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Wear resistance investigation of thin film deposition technology

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Foreword

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Yu Qi

Abstract

This article focuses on the popular thin-film deposition technique chemical vapor deposition (CVD) and outlines its latest research in improving wear resistance. The article highlights how the wear resistance of thin film coatings can be improved through material selection, process parameter optimization, post-processing techniques and structural design. The combined application of these methods has a deep impact on thin film deposition technology. In this paper, an experiment was designed to use a Pin on Disk (PoD) machine to explore whether it could be used to verify the wear resistance of atomic layer deposition (ALD) thin films. The samples were mainly deposited on stainless steel substrates using aluminum oxide, silicon dioxide, and hafnium (IV) oxide as ALD coating materials. It was demonstrated that ALD does influence substrate wear resistance, but there is more scope for improving this methodology for verifying the wear resistance of thin films.

Keywords: Thin film deposition, Wear resistance, Atomic layer deposition, Chemical vapor deposition, Pin on disk, Coefficient of friction, Adhesion.

Table of Content

1. Introduction

Thin film deposition (TFD) is an important material preparation method widely used in many fields such as electronic device manufacturing, optical coating, renewable energy field and functional thin film coating. TFD technology has become a ubiquitous part of our daily lives nowadays. For example, the most common deposition-coated cutting tools in industrial production, which are designed to reduce friction and wear during operation to extend tool life. In addition to this, it is also common in copper metallization layers for electronic communication between silicon integrated circuits as well as for corrosion and wear protection of automotive and aircraft engine parts. Functional coating, as the name implies, provides the required functions attached to the substrate material, commonly used functions such as corrosion resistance, scratch resistance, high temperature resistance, etc. It is known that friction occurs whenever two objects in contact with each other, and through different friction mechanisms it can eventually lead to wear. Wear fatigue is frequently found in mechanical devices and engineering structures, especially in equipment that operates under high load and high frequency conditions. Wear leads to material loss, functional degradation, and ultimately failure, so it is critical to control wear in order to extend equipment life and improve reliability. During the journey to find advanced materials and coatings that can withstand high friction and wear environments, TFD has emerged as a key area of research and development. TFD has not been in use for a very long time compared to many existing coating technologies, but nowadays it has many applications in industrial production and academic research. However, the existing research is not sufficient to support the broad market behind TFD, so there is space for wider academic exploration. It can be said that the development of TFD will not only promote the progress of materials science and engineering technology, but also provide strong support for the sustainable development of human society.

This paper focuses on an in-depth study of the wear resistance of chemical vapor deposition (CVD), with a special focus in the direction of atomic layer deposition (ALD). Not only summarizing the existing research, but also designing experiments using the PoD machine to explore the applicability of this methodology for exploring the wear resistance of different coating materials treated with ALD. Optimization and exploration directions are also proposed for future related experimental studies.

The literature summary focuses on the recent progress of CVD technology for the study of thin film materials and substrates. Thin film materials focus on depositing oxide and nitride-based wear-resistant coatings. Substrate materials are mainly concerned with recent developments of iron-based alloys. The review summarizes the methods to enhance the adhesion and wear resistance of TFD coatings to metals by categorizing different principles. The focus is on key parameters such as the technical design of the thin film structure, deposition conditions and post-deposition treatments, and on how these factors affect the overall wear resistance. Approaches to testing the wear resistance of coatings are summarized and evaluation experiments using PoD are conducted to quantify the wear characteristics to assess its effectiveness in investigating the adhesion of ALD films on metallic substrates and testing the wear resistance of the coatings. Aluminum oxide (Al_2O_3), silicon dioxide (SiO_2) and Hafnium (IV) oxide (HFO_2) were used in the experiments for ALD on stainless steel (SS) substrates.

The results of the experimental study are expected to provide information for ALD wear resistance studies, future material selection, and process optimization. By improving the understanding of abrasion resistance in TFD, a path can be opened for innovations that meet the increasingly demanding requirements of modern technology and industrial applications. By utilizing a variety of deposition techniques, scientists and engineers can create materials with precise properties for specific applications. Sustained research and development in this field continues to push the boundaries, bringing new innovations and higher performance to a wide range of industries.

2. Literature review

2.1. Thin film deposition

Thin films, coatings and surface treatments cover a wide range of industries and applications, from medical device coatings to semiconductors to metal machinery. In each of these applications a layer of coating is applied to improve the final properties of the product. This may be to improve wear resistance in cutting tools, corrosion resistance in metal materials in special environments, thermal properties in heat sensitive materials, or to provide desired reflective or refractive properties in fiber optic cables. Although having such a wide range of properties and applications, it should be emphasized that there is no strict definition of film thickness [1]. Usually, however, thin films are deposited at thicknesses in the nanometer to micrometer range. Thin film deposition technique is the process of depositing thin films on solid surfaces by physical or chemical methods. The common thin film deposition methods include CVD and ALD. Regardless of which technique is used, it is often the final step in the manufacturing process. The nano-scale structure of a thin film can significantly improve the physical and chemical properties of a material, and its deposition on the surface of a substrate can provide it with a wide range of functions such as abrasion resistance, electrical conductivity, insulation, anti-reflectivity, and corrosion resistance. Its thickness and structure can be precisely controlled, suitable for high-precision requirements of the implementation. And the importance of wear resistance to thin film deposition technology should not be ignored. It not only extends the life of equipment and devices, improving performance and reliability, but also reduces maintenance costs and improves economic efficiency. With the development of science and technology, the demand for abrasion resistance of thin films will be further increased, and the research on abrasion resistance requires more research and exploration.

2.1.1. The history of TFD technology

Coating technology is believed to have a documented history of more than 5,000 years [2], but over the years advances and new areas of application continue to evolve. The concept of thin film deposition dates back to ancient civilizations, with early meteorological processes involving primary forms of coated materials [2], [3]. The first production of thin metal films by CVD occurred in the middle of 17th century, when a German apothecary, Johann Schroeder, documented how to restore arsenic oxide (As_2O_3) from carbon by a total adsorption reaction [4], [5]. However, the significant scientific improvements in thin-film technology began in the late 19th and early 20th centuries. Physical vapor deposition (PVD) techniques of sputtering and evaporation were developed in the middle and late 19th century [2]. The development of vacuum technology, which plays a crucial role in the development of thin-film technology, is attributed to Evangelista Torricelli, who invented the barometer for the measurement of atmospheric pressure in approximately 1640 [6]. TFD technology was rapidly industrialized and commercialized in the middle of the 20th century, driven by the booming development of the electronics and optics industries [7]. As for ALD the most documented beginnings can be traced back to the "atomic layer epitaxy (ALE)" technology originally developed in Finland for electroluminescent (EL) flat panel displays in the 1970s [8], [9]. The original name of the process was atomic layer epitaxy, however this name faced persistent resistance from the semiconductor industry, who had been using the term epitaxy to indicate only single-crystal growth. Eventually the name of the technology was changed to ALD as it is often mentioned today. By 2024, it will have been 50 years since ALE was globally patented as a method of growing compounds as thin films [10]. Today, many industries rely on precise atomic layer deposition to produce multifunctional, high purity deposited films.

2.1.2. Chemical vapor deposition

Chemical vapor deposition (CVD) is the process of depositing a solid layer of material from a vapor by chemical reaction on or near the surface of a heated substrate. The solid material obtained presents as thin films, powders or single crystals [11]. The chemical reaction can be expressed as releasing a gaseous reactant into a reactor for a deposition reaction to obtain solid material and gaseous products. Materials with various physical, tribological and chemical properties can be grown by varying the reaction conditions, including substrate material, substrate temperature, composition of the reactant gas mixture and total pressure gas flow rate. CVD technology has an excellent ability to produce homogeneous coatings with uniform thickness, properties and low porosity, especially on substrates with complex shapes. Substrates can also be selectively localized for deposition [11].

Producing adherent coatings with the desired properties is the goal of all CVD operations. In order to have a better explanation of the adhesion between the coating and the substrate, Jan-Otto Carlsson summarized five important reaction zones of the surface microstructure in the CVD process, which are divided according to the gas flow and the temperature, as shown in Figure 1. In the CVD process, the reactive gas mixture flows continuously at high temperatures over the substrate and the gradually forming coating surface. Zones 1 to 5 define the areas located away from the substrate, the coating area and the substrate area, as well as the mixing layer of the neighboring areas. In the reaction zone 1 homogeneous reactions may occur in the vapor, which may lead to undesirable homogeneous nucleation and ultimately result in a coating that is easily peelable and poor adhesion. Conversely, if the reactions do not accompany homogeneous nucleation, the CVD process can benefit from it. Non-homogeneous reactions occur at the boundary between the vapor and the coating (Zone 2). These reactions generally determine the properties of the coating and the speed of deposition. Solid-state reactions such as grain growth, phase transitions and recrystallization may occur in Zones 3 to 5. Zone 4 is critically important as the diffusion zone, where various intermediate phases may form, which determine the adhesion of the coating to the substrate [11]. It is important to note that hydrogen （H_2） is often used in cleaning procedures before deposition. While some metallic materials can absorb large amounts of H_2, this may lead to difficulties in adhesion of solids and coating breakage. This is known as substrate hydrogenation, which can lead to poor coating bonding. Hydrogenation can be eliminated by using other cleaning procedures or by heating the substrate in a vacuum or inert gas [11].

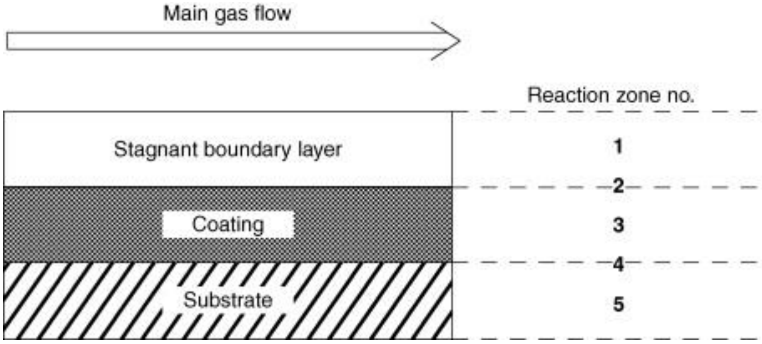

Figure 1: *Important reaction zone in CVD [11].*

CVD is the foundation of a series of processes that can be used for a variety of thin film applications including dielectrics, conductors, conductive oxides, friction and corrosion resistant coatings, and heat resistant coatings. Other CVD applications include the preparation of high temperature materials (tungsten, ceramics, etc.) as well as solar cells and high temperature fiber composites [12], [13], [14].

