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Article

Fabrication of Cu-Doped Diamond-Like Carbon Film for Improving Sealing Performance of Hydraulic Cylinder of Shearer

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Abstract: During shearer operation, piston rod is susceptible to wear from invasion of pollutants, and then ruins sealing ring in hydraulic cylinder. This work attempts to conduct a systematical investigation of Cu-DLC (Cu-doped Diamond-like Carbon) film to improve the seal performance. Failure process of hydraulic cylinder was analyzed, and relevant parameters were determined. Several Cu-DLC films were deposited on the substrate of piston rod in a multi-ion beam assisted system, and their structures and combined tribological performances were investigated. As a result, dust particles attached to the piston rod may enter the cylinder, and damage the sealing ring under the radial force. Cu grains prefer to orientate (111) crystal plane with increase of Cu content, and may induce drop of hardness and internal stress for the film. Friction coefficient of the film ranges from 0.04 to 0.15, and rise of IG/ID ratio from 1.4 to 2.3 in Raman data indicates that bond transition from sp3-C to sp2-C may lower the friction coefficient. Modification mechanism of Cu-DLC film for the seal performance may come from synergistic effects of (i) contact force and friction heat induce the film graphitization, (ii) Cu doping improves toughness of the film and acts as a solid lubricant, and (iii) transfer layer plays a role of self-lubrication.

Keywords: diamond-like carbon film; Cu doping; sealing performance; hydraulic cylinder; Shearer

1. Introduction

Coveted clean power such as photovoltaic and wind power remains up in the air, and the shortage of electricity in the hot summer has prompted attention to the reliable mining of coal for thermal power [1]. Hydraulic cylinder is a critical height control device, and whose sealing performance directly related to operational stability and efficiency of shearer [2]. Invasion of extrinsic pollutants during shearer operation tend to induce wear of vulnerable friction pair of hydraulic cylinder, and the induced seal failure may endanger shearer operation and operator safety [3]. Film modification for the vulnerable component is a preferred approach to improve the seal performance [4]. Diamond-like carbon (DLC) films of carbide metals like W or Cr doped are commonly used, but subject to early failure due to poor toughness [5,6]. None carbide metals like Cu hold promise for replacing their carbide counterparts and avoiding early failure. Service conditions of shearer are rather complex and rigid, and related interactions of working loads and media like grave are involved [7]. Consequently, systematic investigation is rare to see in literature to date for using Cu doped DLC (Cu-DLC) to improve seal performance of hydraulic cylinder for reliable shearer operation.

In view of the seal issue under service conditions, such systematic investigation is expected to have three progressive links of (1) failure analysis and film solution; determine component to be worn, and clarify performance requirements of the film under service conditions. (2) film fabrication and verification; fabrication of Cu-DLC film and verification of the film requirements. (3) film modification mechanism, reveal modification mechanism of the film applicable for hydraulic cylinder of the shearer.

This work endeavors to fabricate Cu-DLC film applicable for improving sealing performance of hydraulic cylinder of shearer under service conditions. Failure of hydraulic cylinder is analyzed, and performance requirements are proposed for the film on vulnerable component. Cu-DLC film with different Cu contents are deposited on the substrate of piston rod in multi-ion-beam assisted system, structure of the film is characterized, and the combined tribological performances are evaluated. This allow us to propose modification mechanism how Cu-DLC film overcome the seal issue.

2. Performance Requirements and Preparation of thin Films

2.1. Performance Requirements

2.1.1. Analysis of the Structure and Working Principle of the Hydraulic Cylinder

Figure 1 shows structure diagram of hydraulic cylinder [8]. The cylinder takes on important role on raising and lowering cutting transmission part of shearer drum in fore and aft motion. As shown in Figure 1, the cylinder is composed of guide ring 1, seal ring 2, piston rod 3, rod chamber 4, cylinder block 5, piston 6 and head port 7. Piston 6 is driven by hydraulic oil, piston rod 3 and sealing ring 2 rub against each other at the sealing component. The cylinder withstands load pressure by pushing flow of hydraulic oil, flow speed and flow rate of the oil determine the reciprocating speed and frequency of piston rod 3 and the cylinder block 5. In case of drum raising, high-pressure hydraulic oil in head port 7 pushes the piston rod out. In case of drum lowering, high-pressure hydraulic oil in rod chamber 4 pushes the piston rod back.

