Nanoindentation study and mechanical property analysis of nickel-based single crystal superalloys

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Keywords: nanoindentation, TCP phase, young’s modulus, hardness, finite element analysis

Abstract

Based on uniaxial tensile and compressive creeps, the mechanical properties of the third generation nickel-based single crystal superalloy were studied via the combination of nanoindentation, crystal plasticity theory, and finite element analysis. The Young’s moduli and hardness of γ, γ’ and the topologically close-packed (TCP) phases are related to chemical composition and applied stress. The maximum values of the Von Mises equivalent stress and damage are at the tip of the TCP phase. These values are inversely proportional to the distance to the TCP phase. Furthermore, the rafting of the γ’ phase can be predicted according to the distribution of the TCP phase and stress.

1. Introduction

Nickel-based single crystal superalloys have been widely used in the hot parts of aero engines and gas turbines because of their excellent mechanical properties. These properties are related to their two-phase microstructure with the long-range ordered L12 γ phase (close to 70%) that occurs in the form of cubes coherently embedded in a face-centred cubic solid solution γ’ matrix phase [1, 2]. The creep response of the superalloy largely determines its service life, and the addition of some insoluble elements, such as Re, Ru, Mo, W, enhances its mechanical properties [3]. Furthermore, Al, Ti and Ta achieve precipitation strengthening by forming Ni3 (Al, Ti, Ta). The addition of Cr and Co can improve the performance of superalloys to cope with environmental deterioration [4].

During the development of the γ phases, the solute atom (Re) accumulates in the γ phase and the γ/γ’ phase interface. The accumulation of Re hinders the movement of dislocations [5, 6]. Moreover, Re strengthens the creep properties of nickel-based single crystal superalloy by impacting the lattice misfit and decreasing the rafting rate of the γ’ phases [7, 8]. However, as more Re is added, segregation becomes increasingly severe. Nevertheless, Re is one of the slowest diffusion elements, which can lead to the precipitation of the TCP phase during high-temperature long term aging or endurance tests. The TCP phase has a detrimental effect on mechanical properties such as tensile plasticity, impact toughness, corrosion resistance, and creep strength of single crystal superalloys [9, 10]. The damage mechanism is mainly reflected as follows [11–13]. (1) The TCP phase is a brittle inclusion that decreases plasticity, while facilitating the initiation and propagation of cracks and finally causing failure. (2) The content of insoluble elements such as Re in the γ phase located near the TCP phase is lowered, that is the γ-matrix-softening damage mechanism. (3) The TCP phase causes the local distortion of γ’ phases during the high temperature creep.

The nanoindentation test has shown unique superiority in characterizing the single-phase mechanical properties of the nickel-based single crystal superalloys. Göken et al. [14] who measured the mechanical properties of the γ and γ’ phases of nickel-based superalloys using a nanoindenter with atomic force microscopy believed that the Young’s modulus and hardness of the γ phase are smaller than that of the γ’ phase. The nanoindentation test was subsequently adopted by Durst et al. [6, 15] and Neumeier et al. [16] in their study of the solution strengthening effect of insoluble elements on the γ phase. Durst et al. [6, 15] studied the mechanical properties of a single phase of nickel-based single crystal superalloys with different Re and Tu contents. The
results showed that the hardness of the $\gamma$ phase augmented gradually with the increase in the content of insoluble elements, while that of the $\gamma'$ phase basically remained unchanged, and the Young's modulus of the $\gamma$ and $\gamma'$ phases both augmented gradually. Neumeier et al. [16] researched the effect of Re and Ru on the hardness of the $\gamma$ and $\gamma'$ phases; they believed that Re had a strong solution strengthening effect on the $\gamma$ phase, while Ru strengthened both the $\gamma$ and $\gamma'$ phases. Rehman et al. [17] studied the mechanical properties of the $\mu$ phase in the creep of Re containing nickel-based superalloys; they indicated that the hardness of the $\mu$ phase was greater than those of the $\gamma$ and $\gamma'$ phases, and that the hardness of the dendritic region in which Re aggregated was basically the same as that of the interdendritic region, indicating that Re was consumed by the TCP phase in the dendritic region.

