Effect of interfacial friction on wear behavior and microstructure during extrusion of Pure Nickel N6

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Original Article

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Abstract

This paper combines field extrusion and finite element simulation to study the influence of the friction state on the extrusion deformation of pure nickel. The use of lubricant reduces the shearing force of the billet-die interface, resulting in lower heat generation at the interface, greater forming stress and strain, and reduced uneven deformation of the billet. As a result, the surface quality of the workpiece is relatively good, but uneven lubrication is the main cause of tearing damage on the surface of the billet. The grain morphology, grain boundary distribution, and geometrically necessary dislocations distribution on the surface of the lubricating extruded bar are uniform, the microtexture strength is weak, and the Schmidt factor is large.

1 Introduction

N6 grade pure nickel is a single austenitic non-ferrous metal. Due to its excellent corrosion resistance, it is widely used in petrochemical, coal chemical, alkali production and other fields [1–4]. Currently, pure nickel and nickel alloys are mainly formed by extrusion, rolling, and drawing processes, among which extrusion is the best process for obtaining tubes/bars [5–7]. In the extrusion process, the interface friction caused by the relative movement between the billet and the die strongly affects the surface quality, the mechanical properties of the workpiece and the service time of the die [8, 9]. The use of lubricants for improving the friction state of the billet-die interface and reducing wear has attracted significant research attention, with great progress made in research on the extrusion friction and lubrication of lightweight alloys (magnesium, aluminum) [10, 11]. By contrast, for the process of pure nickel extrusion, there is a lack of comprehensive understanding of the friction and lubricants and their wear characteristics and the structure of the workpiece surface, as well as die wear. Under normal conditions, the die cannot be reused the surface of the workpiece is uneven, and the surface structure is different from the internal structure. This greatly hinders the development of extrusion technology for pure nickel and nickel alloys.

Pure nickel extrusion is a plastic deformation process in which the billet is subjected to three-dimensional compressive stress and multi-factor coupling [12]. A large amount of material flows through the die, making the working environment of the die and the billet highly complicated. It is impossible to directly measure friction and observe wear during the extrusion process, and laboratory extrusion tests can only provide indirect information regarding the friction between the billet and the die [13]. Therefore, the study of the billet-die interface friction in the pure nickel extrusion process is a highly challenging.

The research methods used in the past two decades for the investigations of billet-die interface friction in the extrusion production process can be divided into three categories of field tests, physical simulation tests, and tribological tests [14]. Hild [15] analyzed the friction and wear phenomenon in the forward extrusion process under dry friction conditions, and found that the maximum wear of the die does not occur in the area with the highest pressure, also pointed out that no suitable wear model is currently available. The effect of lubricant quantity on friction was studied by combining field extrusion test and finite element simulation [16]. Dang et al. used the finite element method to analyze the interface

temperature changes during the extrusion of a large thick-walled nickel-based superalloy tube [17]. Hu et al. [18] used the ABAQUS software to study the effect of glass lubricant on the stress, strain and burst phenomenon of the workpiece during the hot extrusion of nickel alloys. The ball-to-disk test can accurately control the temperature, load, sliding speed and wear time, so that the evolution law of a single working condition and friction can be obtained [19–21]. Bonnet et al [22]. analyzed the wear mechanism between the billet and the die using the ball-disk test that mainly measures adhesive wear and abrasive wear, and the sliding speed and contact load are the main factors that determine the friction coefficient. Although the ball-disk test is easy to implement, it is quite different from the complex contact formed between the billet and die during nickel alloy extrusion. The ball-to-disk specimen is partially deformed, and single contact is formed at each stage of the ball-disk test. By contrast, extrusion involves the overall deformation of material in the same stage and the contact is complex and diverse. Therefore, the ball-to-disk test can only be used for a qualitative study of the state of the interface during the extrusion process. Since the forming and the structure change are carried out at the same time during the extrusion process, the interface state affects the microstructure to a certain extent. Guo et al. [23] observed the microstructure of the head, center, and tail of the nickel alloy pipe, and found that their structures were inconsistent. This strongly affected the service performance of the pipe. To summarize, investigations of the friction and wear phenomenon, theory, lubrication design, and microstructure evolution between billet and die in the pure nickel extrusion process have been rare, and a complete basic understanding of this system is far from being achieved.