2.1.3. Atomic layer deposition

Atomic layer deposition (ALD) is a chemical vapor deposition method for TFD based on sequential, alternating saturated surface reactions [15]. Two or more precursors are introduced separately and sequentially onto the substrate surface (each precursor chemical contains a different element of the deposited material). Each precursor sequentially saturates the surface to form a single layer of material. As shown in Figure 2, each ALD cycle can be described as pulse introduction of the first precursor-purge reaction chamber-pulse introduction of the second precursor-purge reaction chamber and repeat [16]. Most ALD deposition systems require the use of an inert carrier gas, such as the commonly used nitrogen. A certain amount of precursor chemistry is pulsed into the flowing inert carrier gas and separated by short purge or evacuation cycles. In a single deposition, a certain amount (usually a monolayer of about 1 Å) of thin film material is grown [16]. Typical cycle times are one to several seconds. Process temperatures range from room temperature to above 500 °C. Most ALD processes operate at temperatures from 200 °C to 400 °C [17]. The choice of precursor plays a crucial role in the quality of the coating for ALD growth, and the precursor must be thermally and chemically stable to prevent self-decomposition in the highest temperature range of the reaction [15]. It is desirable to be able to adsorb and saturate quickly on the surface of the material or to react quickly and efficiently with the surface of the material without producing undesirable products such as toxic or corrosive effects. ALD offers more precise film thickness control and better uniformity than CVD [18]. In comparison, the higher deposition temperature of CVD may limit the choice of substrate [16]. ALD is a slower reaction and the equipment is more expensive. The choice between CVD and ALD depends on specific requirements such as film thickness, uniformity, substrate compatibility and application needs.

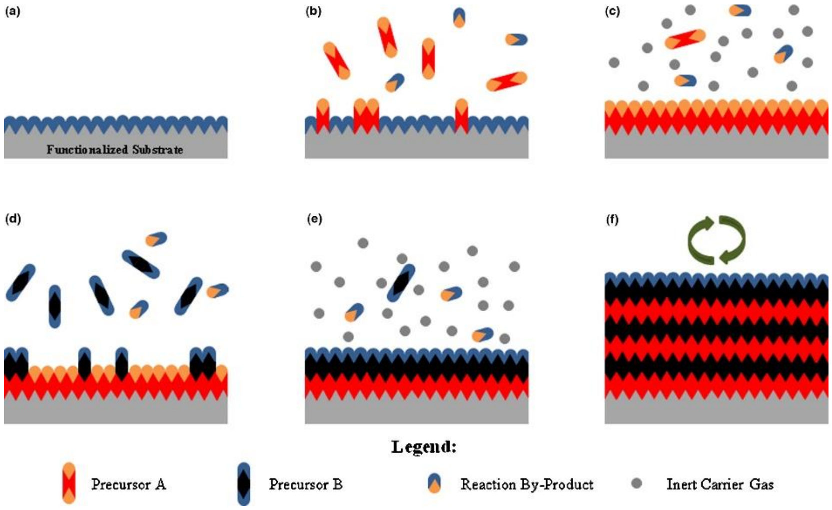

Figure 2: *Illustration of the ALD process. (a) The substrate surface is prepared for deposition. (b) Precursor A reacts with the surface in pulses. (c) Purge the excess precursor A and side products of the reaction with an inert carrier gas. (d) Precursor B reacts with the surface in pulses. (e) Purge the excess precursor B and side products of the reaction with an inert carrier gas. (f) Repeat steps 2-5 until desired material thickness is achieved [16].*

Atomic Layer Deposition (ALD) technology produces films with excellent wear resistance due to its high precision, uniformity and good coverage. By selecting the right materials, optimizing deposition parameters and implementing post-treatments, ALD coatings provide excellent performance in high-load and high-wear environments. This enables ALD to be used in a wide range of applications such as cutting tools, biomedical implants and optoelectronic devices.

2.1.4. Film materials

TFD utilizes a wide range of deposited coating materials, each with specific properties suitable for different applications. These materials include metals to semiconductors, oxides to nitrides, and advanced materials such as graphene and chalcogenides [19], [20]. The choice of materials is critical to achieving the performance characteristics required in electronic, optical, energy storage and mechanical systems. Based on the experimental design in the later part of this paper, an emphasis is hereby placed on the three coating materials used and their adhesion to the substrate.

One of the most common thin-film materials is alumina, which is highly resistant to abrasion due to its high hardness. It is chemically stable and resistant to reacting with other materials, which makes alumina coatings very durable and corrosion resistant. Mechanical component parts are often coated with aluminum oxide to protect against wear and corrosion and to extend the service life of the parts. However, the films may be subjected to stresses during the deposition process, which can lead to potential cracking or delamination that reduces their adhesion to the substrate [21]. Experiments by G. H. Meier et al. have shown that surface roughness can be increased by techniques such as sandblasting and chemical etching to promote mechanical interlocking and thus enhance adhesion. Chemical bonding and compatibility can also be improved by using adhesion-promoting intermediate layers such as titanium or chromium. Experiments were conducted on the adhesion strength of aluminum oxide films on different metal substrates (stainless steel, titanium alloys, and nickel-based high temperature alloys). It was concluded that the film adhesion strength depends largely on the deposition method, surface treatment and substrate properties [22].

Silicon dioxide has to be mentioned as a common film material for TFDs in the semiconductor industry, which is widely used in integrated circuits and microelectronic devices as gate oxide, passivation and isolation layers [23]. Common precursors used in the CVD preparation of silicon dioxide films include silane (SiH_4) and oxygen (O_2) [24]. It is worth noting that internal stresses may be generated during the deposition process, affecting the adhesion and stability of the film. In addition to this, the coefficient of thermal expansion of SiO_2 is significantly lower than that the most common metals, and this difference may lead to cracking or peeling of the film during temperature changes [25]. S. Vallon et al. demonstrated through conventional thermal adhesion tests on SiO_2 films that preliminary plasma treatment of the polymer substrate prior to the growth of the film improves the adhesion of the first monolayer of the film, thus improve its adhesion [26]. Y.M. Lian et al. through experimental design study concluded that glow discharge treatment has a greater effect on the adhesion of silica films while vacuum pressure and deposition rate have a lesser effect on the adhesion [27].

Ultra-thin hafnium dioxide (HfO_2) is commonly used as a replacement for silicon dioxide to meet the requirements of transistor feature size in the advanced semiconductor industry. The reliability criteria such as wear resistance, thermal fatigue and stress-driven failures are heavily reliant on film adhesion [28]. Wei-En Fu et al. analyzed the mechanical properties of annealed and untreated HfO_2 films, the annealing process increased the surface hardness of the crystalline HfO_2 films, which in turn improved the resistance to nano-scratches. The annealed samples exhibited lower coefficients of friction (COF) and wear rates under the same normal force conditions compared to the untreated samples, which is attributed to the increased abrasion resistance induced by crystallization and is less affected by substrate effects [29].

Thin films of nitride usually have excellent hardness and wear resistance, making it suitable for improving the wear resistance and lifetime of the component surfaces [30]. For example, carbon nitride in the form of thin films is a good material for hard coatings, but it has poor adhesion on tool steels due to the diffusion of nitrogen or carbon atoms into the substrate under the deposition temperatures. A.K.M.S Chowdhury and D.C Cameron saturated the surfaces of AISI H13 and AISI/SAE8620 tool steels with nitriding technique and evaluated the carbon nitride film adhesion on it. It was shown that the film material had significantly improved adhesion properties on the steel surface [31]. Bonding problems are usually encountered when diamond is deposited directly on ferrous materials due to the formation of a graphite layer at the interface between the two. O. Glozman and A. Hoffman successfully solved this problem by applying an intermediate layer consisting of a 20μm thick chromium nitride film. The initial stage of diamond deposition leads to partial carbonization of the chromium nitride interlayer and deposition of a continuous diamond film on top of it. The adhesion of the diamond film to the substrate is enhanced by the complex mechanical structure and chemical bonding in the interfacial region [32].

Progress in thin film deposition technology continues to expand the possibilities for material applications. More emerging materials are being explored, which contributes to increased TFD upgrades to accommodate the changing needs.

2.1.5. Substrate materials

The selection of substrate materials for TFD requires a combination of flatness, chemical stability, thermal stability, thermal conductivity, mechanical properties, dimensional stability, and cost to ensure that the final TFD process and finished product meet the requirements of the specific application. Common substrate materials are listed below:

1. Metals (e.g., stainless steel, aluminum, titanium): Metals are commonly used as substrates in TFD processes for applications requiring high mechanical strength, electrical conductivity, or corrosion resistance. On most steels, the adhesion of the film increases with increasing substrate temperature, reaching a maximum between 400 and 500°C. The adhesion of the film increases with increasing substrate temperature. The increase in adhesion is related to changes in the oxide layer on the steel substrate. As the temperature increases, Fe_2O_3 and Fe_3O_4 decompose to FeO. Sputter etching of the substrate prior to deposition improves film adhesion, even if it does not completely remove the oxide layer [33].

2. Silicon (Si): Silicon and its compounds are widely used as substrate materials in a variety of TFD applications, especially semiconductor device manufacturing. Properties such as its high thermal conductivity and compatibility with various thin film materials make it suitable for a range of applications [34].

3. Ceramics (e.g., alumina, silicon carbide): ceramic substrates are used in the TFD process for applications requiring high-temperature stability, electrical insulation, or chemical resistance. k. Kishitake et al. heated ceramic substrates with alumina coatings in air at a specific temperature, to find that as the thickness of the bonding coating was reduced, the bond strength of the coating increased [35].

4. Polymers (e.g., polyimide, polymethylmethacrylate): Polymer substrates are used in the TFD process for applications requiring flexibility, light weight, or transparency. Films deposited on polymer substrates can provide barrier, conductivity [36], [37].

2.2. Wear resistance testing methods

2.2.1. Methods for testing the abrasion resistance of coatings

Friction is related to the tangential force between two objects at a junction, which move relative to each other when in contact, and is controlled by three basic mechanisms: adhesion, plowing, and roughness deformation [38]. As for wear, it is the removal of material due to adhesion, abrasion, surface wear fatigue and chemical reactions during the contact between two objects that move relative to each other. The coefficient of friction is used as a metric to quantify it, and the coefficient of friction represents the ratio of tangential (frictional) force to normal force [38]. Wear testing is an important means of assessing the wear resistance and life of a material in practical situations. Different wear testing methods can simulate the behavior of materials under different wear mechanisms such as abrasive wear, adhesive wear, and fatigue wear.

To determine tribological properties such as friction coefficients and specific wear rates, several different characterization techniques are used, Pin on disk test being the most simple and straightforward method to simulate wear conditions. In parallel to this, Energy Dispersive X-ray Spectroscopy (EDX) and X-ray Photoelectron Spectroscopy (XPS) are also commonly used in conjunction with the Pin on disk machine. (Jiri Nohava et al., 2015) conducted Pin on disk testing of nitride, wear-resistant nitride and oxide coatings made by PVD technology at different temperatures, and the SEM-EDX was used in combination with Leigh to check the surface morphology and cross-sectional structure of the wear tracks [39]. A similar experimental approach is the study by (O. Jantschner et al., 2015), where the researcher performed Pin-on-disk tests on Si containing amorphous carbon films, followed by using XPS to check for the presence of different Si chemistries in the wear tracks [40].

