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Study on Low-Temperature Deposition of Diamond-like Carbon Film on the Surface of Bionic Joint Thread and Its Properties

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Abstract: The double-connection structure of bionic joints of mining drill pipes has solved the problem of drill drop caused by fatigue cracks. However, with low-melting-point elastic–ductile alloy filling in the bionic joint, the thread on the joint cannot be hardened by high-temperature surface hardening treatments such as quenching and nitriding, making it prone to thread gluing or excessive wear. In this paper, the feasibility of diamond-like film deposition on the surface of a bionic drill pipe thread was studied. A tungsten transition film was used to improve the thickness of the film and the interfacial bond strength between the film and the substrate. The test results show that the total thickness of the DLC film is about 3~5 μm, the roughness is less than 2 μm, the hardness of the film reaches 24.4 GPa, the friction coefficient is 0.04, and the critical load is 56 N. SEM and EDS analyses show that the tungsten film and the bionic joint thread form a metallurgical structure. The morphology of the diamond-like carbon film is uniform and dense, and there is no obvious stratification between the substrate material. The joint with a diamond-like coating treatment has a longer service life than joints receiving conventional high-temperature nitriding treatment.

Keywords: bionic joint; thread gluing; low-temperature deposition process; diamond-like carbon film

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1. Introduction

The hidden dangers of gas and water inrush directly affect the safe and efficient operation of coal mines, and drilling is the main technical means for gas and water disaster control. Limited by the size of the roadway, a single drill pipe is short, its number of connections is large, the rigidity of the drill pipe string is strong, and coal mines mainly require near-horizontal drilling, which can only rely on the feed force of the drilling rig for drilling, so the drill pipe string is seriously bent in the hole and the root of the external thread is easily broken.

Based on the theory of engineering bionics, bionic joints (Figure 1) and bionic double-connection technology (Figure 2) were originally created, which significantly reduced the risk of fracture. Even if the thread of the male joint is broken under bad working conditions, the drill pipe can still maintain the axial connection between drill pipes, and the effect of "breaking bones and connecting ribs" is realized. The drill tool is taken out of the hole at the breakpoint depth, so as to avoid drilling scrap and huge economic losses [1,2].

The developed Φ73 mm bionic double-connected drill pipes were tested in a mine operated by the Xishan Coal and Electricity Company. A total of five fracture accidents occurred, all of which were located at the root of the thread of the male joint. Four of them successfully put forward all the drill pipes to the depth of the breakpoint, and the other one was not put forward due to the serious buried drilling in the hole, which achieved the expected effect [3,4]. At present, the bionic double-connected drill pipes have been applied in many mining areas.

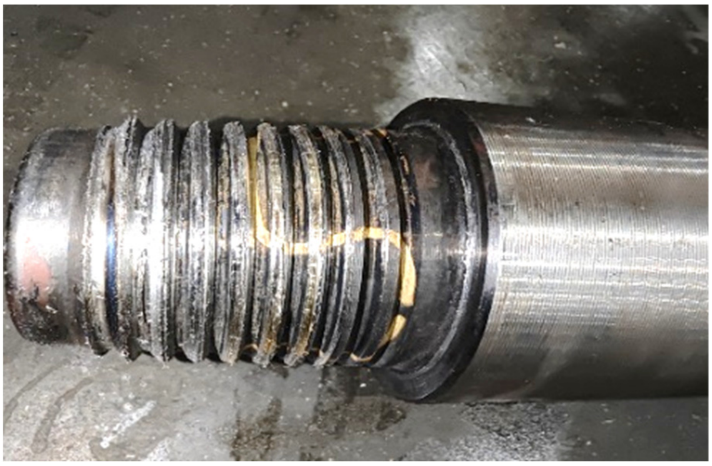

Figure 1. Bionic joint after wear.

