Enhancement of wear and erosion-corrosion resistance of Inconel 718 alloy by liquid nitriding

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Abstract

Inconel 718 alloy is often used for the oil and gas industry, which usually failed due to the erosion-corrosion of multi-phase flow. In this study, we expect to enhance the wear and erosion-corrosion resistance of Inconel 718 alloy by liquid nitriding and a high-hardness nitrided layer with the thickness of about 9.5 μm on the surface of Inconel 718 alloy was formed by liquid nitriding. Wear and erosion-corrosion tests were performed on untreated and nitrided Inconel 718 alloys. Results show that abrasive wear and adhesive wear occurred in both nitrided and untreated samples at different temperatures (25 °C, 100 °C and 200 °C). The width and depth of the wear track and the wear rate of the nitrided samples are all smaller than that of the untreated samples. Liquid nitriding significantly improves the wear resistance of Inconel 718 alloy because of the high-hardness nitrided layer. The nitrided Inconel 718 alloy exhibited an improvement of almost 80.3% in the erosion-corrosion resistance compared to their untreated counterparts. The weight loss rate of the erosion-only shows an obvious reduction after liquid nitriding. The surface nitrided layer with high hardness effectively protects the substrate, and prevents the material from the erosion-corrosion of multi-phase flow consisting of the SiO2 particles and the acidic solution, so that the total weight loss of nitrided Inconel 718 alloy decreased evidently.

1. Introduction

Inconel 718 alloy is one of the most important nickel-based superalloys, and has wide applications in many special fields that require high comprehensive performance of materials, such as aerospace, industrial gas turbines, nuclear engineering, etc, due to its excellent mechanical properties as well as good corrosion resistance, outstanding strength, superior fatigue and radiation resistance together with good oxidation resistance at high temperature [1–6]. Also, Inconel 718 alloy is the preferred material for manufacture of wellhead components, downhole equipment and auxiliary tools in the oil and gas industry, such as valve systems, tubing hangers, packers, safety valves, fasteners, and drill tools [7–11]. As we all know, the environment of the acidic oil and gas field is very complex, and there are multi-phase media such as water, gas, hydrocarbons, and solids that move with each other. High-speed moving solid-phase particles (such as sand particles) can easily wear the material surface, thereby making the metal material occur serious erosion-corrosion in liquid-solid or multi-phase flow corrosive environments, and the erosion-corrosion will promote the premature failure of materials when exposed to erodent in a corrosive environment [12]. Further, some of these environments are at high pressure and temperature, more than 200 °C [13], and the materials are more likely to fail especially in high-temperature oil and gas fields. The service conditions of nickel-based 718 alloys for acidic oil and gas fields determine that it requires better wear and corrosion properties. Despite its otherwise excellent properties, Inconel 718 alloy has unsatisfactory wear characteristics [14]. This disadvantage may be eliminated by surface treatment such as thermal spraying [15], physical vapor deposition (PVD) [16], chemical vapor deposition (CVD) [17], electrolytic plasma processing [18, 19] and nitriding [20] without compromising the other properties of the material.
Nitriding is one of the most widely applied thermochemical surface treatment to enhance the wear and corrosion resistance of metallic materials [21]. And researchers have done a lot of research on the use of nitriding technology to improve the wear and corrosion resistance of metal materials in recent years. In [22], the wear and corrosion resistance of Inconel 625 nickel alloy was improved by glow discharge assisted nitriding. Literature [23] demonstrated that plasma nitriding of alloy Inconel 718 provides substantial improvement of its wear resistance. Besides, Qin et al [24] illustrated that the erosion-wear resistance of 304L austenitic stainless steel was improved by plasma nitriding due to large compressive stress caused by the formation of the \( \gamma_N \) phase. Lu et al [25] reported that the surface hardness, wear and corrosion resistance of 304 austenitic stainless steel was increased dramatically after plasma nitriding. Baranowska et al [26] found that the wear resistance of AISI 321 steel in a corrosive liquid environment can be improved by gas nitriding. In [27], the erosion-corrosion and corrosion properties corrosion resistance of AISI 316 stainless steel was enhanced by gas nitriding. And in [28], the liquid-nitrided AISI 316 stainless steel exhibited superior resistance to localized corrosion. The liquid nitriding treatment was regarded as an effective, low-cost method with many advantages, such as low treatment temperature, short treatment time, high degree of shape and dimensional stabilities, and reproducibility [29]. However, liquid nitriding of Inconel 718 alloy and whether this treatment can improve the wear and erosion-corrosion resistance were rarely researched. Therefore, liquid nitriding has applied to Inconel 718 alloy in this study.