Nanoindentation testing determines the hardness of materials on a nanometer scale and is mainly applied to determine the hardness of thin films. It is based on the principle that a sharp tip (usually made of diamond) is indented into the surface of a material with a controlled force. During the indentation process, the depth of penetration and the applied force are continuously recorded. By analyzing the resulting load-displacement curves, various mechanical properties can be determined [41]. Suraj Maganty et al. used nanoindentation to determine the relationship between thickness and hardness of polyurethane (PU) films. It was concluded that the hardness of all components steadily decreases with increasing indentation depth in the depth range starting from 500 nm of the film. In order to minimize the error, they applied five times of nanoindentation test at different locations [42].

The ball-cratering test is an effective method of evaluating the abrasion resistance of thin films by accurately measuring the dimensions of the wear pits to obtain key parameters such as the thickness and wear rate of the coating. The procedure consists of a spherical probe that encounters the coating surface under load and rotates to create a wear. By measuring the diameter and depth of the wear, the thickness of the coating can be calculated. The wear rate of the material can be calculated from the volume of the wear and the applied force. By observing the morphology and structure of the wear, the wear mechanism of the coating can be analyzed [43]. Experiments by

Felipe Carneiro da Silva et al. have shown that the ball-cratering micro abrasive wear test is well suited for investigating the abrasion resistance of homogeneous and functionally graded TiN films [44].

2.2.2. Pin on disk machine

The Pin on Disc (PoD) machine is a popular tribological testing device intended for evaluating the friction and wear characteristics of materials. The PoD used in the experiment is shown as Figure 3. It simulates contact between a fixed pin and a rotating disk to generate wear, providing data on wear rates, coefficients of friction and material migration under controlled conditions. As the name implies, the components are simple and straightforward as shown in the figure 4 and generally consist of the following:

1. Pins

Pins are usually made of a hard material such as steel, tungsten carbide or ceramic and are used as a means of holding the sample in place [16]. Pins can have a variety of shapes but are usually cylindrical or spherical to ensure a consistent contact area.

2. Discs

The disk is coated with or made of the material to be measured. It is mounted on a rotating table with controlled speed.

3. Load application system

The load mechanism applies a constant normal load to the pin to maintain contact with the disk. The load can be changed to simulate different pressure conditions.

4. Rotating Table

The disk is attached to a motorized table that rotates at a preset speed. The speed can be adjusted to simulate different sliding speeds.

5. Data Collection System

Sensors measure friction and normal loads during testing. Data is recorded and analyzed to calculate the coefficient of friction.

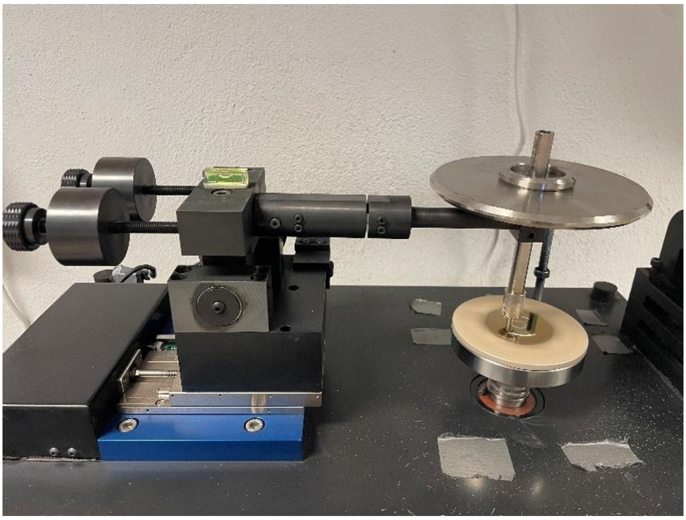

Figure 3: *Pin on disk machine in workshop.*

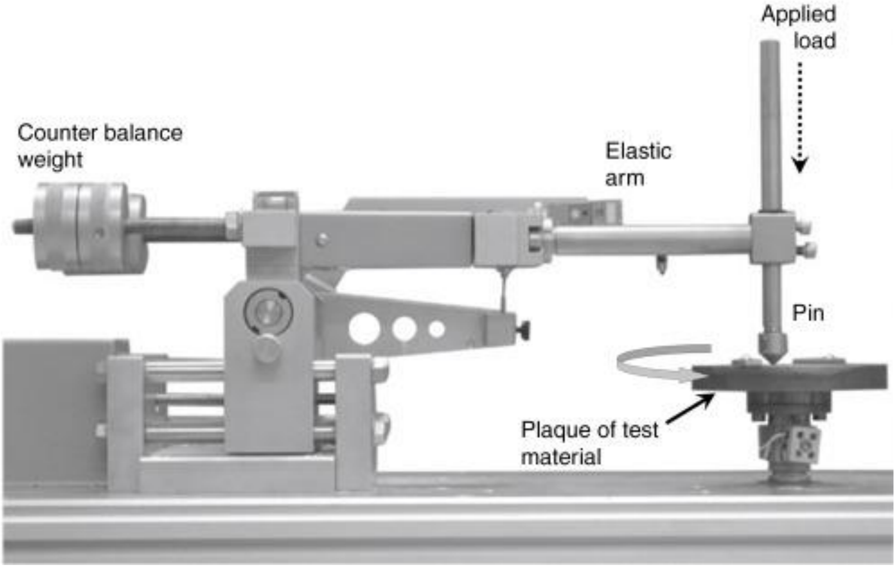

Figure 4: *Pin-on-disk tribometer equipment.* (*Photo courtesy of Nanovea Corporation.)*

The Pin on Disc Machine can be adjusted to simulate a variety of wear conditions by adjusting load, speed, and test duration. It allows precise control of the test parameters and multiple repeated tests [45]. Simple setup and operation make it easy to use for a variety of research and industrial applications without extensive precise specialized knowledge. Typically, this machine is used to evaluate the wear resistance of new materials and coatings, to study basic wear mechanisms and tribological behavior. By simulating realistic conditions and thus predicting the lifetime of production tools, the Pin on Disc machine is an important tool for tribological testing, providing valuable insights into the wear and friction characteristics of materials. Its ability to simulate real wear conditions in controlled laboratory settings makes it ideal for materials development, quality control and tribological research. J.H. Hsieh et al. performed PoD tests on alumina spheres along with a surface profiler (KLA Tencor P-10) to measure the wear tracks of each coating. The amount of wear of the sample was evaluated based on the cross-sectional area of the wear track. The wear rate can be obtained by dividing the sample wear by the total sliding distance [46]. T. Polcar et al. tested W-S-C films by the PoD method to analyze the variation of the coefficient of friction with temperature. It was concluded that the tribological behavior of the coating with increasing temperature depends on the carbon content in the film [47].

2.3. Methods to improve adhesion and wear resistance of CVD and ALD

To enhance the adhesion and wear resistance of CVD coatings to metals can be accomplished in a variety of aspects involving substrate surface treatment, coating material selection, process parameter optimization, and post-processing techniques.

Carbon coatings, especially diamond-like carbon (DLC) coatings, have a wide potential for industrial applications due to their high hardness, low friction, and good chemical stability. However, these coatings are often limited in practical applications by poor adhesion and insufficient wear resistance. A paper describes the solution to this problem by the deposition of Me:C (metal containing) gradient layers (M. Cłapa and D. Batory, 2007). A Ti:C gradient layer is a transition layer between the substrate and the carbon coating, with a gradual transition in composition from titanium (Ti) to Carbon (C). According to experiments, the Ti:C gradient layer promotes the formation of strong chemical bonds between the substrate and the coating, creating a mechanically interlocking interface and reducing the risk of peeling. It also relieves thermal and residual stresses to prevent coating loss. By improving the three mechanisms of chemical bonding, mechanical interlocking and stress relief, the adhesion and wear resistance of the carbon coatings were significantly enhanced. It was shown that the Ti:C gradient layer significantly improved the adhesion of the carbon coatings on different substrates, with higher critical loads and adhesion strengths demonstrated in scratch and pull-off tests. The incorporation of the Ti:C gradient layer improved the wear resistance of the carbon coatings, which was attributed to the improved adhesion and the hardness and toughness of the composites themselves. In terms of tribological properties, the Ti:C gradient layer reduces the coefficient of friction and wear rate of the carbon coating, making it suitable for high stress environments [48].

Transition metal nitrides, such as CrN, are highly valued for its high hardness, excellent oxidation resistance and thermal stability [49]. A study introduced a multilayer composite design approach to DLC coatings to improve the tribological properties of transition metal nitride coatings (X. Sui et al., 2018). The authors successfully deposited CrN/DLC/Cr-DLC multilayer coatings and investigated its mechanical and tribological properties. It was demonstrated that the friction and abrasion of the CrN/DLC/Cr-DLC multilayer composite coatings were reduced by multilayer design. The CrN/DLC/Cr-DLC multilayer coatings consisted of chromium-nitride (CrN), DLC, and chromium-doped DLC (Cr-DLC) layers deposited alternately. By controlling the deposition parameters, a well-bonded and uniform layer thickness between the layers can be achieved. Nano-hardness tests show that the hardness of the multilayer coatings is significantly higher than single-layer DLC coatings. The modulus of elasticity test shows that the multilayer coating not only has high hardness, but also maintains a certain degree of elasticity, which helps to resist external impacts and wear. PoD tests showed that the multilayer coating exhibited a low and stable coefficient of friction, especially under high loads and high sliding speeds. Wear rate tests show that the multilayer coatings have very low wear rates, and the combination of the CrN and Cr-DLC layers effectively distributes the wear load and improves the overall wear resistance. The CrN/DLC/Cr-DLC multilayer coatings significantly improve the overall mechanical properties and wear resistance by combining the advantages of each layer [50].

A study (Z. Hsain et al., 2017) showed the influence of annealing on the wear resistance of ALD deposited alumina films. The researchers used ALD to deposit alumina thin films on silicon substrates and annealed the deposited films in an inert atmosphere at different temperatures to study the effect on their microstructure and mechanical properties. According to the results of wear tests performed in a pin-and-disk friction and wear tester, the annealing of the ALD alumina coatings at 1000 °C resulted in a significant reduction of wear compared to their counterparts in the as-deposited state. SEM images showed that annealing resulted in a smoother surface topography of the material. In this study, it is shown that annealing significantly improves the wear behavior of ALD deposited alumina films. The crystallization and densification of the films during annealing leads to an increase in hardness and elastic modulus, leading to excellent wear resistance [51]. The results of this study highlight the potential of annealed ALD-deposited alumina films as high-performance wear-resistant coatings. However, the amount of experimental data is not sufficient to fully support this argument, and more experiments are needed to select better annealing temperatures and lengths of time. For example, the implementation of additional wear resistance tests could simulate conditions similar to those of actual use. Experimental methods and results

3. Sample preparation and layout

The experimental procedure consisted of three steps: sample preparation, ALD treatment and PoD wear test. Sample preparation and PoD were processed in the workshop of the Department of Production and Manufacturing, and ALD was done in the clean room of the Physics Center in Lund University. Three different materials were selected for deposition in the experiment, Al_2O_3, SiO_2 and HFO_2. Three samples were prepared for each group of the three materials, for a total of nine pieces of material.

