RESEARCHING THE OPTIMIZATION OF TECHNOLOGICAL PARAMETERS FOR COATING HARD CrN ON SKD11 STEEL SUBSTRATE

NGHIÊN CỨU TỐI ƯU HÓA BỘ THÔNG SỐ CÔNG NGHỆ ĐỂ PHỦ MÀNG CỨNG CrN TRÊN NỀN THÉP SKD11

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ABSTRACT

Hard chromium nitride (CrN) coating is a type of hard coating used to cover the surface of machine components to enhance the quality of the coated surface. CrN coatings are widely used in precision mechanical engineering, cutting tools, automotive industry, metal casting industry, and medical applications. The properties of CrN hard coatings are determined by the technological parameters during the coating process. The content of this research article focuses on optimizing the technological parameters to coat the hard CrN layer on SKD11 steel substrate based on the criterion of ensuring the maximum adhesion strength of the CrN hard coating to the SKD11 steel substrate. The results of investigating other characteristics of the CrN hard coating on SKD11 steel substrate with the optimized parameters show relatively promising outcomes.

Keywords: *SKD11 steel; Hard coating; CrN; Adhesion strength.*

TÓM TẮT

Màng cứng CrN (chromium nitride) là một loại lớp phủ cứng dùng để phủ lên bề mặt chi tiết máy nhằm cải thiện chất lượng bề mặt chi tiết được phủ. Màng CrN được sử dụng phổ biến trong ngành công nghiệp cơ khí chính xác, dụng cụ cắt gọt, ngành công nghiệp ô tô, công nghiệp đúc kim loại và ứng dụng y tế. Đặc tính lớp phủ cứng CrN được quyết định bởi thông số công nghệ trong quá trình tạo phủ. Nội dung của bài báo nghiên cứu tối ưu hóa bộ thông số công nghệ để phủ lớp phủ cứng CrN trên nền thép SKD11 dựa trên tiêu chí đảm bảo lực bám dính của lớp phủ cứng CrN với lớp nền thép SKD11 là lớn nhất. Kết quả khảo sát các đặc tính khác của lớp phủ cứng CrN trên nền thép SKD11 với bộ thông số tối ưu cho kết quả tương đối khả quan.

Từ khóa: *Thép SKD11; Màng cứng; CrN; Độ bám dính.*

1. INTRODUCTION

Today, around the world, hard coatings (with thickness measured in nanometers or micrometers) capable of resisting wear, corrosion, etc., are being applied to the surfaces of cutting tools, molds, medical instruments, etc., to improve the quality of the coated surfaces [1]. Companies in the United States, Japan, Germany, and South Korea such as Kennametal, CVD Equipment Corporation, Mitsubishi Materials, Oerlikon Balzers Hyundai Wia, Doosan, and research organizations like the Korea Institute of Materials Science, are pioneering research and application of hard coatings. Countries in the region such as Taiwan, China, and Thailand are also heavily investing in surface technology, including manufacturing protective surface coatings, and have achieved significant milestones. For example, companies like Fujilloy Co., Ltd. in Thailand and Zhejiang Huijin in China have successfully produced and commercialized various types of hard coatings.

The technology for creating hard coatings through physical vapor deposition (PVD) involves depositing material vapor onto the surface to be coated in a vacuum environment. The PVD process consists of three main stages: material evaporation from the source; material transport to the surface to be coated; and deposition to form a film layer on the substrate surface. PVD technology has been widely applied to produce hard coatings such as TiN, CrN, TiAlN, DLC, AlTiN, protecting detailed surfaces and tools in industrial and everyday life. The CrN hard coating layer has high hardness (approximately 1800-2100 HV), low friction coefficient, good thermal resistance (stable up to 700°C), good wear and corrosion resistance [2; 4; 5], non-reactive (does not react with most chemicals), stable, good adhesion to substrate material, suitable for coating on various substrates (metals, and non-metals). The properties of the CrN coating layer (adhesion of the coating layer to the substrate, friction coefficient, thickness, coherence of the coating layer, etc.) are determined by the method and technological parameters during the coating process [5, 7]. The DC pulsed magnetron sputtering method is a suitable PVD technique to produce CrN hard coatings on detailed surfaces and tools. SKD11 steel is an alloy steel, achieving hardness of about 58-60 HRC after heat treatment, suitable for use as cold work dies to improve fatigue, cracking, and elastic deformation phenomena during the operation of cold dies [10].