The goal of the present work is to improve the wear and erosion-corrosion resistance of Inconel 718 alloy. The main research contents include three parts: (1) analyzes the thickness, phase, microhardness, and microstructure of the nitrided layer; (2) researches the wear behavior of untreated and nitrided Inconel 718 alloy; (3) investigates the erosion-corrosion resistance and mechanism of untreated and nitrided Inconel 718 alloy during liquid-solid dual-phase flow.

### 2. Experimental

#### 2.1. Materials

The raw material used for investigation is a hot-rolled Inconel 718 alloy, and its chemical compositions are summarized in Table 1. All samples were machined into a size of 30 mm \( \times \) 15 mm \( \times \) 3 mm. The surfaces of the samples were polished step by step with abrasive SiC papers of 240, 600, and 1000 grits, then ultrasonically cleaned in anhydrous ethanol for 10 min followed by air drying, before being subjected to nitriding.

#### 2.2. Nitriding

Raw materials used for liquid nitriding are nontoxic cyanate (KCNO and NaCNO), chloride (KCl and NaCl), and carbonate (K\(_2\)CO\(_3\), Na\(_2\)CO\(_3\), and Li\(_2\)CO\(_3\)). The compositions of these components were described in detail in our previous study [30]. The Inconel 718 alloy were suspended with steel wire and immersed in nitriding salt bath at 500 °C for 16 h. The nitriding process is mainly the decomposition of CNO\(^{-}\) in the salt bath to generate active nitrogen atoms [N]. [N] diffuses into the surface of the sample to form a nitrided layer. The main reaction can be described as following equations:

\[
4\text{CNO}^- \rightarrow \text{CO}_2^2- + 2\text{CN}^- + \text{CO}_2 + 2[\text{N}] \tag{1}
\]

\[
[\text{N}] + \text{Cr} \rightarrow \text{GrN} \tag{2}
\]

After the liquid nitriding was completed, the sample was air-cooled to room temperature and then immersed in clean water until the nitriding salt attached to the surface was removed by hydrolysis. Finally, it was ultrasonically washed with anhydrous ethanol for 10 min and dried.

#### 2.3. Characterizations

The cross-sections of the sample were etched using a solution containing 20 ml hydrochloric acid, 20 ml anhydrous ethanol, and 1.5 g anhydrous copper sulfate etchant. An Olympus CK40M optical microscope was used to observe the metallography and measure the thickness of the nitrided layer. The phase constituents were determined by EMPYREAN x-ray diffraction (XRD) using Cu-K\(\alpha\) (\(\lambda = 1.54 \text{ Å}\)) radiation over the 2\(\theta\) range 20–80°. Hardness measurement was made by an HXD-1000TMC Digital Intelligent microhardness tester, with a test load of 10 g and the holding duration of 15 s. Surface and cross-sectional morphologies were analyzed by

| Elements | C | Ni | Cr | Mo | Nb | Ti | Co | Mn | Si | Al | Fe |
|----------|---|----|----|----|----|----|----|----|----|----|----|
| Content  | 0.08 | 55 | 21 | 3.3 | 5.1 | 1 | 0.3 | 0.35 | 0.15 | 0.8 | Bal. |
scanning electron microscope (SEM; JSM-6510). The 3D images and roughness were determined by an atomic force microscope (AFM; MFP-3D-BIO) using tapping mode. The weight loss of the samples was measured with an optical analytical balance accurate to 0.1 mg.