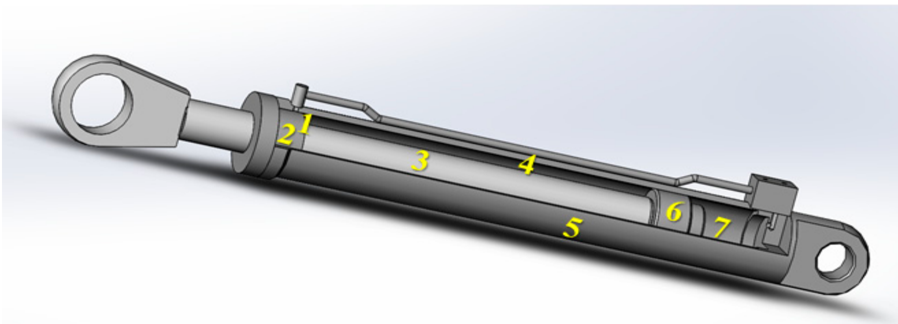

Figure 1. Structure diagram of hydraulic cylinder of shearer.(1) guide ring; (2) seal ring; (3) piston rod; (4) rod chamber; (5) cylinder block; (6) piston; (7) non-rod chamber.

2.1.2. Cylinder Failure Analysis

Process of sealing failure of hydraulic cylinder [9,10] can be divided into three stages during shearer operation. Stage I: Sealing ring is an important component to prevent extrinsic pollutants from invasion by means of its elastic deformation. Retractable deck can prevents large-size pollutants from entering the cylinder, but large-size pollutants may enter into the cylinder or mix into hydraulic oil with movement of piston rod, and stay in gaps between piston rod and sealing ring, resulting in hidden trouble for the seal performance. Stage II: Sealing ring expands when hydraulic oil enters the cylinder, sealing area at both ends of piston increases and lead to friction force increase. Solid pollutants have higher hardness than rubber of sealing ring, and are likely to embed in the cylinder under high internal pressure, resulting in wear. Stage III: The pollutants embedded on sealing ring act as abrasives, repeatedly rub piston rod during reciprocating movement of piston rod. This induces wear scar formed on piston rod. The abrasion aggravates over time and result in the seal failure. In this way, the oil leakage occurs in the cylinder, and eventually lead to failure of the cylinder as well as shearer accident.

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2.1.3. Film Performance Parameters

Accordingly, fabrication of Cu-DLC film on the piston substrate is proposed to improve the seal performance. The film should have good combined tribological performance, i.e., high hardness, low internal stress, and low friction coefficient. The high hardness, over 7 GPa of solid pollutants, is important to avoid wear scar formed on the piston. The low internal stress may enable the film to have good toughness so as to resist early failure of the film. The low friction coefficient, less than 0.2, may enable the film to have lubrication action so as to resist adhesion of the pollutants on the piston.

2.2. Film Preparation

The substrate was two wafers, silicon was used for structural and mechanical analyses, and 40CrNiMoA (raw material of piston rod) was used for tribological analysis. The substrate was cleaned with acetone in an ultrasonic bath for 20 min. After drying with clean nitrogen, the substrate was placed in vacuum chamber ready for deposition.

Cu-DLC film [11]was deposited on the substrate in multi-ion beam assisted system. The base vacuum was set to 2.0×10^{-4} Pa, and the deposition pressure was fixed at 2.5×10^{-2} Pa. The substrate was initially bombarded with an Ar^+ beam of 10 kV/20 mA (ion source voltage/ion beam current) for 10 min to obtain a clean surface. Subsequently, a 0.2 μm-thick Cu interlayer was deposited by singular sputtering of Cu target at 1100 eV/40 mA. A 0.8 μm-thick layer (a:C-Cu_x) was then synthesized by simultaneous co-sputtering of Cu and C targets. The co-sputtering was performed by: (i) Cu sputtering current was respectively set to 0, 18, 35, 50, 70 and 90 mA, while maintaining the energy constant at 900 eV; and (ii) constant C sputtering current and energy of 50 mA and 1200 eV, respectively. Six samples of Cu-DLC film (approximately 1 μm-thick) were prepared and denoted as A_0–A_5 in Table 1.

Table 1. Atomic percentage content of copper in six samples of a: C-Cu x%.

Sample	Film	Cu sputtering current (mA)	Cu (at%)
A_0	a:C-Cu0%	0	0
A_1	a:C-Cu6.8%	18	6.8
A_2	a:C-Cu9.2%	35	9.2
A_3	a:C-Cu15.4%	50	15.4
A_4	a:C-Cu23.7%	70	23.7
A_5	a:C-Cu32.6%	90	32.6