3.1.Sample specifications and processing

A common material chosen for the substrate sample was stainless steel 316L, an austenitic chromium-nickel stainless steel that contains molybdenum to enhance its corrosion resistance [52]. This material was chosen not only for its high machinability, but also because it is a common and readily available material in the workshop.

The size of the substrate was chosen as 50×50 mm due to the size of the PoD rotary table, which made it inappropriate to choose an oversized disk material. The original material was a 1.5mm thick piece of stainless steel, which was cut to the design size using a cutting machine. To fix the disk material on the rotary table, two holes were drilled diagonally in the square material using the drilling machine. The size of the drill holes is matched to the size of the screws of the rotary table. Precision is not highly required, to ensure that the material can be fixed on the rotary table without displacement.

The processed material needs to be polished, in which case it was done by manual polisher as shown in Figure 5, which is a time-consuming and labor-intensive part of the process. Due to the size of the base material, it was important to ensure that the polishing surface is flat enough. The order of polishing precision used is: 9 microns - 3 microns - 1 micron - 0.25 microns. The surface of the polished material is virtually scratch-free, mirror-like and reflective.

Figure 5: *Manual polishing machine.*

Polished substrates need to be thoroughly cleaned before ALD to remove any particles that may be carried. As shown in Figure 6 ethanol and isopropanol (IPA) are used in turn for 3min soaking respectively in an ultrasonic machine, and air pumps are required to blow dry between the two liquids. The Ethanol solution is effective in removing dirt, grime and stains from a variety of surfaces including plastics, glass, ceramics and metals [53]. IPA is a good general metal cleaner and will remove most of the cutting oils and grime remaining from the cutting process conducted [54]. Polishing and cleaning the material is done to obtain a purer surface, which is a requirement for ALD to be able to adhere strongly.

Figure 6: *Ethanol(left) and isopropanol (right) cleaning liquid in ultrasonic machine.*

The pin material is processed by Electrical discharge machining (EDM), using the stainless-steel round bar material that is readily available in the workshop. The shape of the pin was chosen to be a flat cylinder, which is easy to process and reduces the waiting time for the laboratory, considering the difficulty of processing the material. The specification is 6mm diameter and 17cm length stainless steel round bar.

3.2. Thin Film Depositions process

Due to time constraints, three different sets of materials were deposited by two different ALD machines. The machines work on the same principle, despite the different size of the reaction chamber. The first set of Al_2O_3 films and the third set of HFO_2 film were deposited by Savannah100, the second sample set of SiO_2 film was deposited by Fiji F200. Table 1 summarizes the specifications and the naming of the nine samples prepared, all of which are referred to by name in the following text.

Table 1: *Sample Specifications.*

Material	Thickness	Amount	Sample number
Al_2O_3	60nm	3	S_1, S_2, S_3
SiO_2	100nm	3	S_4, S_5, S_6
HFO_2	100nm	3	S_7, S_8, S_9

Figure 7 shown the Savannah S100 ALD system in the lab. It provides excellent deposition results, and each Savannah system can be digitally controlled for thin film deposition. The machine is suitable for depositing Al_2O_3, SiO_2 and HfO_2 at deposition temperatures from 80 to 200°C. The maximum film thickness that can be deposited is 100 nm. The deposition rate is approximately 1 angstrom per cycle. As shown in the figure 8, the three substrates prepared in the first and third groups were placed into the reaction chamber in two times according to the different deposition materials, and Al_2O_3 and HfO_2 were used as the deposition materials, with the deposition thicknesses of 60 nm and 100 nm, respectively. The process lasted about 9 hours for each group.

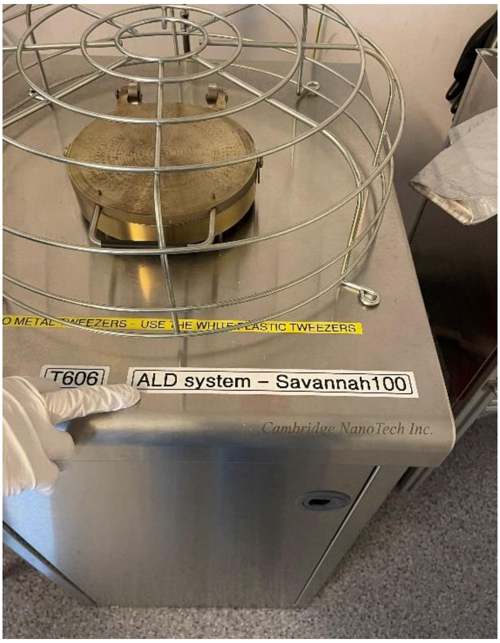

Figure 7: *Savannah 100 ALD machine.*

Figure 8: *Three samples were placed in the reaction chamber to await deposition for Savannah 100 ALD machine.*

The Figure 9 shown the Fiji F200, which has a larger reaction chamber than the Savannah S100. This is a modular high-vacuum ALD system with a flexible system architecture and multiple configurations of precursor and plasma gases to accommodate multiple deposition modes. As shown in the Figure 10, the second set of three prepared substrates was laid flat into the reaction chamber and deposited with SiO_2 as the deposition material with a thickness of 100 nm. The process lasted about 9 hours.

Figure 9: Fiji F200 ALD machine.

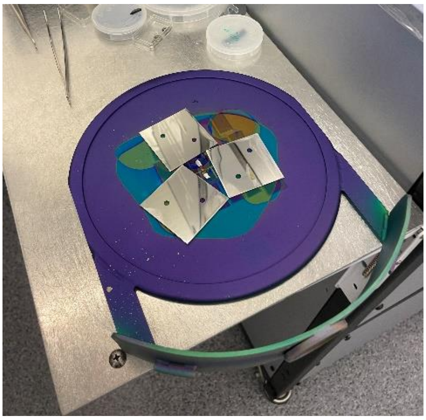

Figure 10: Three samples were placed in the reaction chamber to await deposition for Fiji F200 ALD machine.

When placing into the reaction chamber make sure that the substrates do not overlap or interfere with each other and should be laid flat to prevent them from being placed on the plate. The deposited material should be removed with care because of its high temperature and may be cooled with an Air Pump before being carefully wrapped in clean non-woven fabric for storage.

3.3.Experiment procedure

The initial version of the experimental design encountered some difficulties in its operation, and after certain adjustments, the specific abrasion test procedure is described below.

1. Sample and needle mounting

Place the deposited SS sample disk on the rotary table and fix it to the sample holder using two screws through the pre-drilled holes. Install the prepared pin in the chuck and change it after every test track change of the sample. Use lab gloves throughout to contact the samples and pins to ensure that the surfaces of the pins and samples are clean, flat, and smooth, to minimize factors affecting the experiment.

2. Load Setting

The pin was gently touching the surface of the sample disk, zeroing the load and using the loading page in the software to set the required normal load to 11 N. Ensure that the pin is in contact with the disk at the specified load and check the value in the application. It is important to note that the load only needs to be zeroed once before the start of the formal test, it does not need to be zeroed again after subsequent operations such as changing samples. The reason for choosing the value of this load will be explained in more detail later.

3. Setting test parameters

Set different rotation speeds for the inner and outer orbitals of the sample disk and define the duration of the test to be 3 min, which is decided after completing the pre-test, and is sufficient to remove all the film coating on the track surface. For each sample, testing was performed first on the inner track and then on the outer track.

4. Run the test

Press the "Start" button in the software to start rotating the sample disk. While the disk start rotating, the pin maintains its position and produces a continuous sliding motion between the contact surfaces. Friction and normal load data are continuously recorded. Although the sliding speeds and load selected for this experiment are not high, it is advisable to cover the PoD machine with a protective shield before start in order to accomplish the experiment in a safer environment. A metallic grinding sound can be heard as soon as the sample disk begins to rotate after the test begins. Depending on the material deposited on the sample, it is reasonable to assume that some of the samples will show a difference in the noise after a short period of time.

5. Post-test analysis

Once the test is complete the rotating disk will automatically stop, make sure the PoD is completely stopped then open the shield and remove the sample disk and the pin for recording and storing. Observe the wear marks on the disk and the wear marks on the pin. Obtain COF data through the software connected to the sensor, analyze the data and plot graphs for further analysis.

Since the material was large enough, both inner and outer tracks were selected for wear testing, with distances from the center point of the inner and outer being 4mm and 19mm, respectively. The specific experimental data were selected according to the experimental data of (L. Natrayan and M. Senthil Kumar, 2021) [55], and 11N was selected as load with reference to the conditions of Level 2. In the pre-test experiment, we selected 1m/s and 0.5m/s as the linear velocity, but the wear occurs too fast, so in the formal test, the linear velocity defined as 0.25m/s. According to the Equation 1, the rotational speeds are about 1200rpm and 253rpm by inputting 4mm and 19mm as the inner and outer track radius respectively.

$$N(rpm) = \frac{60}{2\pi \times r} v(m.s^{-1}) \qquad \textit{Equation 1}$$

The specific setup parameters are shown in Table 2.

Table 2: *Considered factors.*

Process	**Unit**	**Inner track**	**Outer track**
Load	N	11	11
Rotating velocity	rpm	1200	253
Sliding time	min	3	3
Distance from center point	mm	4	19

4. Results and analysis

4.1. Al_2O_3 sample

Thin films of aluminum oxide (Al_2O_3) are valued for their excellent wear resistance in many high-tech and industrial applications. Aluminum oxide in thin film form combines the material's inherent high hardness, chemical and thermal stability to provide effective protection and enhanced performance under a wide range of challenging conditions [56]. Its high hardness, good mechanical strength and chemical stability make it an ideal wear-resistant material.