2.4. Wear tests

Wear tests were performed with a pin-on-disc tribometer (HT-1000 wear tester; Lanzhou Zhongkekaihua Technology Development Co., Ltd, China). During the test, samples were rotating against a stationary grinding ball (Si₃N₄) of 5 mm diameter at a speed of 1000 rpm. The normal contact load was 2 kg, and the total sliding time of each sample was 30 min. The Si₃N₄ ball contacted the sample surface at 2.5 mm off the axis of rotation to produce a wear track of 5 mm in diameter on the sample surface. Tests were unlubricated and performed at different temperatures (25 °C/100 °C/200 °C). The total sliding distance is 471.2 m. After wear testing, the worn surfaces were examined using a scanning electron microscope (SEM, JSM-6510), and the three-dimensional wear track was studied by an optical profilometry (ContourGT-K Optical profiler). Then, the wear rate of each sample was determined based on the following equation [31]:

\[
W_R = t \times (3r^2 + 4b^2) \times 2\pi r / (6b \times F \times S)
\] (3)

where \(W_R\) is the wear rate (mm³·N⁻¹·m⁻¹), \(t\) is the depth of the wear track (mm), \(b\) is the width of wear track (mm), \(r\) is the radius of wear track (mm), \(F\) is the load pressure (N), \(S\) is the total sliding distance (m), respectively.

2.5. Erosion-corrosion tests

The schematic diagram of the erosion-corrosion experimental device is shown in figure 1, and the device is mainly composed of a polymer container, an agitator and a controller. The erosion-corrosion test was performed in 3.5 wt.% sodium chloride (NaCl) and 5 vol.% hydrochloric acid (HCl) solutions with the addition of 100 g l⁻¹ sand particles (SiO₂). The erosion-only test was performed in an aqueous environment consisting of only 100 g l⁻¹ sand particles (SiO₂). The average size of the sand particles used in this work was approximately 50 μm. The stirring speed was maintained at 500 rpm by using the controller. The process was carried out for 168 h (7 days) at 25 °C. The weight loss was determined for the erosion-corrosion and erosion-only samples every 24 h. The weight loss rate of erosion-corrosion and erosion-only of samples was calculated as the following equation [32]:

\[
R = (M_i - M_f) / (S \times T)
\] (4)

where \(R\) is the weight loss rate (mg·cm⁻²·min⁻¹), \(M_i\) is the initial weight (mg), \(M_f\) is the weight after immersion test (mg), \(S\) is the surface area before the tests (cm²), \(T\) is the test time (h), respectively.

3. Results and discussions

3.1. Characteristics

Figure 2 shows the cross-sectional microstructure of the Inconel 718 alloy after liquid nitriding treatment. It can be seen that a homogeneous and dense nitrided layer on the sample surface, with a thickness of about 9.5 μm.
There is an obvious boundary between the nitrided layer and matrix, this phenomenon should due to the difference in microstructure and corrosion resistance between the nitride layer and matrix.

The typical x-ray diffraction (XRD) patterns for untreated and nitrided Inconel 718 alloy are shown in figure 3. The untreated sample shows only the austenite phase ($\gamma$), while the nitried sample shows the austenite phase and CrN phase due to the infiltration of the N element during the nitriding process. The $\gamma$ peaks of the nitried sample are lower in intensity than that of the untreated sample, and this is understandable since the presence of the nitried layer weakens the matrix signal collected by the x-ray. When the temperature is higher than 450 °C, the negative enthalpy of CrN is high, and the diffusion rate of Cr in the matrix is low, so it is easier to form Cr nitrides [33]. This process was performed at 500 °C, so the main component of the nitried layer is CrN, which is consistent with the research results of Yan et al [34].

Figure 4 shows the evolution of the cross-sectional microhardness of the nitried sample. It is obvious that the liquid nitriding treatment significantly increased the surface hardness of Inconel 718 alloy. The surface hardness value (1810 HV0.01) is the highest, about 4.5 times that of the matrix hardness (400 HV0.01). From the nitried layer to the substrate, the microhardness value decreases sharply, and then gradually decreases to the substrate hardness. The high hardness of the surface layer is mainly due to a supersaturated solid solution containing N element was formed on the surface of the sample by nitriding. Meanwhile, the continuous diffusion of N atoms into the austenite caused severe lattice distortion, which increased its dislocation density [35]. Also, in the tensile stress region of the dislocation, N atoms are more prone to segregation, which makes pinning dislocations more likely to occur, and then promotes solid solution strengthening [36]. At the same
time, the medium-strong nitride-forming elements such as Mo and Ti contained in the Inconel 718 alloy structure can be combined with N atoms in the nitriding process to form nitrides, which is hard, fine and stable, and is uniformly distributed at the grain boundaries, can result in dispersion strengthening [37].