The finite element analysis is an effective way of evaluating material properties and the indentation behaviour of elastoplastic materials [18]. Nanoindentation and finite element analysis were combined to study the mechanical properties of Inconel 617 surface oxide by Salari et al. [19]. Durst et al. [20] studied the reliability of the material mechanical properties obtained from the load-displacement curve through finite element analysis, and verified the experimental results by deriving the load limit of the two-phase indentation test. The finite element analysis is also used to study the effect of the TCP phase on the microstructural evolution of the $\gamma$/$\gamma'$ phases of nickel-based single crystal superalloys. Based on the viscoplastic theory, Graverend et al. [13] studied the local stress-strain field near the TCP phase, and especially stress concentration via the finite element analysis to illustrate the microstructural evolution by observation-based testing.

In this paper, a nanoindenter with scanning electron microscopy (SEM) was used to measure the mechanical properties of the $\gamma$, $\gamma'$ and TCP phases under different test conditions. The effect of the TCP phase on the mechanical properties of nickel-based single crystal superalloys was studied through the combination of crystal plasticity theory and finite element analysis.

2. Experiments

The nickel-based single crystal superalloys, which are classified as a third-generation nickel-based superalloy, have been used as the starting materials. The chemical compositions are listed in Table 1. The heat treatment regime of the alloys was as follows: 1340 °C, 6 h, air cooling (A.C.), +1120 °C, 4 h, A.C. +870 °C, 32 h, A.C. All samples were prepared from [001] oriented along the casting direction, which was detected using the X-ray Lauer diffraction method. The geometrical details and image of the tensile creep specimen are shown in Figure 1 along with the major dimension. The compressive test specimen is a cube measuring 2 mm × 2.5 mm × 3 mm, as shown in Figure 2(a). The fixture was designed and processed for the compressive test, as shown in Figure 2(b).

The creep tests were performed on a CSS-RDL 100 creep testing machine. The loading rate was 1 kN min$^{-1}$, and the loading direction was in line with the [001] axis. The uniaxial constant load creep tests along the [001] direction were conducted in air at 1100 °C and 140 MPa. The temperature was monitored using a thermocouple with an error of ±3 °C. The temperature approached the target value and was maintained for 0.5 h before the...
experiment. There were at least three valid experimental data for tensile and compressive creeps. All tests were performed on the same creep testing machine to ensure consistency in the results. After the creep test, the sample was cut, mechanically ground, polished and chemically corroded using 5 ml nitric acid, 10 ml hydrofluoric acid and 15 ml hydrogen peroxide for SEM analysis. The SEM investigation was performed using the JSM-6390 working at 30 kV. The spot size was 40 μm. The size of the γ′ phase was measured using Image J after binary processing.

The nanoindentation test was performed on a nanoindenter PI 87 equipped with SEM and a triangular pyramid Berkovich diamond indenter. The indentation load which was related to displacement was measured and controlled by a strain gauge. The maximum displacement was 300 nm. The indentation process was automatically controlled using a computer. To eliminate the effect of loading and unloading rates, the loading and unloading time was 5 s respectively, and the holding time was 2 s. According to the Oliver-Pharr method [21], the hardness $H$ and reduced elastic modulus $E_r$ are calculated by

$$H = \frac{P_{max}}{A_c}$$

$$E_r = \frac{S \sqrt{\pi}}{2 \sqrt{A_c}}$$

where $A_c$ is the projected area of contact surface under the load $P_{max}$, and $S$ is the contact toughness.

The Young’s modulus value can be derived from the reduced modulus using the following relation,

$$E_r^{-1} = \frac{1 - v_i^2}{E_i} + \frac{1 - v_s^2}{E_s}$$

where $E_s$ and $v_s$ are Young’s modulus and Poisson’s ratio, respectively; $E_i$ and $v_i$ are Young’s modulus and Poisson’s ratio of indenter, respectively; and $E_i = 1141$ GPa and $v_i = 0.07$ are the values of the diamond indenter [22].

