Mechanism of gradient strengthening layer formation based on microstructure and microhardness of Inconel 718 grinding surface

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Abstract
Microstructure and microhardness are interrelated variables, which remarkably affecting the wear resistance and fatigue life of materials. This work aims to establish a relationship between the microstructure evolution and microhardness in the grinding surface layer of Inconel 718. The microstructure of the grinding surface layer was characterized by optical microscopy, scanning electron microscopy, transmission electron microscopy, and electron backscattered diffraction, while the microhardness was measured using the Vickers microhardness test. The experimental results show that the grinding surface layer consists of a refined grain region and a high-density dislocation region. Original grains in the refined grain region were refined to form nanograins and elongated grains, owing to the high temperature-induced dynamic recrystallization. The high-density dislocation region contained a large number of dislocations, resulting in the maximal hardness (438.6 ± 11.1 HV0.01) in this region. The microstructure increased the microhardness first and then decreased it to the inner matrix value, and the maximal value was obtained at the high-density dislocation. The microhardness variation is discussed in terms of two strengthening mechanisms (namely grain boundary strengthening and dislocation strengthening), and the strengthening mechanisms in the different regions of the grinding surface layer are revealed.

Keywords Inconel 718 · Grinding · Microstructural evolution · Microhardness · Strengthening mechanisms

1 Introduction
Inconel 718 is a high-strength superalloy with excellent high-temperature resistance, contributing to reliable workability in 23 – 973 K temperature range; thus, it has been widely used in engines and gas turbines [1–3]. This alloy is a nickel-based precipitation hardening wrought superalloy, which is composed of a solid solution γ matrix and a strengthening phase (γ’-Ni3(Al, Ti), γ”-Ni3Nb). The γ” phase is a metastable phase with an ordered body-centered tetragonal DO22 structure, which determines its precipitation as the primary strengthening mechanism of the alloy [4]. The γ’ phase only partially strengthens the alloy, owing to its low proportion. In addition, primary carbide particles (e.g., TiN and NbC) and orthorhombic δ phase (Ni3Nb) exist in the γ matrix [5–7].

The removal of workpiece surface materials by cutting tools significantly changes the mechanical and thermal properties, as well as the surface morphology and microstructure. Especially for precision machining, these changes in the materials’ surfaces are essential for regulating and controlling the machining accuracy. Grinding is a key machining method that guarantees the accuracy of the components’ machined surfaces [8]. It is a complex and dynamic process when metal and alloy materials on the workpiece surface is removed at a high speed by the interaction between the grit and the machined material [9]. In the grinding process, the material in the grinding surface can experience a deformation with a high strain, temperature, and strain rate [10, 11], which can change the microstructure of the material in the grinding surface layer [12]. Grinding in the ductile regime has received special attention as a means of producing high-performance surfaces with minimal residual damage. For this reason, it is important to focus on the microstructural complexion of the material and how it influences the ease and mode of material removal [13].
Inconel 718 has a poor grinding performance, marked by a severe wheel wear [14, 15] and high grinding temperature [16, 17], which contribute to the obvious changes in the microstructure and microhardness of the grinding surface [18–20]. Furthermore, the microstructure and microhardness alterations in the surface layer are especially noteworthy, owing to their direct effects on the fatigue performance of components [21, 22] in the aerospace industry, where safety is very important. Huang et al. [13] mentioned the prospect that future studies will be required to systematically delineate how microstructural features and grain–grain interactions affect material removal. Therefore, it is necessary to investigate the surface microstructure and microhardness changes of the Inconel 718 superalloy induced by grinding for improving the fatigue life and stability of aviation components.

The microstructure of the machined surface and sub-surface directly determines the dimensional accuracy of the workpiece and the ability to resist stress corrosion cracking, fatigueing, and other failure forms in the process of use. Many researchers have studied the effects of machining on the microstructure of the Inconel 718 surface layer. Du et al. [23] studied the surface metamorphic layer after grinding Inconel 718. It was found that the surface metamorphic layer can be divided into three regions with different characteristics: (1) the surface amorphous layer, (2) the micro-shear zone, and (3) the nano-grain layer. Bushlya et al. [24] studied the appearance of a surface white layer, plastic deformation, and recrystallization of the surface. After grinding, a white layer was formed on the grinding surface, which was attributed to the dynamic recrystallization and grain subdivision by severe plastic deformation of the region during the grinding process.

