatures, the dominant deformation mechanism has been reported to be $\gamma'$ shearing by dislocation pair $(a/2 \langle 110 \rangle)$ dislocations) and slip bands. At high temperatures, the deformation has been reported to be dominated by $\gamma'$ by-pass. At intermediate temperatures it showed a transition from $\gamma'$ shearing by-passing. In the conventionally cast (CC) and the directionally solidified (DS) superalloy CM247LC [27], the tensile strength of the CC and the DS alloys is dependent upon ageing and solution treatment condition. Shearing of $\gamma'$ by coupled dislocations is a principal deformation process at low temperatures, and cutting of fine secondary $\gamma'$ plays an important role at an early stage of deformation in the CC specimen. However, the investigation on the deformation and fracture of K403 alloy is still very limited [12,22].

The present investigation is aimed to elucidate the effects of the deformation temperatures, the strain rates and the stress states on the deformation and fracture behaviors of K403 Ni-based superalloy. Thermal compression and tensile simulating tests using Gleeble-1500 thermo-simulation machine were used to measure the compression and tensile properties at the temperature ranges from 850 °C to 1000 °C and strain rates ranges from 0.01 s−1–10 s−1. The microstructure evolution and fracture surface was also investigated using scanning electron microscope and transmission electron microscope, with the aim to elucidate the deformation and fracture mechanism. Furthermore, the fracture mechanism and the high temperature fracture model of K403 alloy was established in terms of strain and stress, respectively.

2. Experimental method

K403 Ni-based superalloy was melted in a vacuum induction furnace. The alloy melting was poured into test bars in vacuum. The chemical composition is listed in Table 1. Thermal compression and tensile simulating tests were performed using Gleeble-1500 thermo-simulation machine. In the case of thermal tensile simulating tests, two different types of testing samples were used. One type is a smooth tensile sample with 6 mm in diameter and 34 mm in length. The other type is a notch tensile sample with 4 mm in diameter and 34 mm in length. The notch size and the max stress triaxiality are listed in Table 2. It should be noted here that the max stress triaxiality ($\eta_{\text{max}}$) is calculated using Equation (1) [28]:

$$\eta_{\text{max}} = \frac{\sigma_{m}/\sigma_{eq}}{\max} = 1/3 + \ln \left(1 + R_{f}/2R\right)$$

Table 1

<table>
<thead>
<tr>
<th>C</th>
<th>Cr</th>
<th>Co</th>
<th>W</th>
<th>Mo</th>
<th>Al</th>
<th>Ti</th>
<th>Fe</th>
<th>B</th>
<th>Zr</th>
<th>Ni</th>
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<td>0.14</td>
<td>11.32</td>
<td>5.41</td>
<td>5.17</td>
<td>4.24</td>
<td>5.32</td>
<td>2.61</td>
<td>≤1.0</td>
<td>0.017</td>
<td>0.06</td>
<td>Bal</td>
</tr>
</tbody>
</table>

Fig. 1 shows true stress-strain curves of K403 alloy as a function of strain rates (0.01 s−1, 0.1 s−1, 1 s−1, 10 s−1) at different temperatures of 850 °C (Fig. 1a), 900 °C (Fig. 1b), 950 °C (Fig. 1c) and 1000 °C (Fig. 1d), respectively. Clearly, the true stress increases significantly with increasing the true strain during the early stage of deformation. Then, the true stress becomes stable with further increasing the true strain, which is similar to the dynamic recovery curve [22].
It should be noted here that, at a low deformation temperature (i.e. 850 °C, 900 °C), the true stress still increases slightly with increasing true strain, as shown in Fig. 1a and b. However, at a high deformation temperature (i.e. 950 °C, 1000 °C), no significant increase was observed when the true strain is higher than 0.1 s⁻¹. Instead, the true stress decreases when the true strain is 0.01 s⁻¹, as shown in Fig. 1c and d. This observation can be interpreted by the fact that the deformation at elevated temperatures is dependent on the two aspects, including working hardening and softening effect of dynamic recovery recrystallization [22,23,29]. During the early stage of deformation, working hardening effect is dominated, and then dynamical recrystallization softening effect is enhanced with the deformation process. Finally, a dynamic balance is obtained between working hardening and dynamic recovery recrystallization softening [29,30]. Furthermore, at a defined deformation temperature, increasing strain rates increases the stress, which can be attributed to the increase of the flow stress, the shear stress and softening effect of dynamic recovery recrystallization [30].