### 3. Finite element analysis

#### 3.1. Crystal plastic creep constitutive model

The creep shear strain rate $\dot{\gamma}^{(\alpha)}$ of slip system $\alpha$ can be expressed as [23]

$$\dot{\gamma}^{(\alpha)} = \dot{\gamma}_0^{(\alpha)} \left( \frac{1}{1 - \omega^{(\alpha)}} \right)^n S^{(\alpha)}$$

where $n$ is the parameter related to the temperature, $\dot{\gamma}_0^{(\alpha)}$ is the initial creep rate, $S^{(\alpha)}$ denotes the material degradation. $\omega^{(\alpha)}$ which is the cavity damage in the creep damage mechanism [24] is used to reflect the degree of material deterioration. The evolution equation of $\dot{\gamma}_0^{(\alpha)}$, $\omega^{(\alpha)}$ and $S^{(\alpha)}$ can be expressed as

$$\dot{\gamma}_0^{(\alpha)} = A \cdot \exp \left( -\frac{Q}{RT} \right) \cdot \left( \tau^{(\alpha)} \right)^m$$

$$\dot{\omega}^{(\alpha)} = \dot{\omega}_0 \left| \dot{\gamma}^{(\alpha)} \right|^m$$

$$S^{(\alpha)} = C \left| \dot{\gamma}^{(\alpha)} \right|^p$$

where $\tau^{(\alpha)}$ is the resolved shear stress, $\dot{\omega}_0$ is the initial damage rate, $A$, $n$, $m$, $C$ and $P$ are the material parameters that are dependent on temperature. $R$ is the gas constant, $T$ is the absolute temperature, $Q$ is the activation energy, and the octahedral slip system activation energy is $Q_{oct} = 6.97E - 19$ J atom$^{-1}$ [25].
According to Schmid’s law, the formula between the macroscopic stress and the resolved shear stress is given as

$$\tau^{(\alpha)} = S^{(\alpha)}_f \sigma$$

(8)

where $S^{(\alpha)}_f$ is the Schmidt factor, the specific values are listed in table 2.

The transformational relationship between the shear strain and the axial strain of the slip system is given as

$$\varepsilon^{(\alpha)} = S^{(\alpha)}_f \gamma^{(\alpha)}$$

(9)

where $S^{(\alpha)}_f$ is the transformational coefficient between the shear strain and the axial strain, as shown in table 3.

The nickel-based superalloy has three slip family groups that may be activated, namely, the octahedral slip family with 12 slip systems, the dodecahedral slip family with 12 slip systems, and the hexahedral slip family with 6 slip systems [26]. Based on the fracture morphology observation after the experiment [27], only the octahedral slip system $\{111\} \langle 110\rangle$ is assumed to be activated in this study. The model parameters are calibrated by the creep. The simulation parameters are fitted as shown in table 4, and the fitting curve is consistent with the trend of the one in the test, as shown in figure 3. The creep constitutive model and parameters can evidently describe the creep behaviour of single crystal superalloys at 1100 °C and 140 MPa accurately.

### 3.2. Finite element model

The ABAQUS software is used to study the effect of the TCP phase on the microstructural evolution of nickel-based single crystal superalloys. The finite element model is consistent with the creep microstructure, as shown in figure 4. The axial symmetry boundary condition is applied to the model. A displacement constraint along the load direction is applied on the left side of the model, that is, $U_1 = UR_2 = UR_3 = 0$. The central point of the left side is completely fixed, that is $U_1 = U_2 = U_3 = UR_1 = UR_2 = UR_3 = 0$. The top, bottom and left side of the model are defined as displacement compatibility. The stress on the right side is 140 MPa. The model is meshed into the C3D4 cell with a total of 244 483. The crystal plasticity constitutive model is imported to ABAQUS via the user subroutine (UMAT). The Young’s modulus and hardness of the TCP phase are measured by nanoindentation, and the other parameters are calibrated in table 4.