3.2. Wear

Figure 5 shows the cross-sectional profiles of the wear tracks for untreated and nitrided samples at different test temperatures. The wear trace of the nitrided samples is shallower and narrower than that of untreated samples at the same temperature. Table 2 shows the size of wear track and wear rate for each sample. It can be seen that the wear rate of nitrided sample is much smaller than that of the untreated sample at the same temperature. This indicates that the liquid nitriding treatment can significantly improve the wear resistance of Inconel 718 alloy at the temperatures of 25 °C, 100 °C and 200 °C. This is because a nitrided layer with high hardness was generated on the surface of the sample by liquid nitriding treatment. The high-hardness nitrided layer has a good protective effect on the matrix during the wear process, which greatly reduces the wear rate. The results are agreed very well with the research conclusion of Aksoy et al [38], the harder the sample, the more resistant it is to wear.

Figure 6 shows the 3D wear morphologies of untreated and nitrided samples. Figure 7 shows a detailed view of the wear tracks by SEM morphologies. The worn surface of each sample is rough with obvious delamination and plastic deformation. For the untreated samples, the tracks were relatively deeper and wider, and many deep grooves along the sliding direction can be observed from the 3D topography as shown in figures 6(a), (c) and (e). This consistent with the SEM morphologies in figures 7(a), (c) and (e), respectively, which shows many clear grooves and severe spallation. This phenomenon indicates that abrasive wear and adhesive wear occurred on
untreated samples. On the other hand, a smooth wear track appeared on the nitrided samples from the 3D topographies in figures 6(b), (d) and (f), which indicates that the nitrided samples suffered much less damage than the untreated samples during the wear test. As shown in figures 7(b), (d) and (f), it can be seen that some lamellar spalling appeared on the worn surface, and this is a typical characteristic of adhesive wear. Also, some grooves can be found by carefully observing the worn surface of the nitrided sample, and this is the evidence of abrasive wear.

During the wear tests, the Inconel 718 alloy and the Si₃N₄ grinding ball will appear plastic deformation, which makes the contact surface of the friction pair appear local adhesion phenomenon. The adhesion point was destroyed under the relative sliding of the frictional pair, and it will cause the material to peel off from the sample surface. Therefore, tracks of tearing and peeling appear on the worn surface, showing typical adhesive wear characteristics. At the same time, the Si₃N₄ grinding ball has a very high hardness, and its hard particles will continuously scratch the sample surface during grinding, which will cause grooves, so the worn surface also shows the typical characteristics of abrasive wear.

### 3.3. Erosion-corrosion behavior

Figure 8 shows the relationship between weight loss and time for untreated and nitrided Inconel 718 alloy samples. The weight loss of the two materials gradually increases with the increase of time in the same test environment. As shown in figure 8(a), during the same test time of the erosion-corrosion process, the weight loss of the nitrided sample is much lower than that of the untreated sample. Also, it can be seen that the weight loss of the nitrided sample during 0–24 h is the largest. This may be because liquid nitriding formed a thin oxide film on the outermost surface of Inconel 718 alloy. The outermost oxide film has a lower hardness than the nitrided layer and is more likely to be damaged when it was impacted by the hard particles of SiO₂, and the cracked oxide film

| Depth (µm) | Width (mm) | Wear rate (mm³N⁻¹m⁻¹) |
|-----------|------------|------------------------|
| 25 °C     | Untreated  | 114.07                 | 2.05 | 2.60 x 10⁻⁴ |
| Nitrided  | 0.22       | 1.08                    | 2.64 x 10⁻⁷ |
| 100 °C    | Untreated  | 100.37                 | 1.99 | 2.22 x 10⁻⁴ |
| Nitrided  | 0.24       | 1.15                    | 3.07 x 10⁻⁷ |
| 200 °C    | Untreated  | 114.53                 | 1.99 | 2.54 x 10⁻⁴ |
| Nitrided  | 0.25       | 1.15                    | 3.19 x 10⁻⁷ |