2.3. Characterization

As for structure of the film, elemental composition was detected using energy dispersive spectroscope (EDS, JSM 6301F, JEOL, Tokyo, Japan), phase structure [12] was evaluated using X-ray diffraction (XRD, Model XD-3, Rigaku, Tokyo, Japan), and bonding structure was analyzed using Raman spectroscopy (LabRAM HR Evolution, HORIBA, Kyoto, Japan). As for mechanical performance of the film, hardness was measured in nanoinductor (MTSXP, MTS, Minnesota, USA) under a load of 2 mN, in a continuous stiffness measurement test pattern; intrinsic stress was calculated using the Stoney formula. The tribological performance of the films was evaluated via reciprocating friction on a reciprocating friction and wear tester (MFT 4000, Huahui, Lanzhou, China) under condition of (i) the coated sample was fixed, and steel ball with diameter of 6mm was used as counterpart; (ii) subsequently conducted a reciprocating motion on the sample surface at a load of 5 N; (iii) the wear morphology was observed on an optical microscope (OM, BX51M, OLYMPUS, Tokyo, Japan) provided with an attached digital camera. To ensure reliability, the obtained values for each sample were the average of six measurements, and the accuracy was within a ± 5% error range.

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3. Results and Discussion

3.1. Structures and Mechanical Properties of Cu-DLC Film

3.1.1. Structures of Cu-DLC Film

Figure 2 shows XRD spectra of Cu-DLC film with six Cu contents. Without Cu doping, only amorphous carbon peak can be seen for the pure DLC film (a:C - Cu 0%). With Cu doping, Cu-DLC film presents two characteristic peaks of Cu, crystallographic peak of Cu (111) around $2\theta = 43.3°$ and Cu (200) near $2\theta = 50.4°$. Intensity of Cu diffraction peaks increases with increase of Cu doping content. Intensity of Cu (111) peak is higher than that of Cu (200), and suggests that dope Cu grains may have preferred orientation at tend to crystallographic plane (111) [13]. On the other hand, diffraction peak of Cu (111) is widened with decrease of Cu content, and this may be involved with existence of Cu grains with small size [14]. In this way, microstructure of Cu-DLC consists a mixture of amorphous carbon and crystalline Cu. Size of Cu crystals may be varied with doping content of Cu crystals, and may affect the film performances in turn.

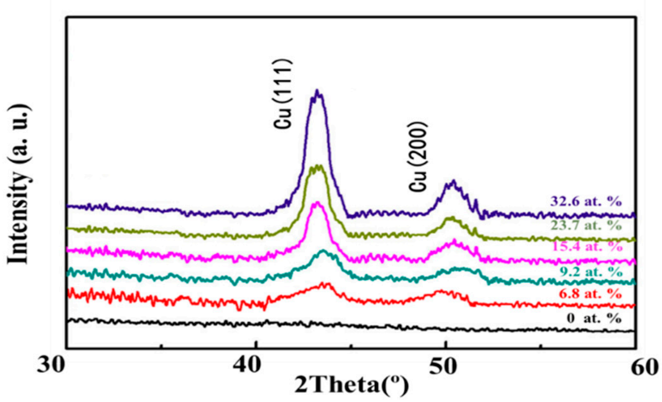

Figure 2. XRD spectra of Cu-DLC film with six Cu contents.

3.1.2. Hardness of Cu-DLC Film

Figure 3 shows hardness values of Cu-DLC film with six Cu contents. As shown in Figure 3, the hardness value is in a range of 14.8-27.6 GPa, and drops with rise of doping content of Cu. The film hardness is higher than maximum working stress (10 GPa) of the cylinder, and is much higher than that of the piston substrate (7 GPa). Such film may enable piston thereon to have a superior wear resistance against the pollutants.

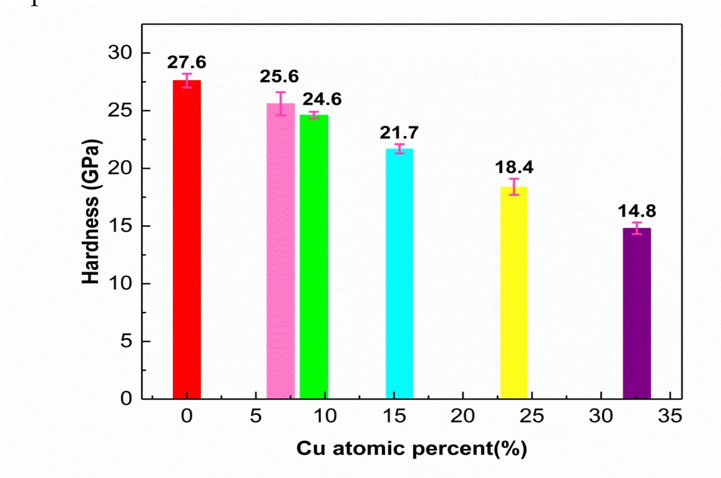

Figure 3. Hardness of Cu-DLC film with six Cu contents.