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**Table 2. The Schmidt factors of three different orientations.**

| Orientation | Number | $S_f$  |
|-------------|--------|--------|
| [001]       | 8      | 0.4082 |
|             | 4      | 0.0000 |
| [011]       | 4      | 0.4082 |
|             | 8      | 0.0000 |
| [111]       | 6      | 0.2722 |
|             | 6      | 0.0000 |

**Table 3. The $S_f^{(\alpha)}$ of three different orientations.**

| Orientation | $\{111\} \langle 110\rangle$ | $\{111\} \langle 112\rangle$ | $\{100\} \langle 110\rangle$ |
|-------------|-----------------------------|-----------------------------|-----------------------------|
| [001]       | $8/\sqrt{6}$                | $8/3\sqrt{2}$              | 0                           |
| [011]       | $4/\sqrt{6}$                | $4/3\sqrt{2}$              | $\sqrt{2}$                 |
| [111]       | $2/\sqrt{6}$                | $4/3\sqrt{2}$              | $\sqrt{2}$                 |

**Table 4. Creep parameters of [001] orientation at 1100 °C.**

| Temperature/°C | $E$/GPa | $G$/GPa | $\mu$ | $A$  | $n$  | $\omega_0$ | $m$  | $C$  | $P$  |
|----------------|---------|---------|-------|------|------|-------------|------|------|------|
| 1100           | 69.8    | 80.0    | 0.42  | 1e-15| 6.08 | 0.5187      | 0.5609| 0.000| 0.001|
4. Results and discussion

4.1. Nanoindentation measurements

4.1.1. Young’s modulus measurements

The Young’s modulus measurements were performed on the $\gamma$, $\gamma'$, and TCP phases after tensile and compressive creeps. The Young’s moduli and load-displacement curves of the $\gamma$, $\gamma'$, and TCP phases are shown in figure 5. In order to decrease the measurement deviation, three sets of data with relatively little dispersion were selected and their average was obtained. Figure 5(a) shows that the Young’s modulus of the $\gamma'$ phase is higher than that of the $\gamma$ phase, and the Young’s modulus of the TCP phase is higher than that of the $\gamma$ phase and lower than that of the $\gamma'$ phase. The Young’s modulus after the tensile creep was higher than that of the compressive creep. At the same indentation displacement, the required indentation load was $P_{TCP} > P_{\gamma} > P_{\gamma'}$, as shown in figure 5(b).

Young’s modulus is the ability of a material to resist elastic deformation, that is, the ability of an applied load to overcome the force between atoms and change the atomic spacing. To understand the difference in the Young’s modulus in figure 5(a), the chemical composition and applied stress have to be discussed. Furthermore, Co, Cr, Re, and Mo segregate to the $\gamma$ phase; Ni, Al, W, Ta, and Ti segregate to $\gamma'$ phase; and W, Re, Mo, and Cr segregate to the TCP phase [28, 29]. The different phase elements cause different atom bonding forces, which influence the change in atomic spacing. After the creep test, a large amount of dislocation networks aggregate in the $\gamma$ phase and $\gamma'/\gamma'$ interface [30], with the Re atoms hindering the motion of dislocations. The large amount of dislocations in the $\gamma$ phase changes the interaction of atoms. The diffusion activation energy of the atom along
the dislocation line is approximately 1/2 of the bulk diffusion [31, 32]. The differences in chemical composition and dislocations are associated with the Young’s modulus of the γ and γ’ phases. The difference between the atomic configuration of the TCP phase and single crystal superalloys leads to the difference in atomic interaction. Instead of the γ and γ’ phases, the topologically close-packed structure should be associated to the difference in Young’s modulus.

As seen in figure 5(a), the Young’s modulus depicts ‘tension-compression asymmetry’. The Young’s modulus after the tensile creep is higher than that observed after the compressive creep. The applied tensile and compressive stresses along the [001] direction increase and decrease the resistance to the motion of dislocations, respectively [26]. Atoms, such as Re, diffuse along the dislocation line [29] and cause changes in chemical composition and atomic interaction, which is related to the difference in the Young’s modulus after the tensile and compressive creeps.