The above research focused on changes in the surface microstructure during material processing. However, microhardness changes affect the processing performance of materials, and the surface microhardness directly determines the wear resistance and fatigue performance of the machined surface. Karabulut et al. [25] proposed that microhardness and wear resistance can be remarkably induced from the final state of the microstructure. The phase state was affected by the heat treatment temperature, which increased microhardness. Sharma et al. [26] studied and compared the material surface properties of Inconel 718 machined with 2-kW continuous wave (CW) Yb-fiber laser. The coarsening of the microstructure (grain size change from 20.5 μm to 47.6 μm) and the increase in the microhardness value (weld zone, 296–319 VHN; base metal, 278 VHN) were observed for the machined surface. Yao et al. [27] found that the surface microhardness decreased after grinding Inconel 718. The main reason was that the γ" phase changed to the δ phase when the temperature rose. Ren and Liu [28] investigated the work hardening phenomenon after machining Inconel 718, and found that the reason can be attributed to refined grains.

However, changes in the surface microstructure and microhardness are not independent of each other, and microhardness embodies the microstructural evolution of strength aspect. Therefore, it is necessary to further analyze the relationship between the sample’s microstructural evolution and the change in the microhardness. Although some researchers have studied the change of surface microstructure and microhardness after machining Inconel 718, the relationship between them has not been discussed in depth. Therefore, the present work aimed at investigating the changes in the surface microstructure and microhardness from the grinding surface to the inner matrix. The relationship between the microstructural evolution and the change in the microhardness are investigated in this study. In addition, the strengthening mechanisms of different regions near the grinding surface are discussed.

### 2 Experimental procedures

The material is Inconel 718 superalloy rod with a diameter of 80 mm, the chemical composition of which is given in Table 1. The heat treatment process for Inconel 718 including solid solution and age-hardened treatments. The solution heat treatment was performed at 1233 K for 1 h followed by air cooling. The double aging was conducted at 993 K ± 5 K for 8 h, followed by furnace cooling to 893 K ± 5 K at a rate of 50 K/h, and finally air cooling to the room temperature.

As shown in Fig. 1a, the grinding experiments in this study were carried out at the three-axis machining center. A cooling nozzle was used to provide wet conditions using a sprayed water-based coolant. The spindle power of the machining center was 5.5 kW, the maximal spindle speed was 18,000 r/min, and the repeated positioning accuracy was ±0.002 mm. The grinding wheels used in the experiments were a resin cubic boron nitride (CBN) cup-shaped wheel and with a grit size approximately 200 μm. The rotation speed of the grinding wheel was n = 4000 rev/min with a radial feed speed f = 100 mm/min and an axial cutting depth a_p = 100 μm, and the grinding experiments were repeated at least three times.

After grinding, the microstructures of Inconel 718 were examined using an ultra-depth of field optical microscope (OM) (VHX-600E, Keyence), a scanning electron

| Table 1 Chemical components of Inconel 718 (wt %) |
|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|-----------------|
| Al | Co | Cr | C | Fe | Mo | Nb | Ti | Ni |
| 0.55 | 0.32 | 17.6 | 0.04 | 20.7 | 2.92 | 5.21 | 1.05 | Balance |

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microscope (SEM) (Supra55, Zeiss), a transmission electron microscope (TEM) (G2, Philips Tecnai) equipped with selected-area electron diffraction (SAED) analysis, and a Tecnai F30 scanning transmission electron microscope (STEM). Cross sections of the samples were prepared for observing the samples’ microstructural evolution, from the grinding surface to the inner matrix. To prevent the edges of the samples from being destroyed when ground by SiC papers, the sample margin was protected by an AB-type epoxy resin (CM4). For OM and SEM examinations, samples after mechanical polishing (0.05-μm colloidal silica) were subjected to electrolytic corrosion, and the etching solution consisted of 80% HCl + 13% HF + 7% HNO₃ (volume fraction). For the TEM analysis, the samples near the grinding surface were prepared by the focused ion beam (FIB) lift-out method, and the TEM samples of the matrix were prepared by twin-jet electro-polishing in a solution of 90% CH₃OH + 10% HClO₄ (volume fraction) at approximately 243 K. For the electron backscattered diffraction (EBSD) examinations, the samples were polished by the cross-section angle polishing method, as shown in Fig. 1b, which is commonly used for magnifying the size of the sample cross section for easier observations [29]. Using this method, the thickness (H) was calculated using the following equation:

\[ H = L \times \sin \theta \]  

where \( L \) is the length after magnified, \( \theta \) is the bevel angle.

In the experiments, \( \theta = 6.4^\circ \), the thickness (H) was increased approximately nine times in the region near the grinding surface. The samples were polished to a final finish by 0.05-μm colloidal silica and argon ion polishing. The EBSD was conducted using a Zeiss Crossbeam 540 equipped with a NordlysMax2 detector and an HKL Channel 5 data analysis system [30], with a scanning step of 1 μm.

A Vickers microhardness measurement apparatus (Feima) was used for measuring the samples’ microhardness, and the test force and keeping time were set to 10 g and 10 s, respectively. It is worth noting that the samples were polished using the cross-section angle polishing method before the Vickers microhardness test. Each region along the radial direction of the grinding surface was tested five times.

An Empyrean X-ray diffractometer (PANalytical, Netherlands) was used for measuring the dislocation density of the grinding surface layer. The XRD measurements were conducted between 20° and 130° at room temperature, using a diffractometer with a Cu target and a step size of 0.008°.

## 3 Results and discussion

### 3.1 Microstructure analysis in the affected layer of the grinding surface

The microstructure analysis of the grinding-affected layer mainly included the grain size, dislocation characteristics, and evolution mechanisms. A certain depth of layer is formed from the grinding surface to the inner matrix. The properties of the affected layer are different from those of the inner matrix, which depend on the processing method and conditions [31]. The microstructural characteristics of the affected layer of the grinding surface, which were analyzed using OM, SEM, and EBSD, are shown in Fig. 2. The inner matrix exhibited no obvious texture, and was composed of equiaxed grains, approximately 15 μm in size, and a small number of annealing twins and NbC particles (Fig. 2a, OM image). As seen in Fig. 2b (SEM image), the average size of the matrix grains was 18 μm, as inferred from the SEM analysis, and consistent with Fig. 2a. In addition, a thin refined grain layer was observed in the near grinding surface (< 5 μm). After angle polishing, there was an obvious 3–4-μm-wide refined-grain layer with, as shown in the EBSD image in Fig. 2c.

To further investigate the microstructure variation of the grinding surface, TEM observations were performed,
and the results are presented in Fig. 3. From the SAED pattern (Fig. 3a-1) and high-resolution TEM (HRTEM) image (Fig. 3a-2), it was found that the inner matrix far from the grinding surface was composed of the $\gamma$ phase and $\gamma''$ phase. It should be noted that the $\gamma'$ phase could not be directly observed because all $\gamma'$ reflections overlapped with $\gamma''$ reflections. In the region within 2.4 μm of the grinding surface, the grains changed from coarse grains ($d_b = 15$ μm) of the inner matrix to ultra-fine equiaxed grains according to the TEM results (Fig. 3b-1). The size of the refined grain size was in the 21–270-nm range. Moreover, nanotwin lamellae of the $\gamma$ phase were observed in this region, which intersected the retained grain or sub-grain boundaries, thus enhancing the strength and thermal stability of Inconel 718 [32]. Within 2.5–4 μm of the grinding surface, unlike the nano-grains mentioned above, many elongated grains parallel to the grinding direction were observed (Fig. 3c). The circumferential lengthening of the diffraction spots in the SAED pattern in this region was obvious, indicating that the material here experienced a certain plastic deformation. By further increasing the distance from the grinding surface to approximately 3.5–5 μm, instead of the nano-grains and elongated grains, a large number of dislocations appeared (Fig. 3d), including dislocation cells and network dislocation, formed during a high-strain plastic deformation.