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Hardness of DLC film is 27.6 GPa for pure DLC film without doping. Hardness of Cu-DLC film drops to 24.6 GPa when Cu content is 9.2%, but the hardness drop is not significant. Hardness of Cu-DLC film continues to drop to its minimum of 14. 8 GPa when doped Cu [15] content reaches its maximum content of 32. 6%. This means that adjusting Cu content can change the film hardness.

Figure 4 shows variation curve of internal stress for Cu-DLC film. Internal stress of Cu-DLC film is lower than that of pure DLC film without Cu doping, and indicates that the internal stress can be varied with Cu doping. When the Cu content increases from 0% to 9.2%, the internal stress decreases rapidly from 3500 MPa to 1750 MPa. When Cu content continues to increase, the internal stress decreases slowly. When the content of Cu is 32.6%, the internal stress decreases to minimum of 1250 MPa. The reduction of the internal stress may avoid the film peeling during the movement of the components [16,17].

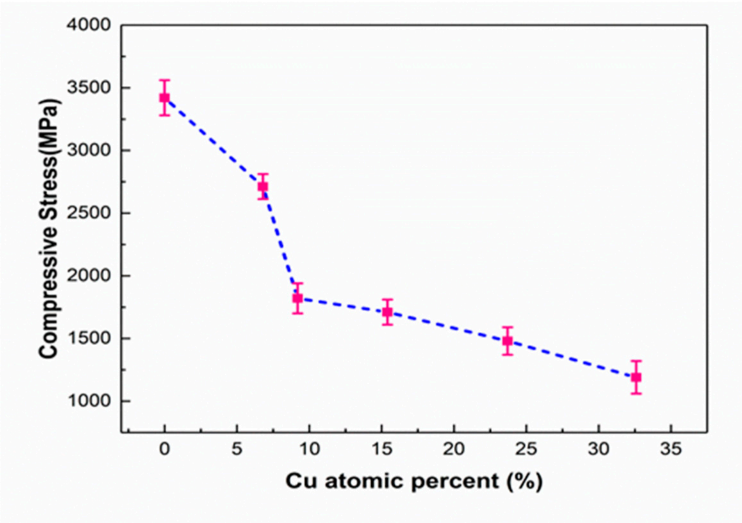

Figure 4. Variation of internal stress of Cu-DLC film with six Cu contents.

The mechanical results exhibit that, Cu-DLC film meet the hardness requirement of actual working conditions, and an appropriate doping content may get the film have both high hardness and low internal stress.

3.2. Tribological Performance of Cu-DLC film

3.2.1. Friction Coefficient and Wear Rate

Invasion of extrinsic pollutants induce deterioration of seal performance of the cylinder. Typically, the performance deterioration is involved with seal ring and piston rod. During the reciprocating motion, pollutants adhered on piston rod can move to seal ring, and abrasive wear between seal ring and piston rod may lead to the seal failure.

Figure 5 show steady-state coefficient of friction (COF) of Cu-DLC film after reciprocating sliding for 45 min. COF of Cu-DLC film fluctuates between 0.04 and 0.15 with different Cu contents, and is lower than that of undoped DLC (0.18). Among them, COF of Cu-DLC film with Cu content of 9.2 % (A2) is the lowest, as low as 0.04. In this experiment, all friction coefficients of Cu-DLC film is much lower than 0.2, indicating that the film can play a good lubricating role.

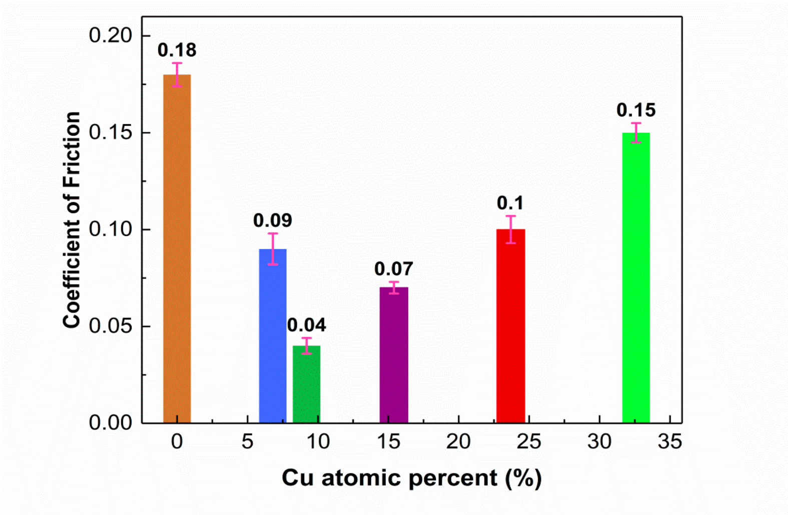

Figure 5. Friction coefficient of DLC Film with six Cu contents.