The author has previously conducted some studies on the friction, wear and lubrication in the extrusion process of pure nickel and its alloys. These studies elucidated the influence of the extrusion temperature and speed on the wear macroscopic morphology, friction coefficient and wear rate [24]. In the process of nickel alloy extrusion, when the interface friction is too large, the local grains of the billet will grow quickly, and in severe cases, this growth will directly cause the workpiece to burst [25]. However, the interface wear mechanism during the extrusion process and the influence of friction on the structure of the workpiece still need to be further studied.

Therefore, in this work we study the wear behavior of the billet and the die surface and the evolution of the microstructure of the billet by adjusting the billet-die friction state (lubrication state). In the investigations of the friction between the billet and the die during the extrusion of pure nickel N6, it is important to analyze the surface and internal structure and the surface morphology of the workpiece after extrusion, as well as the die wear mechanism in order to study the plastic deformation and wear characteristics of the billet. We use the field extrusion test and finite element simulation to jointly describe the interface friction behavior during pure nickel extrusion. This specifically obtains the macro and micro morphology characteristics of the wear surface of the workpiece and the die, as well as the microstructure of the workpiece. This work reveals the wear behavior and structure evolution mechanism of the workpiece surface under different interface friction during the extrusion process, and also reveal the failure behavior of the die.

2 Experiment

This experiment uses commercial pure nickel N6, and its chemical composition is shown in Table 1. Pure nickel is processed into a cylinder of $\phi 10.5 \times 20$ mm using a wire cut electric discharge machine. Prior to extrusion, the sample was annealed in vacuum at 700°C for 4 h to eliminate deformed grains and obtain approximately equiaxed grains, as shown in Figure 1(a). The orientation of the recrystallized grains and substructures tends to be liberated during the annealing process, so that the original sample displays a weak texture [26, 27], as shown in Figure 1(b). The extrusion test was carried out with a vertical extruder at 400°C using the forward extrusion method, and the die material was H13 steel. The diameter of the workpiece after extrusion is 7 mm, so that the extrusion ratio and the reduction of area are 2.469 and 59.5% respectively. To study the interface wear during the pure nickel extrusion process, solid powders graphite lubricant was used to improve the friction state.

Table 1
Pure nickel N6 ingredients (wt%)

Ni + Co	Cu	Fe	Mn	C	Si	S	Al	Ti
99.6	0.064	0.11	0.05	0.047	0.018	0.045	0.046	0.018

After extrusion, Zeiss laser confocal microscopy (LSM 800), thermal field scanning electron microscopy (SEM, Quanta FEG-450), and stereo microscopy (SOPTOP) were used to analyze the three-dimensional morphology, micro-morphology, and macro-morphology of the workpiece and die surface, respectively. Electron backscatter diffraction (EBSD, JSM-6700F) was used to observe the texture and microstructure. The dislocation structure was characterized by transmission electron microscopy (TEM, JEM-2010). The EBSD and TEM samples were prepared using the conventional method. First, mechanical grinding was performed. Second, the EBSD and TEM samples were electropolished and double-sprayed in a mixed solution of 90% ethanol and 10% HClO_4 , respectively. The polishing voltage of EBSD sample was 50 V, and the temperature was -20°C , while the voltage of TEM sample was 22 V, and the temperature was 0°C . EBSD data were processed using Channel 5 software. In this work, EBSD was used to count low angle grain boundaries (LAGBs, $3^\circ < \theta < 10^\circ$), medium angle grain boundaries (MAGBs, $10^\circ < \theta < 15^\circ$), and high angle grain boundaries (HAGBs, $\theta > 15^\circ$).