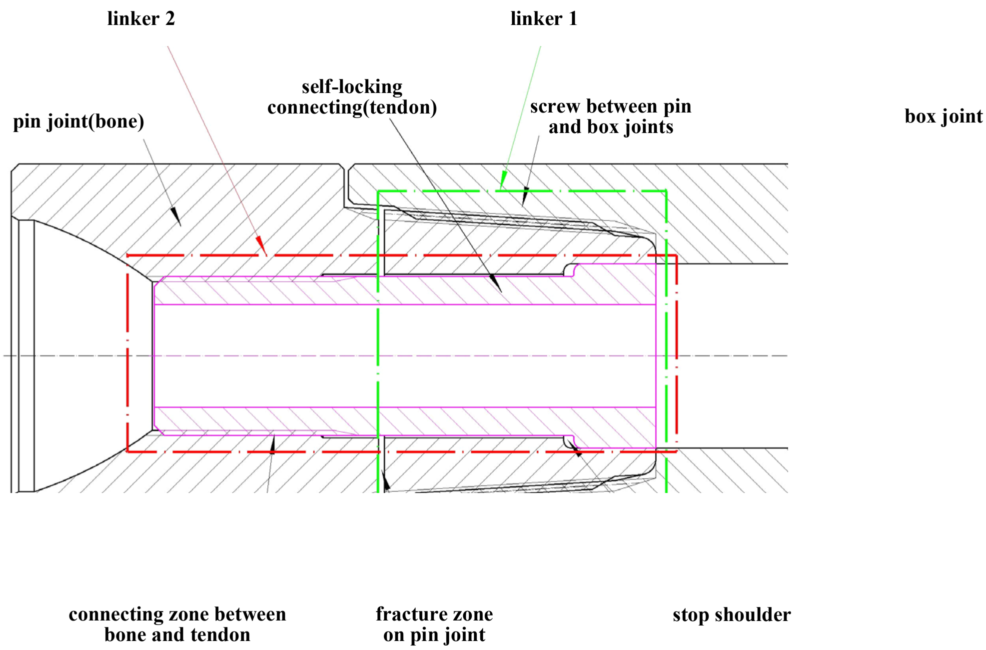

Figure 2. Bionic double-connection structure.

The bionic double-connected drill pipe solves the problem of drilling loss caused by joint fracture. However, due to the low melting point of the elastic–ductile alloy that fills in the bionic drill pipe joint, the thread cannot be subjected to quenching, nitriding, or other high-temperature surface-hardening treatments. The thread wears too fast, and even the phenomenon of sticking occurs, resulting in a short service life and high overall cost [5–8]. The main reasons for excessive thread wear and gluing are as follows [9–11]: (1) In order to maintain the high toughness of the drill pipe joint, the quenching and tempering hardness is generally maintained between 30 and 34 HRC, and if the quenching and tempering hardness is too high, the fracture risk is obviously increased. (2) Due to the harsh down hole conditions, the drill pipe is screwed and unloaded by the drilling rig, and the drill chuck, gripper, and drilling hole have poor coaxiality, resulting in eccentric wear during screwing and unscrewing. (3) With the increase in hole depth, drill pipe bending intensifies, and the hole wall friction increases, resulting in the drilling rig experiencing an overload phenomenon, the torque becoming too large, etc. It can be seen that the key to improving the service life of the bionic double-connection drill pipe is to improve the hardness and wear resistance of the thread surface on the premise of maintaining the comprehensive performance of the bionic joint.

To solve the problem of drill pipe thread failure, scholars at home and abroad have put forward many solutions, such as surface hardening, gas nitriding, soft nitriding, and other technologies, and achieved good results. The above technology cannot be introduced into the bionic double-connection drill pipe thread treatment, however, because the heating temperature of the bionic joint filled with elastoductile alloy cannot exceed 400 °C; otherwise, the elastoductile alloy will undergo secondary softening, seriously affecting the comprehensive performance of the bionic joint.

2. Diamond-like Carbon Films and Preparation Technology

Diamond-like carbon films are metastable amorphous materials containing diamond structures (sp3 bond) and graphite structures (sp2 bond). Carbon atoms are mainly combined with sp3 and sp2 hybrid bonds, which have the excellent properties of both diamond and graphite [12]. Because of its high hardness, high elastic modulus, low friction coefficient, corrosion resistance, wear resistance, and good tribological properties, diamond-like carbon films are a new type of thin film material with very good application prospects [13]. The United States has made diamond-like carbon film material one of their national strategic materials in the 21st century.

Vapor deposition is widely used in the preparation of diamond-like carbon films. The most commonly accepted deposition theory is the sub-surface implantation model. This theory holds that the essence of the preparation process of diamond-like carbon films is that C+ is implanted into the sub-surface and grows in the film [14,15]. The vapor deposition process can be divided into three categories: physical vapor deposition, chemical vapor deposition, and liquid-phase deposition.