Through the above analysis of microstructure characteristics observations (Figs. 2 and 3), the affected layer of the grinding surface of Inconel 718 can be divided into three regions: (1) the refined-grain region, (2) the high-density dislocation region, and (3) the inner matrix.

The refined-grain region is the most superficial region of the grinding surface with a width of approximately 3–4 μm. It is subdivided into the nano-grain region and the elongated-grain region, respectively. The nano-grain region is within 2–3 μm of the grinding surface and consists of equiaxial nano-grains and nanotwin lamellae. In the process of grinding, abrasive particles remove the material at a high speed, which leads to the force thermal coupling effect on the surface. Many dislocations appear owing to the plastic deformation on the surface of the material, and dynamic recrystallization of the original structure occurs under high temperature conditions [10]. Thus, these abundant dislocations create new sub-grain boundaries, resulting in the formation of nano-grains. Moreover, because of the low-stacking-fault-energy [33] of Inconel 718, the plastic deformation induces the formation of nanotwin lamellae in this region. The elongated grains are close to the nano grains and exist in the region about 2.5–4 μm away from the grinding surface, which are parallel to the grinding direction. It is worth noting that compared with the nano-grain region, the dynamic recrystallization in the elongated-grain region is not sufficient, owing to the lower temperature.

Then, increasing the distance up to approximately 3.5–5 μm from the grinding surface, the refined grains disappear. From the TEM results (Fig. 3d), high-density dislocation exists in this region. Plastic deformation leads to dislocations with a high density occurring in the surface layer during the grinding process. The dislocations, which are thermodynamically unstable, tend to transform to grain boundary with thermal activation [34]. Therefore, with the temperature increasing, more thermal energy is imported, leading to recrystallization happened. Further, refined grains are formed in the near grinding surface. With the distance from machined surface increasing, the temperature decreases gradually, recrystallization is restrained, where the dislocations with the high density is retained and high-density region is occurred.

The properties of the affected layer in the grinding surface are determined by micro characteristics such as grain shape, size, distribution, and dislocation. To further analyze the mechanism of the microstructure characteristics and
surface material properties, the EBSD local misorientation map is shown in Fig. 4. It can be observed that the local misorientation of the refined-grain region is small. The main reason for this phenomenon is dynamic recrystallization, which indicates a small residual strain in the refined-grain region. The grain size and release of internal stress during dynamic recrystallization can improve the fatigue properties and fatigue life of materials [35]. Compared with the grain refinement zone, high-density dislocation region exhibits significant local misorientation, and which leads to severe residual strain. High-density dislocation can improve the wear resistance of grinding surfaces. However, the residual strain in this region induces microcracks when a load is applied, which damages the corrosion resistance and fatigue property of materials [36].

3.2 Microhardness variation of affected layer

As shown in Fig. 5, the microhardness variation below the grinding surface with increasing depth. The microhardness decreases with increasing distance from the surface and is the lowest (306 ± 7.2 HV0.01) at 40 μm from the grinding surface. The maximal microhardness value is observed at a distance approximately 2–4 μm from the grinding surface. The microhardness varies from 405 ± 12.7 HV0.01 to 438.6 ± 11.1 HV0.01 in this region, and the average microhardness value is 420 ± 9 HV0.01. The microhardness results are similar to the microstructure results, and the main characteristics of the three regions (the refined-grain region, the high-density dislocation region, and the inner matrix) are different. The results of the microhardness analysis show that there
are different degrees of strengthening in the affected layer of the grinding surface. This phenomenon is defined as a gradient-hardening layer that is related to the evolution of the microstructure. The strengthening mechanisms are different in each region, and will be discussed in detail in the following section.

### 3.3 Mechanism of the gradient strengthening layer

As shown in Fig. 4, the microstructural evolution from the grinding surface to the inner matrix affects the microhardness in these regions. It increases gradually throughout the refined-grain region, then peaks in the high-density dislocation region, after which it decreases gradually and finally reaches the level of the inner matrix. The variation in the microhardness is owing to the different strengthening mechanisms in different regions, that is, grain boundary strengthening and dislocation strengthening.