Al_2O_3 coating materials inner track PoD wear testing:

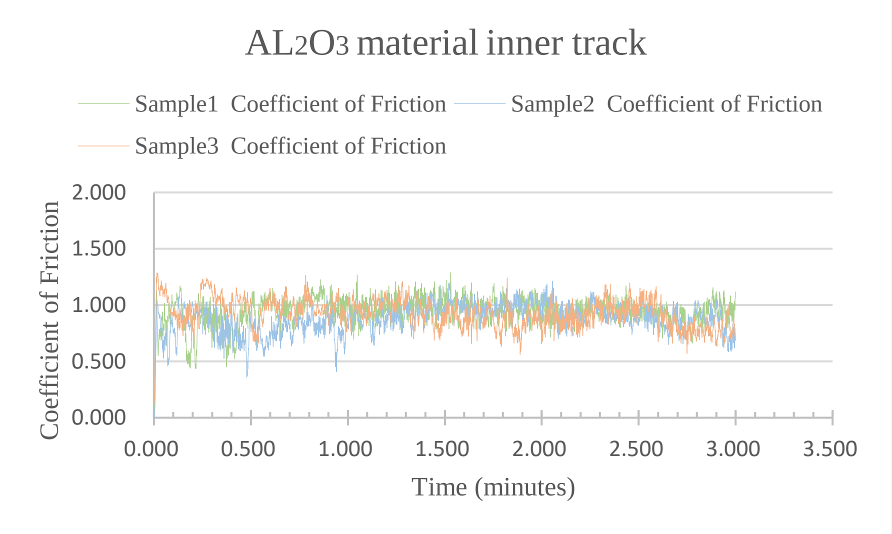

Figure 21: *COF of Al_2O_3 coating S_1、S_2、S_3 of inner track.*

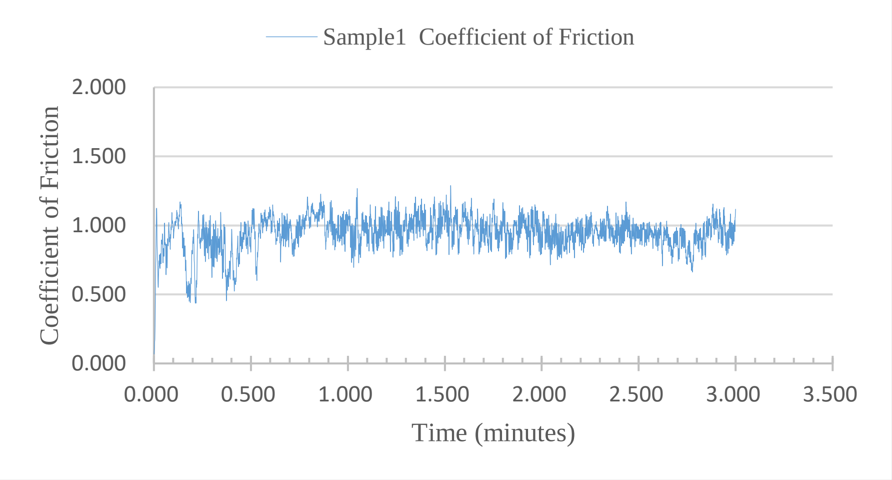

Figure 12: *COF of Al_2O_3 coating sample 1 of inner track.*

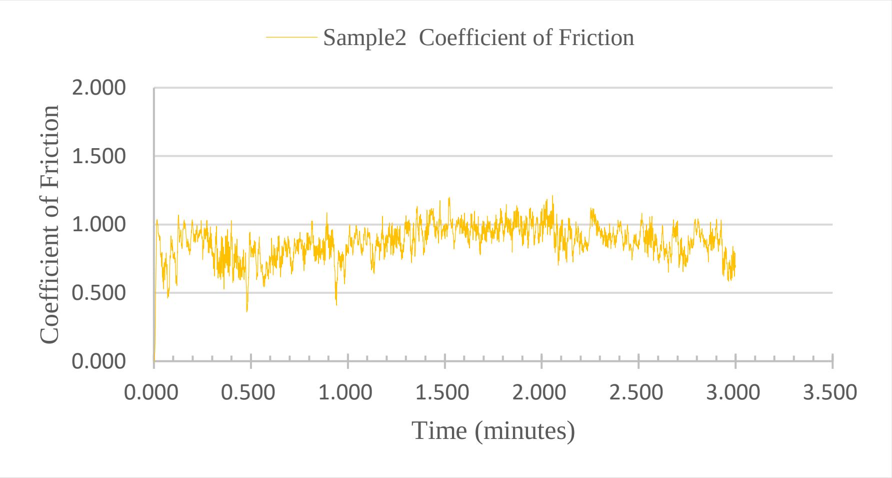

Figure 33: *COF of Al_2O_3 coating sample 2 of inner track.*

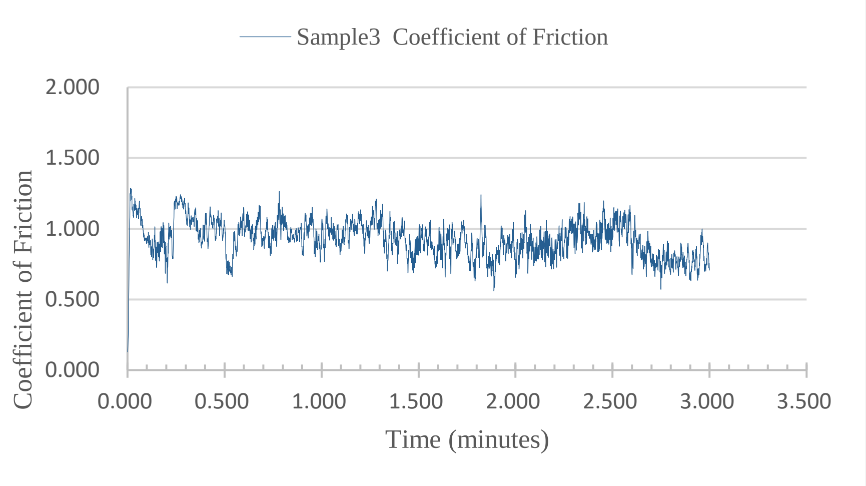

Figure 43: *COF of Al_2O_3 coating sample 3 of inner track.*

From the data of the internal track wear test as shown in Figure 11, the COF curves of the three samples are disordered lines and almost irregular. As shown in Figure 12 and 14, S1 and S3 produced a numerical change in COF close to 0.5 minutes, after which it stabilized. It is hypothesized that the films of these two samples were completely worn out at the 0.5 min time point. This rate of wear is very fast and they are not very resistant to abrasion. While S2 produced a change around 1 min, as shown in Figure 13, which is 0.5 min more than the other two groups. This reason could be due to the uneven coating.

Al_2O_3 coating materials outer track PoD wear testing：

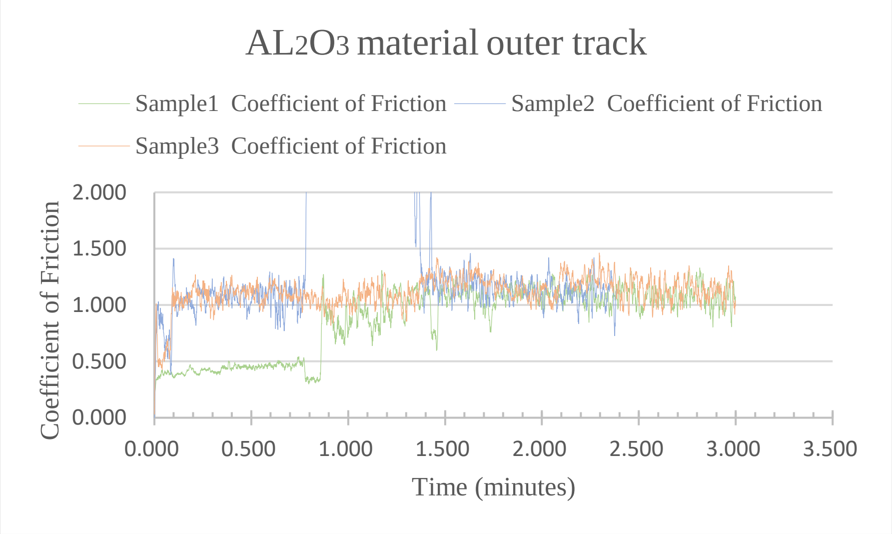

Figure 54: *COF of Al_2O_3 coating S_1、S_2、S_3 of outer track.*

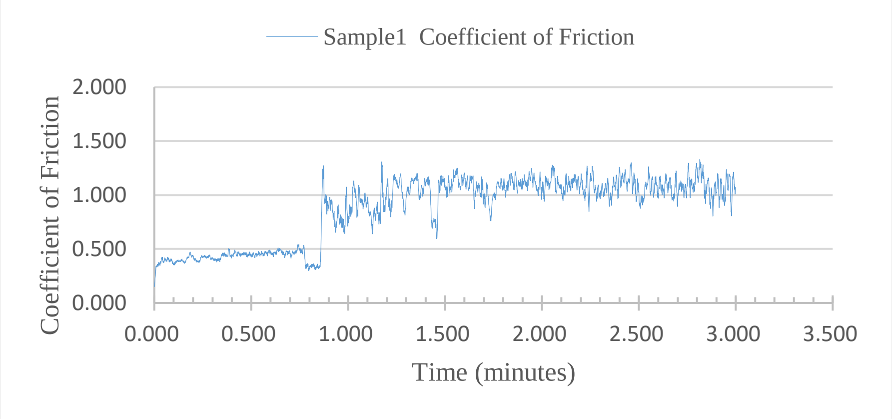

Figure 65: *COF of Al_2O_3 coating sample 1 of outer track.*

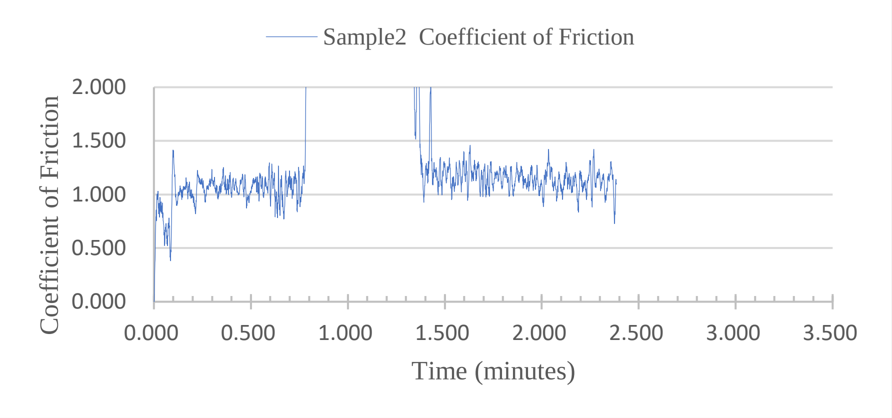

Figure 76: *COF of Al_2O_3 coating sample 2 of outer track.*

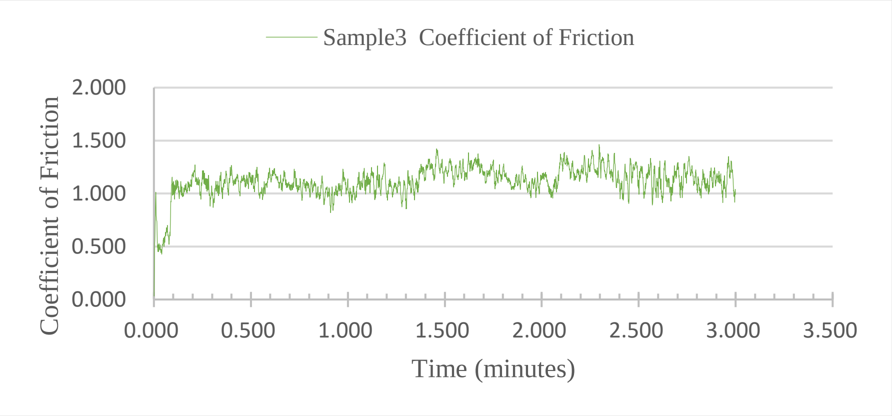

Figure 87: *COF of Al_2O_3 coating sample 3 of outer track.*

As shown in Figure 14 the results of S1 get the ideal data we pre-guessed when doing the outer track measurement. As shown in Figure 15, the COF value of S1 consistently stays around 0.5, then decreases slightly at 0.8 min then increases and stays around 1. It can be presumed that the alumina film is completely worn out at this point, and the process thereafter is the COF of the pin material and the substrate itself wearing out. The lower COF value in the first stage indicates that the surface of the sample is smoother and the friction is weaker, and the higher friction value in the later stage indicates that the friction increases, and it is maintained at around 1 because the pin and the substrate belong to the same SS.