Figure 6. 3D wear morphologies of untreated and nitrided samples: (a), (b) 25 °C, (c), (d) 100 °C, (e), (f) 200 °C.
will soon leave the sample surface under the synergy of erosion and corrosion. As shown in figure 8(b), during the erosion-only process, the untreated sample has more obvious weight loss, and the curve was steeper than that of the nitrided sample. It is well known the untreated Inconel 718 alloy has good corrosion resistance, but its matrix hardness (about 400 HV0.01) is relatively low, resulting in poor resistance to the impact of SiO2. On the

| Temperature | Untreated | Nitrided |
|-------------|-----------|----------|
| 25°C        | ![Untreated 25°C](image) | ![Nitrided 25°C](image) |
| 100°C       | ![Untreated 100°C](image) | ![Nitrided 100°C](image) |
| 200°C       | ![Untreated 200°C](image) | ![Nitrided 200°C](image) |

**Figure 7.** SEM morphologies of the worn surfaces of untreated and nitrided samples: (a), (b) 25 °C, (c), (d) 100 °C, (e), (f) 200 °C.

**Figure 8.** The relationship between weight loss and time for untreated and nitrided Inconel 718 alloy samples: (a) erosion-corrosion, (b) erosion-only.
contrary, the high hardness (about 1810 HV0.01) nitrided layer of the nitrided sample has excellent wear resistance and can effectively resist the mechanical erosion of the hard particles to the material surface, and this is consistent with the results of the wear tests in section 3.2. By further analysis and calculation, after the material has been tested for 168 h, the weight loss rate of the erosion-corrosion (0.0037 mg·cm$^{-2}$·h$^{-1}$) and the erosion-only (0.0015 mg·cm$^{-2}$·h$^{-1}$) of the nitrided sample is much lower than that of the untreated sample, and the nitrided Inconel 718 alloy exhibits an improvement of almost 80.3% in the erosion-corrosion resistance compared with its untreated counterpart.

Figure 9 shows the 3D surface morphologies of the untreated and nitrided Inconel 718 alloy samples after 168 h of erosion-corrosion and erosion-only. Figures 9(a) and (b) show the 3D surface morphologies of samples after erosion-corrosion. It can be seen that the surface morphologies of the materials in the two different states are completely different. The surface of the untreated sample is uneven, with very obvious erosion tracks and shear lips caused by plastic deformation of the material. Compared with untreated samples, the surface of nitrided samples is relatively flat, and there are fluvial erosion tracks on the surface, but the fluctuation is not obvious. Also, the surface roughness of the untreated sample (127.4 nm) is higher than that of the nitrided sample (220.2 nm). Figures 9(c) and (d) show the 3D surface morphologies of samples after erosion-only. The nitrided sample surface appears lots of scratches, and this is because the sample was polished with 1000 grits SiC paper before nitriding. Nitriding treatment greatly increased the surface hardness and wear resistance of sample, so the surface was less worn when impacted by SiO$_2$ hard particles, thus the scratches were still clearly visible after the erosion-only. Compared with the nitrided sample, the surface roughness of the untreated sample is lower after the erosion-only. This is because the untreated sample with low surface hardness is prone to be damaged by the impact of SiO$_2$ hard particles, which causes the surface scratches to be gradually flattened, and finally only some burr-like traces appear.