4.1.2. Hardness measurements
Hardness measurements were performed on the γ, γ’, and TCP phases after tensile and compressive creeps, as shown in figure 6. Three sets of data with relatively little dispersion were selected in order to decrease the deviation in measurements. As seen in figure 6, the hardness of the γ’ phase is higher than that of the γ phase; further, the hardness of the TCP phase is higher than those of the γ and γ’ phases and the hardness after the compressive creep is higher than that after the tensile creep. These hardness measurements are consistent with the findings of the research by Rehman et al [17].

![Figure 5](https://example.com/figure5.png)
**Figure 5.** Young’s moduli and load-displacement curves: (a) Young’s moduli of the γ, γ’, and TCP phases at different test conditions, (b) load-displacement curves at different test conditions.

![Figure 6](https://example.com/figure6.png)
**Figure 6.** Hardness of γ/γ’/TCP phases at different test conditions.
The TCP phase is a brittle inclusion with a close packed crystal structure. The close packed crystal structure and the presence of refractory elements result in high hardness of the TCP phase. A comparison of the $\gamma$, $\gamma'$ and TCP phases shows that the content of refractory elements is proportional to the hardness. It can be inferred that the refractory elements can increase hardness. The comparison between figures 5 and 6 shows that the 'tension-compression asymmetry' of hardness is antonymous to that of the Young's modulus. The hindrance of applied compressive stress on the motion of dislocations hinders the diffusion of refractory elements, thus increasing hardness after the compressive creep. The greater hardness of the TCP phase results in a reduction in creep life and plasticity [33, 34], and easily leads to material failure. The crack is usually initiated and propagated along the $\gamma$/$\gamma'$ interface and TCP/$\gamma'$ interface due to the major difference between Young's modulus and hardness, which is consistent with the findings from the research conducted by Zhang et al [30]. In the design of single crystal alloys, the advantage and disadvantage of the insoluble element should be taken into consideration, and the substituted element for the insoluble element may be useful for elimination of the disadvantage.

4.2. Finite element analysis

The three-phase finite element model is modelled for numerical simulation to study the stress and damage distribution caused by the TCP phase. The TCP phase is regarded as viscoplasticity in numerical simulation analysis [13]. Based on the mechanical properties by nanoindentation in sections 4.1 and 4.2, the TCP phase is considered to be brittleness rather than viscoplasticity in this study. Therefore, the TCP phase is endowed with elastic constants but the $\gamma$ and $\gamma'$ phases are assigned viscoplasticity for finite element analysis.

4.2.1. Mises stress analysis

The Von Mises equivalent stress distribution and stress curves on the paths (paths 1 to 3) are analysed to study the response between Von Mises equivalent stress and the microstructural evolution of nickel-based single crystal superalloys, as shown in figure 7.

Figure 7. Von Mises equivalent stress distribution: (a) Von Mises equivalent stress distribution in the TCP phase, (b)–(d) Von Mises equivalent stress distribution along paths 1 to 3.
As seen in figure 7(a), stress concentration is clearly present in the TCP phase, and the maximum Von Mises equivalent stress is at the tip of the TCP phase. This is related to the difference between the mechanical properties of the TCP and $\gamma'$ phases. The Von Mises equivalent stress along path 1 on both sides of the TCP phase was different, as shown in figure 7(b). Due to the rightward slant, the TCP phase had a strong effect on the right Von Mises equivalent stress. Therefore, the Von Mises equivalent stress on the right was greater than that on the left. The Von Mises equivalent stress along path 2 was symmetrically distributed around the TCP phase, as shown in figure 7(c). The distribution of Von Mises equivalent stress along path 2 corresponds to that of the TCP phase. The shape of the TCP phase clearly influences the distribution of stress. As seen in figure 7(d), the Von Mises equivalent stress remains approximately the same after 9 $\mu$m along path 3. The influence of the TCP phase on the Von Mises equivalent stress was found to be weak beyond a certain distance. Evidently, the further it is from the TCP phase, the smaller the Von Mises equivalent stress is. However, the distance to the TCP phase is not absolutely proportional to Von Mises equivalent stress, which is related to the morphologies (width and thickness) of the TCP phase.