Figure 6 show curve of wear rate for Cu-DLC film. With increase of Cu content, the wear rate decreases first and then increases, and reaches the lowest ($5.0 \times 10^{-9} mm^3 /N \cdot m$) at 9.2% content. As a soft and ductile metal, Cu grains embedded in amorphous carbon network of DLC can improve brittleness of DLC film. In this way, doping Cu may improve wear resistance of DLC film in a better toughness, and reduce stress concentration and early crack initiation as well.

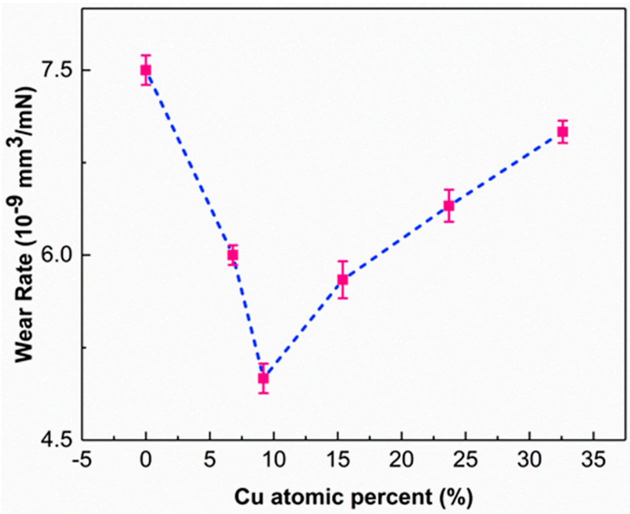

Figure 6. Wear rate of DLC film with different Cu content.

Figure 7 shows optical microscope images of wear scars for a:C-$Cu_{9.2\%}$ film at different sliding times. It can be seen from comparison between Figure 7a,b that the wear marks and debris of a: C-$Cu_{9.2\%}$ film become more with increase of sliding time. On the edge of the wear marks of the ball and disk, the debris accumulation can be seen, accompanied by the formation of the transfer layer. Figure 7c shows that the wear of the film increases and traces of furrows become visible. Abrasive particle accumulation can be observed at the edge, the film subject to damage and the transfer layer becomes obvious. Figure 7d is wear morphology of the counterpart in Figure 7c. The wear rate of counterpart is higher than that of Cu-DLC film in that its hardness is lower than that of the film.