Table 2

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Sample A</th>
<th>Sample B</th>
<th>Sample C</th>
<th>Sample D</th>
</tr>
</thead>
<tbody>
<tr>
<td>R/mm</td>
<td>0.5</td>
<td>1</td>
<td>2</td>
<td>4</td>
</tr>
<tr>
<td>2R/mm</td>
<td>3.94</td>
<td>4.06</td>
<td>3.94</td>
<td>3.95</td>
</tr>
<tr>
<td>ηmax</td>
<td>1.4279</td>
<td>1.03395</td>
<td>0.73379</td>
<td>0.55397</td>
</tr>
</tbody>
</table>

3.2. Thermal tensile simulating tests

Fig. 3 shows tensile properties of smooth tensile samples in K403 alloy as a function of strain rates (0.01 s⁻¹, 0.1 s⁻¹, 1 s⁻¹, 10 s⁻¹) and temperatures (850 °C, 900 °C, 950 °C, 1000 °C), respectively. The breaking strain is calculated using Equation (2):

\[
e_{\text{f}} = \ln(1/Z) = 2 \ln\left(\frac{d_0}{d_f}\right)
\]

where, Z is the reduction of area, \(d_0\) is the diameter of smooth tensile sample prior to testing, \(d_f\) is the diameter of smooth tensile sample after fracture.

As shown in Fig. 3a, at a defined strain, increasing the deformation temperature decreases the peak stress (\(\sigma_p\)), but increases
the reduction of area (Z). For example, at the strain of 0.01 s⁻¹, increasing the deformation temperature from 850 °C to 1000 °C decreases the peak stress (σₚ) from 1151.71 MPa to 677.06 MPa, but increases the reduction of area from 3.95% to 12.31%. At the strain of 10 s⁻¹, increasing the deformation temperature from 850 °C to 1000 °C decreases the peak stress (σₚ) from 1392.34 MPa to 111.72 MPa, but increases the reduction of area from 3.04% to 4.59%. It should be noted here that, at a lower strain (i.e. 0.01 s⁻¹), the deformation temperature has a more significant effect on the peak stress and the reduction of area. On the other hand, at a defined temperature, increasing strain increases the peak stress, but decreases the reduction of area. The same is also true with the breaking stress, as shown in Fig. 3b. Fig. 4 shows the relation curves of σₚ-ln (Fig. 4a) and σₚ-ln (Fig. 4b) at different temperatures (850 °C, 900 °C, 950 °C, 1000 °C).

Fig. 5 shows effects of different stress states on the tensile mechanical properties of K403 alloy. Increasing the max stress triaxiality (hₘₐₓ) increases the peak stress (σₚ) and the breaking stress (σᵤ), as shown in Fig. 5a, but decreases the reduction of area (Z) and the breaking strain (εᵤ), as shown in Fig. 5b.

Fig. 2. True stress-strain curves of K403 alloy as a function of temperatures (850 °C, 900 °C, 950 °C, 1000 °C) at different strain rates of 0.01 s⁻¹ (a), 0.1 s⁻¹ (b), 1 s⁻¹ (c) and 10 s⁻¹ (d), respectively.

Fig. 3. Tensile properties of smooth samples in K403 alloy as a function of strain rates (0.01 s⁻¹, 0.1 s⁻¹, 1 s⁻¹, 10 s⁻¹) and temperatures (850 °C, 900 °C, 950 °C, 1000 °C). (a) peak stress and reduction of area, (a) breaking stress and breaking strain.
3.3. Microstructure after thermal tensile simulating tests

Fig. 6 shows optical microscopy images (Fig. 6a, c, e, g) and TEM images (Fig. 6b, d, f, h) of the microstructure taken from the transverse-section (perpendicular to the tensile direction) in K403 alloy after thermal tensile simulating tests with a strain of 0.01 s\(^{-1}\) at different temperatures of 850°C, 900°C, 950°C, and 1000°C, respectively. At a low deformation temperature (i.e. 850°C), the dendrite structure was observed, as shown in Fig. 6a. With increasing the deformation temperature (i.e. 900°C, 950°C, 1000°C), such type of dendrite structure became less visible, as shown in Fig. 6c, e, g. Furthermore, at a low deformation temperature (i.e. 850°C, 900°C), the dislocation structure with a high number density was observed, as shown in Fig. 6b, d. With increasing the deformation temperature (i.e. 950°C, 1000°C), the number density of such type of dislocation structure appears to decrease, as shown in Fig. 6f, h.