Physical vapor deposition technology refers to the deposition of diamond-like carbon films on the surface of materials by physical means under vacuum conditions. The main preparation processes include ion beam deposition, sputtering deposition, vacuum cathode arc deposition, and pulse laser deposition. Among them, sputtering deposition has a wide range of applications [16–18]. Chemical vapor deposition is used to prepare diamond-like carbon films via the chemical reaction of gaseous compounds or elements on the substrate. The main preparation processes include hot filament chemical vapor deposition and direct photochemical vapor deposition. Among them, plasma-enhanced chemical vapor deposition is widely used [19,20]. The liquid-phase method was discovered by Namba in 1922. Diamond-like carbon films prepared by liquid-phase deposition technology make up for the shortcomings of the vapor deposition method to a certain extent, and the physical properties of the film are more stable. The main processes include electrochemical deposition and polymer pyrolysis. There is no significant temperature difference before and after deposition. At the same time, liquid-phase deposition technology is easier to industrialize, but the deposition principle of this method is not clear [21,22].

3. Test Materials and Methods

3.1. Laboratory Equipment

A vacuum arc ion multi-functional coating system is used for coating, comprising a vacuum system, power supply system, gas control and mechanical transmission system, vacuum chamber heating and temperature measurement system, cooling system, and so on. The working principle of the device is shown in Figure 3. In order to ensure that the thread end face can also achieve uniform coating, the male and female joints are suspended in the sample chamber when placing the sample, so as to ensure that all the end faces of the thread do not make contact with the base of the sample chamber.

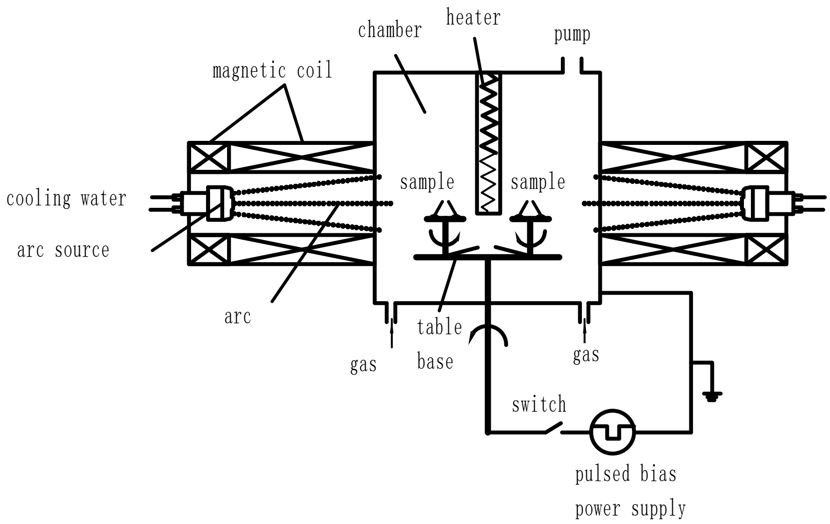

Figure 3. Principle of vacuum arc ion plating.

3.2. Thread Structure Design and Preparation

The Φ89 mm bionic drill pipe joint was used in the test, and the material was 42CrMoA. After quenching and tempering, the internal and external threads were processed by CNC machine tools. The thread tooth type was a trapezoidal structure, and then the diamond-like film was deposited on the surface of the internal and external threads at low temperature to improve the wear resistance life of the thread. The male and female joints and thread structure are shown in Figure 4.

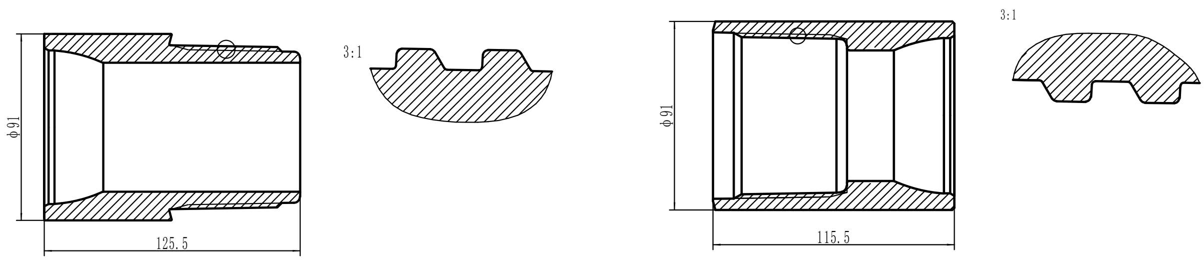

Figure 4. The Φ89 mm joint structure.