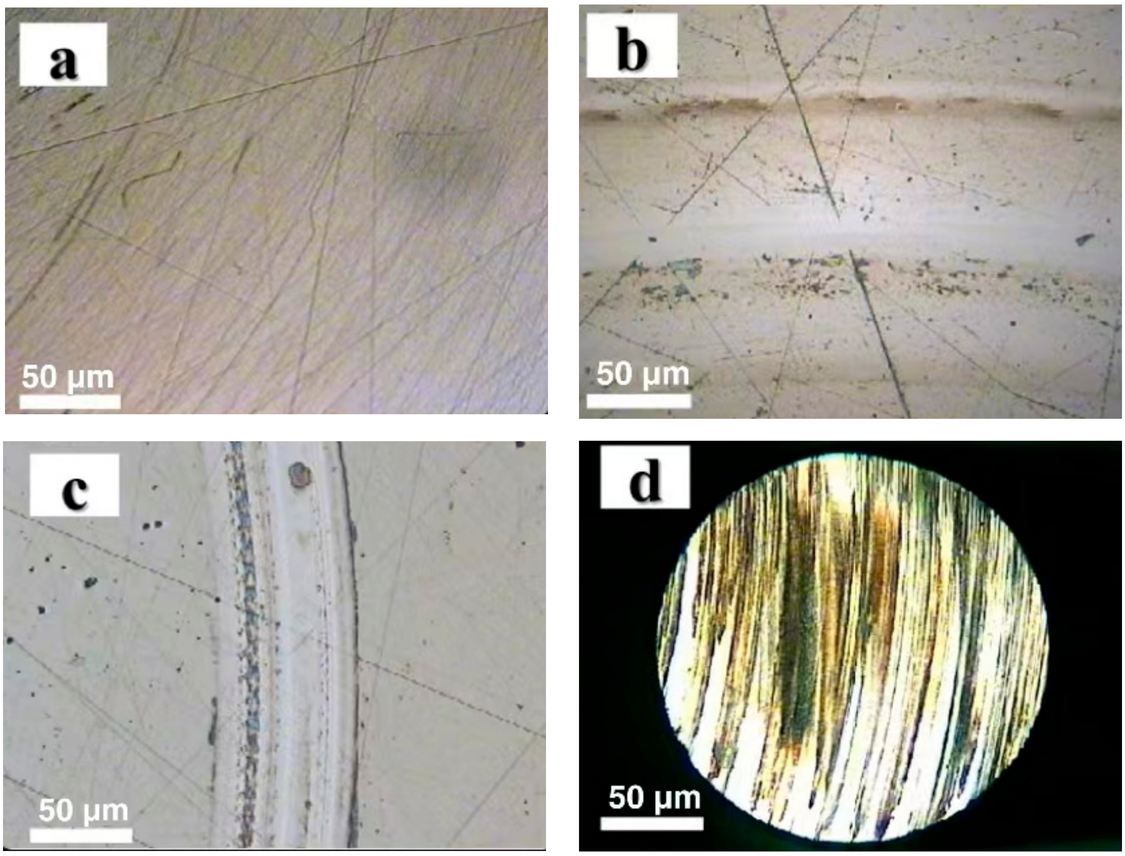

Figure 7. Photographs of wear scar morphology of a:C-$Cu_{9.2\%}$ film during friction test: (**a**) 10 min/film; (**b**) 20 min/film; (**c**) 50 min/film; (**d**) 50 min/steel ball.

EDS analyses of the wear debris of the friction pair indicate occurrence of Fe, Cr, C and Cu elements. Iron and chromium elements come from the steel substrate, and carbon and copper elements come from Cu-DLC film. This implies that the transfer layer is formed from the friction process and is the main reason for decreasing friction coefficient of the film. Among them, a : C-$Cu_{9.2\%}$ film has optimal tribological performance.

3.2.2. Raman Analysis of Cu-DLC Film

Figure 8 shows the Raman spectra of a:C-Cu9.2% film. From top to bottom, they are the Raman spectra for the deposited film (no wear), the wear mark on the surface of the worn film under 5 N load, and the wear debris. There are two obvious characteristic peaks in the Raman spectra of a: C-Cu9.2%: D-peak and G-peak, whose peaks correspond to ~ 1380 cm^{-1} and ~1550 cm^{-1}, respectively. The shift of G-peak to higher wave number and D-peak to lower wave number mean increase of sp^2-C content and decrease of sp^3-C bond content [18]. The reason for this phenomenon is that the heat generated during the reciprocating friction between the film and the counterpart increases the temperature of the contact area [19,20]. After friction and wear test, IG/ID increased from 1.4 (non-wear film) to 1.9 (wear mark) and 2.3 (wear debris). On the one hand, this reflects the change of distribution and content of sp^2-C and sp^3-C bonds. During the experiment, part of the sp^3-C bond in Cu-DLC film is transformed into sp^2-C bond under the action of friction heat, and then sp^2-C bond moves to the film surface under action of contact force. The carbon density decreases, and further leads to the transformation of sp^3-C bond to sp^2-C bond. On the other hand, Cu particles changes the angle of C-C covalent bond and the distribution of carbon atoms [21]. In the reciprocating motion, the Cu formed by non-carbides underwent plastic deformation and diffused to the high temperature region, which are uniformly distributed in the film. Such Cu can fill more d orbitals and convert the sp^3-C bond into a sp^2-C hybrid bond [22].

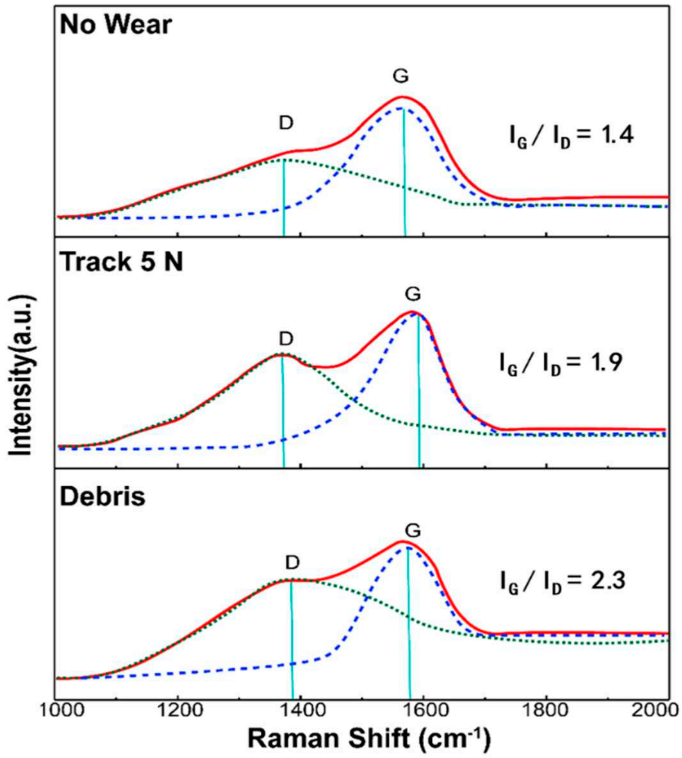

Figure 8. Raman spectra of a:C-$Cu_{9.2\%}$ film.