The commercial Deform-3D finite element software was used to analyze the extrusion deformation of pure nickel. The billet type is plastic body, and the die and extrusion cylinder are rigid bodies. The friction coefficient of 0.5 is defined as the non-lubricated extrusion condition, and the friction coefficient of 0.1 is defined as the graphite-lubricated extrusion condition. The shear friction model is used to define the complex interface between the billet and the die, and the heat exchange coefficient between the billet and the die is $11\text{N}/(\text{sec}\cdot\text{mm}\cdot^\circ\text{C})$. Since the extrusion model is symmetrical, to improve the simulation efficiency, this simulation adopts the 1/4 structure.

3 Results And Discussion

3.1 Wear morphology characteristics

3.1.1 Workpiece wear

The surface morphology of the workpiece has the decisive effect on the initiation of surface defects and secondary processing. Figure 2 shows the surface morphology of the pure nickel N6 bar obtained under unlubricated extrusion conditions. The surface of the bar is uneven, giving rise to furrows with different depths along the extrusion direction (Figures 2(a-c)). Observation of the surface and cross-section through SEM reveals that on the one hand, the surface material peels off and causes abrasion, and on the other hand, lamellar cracks are formed on the surface of the substrate and their propagation will eventually cause the loss of the material, as shown in Figure 2(d). Due to the direct contact between the billet and the die in the case of dry friction, the relative movement is complex and changeable. The old asperity sticks and then tears, and the new asperity circulates again. The billet flowing through the die causes fatigue damage to the surface material, leading to the peeling and cracks on the surface of the workpiece. The lost material acting as the third body increases the shear stress and accelerates the wear [28].

Compared to the non-lubricated extrusion, the three-dimensional morphology of the lubricated extrusion bar is greatly improved, the surface is flat, and the roughness is reduced (Figure 3(a)). An examination of the cross-sectional topography, shows that no peeling occurred on the surface, but pits were present. Figures 3(c) and (d) show the surface microscopic features. At low magnification, the surface material partially resembles a river shape (yellow dashed line in the figure) and flows downward along the extrusion direction, and obvious cracks between the flowing material and the substrate are observed. Under high-magnification SEM, small fragments on the surface and irregular pits in the cracks are observed. The river-like surface material is caused by the inconsistency of the friction coefficient between the billet and the die during extrusion, the poor fluidity of the area with a larger friction coefficient, and the large shear force on the surface material that causes the surface of the billet and the substrate to tear and form cracks and pits. Therefore, uneven lubrication is the main cause of billet wear. However, this kind of wear is observed in the relatively shallow range of the billet, so that the degree of wear is small. Generally, the use of lubricants improves the surface quality of the workpiece.

3.1.2. Die wear

The effective understanding and control of die wear in pure nickel extrusion can significantly improve the surface quality and dimensional accuracy of the workpiece, and reduce die wear. Figure 4 shows the surface morphology of the H13 steel die after a single one extrusion of pure nickel N6 under lubrication conditions. The plastic deformation at the exit of the die bearing can be observed by the naked eye. Since different parts of the die have different wear mechanisms during the extrusion process [29, 30], to better study the wear characteristics of the die, different parts of the die were observed. Oxidized particles were found on the surface of die b (Figure 4(b)). The particle material changes the original surface of the die, indicating that oxidative wear and abrasive wear have occurred. This is different from the adhesive wear

of the extrusion cylinder during aluminum alloy extrusion [31, 32]. Figures 4 (c) and (d) show the transition areas between the bearing inlet and outlet, with the transition line indicated by the red arrow. It is found that a large number of particles are present above the bearing, the bearing surface is plastically deformed, and scratches and cracks are present.