Fig. 7 shows optical microscopy images (Fig. 7a, c, e, g) and TEM images (Fig. 7b, d, f, h) of the microstructure taken from the transverse-section (perpendicular to the tensile direction) in K403 alloy at 950°C at different strain rates of 0.01 s\(^{-1}\), 0.1 s\(^{-1}\), 1 s\(^{-1}\), and 10 s\(^{-1}\), respectively. At a low strain (i.e. 0.01 s\(^{-1}\), 0.1 s\(^{-1}\)), no significant dislocation structure was observed, as shown in Fig. 7b. With increasing the strain (i.e., 1 s\(^{-1}\), 10 s\(^{-1}\)), the dislocation structure with a high number density was observed, as shown in Fig. 7d, f, h.

3.4. Fracture after thermal tensile simulating tests

Fig. 8 shows SEM images of the fracture surface in K403 alloy at a strain rate of 0.01 s\(^{-1}\) as a function of temperatures of 850°C, 900°C, 950°C, and 1000°C, respectively. Fig. 8b, e, h, k are taken from the transverse-section (perpendicular to the tensile direction). At a low deformation temperature (i.e. 850°C, 900°C), brittle quasi-cleavage fracture morphology was observed, as shown in Fig. 8a, b, d, e. However, increasing the deformation temperatures changes the fracture from quasi-cleavage fracture to inter-granular fracture, as shown in Fig. 8g, h, j, k. Viewed from the vertical-section (parallel to the tensile direction) (Fig. 8c, f, i, l), at a low deformation temperature (i.e. 850°C, 900°C), the fracture within the dendrite was observed, as shown in Fig. 8c, f. However, with increasing the deformation temperatures (i.e. 950°C, 1000°C), the fracture along the boundaries of the dendrite was observed, as shown in Fig. 8i, l.
Fig. 6. Optical microscopy images (a, c, e, g) and TEM images (b, d, f, h) of the microstructure taken from the transverse-section (perpendicular to the tensile direction) in K403 alloy with a strain of 0.01 s⁻¹ at different temperatures of 850 °C (a, b), 900 °C (c, d), 950 °C (e, f) and 1000 °C (g, h), respectively.
Fig. 7. Optical microscopy images (a, c, e, g) and TEM images (b, d, f, h) of the microstructure taken from the transverse-section (perpendicular to the tensile direction) in K403 alloy at 950 °C at different strain rates of 0.01 s⁻¹ (a, b), 0.1 s⁻¹ (c, d), 1 s⁻¹ (e, f) and 10 s⁻¹ (g, h), respectively.
4. Discussions

The deformation temperature (850 °C–1000 °C), the strain rate (0.01–10 s⁻¹) and the stress states ($\eta_{\text{max}}$) have an important effect on the deformation behaviors of K403 alloy. As shown in Figs. 1–5, increasing the strain rates increases the peak stress and the yield stress, but decreases the elongation and the yield strain. In contrast, increasing the deformation temperatures decreases the peak stress and the yield stress, but increases the elongation and the yield strain. The deformation temperature (850 °C–1000 °C) also has an important effect on the fracture behaviors of K403 alloy. Brittle quasi-cleavage fracture morphology was observed at the temperatures below 900 °C. However, increasing the deformation temperatures changes the fracture from quasi-cleavage fracture to inter-granular fracture. It should be noted here that the strain rate (0.01–10 s⁻¹) appears to have no significant effect on the fracture behaviors of K403 alloy.