As a result, doping appropriate Cu content into DLC film can improve the toughness of DLC film and effectively improve the tribological performances of piston rod. In this experiment, a:C-$Cu_{9.2\%}$ film can significantly improve the tribological performances of piston rod. It thus provides the basis for revealing improving mechanism of Cu-DLC film for sealing performance of hydraulic cylinder.

3.3. Improvement Mechanism of Cu-DLC Film for the Seal Performance

Figure 9 shows the mechanism of Cu-DLC film [23] improving sealing performance of hydraulic cylinder. Figure 9a shows the front view of the friction pair between the piston rod and solid particles (black blocks) embedded in the sealing ring. Figure 9b shows the side view of the friction pair shown in Figure 9a. Up and down double arrows indicate reciprocating motion. Figure 9c shows the microscopic diagram of the contact interface of the friction pair, from top to bottom, the AISI 440 substrate, transfer layer and solid particles, graphite layer (black ellipse), and Cu-DLC film layer. Figure 9d further illustrates the schema of the transfer layer shown in Figure 9c, showing the composition of the transfer layer from top to bottom, including the transfer layer of copper content (copper-colored balls), sp^3-C (golden balls), sp^2-C (blue balls), Chromium atoms (green balls) and iron atoms (gray balls).

In view of experimental data above, three factors affecting the sealing performance of Cu-DLC film are, (1) appearance of graphitization; (2) doping of Cu; (3) generation of transfer layer. This allows us to suppose that mechanism of improving the sealing performance using Cu-DLC film may be synergistic effect of three mechanisms below.

(1) Graphitization of film surface induced by contact force and friction heat.

As shown in Figure 9c, accumulation of friction heat may lead to local high temperatures in local micro-contact areas. Under the dynamic action, the metastable sp^3-C bond transforms into the sp^2-C bond with high thermal stability at high temperature, which is consistent with the Raman results shown in Figure 8. Under contact force and thermal diffusion, the resulting sp^2-C diffuses onto the surface of Cu-DLC film and aggregates to form the top graphitization layer (black ellipse) on the film. From Figure 9c, it is seen that sp^2-C aggregates in the contact area to form the top graphitization layer.

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Under contact force and thermal diffusion, the resulting *sp*²-C diffuses onto the surface of Cu-DLC film and aggregates to form the top graphitization layer (black ellipse) on the film. The top graphitization layer can play a role of solid lubrication for the friction pair of piston rod-seal ring and reduce friction coefficient.

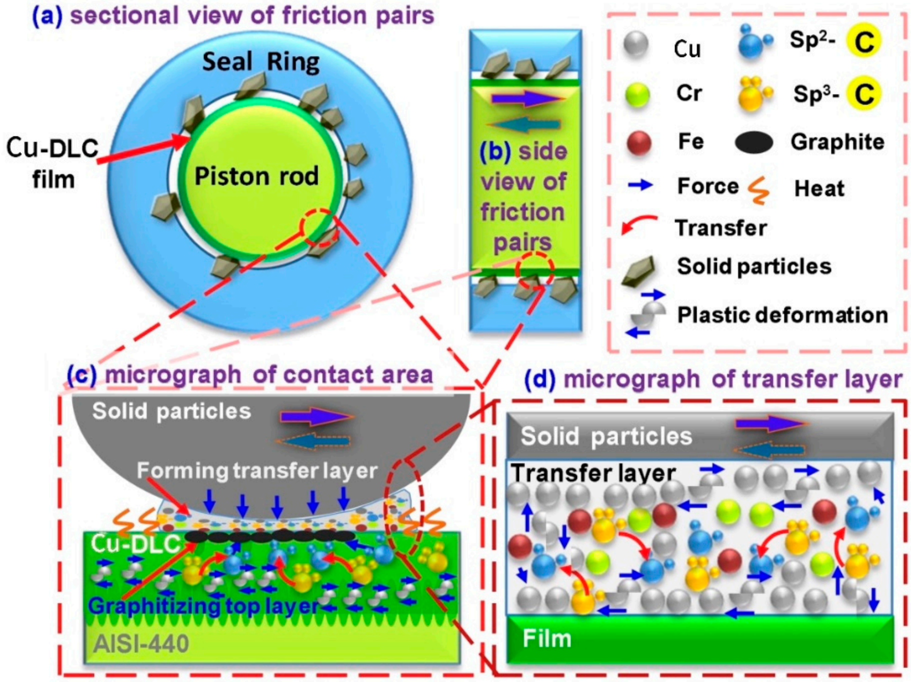

Figure 9. Mechanism of Cu-DLC film improving the sealing performance of the hydraulic cylinder (**a**) sectional view of friction pairs, left top; (**b**) side view of friction pairs, right top; (**c**) micrograph of micrograph of contact area, left bottom; (**d**) micrograph of transfer layer, right bottom.