The cross-sectional morphology of the die bearing is shown in Figure 5. A gradually decreasing oxide layer on the surface of the bearing from the inlet to the outlet is observed, as marked by the green line. Metallographic analysis was performed and compositional changes were examined, as shown in Figure 6. The oxide layer consists mainly of the oxides of Fe, Cr, and Si, and forms two layers of oxides that are structurally different. Cracks are formed on the surface of the bearing entrance along the extrusion direction, which is consistent with the results presented in Figure 4. A step is formed on the bearing surface. Due to the large deformation resistance of pure nickel, the shearing force of the bearing part is complex and varies during the high temperature and high pressure extrusion process, and fatigue wear occurs. However, plastic deformation occurred at the exit, affecting the dimensional accuracy of the workpiece. It is reported in the literature that the plastic deformation at the exit of the H13 steel die is inevitable, and is related to the extrusion ratio, die cone angle, and extrusion parameters. [33–35].

3.2 Microstructure characteristics

In the absence of compositional changes, the structure of the material is the most important factor for determining its performance and properties. The study of the microstructure evolution during the plastic deformation of pure nickel plays an important role in improving the performance and guiding the extrusion process. The action of graphite lubricant strongly affects the normal and tangential forces, thereby affecting the plastic deformation process and the structure.

Figure 7 shows the inverse pole figure within 700 μm of the workpiece surface under different lubrication conditions. The black grain boundaries in the figure are high-angle grain boundaries ($\text{HAGB} > 15^\circ$), and the color of the grains represents different grain orientations. To elucidate the process of the formation and evolution of the surface structure of the bar, a local statistical analysis was carried out for the areas A, B, and C in the figure. The microstructure of the bar obtained by the lubricant extrusion is uniform, and the large and small crystal grains are randomly and uniformly distributed. In the non-lubricated extrusion, regions A, B, and C have different grain morphologies. The area A with a thickness of approximately 50 μm has small grains, and the grains are significantly refined. Region B is the transition zone that consists of lamellar grains parallel to the surface of the bar and contains large and small grains. In region C, some small grains are gathered among the large grains that are approximately equiaxed.

Figure 8 shows the misorientation angle and grain boundary fraction of the different regions in Figure 7. Under non-lubricated extrusion conditions, the average misorientation angle from A to C gradually decreases (Figure 8(a)). Under the lubricating extrusion conditions, the misorientation angles in different regions remain basically unchanged at approximately 20° (Figure 8(c)). This shows that the non-lubricated extrusion causes the LAGB fraction of the bar to increase from the surface to the inside (Figure 8(b)). The high, medium and low grain boundary fractions of the bars obtained by lubrication extrusion

remain stable (Figure (d)). This is because the work hardening caused by the large deformation of the surface material in the non-lubricated extrusion will produce new LAGBs and MAGBs [36]. The dotted line in the figure is the probability distribution curve of the misorientation of the cubic crystal system metal, that is, the Mackenzie distribution [37, 38]. Since the average misorientation angle is much smaller than the maximum misorientation angle (45°) in the Mackenzie distribution, the structure of pure nickel is clearly refined by extrusion. Pure nickel was also found to be similar to the structure obtained by lubricating extrusion after high pressure torsion at 400°C . This phenomenon is more pronounced for higher strain [39].

Figure 9 shows the orientation distribution function ($\varphi_2=45^\circ$ and $\varphi_2=65^\circ$) of the different areas under different extrusion conditions. It was found that some ideal textures were formed during the extrusion of pure nickel, mainly including S $\{123\}\langle 634 \rangle$, Shear $\{111\}\langle 112 \rangle$, Cu $\{112\}\langle 111 \rangle$, Rotated copper $\{112\}\langle 110 \rangle$, Rotated cube $\{001\}\langle 110 \rangle$ [40, 41]. The above-mentioned textures all appear in the case of non-lubricated extrusion, but Rotated copper does not appear in the lubricated extrusion. Since $\{112\}\langle 110 \rangle$, $\{001\}\langle 110 \rangle$, $\{111\}\langle 112 \rangle$ are shear textures [42], it shows that the use of lubricants can effectively reduce the shear force during the extrusion process and promote the uniform deformation of the billet. The results of the quantitative analysis of texture strength of all of the textures under different extrusion conditions are shown in Figure 10. Under non-lubricated extrusion, the texture gradually weakened from area A to area C. However, under lubricating extrusion, the microtextures are relatively weak and remain basically unchanged in different regions.