#### 3.3.1 Strengthening in the refined grain region

According to the TEM results, the size of the refined grains is in the 21-270-nm range, and the average grain size is approximately $d_r = 160$ nm, which is much smaller than the average grain size in the inner matrix ($d_b = 15$ μm). The strength (or microhardness) must be higher, owing to the strengthening of the refined grains. Thus, the yield strength increment ($\Delta \sigma_s$) is [37, 38]:

$$\Delta \sigma_s = kd_r^{-1/2} - kd_b^{-1/2}$$  \hspace{1cm} (2)

where $k$ is the Hall–Petch [39] strengthening coefficient of Inconel 718 and its value is 158 MPa·μm$^{1/2}$ [40, 41], and the calculated yield strength increment $\Delta \sigma_s$ is 354.2 MPa. The yield strength increment $\Delta \sigma_s$ can be converted to the microhardness increment ($\Delta H$) using the following equation [42, 43]:

$$\Delta H = \alpha \Delta \sigma_s$$  \hspace{1cm} (3)

where $\alpha$ is the Tabor factor of Inconel 718 and its value is 2.918 [44], and the microhardness increment between the refined-grain region and the inner matrix is approximately calculated as 105.4 HV$_{0.01}$, which is slightly smaller than the measured result (114 HV$_{0.01}$); this small difference may be attributed to the nanotwins appearing in the nano-grain region providing additional strengthening [45].

#### 3.3.2 Strengthening in the high-density dislocation region

Many tangled dislocations were distributed in the high-density dislocation region (Fig. 3d), leading to the maximal microhardness (438.6 ± 11.1 HV$_{0.01}$). As shown in Fig. 5, the microhardness decreased to the matrix hardness at approximately 40 μm from the grinding surface. Therefore, there was a work-hardening layer with a depth of 36 μm, owing to dislocation strengthening.

The Bailey-Hirsch formula [37, 46] was used for describing the relationship between the yield strength ($\Delta \sigma_D$) and the dislocation density ($\rho$), as follows:

$$\Delta \sigma_D = \alpha \rho^{1/2}$$  \hspace{1cm} (4)

where $\alpha_1$ is a constant associated with the dislocation structure, and its value is 0.52 [47]; $G = 78.5$ GPa is the shear modulus [48]; and $b = 0.254$ nm is the Burger vector of Inconel 718 [49].
The Pantleon method [50] was used for identifying the lattice curvature from local orientation measurements using the EBSD, and the dislocation density tensors were derived. Thus, the dislocation density (ρ) was roughly calculated using the following Eq. (5):

\[
\rho = \frac{1}{b} (|a_{12}| + |a_{13}| + |a_{21}| + |a_{23}| + |a_{33}|)
\]

(5)

where \(a_{12}, a_{13}, a_{21}, a_{23},\) and \(a_{33}\) are the components of the dislocation density tensor, which can be calculated after obtaining the coordinate values and Euler angles of pixels in the EBSD image through HKL Channel5 software processing [51].

In Fig. 6, the dislocation density calculated from the EBSD results (Fig. 2c), and the average dislocation density calculated from the processing of each row in Fig. 6 is shown in Fig. 7. The farther away from the grinding surface, the smaller the dislocation density, and this variation trend is basically consistent with that of the microhardness.

Moreover, the dislocation density of the grinding surface layer was calculated using the Williamson-Hall method [52–54]. The XRD patterns for Inconel 718 before and after grinding are presented in Fig. 8. The dislocation density \(\rho\) of the grinding surface layer was calculated using Eq. (6):

\[
\rho = 14.4\varepsilon^2 / b^2
\]

(6)

where \(\varepsilon\) is the micro-strain, and the value was obtained from the XRD results; the values of \(\varepsilon\) for the grinding surface layer and the inner matrix were \((0.476 \pm 0.0067)\%\) and \((0.261 \pm 0.0102)\%\), respectively. Finally, the calculated dislocation density of the grinding surface layer and the inner matrix were \(5.06 \times 10^9 \pm 1 \times 10^6\) mm\(^{-2}\) and \(1.52 \times 10^9 \pm 2.32 \times 10^6\) mm\(^{-2}\), respectively. Furthermore, the dislocation density increment between the grinding surface layer and the inner matrix was approximately \(\Delta \rho = 3.54 \times 10^9\) mm\(^{-2}\). Therefore, the highest microhardness...
increment between the grinding surface layer and the inner matrix was approximately 189 HV₀.₀₁, close to the experimental value (132.6 HV₀.₀₁). These results indicate that in the high-density dislocation region, the change in the microhardness was mainly caused by dislocation strengthening.