(2) Cu doping improves the toughness of the film and acts as a solid lubricant.

From Figures 9c,d, it can be seen that the action of internal stress resulted in the plastic deformation of Cu in the film, and the strain energy was released. This can avoid the initiation and propagation of early microcracks, and the toughness of the film is improved. Under the joint action of contact force and friction heat, plastic deformation occurs in Cu and some Cu grains diffuse and aggregate to the surface of the film. This part of Cu grains with low shear stress act as a solid lubricant, bearing the deformation energy in the contact zone and reducing the friction coefficient, so that the sealing performance of Cu-DLC film can be improved.

(3) Self-lubrication and long-term protective effect of transfer layer

According to Figure 9c, the transfer layer is formed between the film and solid content under action of contact force and friction heat. This layer can avoid direct contact between two friction surfaces, which has a self-lubricating and long-term protection effect. From Figure 8, it can be seen that due to the accumulation of friction heat, the *sp*³-C bond is transformed into *sp*²-C bond, which diffuses on the surface of the film and forms graphitization. Graphite is produced and accumulated between two contact surfaces and forms a transfer layer with Cu, Fe and Cr. The formation of the transfer layer prevents direct contact between solid content and the film. When the force is applied, solid particles are squeezed into the transfer layer. Local stress concentration causes the plastic deformation of Cu, and local strain energy can be released. So self-lubricating performance of the transfer layer makes the coated piston rod possess a significantly lower wear rate ($5\times10^{-9}mm^3/Nm$, Figure 6) and a predictable long service time.

In summary, the transfer layer, soft Cu and the graphitized top layer play the role of solid lubricant for CU-DLC film, improve the wear resistance of the hydraulic cylinder piston rod and the sealing performance of the sealing ring, thus extending the service life of the friction ring.

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4. Conclusions

In this paper, the sealing failure caused by the wear of the piston rod of the hydraulic cylinder was studied, and the Cu-DLC film was used to improve the sealing performance of the cylinder. Taking the piston rod of the hydraulic cylinder as the research object, the coating sample was prepared according to the working conditions. The structure, hardness and internal stress of Cu-DLC film with different Cu content were analyzed. The friction coefficient, wear rate and tribological performances of different Cu content were analyzed, and the optimum Cu content film was determined. The mechanism of improving the sealing performance of the hydraulic cylinder by Cu-DLC film was revealed to improve the performance of the hydraulic cylinder. Main conclusions are as follows:

(1) The dust particles attached to the piston rod entered the cylinder, and the piston rod tilted to wear the sealing ring under the radial force, which results in the failure of piston rod. The particle hardness is about 7 GPa, and the highest contact force of piston rod is 10 GPa. The film hardness, friction coefficient and wear rate are relevant to the seal performance of piston rod. DLC film with different Cu contents were deposited on 40CrNiMoA by multi-ion beam assisted system.

(2) Intensity of characteristic peaks of Cu(111) of Cu-DLC film is higher than that of Cu (200) with the increase of Cu content, and the Cu grains tend to (111) crystal plane. The hardness of the films ranged from 14.8 to 27.6 GPa, and the hardness decreased with the increase of copper content. The internal stress of the film was reduced from 3500 MPa to 1750 MPa, to avoid the spalling of the film between the moving parts of the mechanism, and the film can effectively protect the wear of the substrate.

(3) The results of friction and wear experiments showed that the friction coefficient fluctuated between 0.04 and 0.15, and the friction coefficient (0.04) and wear rate of a:C-Cu9.2% film are the lowest (5.0×10^{-9} mm^3/N m). The increase of IG / ID ratio from 1.4 to 2.3 by Raman analysis reveals that the transition of sp^3-C to sp^2-C bond, reduces the friction coefficient and effectively improves the tribological performance of piston rod.

(4) The mechanism of improving the seal performance by Cu-DLC film was revealed (i) The metastable sp^3-C bond transforms the sp^2-C bond at high temperature and accumulates at the top layer to form a graphitized top layer. (ii) When the film was in contact with the particles, the local stress concentration was formed, and the plastic deformation of Cu occurred, which acted as a solid lubricant and reduced the friction coefficient. (iii) The graphitized top layer forms a transfer layer containing Cu, Fe and Cr on the contact surface, which plays a self-lubricating and long-term protective effect.

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