The average Schmidt factor (SF) in different regions is shown in Figure 11(a). SF basically remains stable under lubrication conditions. However, the SF of the bar increases gradually from area A to area C under the condition of no lubrication, and is always smaller than the SF of the lubricated-extruded bar. Since SF is the equivalent shear stress of the normal stress on the grain on the slip plan and the slip direction, a larger SF, leads to a weaker orientation and easier movement of dislocations [43]. This shows that the dislocation distribution of the lubricated extruded pure nickel bar is uniform and the plasticity is relatively good. Figure 11(b) shows a partial Kernel Average Misorientation (KAM) diagram. The different colors represent the geometrically necessary dislocations. It is found that a large number of LAGBs appear in large grains. Due to the plastic deformation on both sides of the HAGBs of adjacent grains in the original billet, the grain boundaries are rotated in order to ensure the coordination of grains and the continuity of the grain boundaries. Additionally, subdivision is observed so that the original grain is refined under the action of force. Furthermore, high geometrically necessary dislocations in fine grains and substructures are found. This observations are consistent with the results reported in the previous studies [44].

Due to the spontaneous transition of the material from a high-energy state to a low-energy state, to reduce the stored energy of pure nickel during the extrusion process, the dislocation must move to a new ordered state. Therefore, the existence of dislocations has important implications. Figure 12 shows several dislocation forms in pure nickel extrusion. Dislocations spontaneously rearrange, entangle, and

accumulate to form dislocation cells and dislocation walls, thereby increasing the dislocation density and forming subgrains to reduce the material deformation energy.

3.3 Finite element simulations

Numerical simulation has become an important method for extrusion process optimization. The use of lubricants is related to the stress-strain-temperature relationship during the plastic deformation of the billet [33, 45]. The billet undergoes severe plastic deformation at the exit during extrusion, and the temperature, stress, and strain are the largest. Therefore, the selection of special points is shown in Figure 13(g), and the distance between points P1, P2, and P3 is approximately 0.4mm.

During the lubrication extrusion process, the billet displays a uniform temperature distribution on the same cross-section at a given time. Due to the effects of the heat of plastic deformation and the friction heat of the billet-die interface, the maximum temperature of the billet is 542°C. The material at P1, P2, and P3 heats up slowly when the billet is in the compression stage at the beginning of the extrusion process, and it heats up quickly when it flows through the die bearing. The three-point temperature curves are strongly overlapped (Figure 13(a)). During the non-lubricated extrusion, the temperature at the exit showed a gradient change, the surface temperature was higher than the internal temperature, and the maximum temperature reached 567°C. The curves of points P1, P2, and P3 do not overlap, and the maximum temperature difference between points P1 and P3 is 50°C (Figure 13(b)). This clearly shows that the frictional heat on the surface is present during extrusion in the absence of lubrication. The stress at point P1 under non-lubricated conditions is lower than that at point P3 (Figure 13(d)), while the stress under lubrication remains the same (Figure 13(c)). The strain under non-lubricated extrusion is shown in Figure 13(f). It is found that the surface strain of the blank is greater than the internal strain, and the strain difference between the points is greater than that under lubrication (Figure 13(e)). However, the maximum strain at points P1, P2, and P3 shows opposite trend to that for the maximum stress. The stress and strain in the absence of lubrication are lower than those with lubrication. This is because the billet heats up strongly during the extrusion in the absence of lubrication, which is equivalent to the use of a high extrusion temperature.