The results of the theoretical calculation are summarized and compared in Table 2. Therefore, different strengthening mechanisms affect microhardness increments in the regions near the grinding surface, and this variation tendency is determined by the microstructural evolution. The change in the microhardness and the inner matrix value is caused by the grain boundary strengthening in the refined-grain region, while the microhardness change is caused by dislocation strengthening in the high-density dislocation region.

Fatigue cracks usually originate on the machined surface. Reasonable surface hardening and grain refinement can improve the fatigue life of materials. The refined-grain region improves the fatigue property and fatigue life of materials [35]. However, in the region of high-density dislocations, there exists the large residual strain which consequently decreasing the corrosion resistance and fatigue property the fatigue life of materials [36]. In this work, the relationship between the microstructure and microhardness was established through the analysis of strengthening mechanism, which establishes a theoretical basis for further optimizing the microstructure and surface hardness.

Table 2 Results of the theoretical calculation process and measured

| Region                | d/μm | Δσ/MPa | ΔH/HV₀.₀₁ | ΔH₁/HV₀.₀₁ |
|-----------------------|------|--------|-----------|------------|
| Refined-grain region  | 160  | 354.2  | 105.4     | 114        |
| High-density dislocation region | 5.06 × 10⁶ ± 1 × 10⁶ | 3.54 × 10⁹ | 189 | 132.6 |

ΔH is the microhardness increment by calculated, and ΔH₁ is the experimental value

(2) The strengthening mechanism of the surface gradient hardening layer mainly includes grain boundary strengthening and dislocation strengthening. Grain boundary strengthening in the refined-grain region increases the final microhardness. Dislocation strengthening occurs in the high-density dislocation region. The increment of the dislocation density between the grinding surface layer and the inner matrix is approximately 3.54 × 10⁹ mm⁻², leading to a maximal microhardness (438.6 ± 11.1 HV₀.₀₁).

(3) The variation tendency of the microhardness is closely related to the microstructural evolution, which increases first, reaches a maximum, and then decreases to the matrix value with an increase in the distance from the grinding surface. The calculated microhardness increments through these mechanisms in the refined-grain region and high-density dislocation region are consistent with the measured values.

(4) Controlling the grinding parameters to increase the grain refinement region and reduce the high dislocation density region is an important research direction for the subsequent anti-fatigue machining process. A multi-index coupled surface integrity model can be realized based on the relationship between microstructure and microhardness.

4 Conclusions

The affected layer of the grinding surface of the Inconel 718 superalloy was investigated, which included a refined-grain region and a high-density dislocation region. Compared with the matrix, the affected layer exhibited obvious hardening and formed gradient hardening layer. It can be concluded that the evolution of the microstructure is the fundamental reason for the formation of gradient strengthening layer.

(1) The refined-grain region is the nearest region to the grinding surface, with a width of approximately 3–4 μm, which is composed of equiaxed nanograins and elongated grains. The second-nearest region is distributed by a huge number of tangled dislocations, in which the dislocation density gradually decreases with increasing distance from the grinding surface.

Author contribution Renke Kang contributed to the conception of the study; Lu Han performed the experiment; Zhiqiang Dong contributed significantly to analysis and manuscript preparation; Nianwei Xu and Yuan Zhang performed the data analyses and wrote the manuscript; Xiaofeng Wu and Yan Wang helped perform the analysis with constructive discussions.

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Data availability Data used to support the findings of this study are available from the corresponding author upon request.

Code availability Not applicable.
Declarations

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Consent to participate Not applicable.
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Competing interests The authors declare no competing interests.

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