3.3. Test Materials and Process

In the experiment, methane and argon were used as gas sources to prepare diamond-like carbon films on the surface of bionic joint threads. Considering that the pollution of the substrate surface will reduce the adhesion of the film and lead to a decrease in the quality of the film, the joint is cleaned before the film is prepared, and the joint is ultrasonically cleaned with anhydrous alcohol for 20 min to remove the dirt, oxidation, and roughness of the surface and improve the adhesion of the coating.

Secondly, the surface of the substrate was activated by plasma technology to increase the surface energy and improve the adhesion of the coating. Then, it is suspended in the vacuum chamber for deposition. Before preparation, the vacuum chamber is pumped to 3×10^{-3} Pa, and argon is introduced. The internal and external threads of the joint are cleaned by plasma for 10 min to remove the pollutants and oxides on the surface. Then the coating is polished after deposition to improve the surface finish and accuracy. Finally, the appearance, hardness, adhesion, and wear resistance of the DLC film were tested.

In order to further improve the adhesion between the film and the substrate, a tungsten transition layer with a thickness of about 200 nm was prepared on the surface of the bionic joint thread in advance. During the deposition process, the rate of air supply remained unchanged, the deposition time was 90 min, and the duty cycle was 80%. During the whole film forming process, the temperature does not exceed 200 °C.

3.4. Surface Coating Detection

For the analysis of the microstructure after coating, the thickness of the DLC film was tested using a white light interference three-dimensional topography instrument. The surface roughness of the DLC film was detected using a surface roughness meter. The Raman spectra of the films were measured with a Raman spectrometer (three-stage linkage laser Raman spectrometer, the instrument's plus mode has an ultrahigh resolution of up to 0.14 cm^{-1}, and the strong Rayleigh line suppression ability of the minus mode makes it possible to measure Raman signals above 5 cm^{-1}). The microstructure of the DLC film was measured by scanning electron microscopy (Zeiss Sigma 300 scanning electron microscope, Zeiss, Mainz, Germany; resolution: 1.2 nm@15 kV, 2.2 nm@1 kV; magnification: 10–1,000,000×; probe current: 40 nA or 100 nA optional), and the composition of the prepared DLC film was analyzed based on the energy spectrum.

The mechanical properties of the coating were analyzed, and the nano-hardness of the film surface was tested with a nano-indentation instrument. The friction coefficient of the film was tested by a friction and wear tester. The loads used were 5, 10, 20, and 30 N, respectively. The dual ball was a steel ball with a diameter of 5 mm, the sliding speed was 0.05 m/s, and the test time was 60 min. The film–substrate bonding force under compressive stress state was tested using the scratch method. A diamond indenter with a cone angle of 120° was used to scratch the film surface at a speed of 100 N/min. The critical load of the film was determined by the acoustic signal of the cracking film and the sudden change in the friction force when the indenter scratched the substrate.

4. Performance Testing and Result Analysis

4.1. Film Thickness and Surface Roughness

Diamond-like carbon films were deposited on the thread surface of the drill pipe joint using arc ion plating technology and a tungsten transition layer, as shown in Figure 5. The thickness distribution of the film with tungsten transition is uniform, and the thickness distribution range, tested by white light interference three-dimensional topography, is 3~5 μm. The surface of the inner and outer threads is flat, without scratches, bubbles, and other defects, and there is no obvious local uneven thickness. The surface roughness of the film is less than 2.0 μm. The surface of the joint after hardening treatment is smooth, and there are no obvious defects such as burrs after machining and hardening treatment. The surface after hardening is black, and the film layer is uniform and does not easily fall off.

Figure 5. Joints after DLC coating.

4.2. Surface Nano-Hardness and Friction Coefficient

The surface nano-hardness test results are shown in Figure 6. The average nano-hardness of the matrix alloy steel is 8.3 GPa, and the average nano-hardness of the surface

DLC film is 24.4 GPa. The hardness of the coating was significantly higher than that of the steel joint substrate, and the nano-hardness increased by 1.94 times after coating.