The widths of the $\gamma'$ phase in rectangles 1 and 2 shown in figure 4(a) are 0.963 $\mu$m and 0.982 $\mu$m, respectively. Le Graverend et al [13] found that the respective sizes of the $\gamma'$ phases on both sides of the TCP phase of nickel-based single crystal MC2 are 1.254 $\mu$m and 1.258 $\mu$m which are approximately the same. The $\gamma'$ phase evidently exhibits the same width at the same Von Mises equivalent stress. The smaller the Von Mises equivalent stress, the smaller the width of the $\gamma'$ phase. The $\gamma'$ phase with the maximum width is adjacent to the TCP phase. It is evident that the width of the $\gamma'$ phase is positively related to the Von Mises equivalent stress and negatively related to the distance to the TCP phase. The rafting of the $\gamma'$ phase can be predicted according to the distribution of the TCP phase and stress.

### 4.2.2. Damage analysis

The damage distribution and damage evolution curves along three different paths are shown in figure 8.

Figure 8(a) shows that the maximum damage occurred at the tip of the TCP phase, and the damage concentration originated from the TCP phase. The change trend of damage along path 1 initially increased and then decreased, and the maximum damage occurred in the TCP phase, as shown in figure 8(b). The damage along path 2 was symmetrically distributed around the TCP phase, and the damage in the TCP phase was significantly greater than that of the two sides, as shown in figure 8(c). The damage decreased gradually with...
increase in the distance to the TCP phase, and the maximum damage occurred in the TCP phase, as shown in figure 8(d). A comparison between figures 7 and 8 showed that the distribution and evolution of the damage were similar to that of the stress.

The damage distribution and evolution law show that the damage is closely related to the TCP phase. The difference between the mechanical properties of the TCP and adjacent \( \gamma \) phase (elastoplastic) was the main reason why the greatest damage originated the TCP phase. The crystal structure of the junction region between the TCP and \( \gamma \) phase was disordered. At the same time, the difference between the mechanical properties of the \( \gamma \) and \( \gamma' \) phases also caused damage, but the damage caused by the TCP phase was significantly greater than that caused by the \( \gamma \) and \( \gamma' \) phases. Therefore, the failure occurred in the TCP phase, which was consistent with the results obtained by Latief et al [30, 35, 36].

5. Conclusion

The mechanical properties of the \( \gamma \), \( \gamma' \) and TCP phases, interrelation between stress fields and microstructure evolution, and damage distribution were studied via the combination of nanoindentation, crystal plasticity theory, and finite element analysis. The following related conclusions were drawn:

1. The Young’s moduli and hardness of the \( \gamma \), \( \gamma' \) and TCP phases were related to the chemical composition and applied stress.

2. Stress concentration in evidently present in the TCP phase, and the maximum Von Mises equivalent stress occurs at the tip of the TCP phase. The distance to the TCP phase is not absolutely proportional to the Von Mises equivalent stress, which is related to the morphologies of the TCP phase. The width of the \( \gamma' \) phase is positively related to the Von Mises equivalent stress and negatively related to the distance to the TCP phase. The rafting of the \( \gamma' \) phase can be predicted according to the distribution of the TCP phase and stress.

3. There is obvious damage concentration in the TCP phase, and maximum damage occurs at the tip of the TCP phase. The damage is negatively related to the distance to the TCP phase. The difference between the mechanical properties of the TCP phase and adjacent \( \gamma' \) phase is the main reason why the TCP phase exhibits the greatest damage. The failure occurs in the TCP phase.

Acknowledgments

The research was supported by the National Natural Science Foundation of China (NO. 51875461, 51875462).

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