In this study, the research team fabricated SKD11 steel samples and coated them with a CrN hard coating using the DC pulsed magnetron sputtering method on the surface of the SKD11 steel samples. The study optimized the coating process parameters (pulse frequency, gas flow rate, coating sample temperature) based on the criterion of ensuring the maximum adhesion of the CrN hard coating layer to the SKD11 steel substrate, and investigated other properties of the CrN hard coating layer on the SKD11 steel substrate based on the optimized set of technological parameters.

2. THEORETICAL FOUNDATION/ RESEARCH METHODOLOGY

2.1. The mechanism of Pulsed DC Sputtering

Applying high-frequency pulsed DC voltage (ranging from 0 to 350 kHz) between the cathode and anode (Figure 1), Ar+ ions with high energy in the plasma environment bombard ☞

the surface of the Cr substrate, resulting in the "stripping" of Cr atoms from the Cr substrate. These Cr atoms, after being stripped from the Cr substrate and colliding with other ions in the plasma environment, transform into Cr ions. As they move towards the coating surface (SKD11 steel), they combine with nitrogen ions in the vacuum environment and deposit onto the surface of the SKD11 steel, forming the CrN coating layer [9].

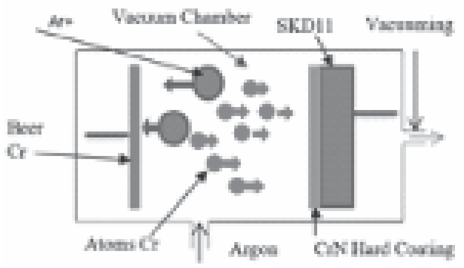

Figure 1. DC Pulse Width Modulation Mechanism

2.2. The CrN coating layer on the SKD11 steel substrate

The SKD11 steel samples with the following chemical composition: C 1.4%, Si 0.275%, Mn 0.39%, Cr 11.24%, Mo 0.83%, V 0.205%, P <0.017%, S <0.0005%, with dimensions: Φ15xL5mm (Figure 2), are heat-treated to achieve a hardness in the range of (58 ÷ 60) HRC. Subsequently, they are ground and polished to achieve a surface roughness of Ra <0.02μm [9].

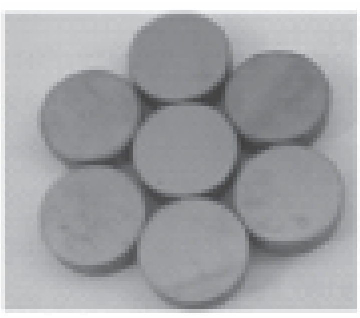

Figure 2. Experimental Sample

The fixed process parameters for creating the hard CrN coating layer are as follows: the distance between the coating surface and the Cr target is 100 mm, the Ar gas flow rate is 12 cm^3/minute, the base pressure is 8 x 10^{-2}Pa, the current density is 1A, and the coating time is 90 minutes. Three process parameters are varied to investigate the optimization of the parameter set for the CrN coating layer on the SKD11 steel substrate, as listed in Table 1 [9].

Table 1. Coating Parameters

No	Parameters	The range of values for the research parameters
1	Pulse Frequency, kHz	50 ÷ 150
2	Nitrogen Gas Flow Rate, cm^3/minute	4 ÷ 8
3	Coating Sample Temperature, °C	100 ÷ 300

Before creating the coating, the steel sample undergoes a rigorous chemical surface cleaning process to ensure that the resulting coating adheres well to the surface [9].

The coating equipment consists of a vacuum chamber with dimensions of 300 x 600 mm connected to mechanical and diffusion pumps. The sputtering target is chromium (99.99%) with dimensions of 100x10 mm. Argon gas (99.99%) and nitrogen gas (99.99%) are used in the coating process, and the gas flow into the vacuum chamber is controlled by two Mass Flow Control 2179A devices from MKS. A pulse power supply device, Pinnacle TM plus, with an oscillation frequency ranging from 0-350 kHz and an output power of 5 kW, manufactured by Advanced Energy, Inc. – USA, is utilized.