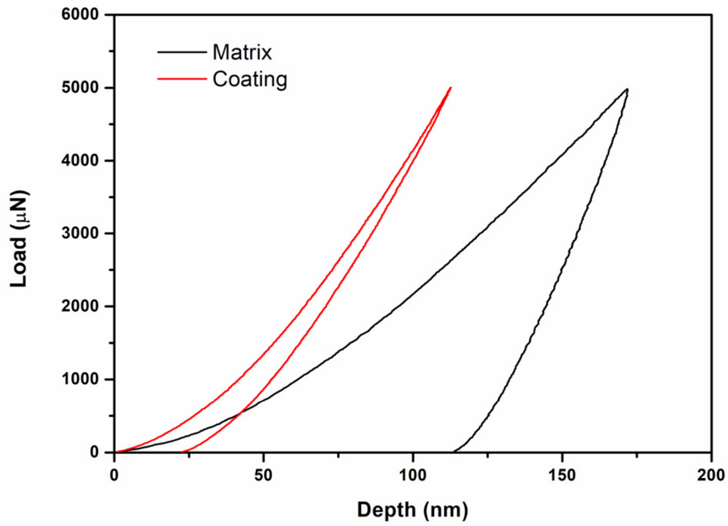

Figure 6. Nano-hardness test.

The friction coefficient of the samples was tested by a reciprocating friction and wear tester. Before coating, the friction coefficient between male and female threads was 0.2, and after coating, the friction coefficient between male and female threads is 0.04. The friction coefficient of the alloy steel after coating is only one-fifth of that before coating, and the tribological performance is greatly improved compared with that before coating.

4.3. Adhesion Force Between Film and Substrate

The bonding strength of the film plays a decisive role in the stability, service life, and comprehensive performance of the film. The critical load of the film is determined by the sudden change in the friction force during the scratch test. The variation curve of the friction force of the W-DLC film with the loading force is shown in Figure 7.

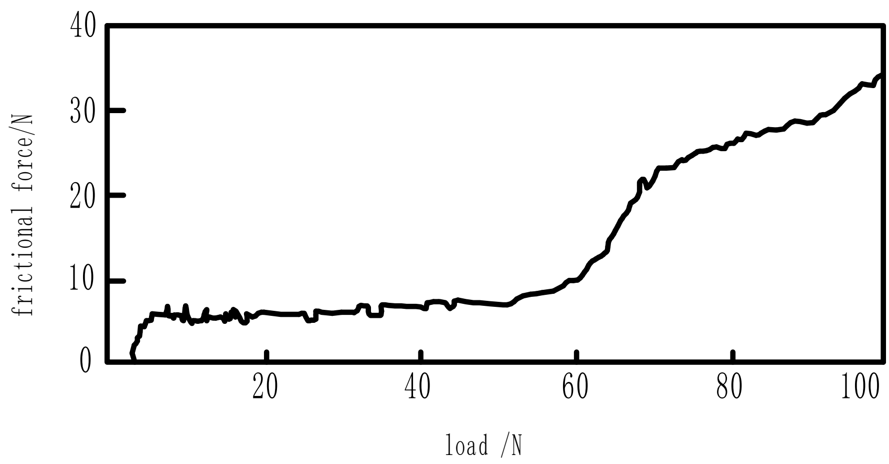

Figure 7. Variation curve of friction force with loading force.

From the diagram, it can be seen that when the loading force is about 60 N, there is an inflection point in the friction force and friction coefficient, which increases sharply with the continuous increase in the loading force and tends to become gentle again at about 70 N. According to the ASTM C1624-2005 standard, the critical load of the film is finally determined to be 56 N.

Chen et al. deposited a Cr-doped DLC film on the outer surface of the torsion arm shaft. The film thickness was 4.3 µm, the hardness was 1600 HV, and the binding force of the film base reached 36.2 N. However, due to the large surface roughness, it was not suitable for the threaded structure. In this work, the bonding force of the DLC film deposited on the surface of the complex thread structure is as high as 56 N, the surface is smooth, and the thread's fit is not affected.

4.4. Determination of Raman Composition and Structure

The characteristic peak of diamond-like carbon film will shift with the changes in the sp2 and sp3 bond content, bond angle, and cluster size. Therefore, the structure of the prepared diamond-like carbon film is analyzed by Raman spectrometry. As shown in Figure 8, in the Raman spectrum, diamond and single-crystal graphite each have a characteristic single peak. The DLC film prepared in the experiment belongs to amorphous carbon film, including two characteristic peaks of graphite "G" and diamond "D", which are located near 1580 cm and 1332 cm, respectively, and the IG/ID peak intensity ratio is about 2.1.