Unfortunately, the S2 outer track test shown in Figure 16 produced an error by the system during the experiment, so the data were unreliable and discarded. In Figure 17, the coating of the outer track of S3 was completely worn out in only 0.1 min, the cause of which may be that the surface of the substrate was not flat enough, resulting in the track that the pin scratched through was not completely horizontal, which aggravated the wear.

4.2. SiO_2 sample

Silicon dioxide (SiO_2) films are one of the most used thin-film materials in modern technology, and are widely used in the fields of microelectronics, optoelectronics, optical devices, and protective coatings. It has a high hardness that makes it superior in abrasive environments. High hardness generally means that the more resistant the film is to abrasive wear and frictional wear. Dense, low porosity films resist wear more effectively. A silicon dioxide film with a smooth surface reduces the coefficient of friction, further improving wear resistance [57]. Silicon dioxide films are commonly used to coat biomedical devices, such as implants and sensors, providing wear resistance and biocompatibility [58].

SiO_2 coating materials inner track PoD wear testing：

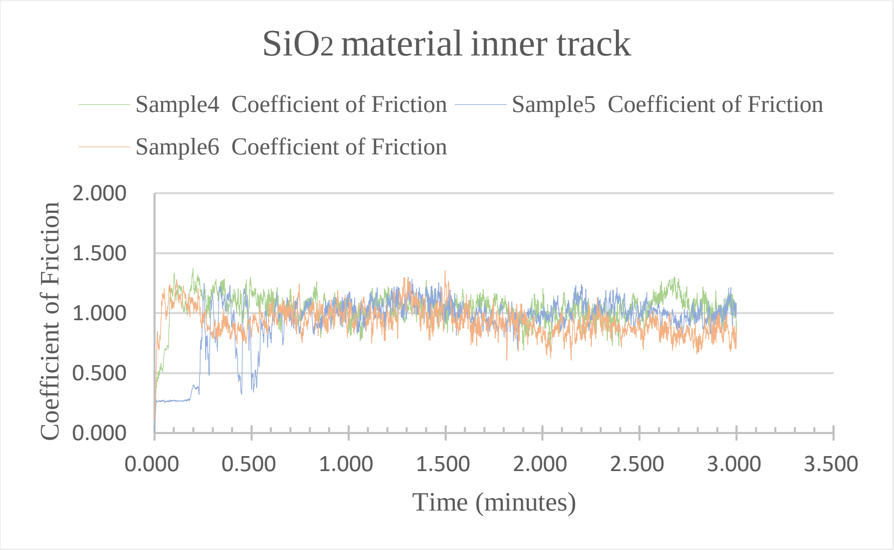

Figure 98: *COF of SiO_2 coating S_4、S_5、S_6 of inner track.*

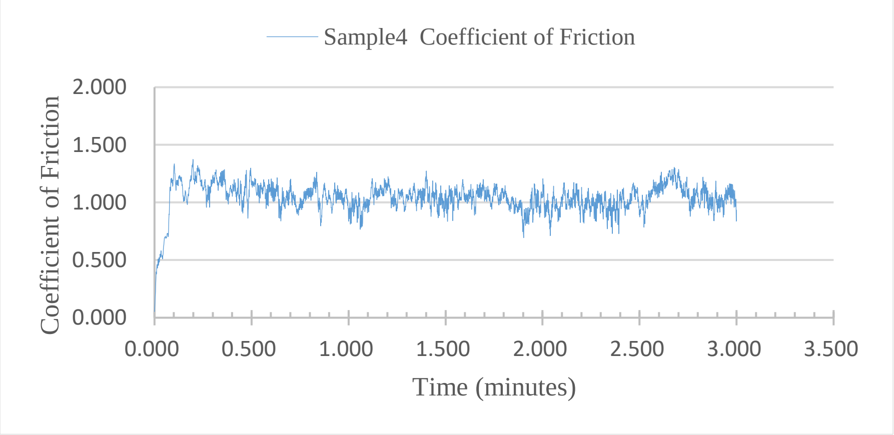

Figure 109: *COF of SiO_2 coating sample 4 of inner track.*

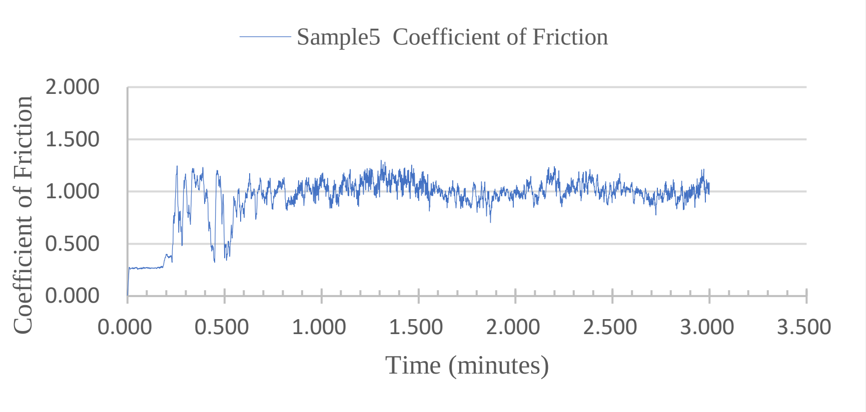

Figure 20: *COF of SiO_2 coating sample 5 of inner track.*

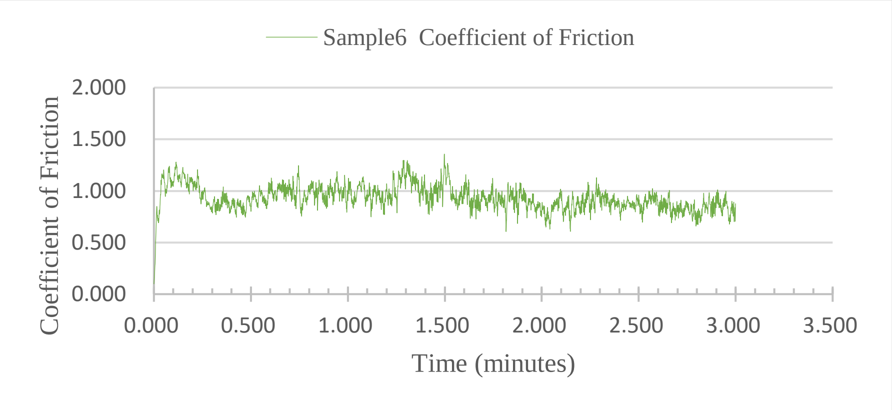

Figure 21: *COF of SiO_2 coating sample 6 of inner track.*

For the inner track wear test on the SiO_2 film, the S5 had ideal data performance, according to Figure 20, the COF stayed around 0.3 from the beginning of the test, then rose at 0.2 minutes, then continued to fluctuate dramatically, and finally stabilized at 0.6 minutes. It is hypothesized that the film began to break down at the first time point, alternating continuous friction with the film and substrate as the pin slid on the track caused the COF amplitude to change dramatically, and the film failed completely at the second time point. Two other sets of COF curves for the internal tracks shown in Figures 19 and 21 did not yield satisfactory data.