2.3. Evaluation Methods for Characteristics of Hard Coating Films

2.3.1. Method for Determining Adhesion of Coating Layer

The adhesion of the coating layer to the substrate was determined using the scratch method [3, 11]. The scratch tip (1) with a radius r_0 moved relatively on the flat sample section AC, creating a cut into the sample that removed both the coating material and the substrate material. During the relative movement of the scratch tip, the applied load F_z increased linearly, while a sound sensor (2) recorded the sound intensity during the scratching process. The adhesion of the coating layer to the substrate was evaluated by the limit adhesion force L_C (N) at the position B on the scratch line where the coating began to delaminate simultaneously with a significant sudden change in sound intensity and the onset of instability. Therefore, based on the time-dependent sound signal (along the scratch path) obtained from the sound sensor attached to the scratch tip, the value of the limit adhesion force L_C of the coating layer to the substrate (Figure 3) was determined. The test to measure the adhesion of the coating layer to the substrate was conducted on a UMT-2 machine, manufactured by the American company CET, with a cutting load of 18 (N), a measurement time of 30 seconds, a cut length of 5 mm, and an acceleration rate of 0.6 (N/s).

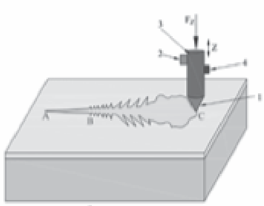

Figure 3. Principle of Adhesion Measurement between Coating and Substrate:
1 - Indentation Tip; 2 - Sound Sensor;
3 - Indentation Force Mechanism; 4 - Position Sensor

2.3.2. Friction and Wear Testing Method

The friction and wear characteristics of the coating layer were evaluated using the block-on-ring sliding motion model on a flat sample [12]. The flat sample was placed underneath, and the ring was placed on top (Figure 4). During the measurement, the sample-holding table moved back and forth perpendicular to the vertical axis, with a constant load (F_Z) applied to the ring along the vertical axis. A 2D force sensor attached to the ring holder determined the friction force (F_X) generated during the sliding of the ring on the flat sample, and the friction coefficient was determined as the ratio of F_Z/F_X. Simultaneously, during the measurement, a position sensor attached to the ring holder determined the depth of wear on the flat sample during the sliding of the ring on the sample. The wear rate coefficient was evaluated using the formula k = z/t, where z (µm) is the depth of wear, and t (s) is the sliding time of the ring on the sample surface. The experiment was conducted on a UMT-2 machine [12], using Al_2O_3 balls with a diameter of 4mm, a load of 5(N), a displacement distance of 600mm, a sliding speed of 10mm/s, a temperature of 240°C, and a humidity of 70% during the measurement.

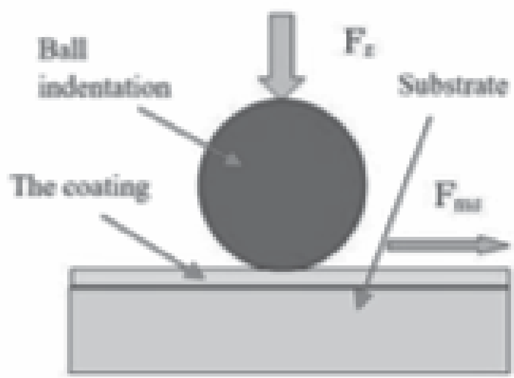

Figure 4. Principle of reciprocating friction and wear measurement, ball on flat sample

2.3.3. Method for evaluating various properties of the coating layer [1-6, 9, 12]

The chemical composition of the coating layer is determined using Energy Dispersive X-ray Spectroscopy (EDX) method. The structure of the coating layer is identified using X-ray Diffraction (XRD) method. The morphology of the coating layer is determined using Atomic Force Microscopy (AFM) method. The thickness of the coating layer is measured using the stylus profilometer method. The hardness of the coating layer is determined using the Vickers hardness method.

3. RESULTS AND DISCUSSION

3.1. Optimizing the parameters of the coating technology

To study the optimization of the coating process parameters (pulse frequency (A), nitrogen gas flow rate (B), and temperature of the SKD11 steel substrate (C)), a total of 15 experiments were conducted using the Box-Behnken experimental design method. The results of measuring the adhesion force of the CrN hard coating layer on the SKD11 steel substrate are summarized in Table 2 [11].

Table 2. Results of measuring the adhesion strength limit of the CrN coating to the SKD11 steel substrate.