Figure 10 shows the microscopic morphologies of untreated and nitrided Inconel 718 alloy samples after 168 h of erosion-corrosion. Figures 10(a) and (b) show the cross-sectional SEM morphologies of untreated and nitrided samples. The untreated sample shows low resistance to erosion-corrosion, and the surface of the material has obvious partial damage and shear lip appeared. In contrast, the surface nitrided layer of the nitrided sample remains intact, with only slight thinning (from 9.5 $\mu$m to 7.1 $\mu$m), which still can effectively protect the substrate. The surface SEM morphologies of the untreated and nitrided samples after erosion-corrosion is shown in figures 10(c) and (d). The surface of the untreated sample is rough, with obvious erosion tracks and many dense corrosion pits. While there are only some scattered corrosion pits and a few small erosion traces on the surface of the nitrided samples, and the degree of damage to it is much smaller than that of the untreated sample. These phenomena can be attributed to the fact that the surface hardness of the nitrided sample is higher.
than that of SiO₂ particles, which can break the particles impacted the material surface, reduce the transmission of impact energy to the material surface, improve the plastic deformation resistance of the Inconel 718 alloy, and finally reduce the influence of the wear and cutting of hard particles on the material surface. Also, some literature [39, 40] shown that the appearance of the nitrided layer can effectively prevent the material surface from being damaged by Cl⁻ and improve the material’s corrosion resistance to Cl⁻. Observing in figure 10(d), we can also find that grain boundaries appeared on the surface of the nitrided sample, which indicates that intergranular corrosion occurred. This is because there are a lot of lattice defects such as dislocations and slips at the grain boundaries, which make the grain boundary has a lower electrode potential relative to other regions so that different parts of the sample form a galvanic cell in the corrosive medium, and the grain boundary is dissolved as the anode.

Figure 11 shows the erosion-corrosion schematic diagrams of the untreated and nitrided Inconel 718 alloy samples. As shown in figure 11(a), the untreated sample suffered from severe mass loss, and its surface is uneven, with a large number of corrosion pits, and some shear lips and deep erosion tracks along the direction of liquid flow. Due to the low hardness (400 HV0.01) of the untreated material, when subjected to the erosion effect of SiO₂ hard particles, the mechanical shear forces can easily damage the sample surface and result in partial crack of the surface passivation film. At the same time, Cl⁻ with strong penetrability is easily attached to the surface of the passivation film, causing local damage to the passivation film. Once this inherent protective film is destroyed, the corrosion rate of the material in an acidic medium will be greatly accelerated [41].

As shown in figure 11(b), the corrosion pits and erosion traces on the surface of the nitrided sample are less and shallower than that of the untreated sample. The surface nitrided layer shows slight thinning, but still can effectively protect the substrate. The nitrided layer has a high hardness (1810HV0.01), which can resist plastic deformation and reduce the cutting damage of hard particles on the material surface [42]. The research of Xi et al [43] indicated that the combined action of microstructure, high surface hardness and high compressive residual stress in the nitrided layer improves the erosion resistance of the material. Simultaneously, the nitrogen in the modified layer has an important effect on improving the corrosion resistance of the alloy in the acidic environment. The rich nitrogen element in the nitride layer can make the austenite phase more stable and effectively prevent the corrosion of Cl⁻ on the material surface [44, 45]. And the nitrogen atom will react with H⁺ in the corrosive solution [46]:

![Figure 10](image-url)
which effectively reduces the concentration of $H^+$ on the surface of the sample, prevents the decrease of the pH in the solution, and thus improves the corrosion resistance of the material. Therefore, the presence of the nitrided layer can prevent material from the erosion-corrosion of multi-phase flow consisting of the SiO$_2$ particles and the acidic solution.

4. Conclusions

In this work, liquid nitriding was used to improve the wear resistance and erosion-corrosion resistance of Inconel 718 alloy. The characteristics of the nitrided layer, the wear properties and erosion-corrosion behaviors before and after nitriding were analyzed and the results can be summarized as follows:

(1) The nitrided layer formed on Inconel 718 alloy surface has the microhardness of up to 1810 HV0.01, which is higher 352% than that of the matrix.

(2) The wear tests conducted at different temperatures (25 °C, 100 °C and 200 °C) show that abrasive wear and adhesive wear occurred in both nitrided and untreated samples. The width and depth of the wear track and the wear rate of the nitrided samples are all smaller than that of the untreated samples, which indicates that the liquid nitriding treatment can significantly improve the wear resistance of Inconel 718 alloy.

(3) Nitrided Inconel 718 alloy exhibits an improvement of almost 80.3% in the erosion-corrosion resistance compared with its untreated counterpart. And the weight loss rate of the erosion-only shows an obvious reduction after liquid nitriding. This proved that the surface nitrided layer with high hardness can effectively protect the substrate, and prevent the material from the erosion-corrosion of multi-phase flow consisting of the SiO$_2$ particles and the acidic solution.

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