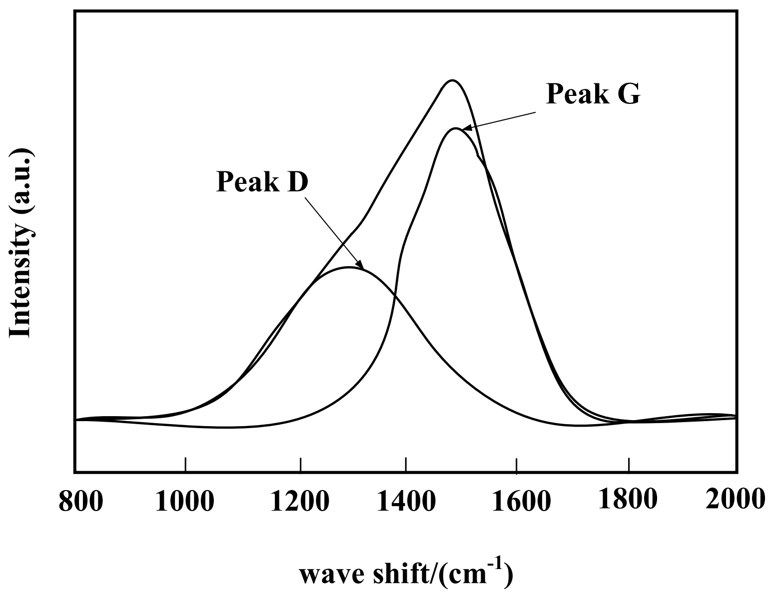

Figure 8. Raman test results.

4.5. Metallographic Structure Analysis

In addition to the characteristics of bionic joint and elastic–ductile alloy material, a diamond-like carbon film was deposited on the surface of the bionic joint thread at low temperature by arc ion plating technology, as shown in Figure 9. The thickness of the DLC film on the top of the external thread is about 5 µm, and the thickness of the film on the bottom of the tooth is about 2.8 µm. The average thickness of the film on the top of the external thread is greater than that on the bottom of the tooth. The thickness of the apical membrane layer of the internal thread teeth is 3.3 µm, and the thickness of the dental membrane layer is about 1.7 µm. Similarly, the average thickness of the apical membrane layer of the internal thread teeth is greater than that of the dental base. For the thread structure position at the same distance from the thread end face (the same as the tooth top or the tooth bottom), the thickness of the outer thread film is greater than that of the inner thread.

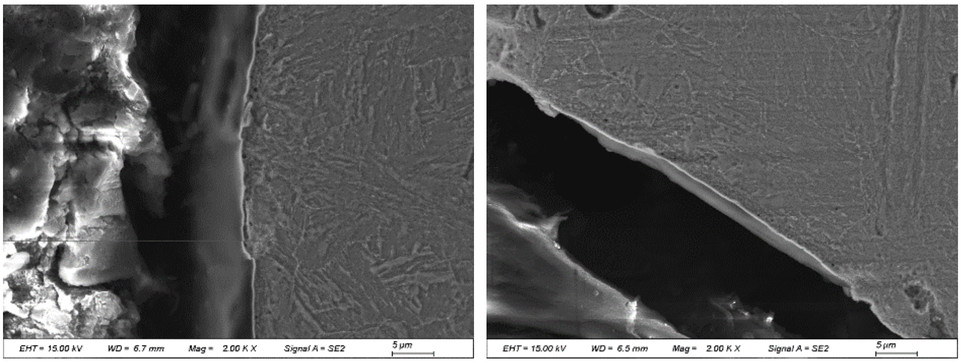

Figure 9. Surface of outer thread and inner thread after coating.

In order to further determine the effect of the transition layer on the adhesion of the film and the substrate, the samples were analyzed by SEM and EDS. It can be seen from Figure 10 that the morphology of the diamond-like carbon film presents a uniform and dense shape, and there is no obvious delamination between the film and the substrate material. The EDS analysis of Line1 shows that C, Fe, W, and other elements fluctuate obviously between the thread surface and the substrate material, indicating that a metallurgical bond has been formed between the tungsten film and the substrate material.