SiO_2 coating materials outer track PoD wear testing：

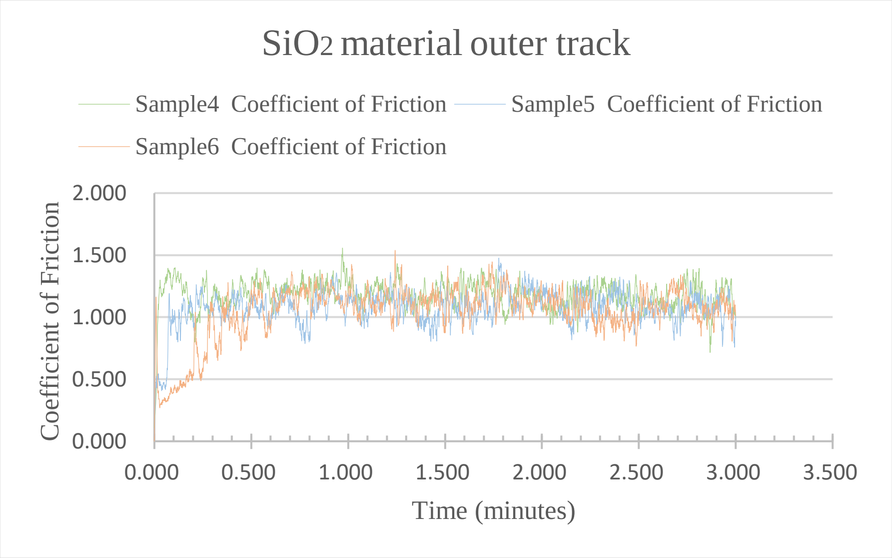

Figure 22: *COF of SiO_2 coating S_4、S_5、S_6 of outer track.*

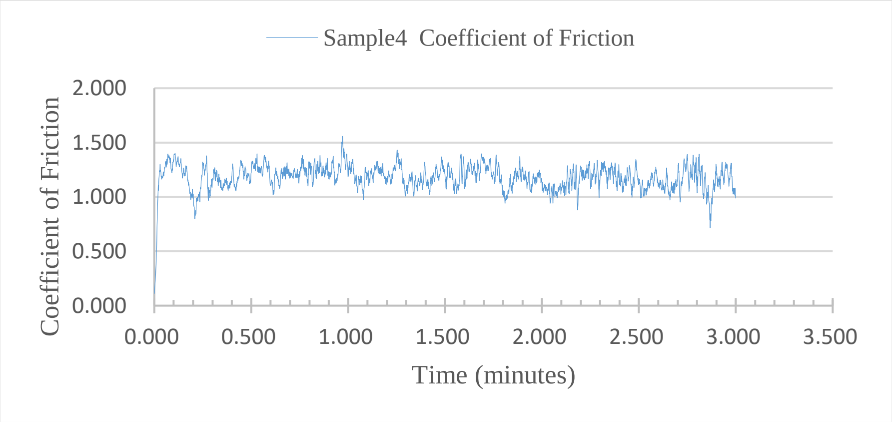

Figure 23: *COF of SiO_2 coating sample 4 of outer track.*

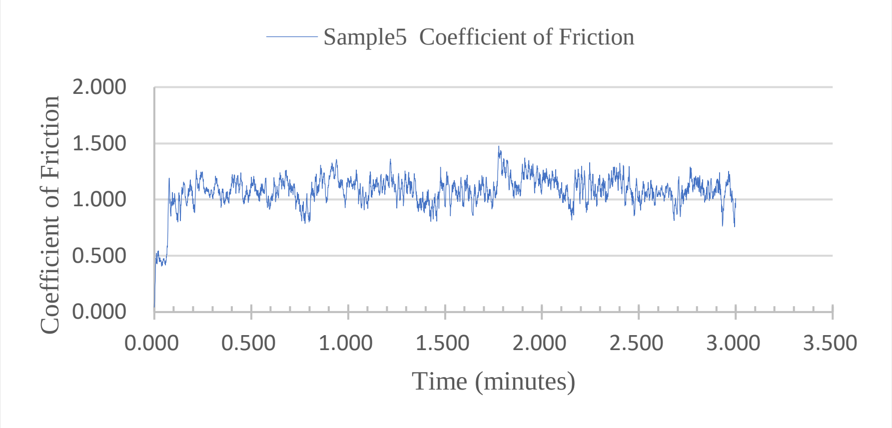

Figure 24: *COF of SiO_2 coating sample 5 of outer track.*

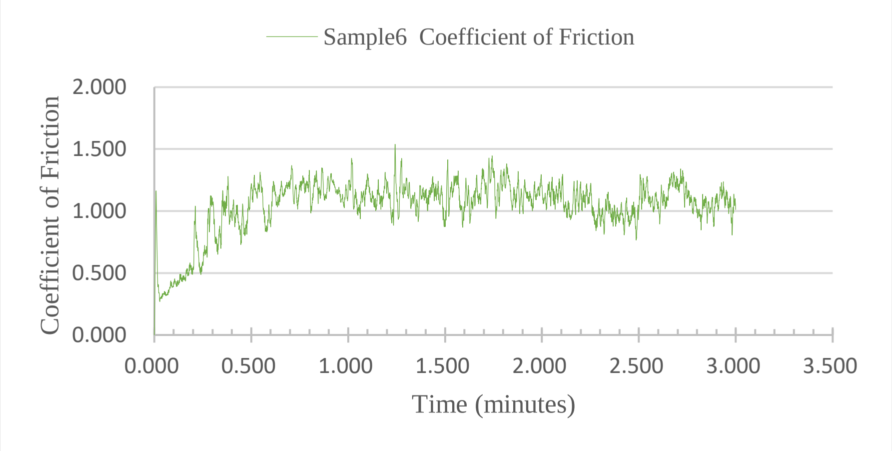

Figure 25: *COF of SiO_2 coating sample 6 of outer track.*

As shown in Figure 22, all three sets of COF values start below 0.5, and as time progresses, S4 and S5 almost immediately undergo complete wear, while S6 undergoes mixed friction (mixing of film and substrate wear) as shown in Figure 25. The final values all slightly fluctuate near 1, indicating that the films all eventually fail completely into mutual friction of the SS materials. It can be said that these three samples were not perfectly deposited near the outer edges area, the reasons for this will be discussed in detail later.

4.3.HfO_2 sample

Hafnium oxide (HfO_2) is widely used in modern electronics, especially as a transistor dielectric in microprocessors [59]. HfO_2 films typically exhibit low coefficients of friction, especially after surface treatment or lubrication, to further reduce frictional wear. ALD and plasma-enhanced atomic layer deposition (PEALD) are the two main methods for depositing high-quality HfO_2 films [60].

HFO_2 coating materials inner track PoD wear testing：

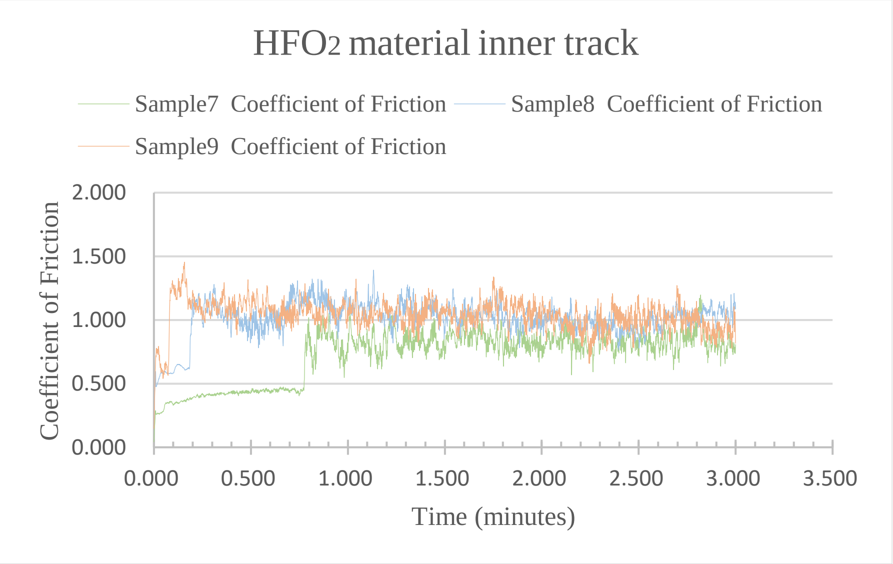

Figure 26: *COF of HfO_2 coating S_7、S_8、S_9 of inner track.*

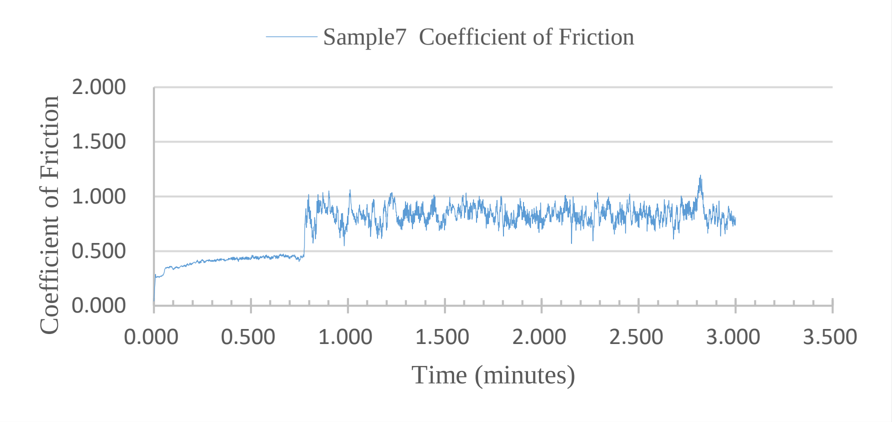

Figure 27: *COF of HFO$_2$ coating sample 7 of inner track.*

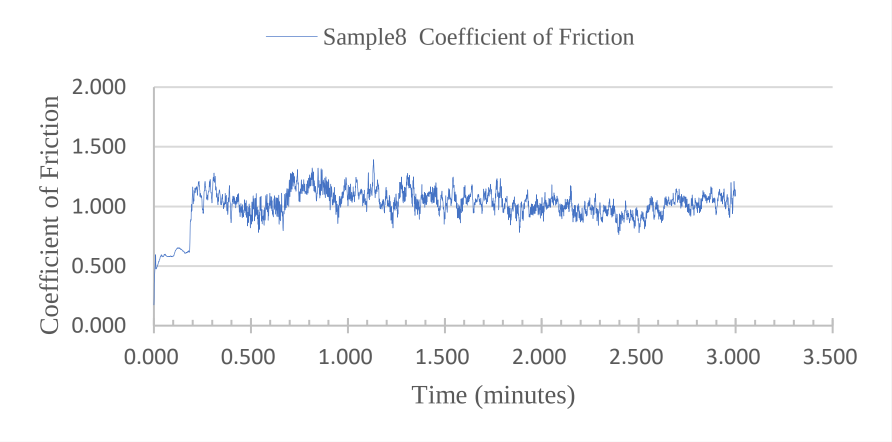

Figure 28: *COF of HFO$_2$ coating sample 8 of inner track.*

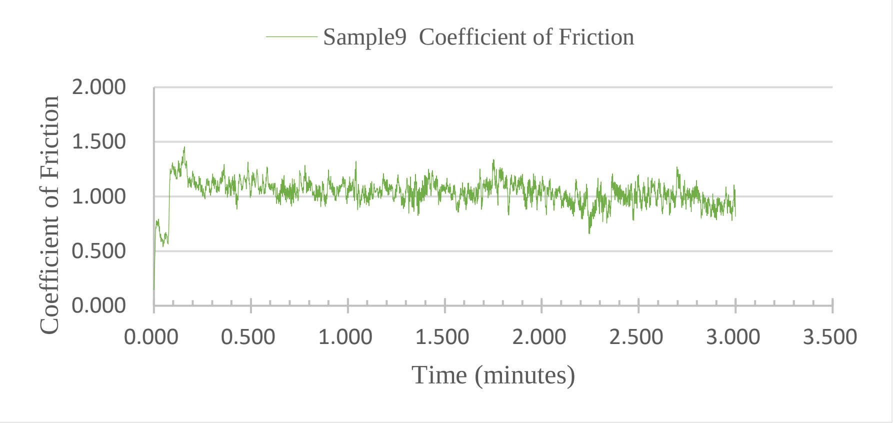

Figure 29: *COF of HFO_2 coating sample 9 of inner track.*

As far as the HfO_2 abrasion test is concerned, this is the most optimized set of experimental data among the three varied materials. As shown in Figure 26, the three samples exhibit the same characteristics, but with different time points for complete wear to occur, which may be due to tolerances in the treatment of the substrate during the material preparation process, which will be analyzed later.