The influence of the interface friction is due to the influence of the contact state of the asperities. Therefore, a schematic diagram of the interface contact between the billet and the die under different conditions is shown in Figure 14. In the lubricating extrusion, the graphite powder is located between the asperities, isolating the direct contact of the asperities, and at the same time increases the force-bearing area of the asperities. By contrast, in the absence of lubrication, the asperities are in direct contact, and the total contact area is smaller than that under lubrication, leading to the higher force. At the same time, the contact form of the asperities has different deformation effects on the different depths of the surface during the deformation of the billet. During lubrication and extrusion, the surface of the worn asperities is smooth and has less wear volume, which is beneficial for the flow and deformation of the material during the extrusion process. In the absence of lubrication, the asperities are torn off, the wear volume is large, the surface of the material is rough, and the stress can easily concentrate, so that the force on the surface of the material is complex and uneven [46].

4 Conclusions

In summary, the use of lubricant has a highly important effect on the plastic deformation and surface morphology of the billet during the pure nickel extrusion process, as well as the wear of the die. The difference of the interface friction state is due to the difference in the shear force of the billet-die interface that, affects the friction behavior and wear mechanism of the billet-die interface. In turn, the friction behavior and wear mechanism affects the interface temperature, stress, strain and microstructure of the billet. Therefore, the following main conclusions are drawn:

- (1) The main wear mechanism of the workpiece surface during non-lubricated extrusion is peeling wear and fatigue wear. For lubricating extrusion, uneven lubrication results in the non-synchronization of the material flow velocity on the surface of the billet, but this non-synchronization is only found on the shallow surface.
- (2) It is determined that the oxidative wear of the die surface during the extrusion of pure nickel N6 is present through the entire extrusion process. The extrusion container is mainly abrasive wear, and fatigue wear and severe plastic deformation are found in the bearing part.
- (3) During the non-lubricated extrusion, the billet deforms unevenly, the interface friction heat, stress, and strain show a gradient change, the friction heat is large, and the stress and strain are relatively small.
- (4) The surface microstructure of the lubricated extruded bar (grain morphology, geometric dislocations, uniform distribution of grain boundaries, Schmidt factor) are evenly distributed in the three regions. By contrast the microstructure of the non-lubricated extruded bar exhibits gradient changes.

Declarations

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Authors' contributions

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Competing Interests

The authors report no declarations of interests.

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Figures

Figure 1

Original sample, (a) microstructure (blue is recrystallized grains, yellow is substructure), (b) pole figure

Figure 2

Surface morphology of the workpiece under non-lubricated conditions, (a) three-dimensional morphology, (b) two-dimensional profile, (c) surface SEM image, (d) cross-sectional SEM image

Figure 3

Surface morphology of the workpiece under lubrication conditions, (a) three-dimensional morphology, (b) cross-sectional SEM image, (c) and (d) surface SEM image

Figure 4

Die wear characteristics, (a) macro morphology, (b) (c) (d) is the corresponding SEM image in (a)

Figure 5

SEM image of the die cross section

Figure 6

Metallographic structure of the die oxide layer and the line distribution of the main elements (the yellow arrow shows the line scan)

Figure 7
Microstructure of extrusion, (a) lubricated (b) non-lubricated

Figure 8

Distribution of misorientation angle, (a) no lubrication, (c) lubrication; (b) and (d) are the corresponding high, medium and low angle fraction

Figure 9

Orientation distribution function, (a) lubrication, (b) no lubrication

Figure 10

Maximum strength of all texture components

Figure 11

Schmidt factor histogram (a), KAM diagram in the white rectangle in Figure 7 (b)

Figure 12

TEM images of the dislocations during pure nickel extrusion, (a) dislocation walls and dislocation rearrangement, (b) dislocation cells and dislocation tangle, (c) dislocation pile-up

Figure 13

Color figure of temperature, stress and strain under different extrusion conditions and quantitative analysis of special points, lubrication (a) (c) (e), non-lubrication (b) (d) (f), selection of special points (g)

Figure 14

Schematic diagram of the interface contact zone