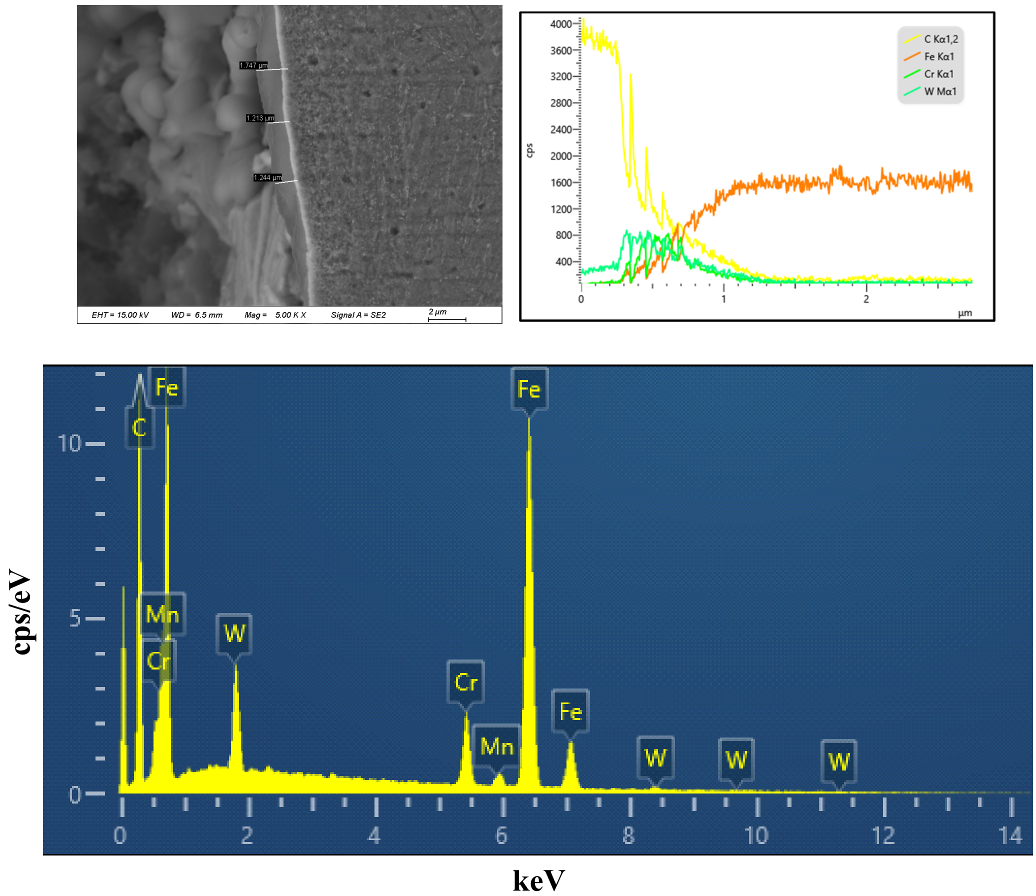

Figure 10. SEM and EDS analysis of diamond-like carbon films.

A ZDY12000LD drilling rig was used to carry out the unscrewing test on the bionic joint. The joint with conventional high-temperature nitriding treatment showed an adhesion phenomenon 30 times after unscrewing, while the joint with diamond-like coating treatment showed a film cracking phenomenon after unscrewing 50 times.

5. Conclusions

(1) In view of the low melting point of the elastic–ductile alloy filled with the bionic joint, high-temperature surface hardening treatments such as quenching and nitriding cannot be carried out on the thread, and the gluing or excessive wear of thread can easily occur. Diamond-like film technology is introduced into the development of the bionic double-connected drill pipe, and the low-temperature deposition of the diamond-like film on the thread surface of the bionic joint is realized, which provides a new idea for solving the problem of the gluing of the bionic double-connected drill pipe and the low-temperature hardening treatment of the special thread surface.

(2) A tungsten transition film was used to improve the film thickness and film–substrate adhesion. The low-temperature deposition process of DLC film on the surface of bionic joint thread was developed. The test results show that the total thickness of DLC film is about 3~5 μm, the roughness is less than 2 μm, the hardness of the film is 24.4 GPa, the friction coefficient is 0.04, and the critical load is 56 N.

(3) SEM and EDS analyses show that the tungsten film and the bionic joint thread form a metallurgical structure, and the morphology of the diamond-like carbon film is uniform and dense, and there is no obvious stratification between the substrate material.

(4) The unscrewing test on the bionic joint using a ZDY12000LD drilling rig shows that the joint with the diamond-like coating treatment has a longer service life than a joint receiving conventional high-temperature nitriding treatment.

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