HFO_2 coating materials outer track PoD wear testing:

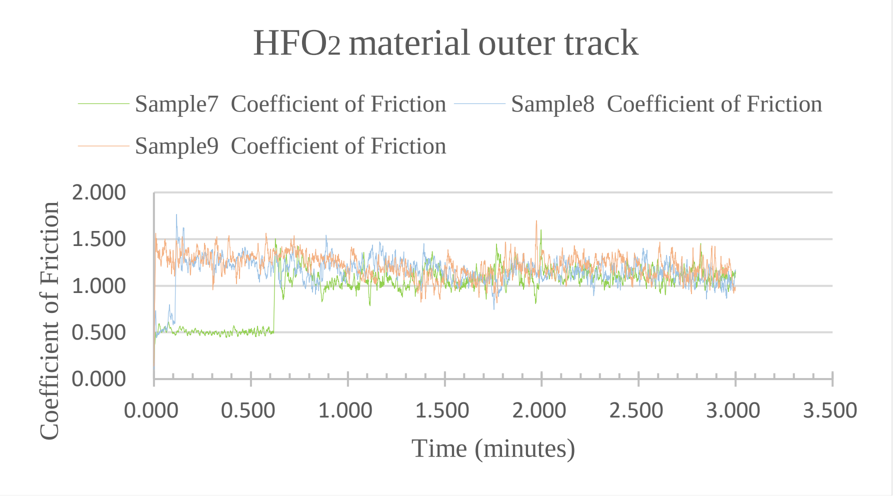

Figure 30: *COF of HfO_2 coating S_7、S_8、S_9 of outer track.*

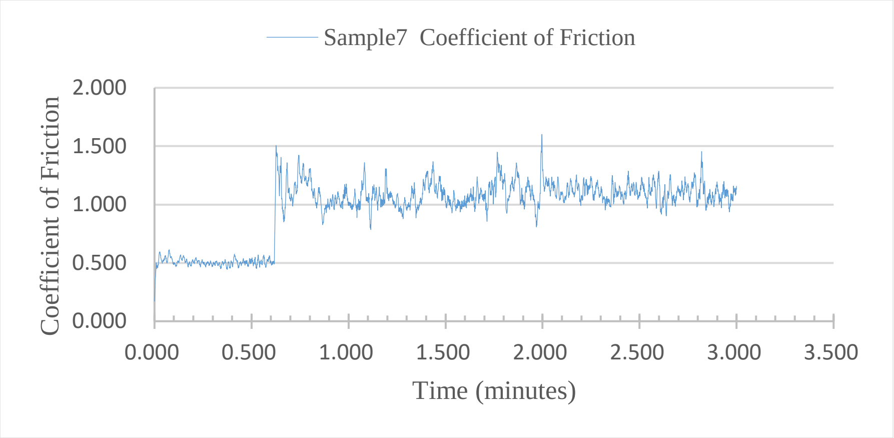

Figure 31: *COF of HFO_2 coating sample 7 of outer track.*

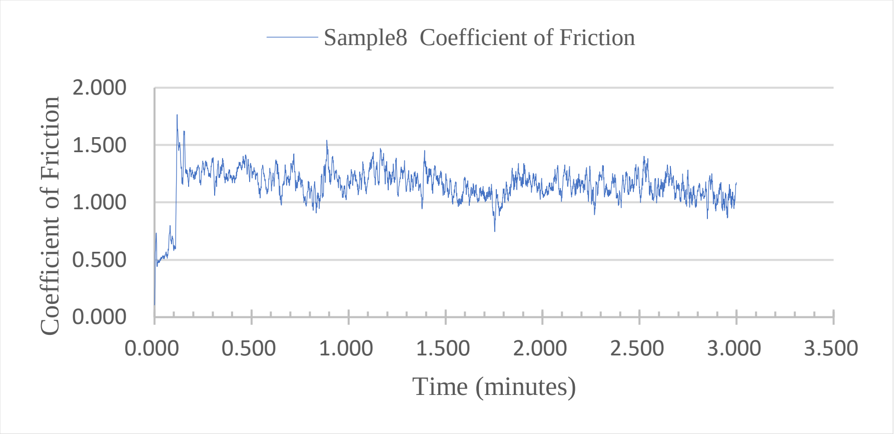

Figure 32: *COF of HFO_2 coating sample 8 of outer track.*

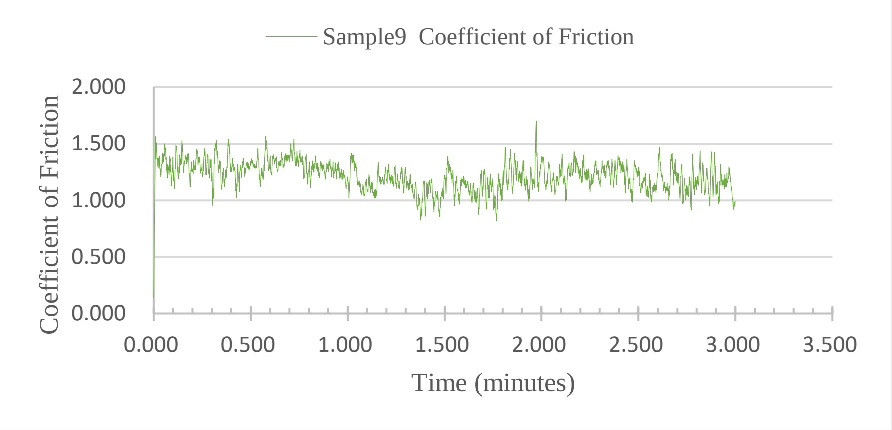

Figure 33: *COF of HFO_2 coating sample 9 of outer track.*

For S7, shown in Figures 27 and 31, wear tests for the inner and outer tracks show consistency. The inner track film wore at 0.8 minutes and the outer track at 0.7 minutes, compared to the outer track which wore faster. This demonstrates that the different areas of a piece of metal do not achieve perfect deposition consistency.

Due to time constraints and the limited number of previous papers to refer to, the reference value of the data from the experiment needs to be further confirmed. There are some unsatisfactory and confusing data in the experiment, for example, Figure 21, S6 does not show a trend change that can be speculated. The reason for this is multiple, the first may be that the samples were not prepared well enough. In fact, manually polishing a material of 50x50mm is not a simple task, it is not possible to ensure that the force is uniform throughout the plane during the polishing process, which results in a plane that is not perfectly horizontal and consistent. During the deposition process, the film thickness on the surface was not uniform, which led to unreliable COF data in the PoD wear test. Because of the unequal thickness of the film worn by the pin on the contact track, the pin is not sliding on a flat surface, and the grooves on the surface itself cause the COF to constantly float. Secondly the number of experimental samples was insufficient, 9 samples were selected for wear testing on the inner and outer tracks, totaling 18 tests. This number of tests is not enough to support the conclusion down. The insufficient number of experimental groups does not allow for many cross-comparisons to be made, and the inferences drawn are one-sided and not trustworthy enough. Table 3 lists the time that the inner and outer tracks of each sample respectively resist coating broke. For the two thin film coating material groups, Al_2O_3 and HFO_2, they were both deposited at 60 nm thickness and ALD was performed using the same machine. in this experiment HFO_2 showed better wear resistance and resisted wear generation for a longer period. The SiO_2 film is 100nm thick and its wear resistance is not as good, most of the wear occurs within 0.2min as shown in Figure 19, 21 and 24. Since there is no comparison group set up with different thicknesses of the same deposited material, it is not possible to conclude whether the film thickness influences the wear resistance of the deposited material or not.

Table 3: *Time until coating broke.*

Coating name	**Sample number**	**inner track coating broke time**	**Outer track coating broke time**
Al_2O_3	S_1	0.2min	0.9min
Al_2O_3	S_2	0.2min	0.1min
Al_2O_3	S_3	0.1min	0.1min
SiO_2	S_4	0.1min	0.2min
SiO_2	S_5	0.2min	0.1min
SiO_2	S_6	0.05min	0.2min
HFO_2	S_7	0.8min	0.7min
HFO_2	S_8	0.2min	0.1min
HFO_2	S_9	0.1min	0.05min

Here I would like to summarize the causes of the data confusion, the first point I consider to be the most important point, the quality of the sample surface is not uniform enough to be perfect, which directly causes some of the fatal errors. The COF measured by POD is very delicate, and slight differences in microstructure can cause different values. Since the surface cleaning process was not done in a clean room, there could be particles and dust left over before the sample was deposited in the reaction chamber, resulting in a non-uniform deposition surface. Secondary the lack of sufficient samples also contributed to the lack of ideal experimental data. As mentioned earlier about the failed alumina samples, I should have made one more sample to compensate for the data. However, due to time constraints and lack of materials, there was no chance to compensate for this set of errors, resulting in a lack of actual data. As the literature reference mentioned on related diamond films, the authors did nearly 20 sets of experiments for comparison and made the point that this amount of data is still not enough to strongly support the argument. In this respect my experiments are inadequate.

The third point is that there are no complement other wear testing methods, which was caused by insufficient samples, and this does not provide strong data to support the arguments. The insufficient samples resulted in the inability to conduct an effective comparison experiment to investigate the effects of different loads, speeds, film thickness and environmental conditions on the film. Lastly, lubricants are not used, the effect of lubricants on POD testing has been investigated in a majority of the papers referenced but due to the fact that I did not want to be influenced by one more factor so did not use them. This resulted in an extremely fast wear rate and the COF curves in the early stages of PoD were fine enough to make a uniform comparison.

5. Conclusion

Improving the wear resistance of thin-film coating technologies is key to ensuring their long-term stability in high-friction and high-wear environments. The currently available research suggests that the wear resistance of thin film coatings can be enhanced by optimizing material selection, process parameters and post-processing techniques. For material selection high hardness and wear-resistant materials can be selected for deposition, such as alumina (Al_2O_3), which are inherently high hardness and wear-resistant, and are suitable for manufacturing wear-resistant thin films. In addition to this the use of composite materials or multilayer structure design can improve the wear resistance of the film. For process parameter optimization the deposition temperature significantly affects the microstructure and properties of the films. The selection of precursors and precise control of the flow rate ensure that the reaction is sufficient to form a dense and homogeneous film. This is essential to achieve coatings with high hardness and low coefficient of friction. Apart from this, the deposition cycle is also vital to improve the wear resistance of the film. Once the deposited thin film material has been prepared, the wear resistance can also be improved by heat treatment and ion implantation techniques. The combination of these methods not only improves the mechanical properties and wear resistance of the film, but also expands its application in high stress and high abrasion environments. Further research into these methods and the development of new materials and processes will further enhance the wear resistance and overall performance of thin film coatings.

For wear experiments using the POD methodology, surface quality is important, the number of samples needs to be sufficient, and such tests cannot be used alone and need to be supplemented by wear mechanism tests and compositional analysis. The use of multiple wear test methods can lead to more comprehensive results.

There are various applications of thin film coating technology, but the ALD technology used in this paper has some limitations due to its high cost and long processing time, which can be explored as a future research direction. Currently the main application is in the field of electronic components, in microelectronics and optoelectronics components, abrasion-resistant coatings can provide excellent protection against surface wear and corrosion of the components to improve the reliability and stability of the equipment. Meanwhile, its anti-wear performance improvement has also attracted attention, which hides a huge potential.

The experiments conducted in this paper are not sufficient for this topic, and more wear tests need to be completed to simulate the wear that occurs to improve the understanding of the wear resistance of thin film deposition technology. There are several ways to improve the experiments that could be considered for future research. Firstly, the process of polishing could be improved by selecting other shapes that are easier to process but more difficult to machine, such as round shape, as well as preparing the appropriate tools for fully automated polishing to achieve a better surface flatness and roughness. This will greatly improve the reliability of the data. Secondly, the same deposition machine can be used to prepare multiple samples with different thicknesses and materials to conduct data analysis, so that the data obtained will be of greater reference value. Future research direction can compare the abrasion resistance of the same coating material with different thickness and the abrasion resistance of different coating materials with the same thickness. Thirdly, different pin shapes can be changed to study the best performance for the wear resistance test. This is a very small experimental study, but the depth of wear resistance for thin film coating is not yet explored enough. There is a huge potential for research into wear resistance that could bring considerable commercial and industrial value.

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