No	Pulse frequency - A (kHz)	Gas flow rate - B (cm^3/minute)	Substrate temperature - C (°C)	L_C [N]
1	100	8	300	12.0
2	100	4	100	9.6
3	50	4	200	9.0
4	100	4	300	11.7
5	150	8	200	10.5
6	50	8	200	9.2
7	100	8	100	10.5
8	150	6	300	12.2
9	100	6	200	13.3
10	150	6	100	11.5
11	50	6	100	9.9
12	50	6	300	12.2
13	150	4	200	9.0
14	100	6	200	13.1
15	100	6	200	12.9

The optimal set of technological parameters selected to coat the CrN hard coating layer on the SKD11 steel substrate must ensure that the adhesion force of the CrN coating layer to the SKD11 steel substrate is maximized. Running the corresponding optimization function of the Minitab software yielded the optimal value of the objective function L_C as: L_{CMAX} = 13.96 (N), corresponding to the optimal values of the coating technological parameters: Pulse frequency A = 99.54 [kHz]; Nitrogen gas flow rate B = 6.18 [sccm]; Substrate coating temperature C = 300 [°C]. Repeating the experiment resulted in an adhesion force limit value of LC = 13.85 [N]. The experimental results indicate that the regression model is suitable, and the adhesion of the CrN coating layer to the SKD11 steel substrate is stable and relatively high.

3.2. Evaluating other properties of the CrN coating

3.2.1. The structure and mechanical properties of the coating layer

The chemical composition of the CrN hard coating layer measured by scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM/EDX) showed that the CrN coating layer was fabricated with a corresponding mass ratio of Cr and N of 76.77% and 18.23%, respectively, which is characteristic of the CrN coating layer. The X-ray diffraction (XRD) analysis results of the CrN-coated sample on the XRD system showed that the coating layer had a well-crystallized structure, with the crystal structure mainly oriented along the faces of the cubic-phase CrN, and the strongest peak intensity corresponding to the orientation along the (200) and (111) faces, which are characteristic orientations of the CrN coating layer. The surface morphology of the CrN coating layer was evaluated using atomic force microscopy (AFM), and the results showed that the particle size of the coating layer was 65 nm, and the average surface roughness was 16 nm (Figure 5), indicating that the coating layer had small particle size and roughness.

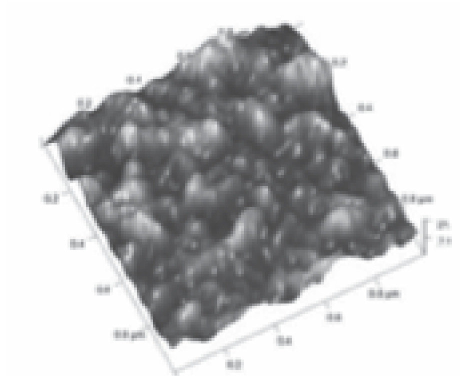

Figure 5. 3D surface measurement image of the CrN coating layer

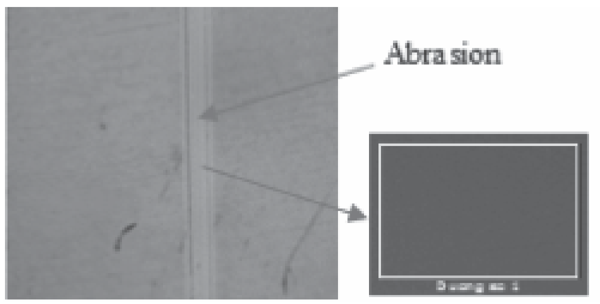

Figure 6. Wear pattern image of the SKD11 steel sample coated with CrN

EDX analysis on the surface of the worn area of the coated sample with increasing wear time: 5 minutes; 10 minutes; 15 minutes, revealed that the percentage of nitrogen by weight decreased gradually as follows: 17.36%; 15.06%; 14.11%. From the SEM analysis results of the EDX analysis region, it was observed that the coating layer was removed from the sample surface through abrasive wear mechanism, rather than delamination, ☞

indicating good adhesion between the CrN coating layer and the SKD11 substrate.

4. CONCLUSION

In this paper, the authors conducted optimization to determine the optimal set of technological parameters (pulse frequency (A), nitrogen gas flow rate (B), and substrate temperature of SKD11 steel (C)) for coating the CrN hard film layer on SKD11 steel substrate based on the criterion of ensuring maximum adhesion force between the coating layer and the substrate. The results obtained during the research process showed that the surface of the CrN-coated steel sample had low friction coefficient and high wear resistance, while also exhibiting high anti-adhesive properties. The reason for this is attributed to the high adhesion of the CrN coating layer to the SKD11 steel substrate, which results from its well-crystallized structure, small particle size, appropriate composition ratio, reasonable thickness, and high hardness. Therefore, the determined technological parameters are considered reasonable for coating CrN on SKD11 steel substrate.❖

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