The fracture mechanism and fracture model of K403 alloy can be discussed in terms of the stress and the strain, respectively. In the term of the strain, Johnson-Cook fracture mode was developed in 1983 [31,32] and described in Equation (3):

$$\varepsilon_f = \left( D_1 + D_2 \exp \left( D_3 \sigma^* \right) \right) \left( 1 + D_4 \ln \varepsilon^* \right) \left( 1 + D_5 T^* \right)$$  (3)

where, $D_1$ and $D_2$ is a constant related to materials, $D_3$ is a factor related to stresses, $D_4$ is a factor related to strains, $D_5$ is a factor related to temperatures, $\sigma^*$ is equal to the stress triaxiality ($\eta$), $\varepsilon^*$ is related to strain rates with Equation (4):

$$\varepsilon^* = \frac{\varepsilon}{\varepsilon_0}$$  (4)

where, $\varepsilon_0$ is the reference strain rate.

$T^*$ is related to temperatures with Equation (5):

$$T^* = \frac{(T - T_r)}{(T_m - T_r)}$$  (5)

where, $T_m$ is the melting temperature of materials, $T_r$ is the reference temperature.

It should be noted here that three parts in Equation (3) are independent and can be related to the stress triaxiality ($\eta$), the strain and the temperature, respectively. The $D_1$, $D_2$, $D_3$, $D_4$ and $D_5$ can be therefore determined by considering the single factor alone. For example, when $\varepsilon$ is equal to 0.01 s⁻¹, $T$ is equal to 950 °C, Equation (3) can be rewritten as Equation (6):

$$\varepsilon_f = (D_1 + D_2 \exp (D_3 \sigma^*))$$  (6)

By least square method nonlinear fitting of the tensile strain data of the smooth samples, as shown in Fig. 9a, $D_1$, $D_2$, $D_3$ can be determined to be 0.025, 0.14, -2.051, respectively. Similarly, when $T^*$ is equal to 0, Equation (3) can be rewritten as Equation (7):
\[ \varepsilon_f = A(1 + D_4 \ln \varepsilon^*) \]  

where, \( A \) is a constant and is equal to \( (D_1 + D_2 \exp (D_3 \sigma^*)) \).

By linear fitting of the tensile strain data of the smooth samples, as shown in Fig. 9b, \( D_4 \) can be determined to be \(-0.095\).

When the strain rate is defined, \( \ln \varepsilon^* \) is equal to 0, Equation (3) can be rewritten as Equation (8):

\[ \varepsilon_f = A(1 + D_5 T^*) \]  

where, \( A \) is a constant and equal to \( (D_1 + D_2 \exp (D_3 \sigma^*)) \).

\( T^* \) can be calculated by Equation (5), where \( T_m \) is 1299 °C (the average value of the melting range of K403 alloy). By linear fitting of the tensile strain data of the smooth samples, as shown in Fig. 9c, \( D_5 \) can be determined to be 2.043.

In total, the fracture mechanism and fracture model of K403 alloy on the basis of Johnson-Cook fracture mode can be described in Equation (9):

\[ \varepsilon_f = \left( 0.25 + 0.14 \exp(-2.05 \sigma^*) \right) \left( 1 - 0.095 \ln \varepsilon^* \right) \left( 1 + 2.043 T^* \right) \]  

On the other hand, in the term of the stress, the breaking stress can be described in Equation (10):

\[ \sigma_f = a + bT \]  

where, \( a \) and \( b \) is a constant related to materials, \( T \) is the deformation temperature. By linear fitting of the tensile strain data of the smooth samples, as shown in Fig. 9d, \( a \) and \( b \) can be determined to be 3770.44, \(-3.102\), respectively.

5. Conclusions

The effects of the deformation temperature (850 °C–1000 °C), the strain rate (0.01 s\(^{-1}\) to 10 s\(^{-1}\)) and the stress states (\( \eta \)) on the deformation and fracture behaviors of K403 Ni-based superalloy were investigated by thermal compression and tensile simulating tests using Gleeble-1500 thermo-simulation machine. The main conclusion can be drawn:

1. With increasing the strain rates, the peak stress and the yield stress increase, but the elongation and the yield strain decrease. In contrast, with increasing the deformation temperatures, the peak stress and the yield stress decrease, but the elongation and the yield strain increase.

2. Various dislocation cells were observed after the deformation at 850 °C. Increasing the deformation temperatures decreases the dislocation density.

3. Brittle quasi-cleavage fracture morphology was observed at the temperatures below 900 °C. However, increasing the deformation temperatures changes the fracture from quasi-cleavage fracture to intergranular fracture.

4. The fracture mechanism of K403 alloy at high temperatures was revealed, and the high temperature fracture model of K403 alloy was established.

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