Review: Physical Vapor Deposition (PVD) Sputtering Process for the production of thin metallic films.

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Abstract: Physical Vapor Deposition (PVD) via sputtering is a versatile thin-film coating technology with broad industrial applications, including aerospace, automotive, cutting tools, medical devices, optics, and textiles. This review presents the fundamental principles of sputtering, where target atoms are ejected by ion bombardment in a vacuum, sometimes with reactive gases, forming adherent metallic or compound films. It details plasma generation, ion bombardment mechanisms, and magnetron sputtering configurations—balanced, unbalanced, and rotatable—highlighting their impact on deposition rate, ion flux, and heat sensitivity. Methodological stages are addressed, including substrate preparation (mechanical, chemical), ion etching for oxide removal, and deposition parameter optimization (target type and purity, temperature, pressure, gas flow, bias voltage). Film performance is analyzed in terms of residual stress, microstructure, corrosion resistance, and wettability, with emphasis on how deposition conditions influence compactness, porosity, and durability. Growth defects, such as particulates and nodules, are examined regarding origins, impacts, and mitigation strategies. Process advancements are discussed, notably High-Power Impulse Magnetron Sputtering (HiPIMS), Deep Oscillation Magnetron Sputtering (DOMS), and hybrid systems combining sputtering with arc evaporation or ECR plasma, enabling denser films with improved adhesion and reduced defects. Industrial aspects include energy efficiency, cost optimization, and the use of Computational Fluid Dynamics (CFD) for reactor design and coating uniformity. Concluding, PVD sputtering emerges as a key and evolving technology, offering precise control over film properties, enhanced performance, and sustainable potential, positioning it as a superior alternative to conventional coating methods.

Keywords: PVD. Sputtering. Thin films. Metallic films. Coating.

1. Introduction

Physical Vapor Deposition (PVD) is a coating process in which thin films are deposited by condensing a vaporized form of the desired material onto the substrate, conducted in a vacuum [1]. This technique has opened up new possibilities in the use of materials, with applications ranging from the aerospace and automotive industries, cutting tools, medical devices, optics, and even textiles[1]. Its diverse applications are due to the ability to control film properties, such as hardness, wear resistance, conductivity, and optical properties [2].

Sputtering, or cathodic spraying, is one of the main PVD methods [3]. Known for decades, it is recognized as a flexible, reliable, and effective coating method. Its evolution has overcome the disadvantages of conventional DC (direct current) discharge systems, such as low deposition rates, by allowing a reduction in operating pressure while maintaining the energy of the sprayed species, often resulting in improved film properties [4]. This article aims to provide an in-depth technical review of the PVD sputtering process for the deposition of thin metal films, addressing its principles, methodology, performance aspects, challenges, and future prospects.

2. Fundamentals of PVD Sputtering

The basic principle of PVD involves the vaporization and subsequent condensation of a coating material on a surface to be coated. In the sputtering method, the solid coating material (target) is evaporated by ion bombardment[1]. The atoms are removed from the target by energetic means and diffuse within a vacuum chamber until they reach a solid surface, where condensation is likely [2]. At the same time, a reactive gas can be introduced, forming a compound with the mixed metal vapor of the target components present in the chamber, and deposited on the substrate as a thin film with a highly adherent coating [1].

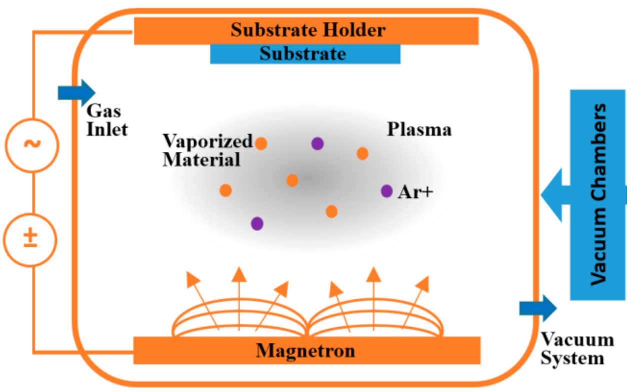

Figure 1: Schematic drawing of the PVD process - Magnetron Sputtering [8]. (Adjusted by author)

2.1. Plasma Generation and Ion Bombardment

The sputtering process is inherently dependent on plasma formation. The working gas, usually a noble gas such as argon, is ionized to create the plasma. The generated ions bombard the target (cathode), ejecting atoms that will form the film[4]. The initial kinetic energy of the sputtered atoms is typically a few eV. Energetic particle bombardment during film growth is crucial, as it can influence adsorption, desorption, nucleation, and diffusion on the substrate surface [5]. The ideal particle energy for activating surface processes without creating volumetric defects for Si, for example, is between [10] and 30 eV. Energies above 30 eV can introduce defects into the film.

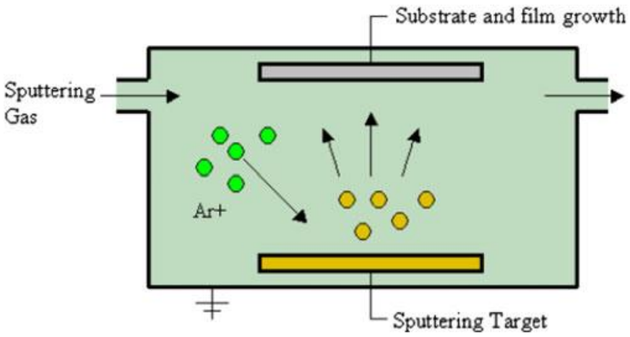

Figure 2: Schematic drawing of the kinetics of the PVD Magnetron Sputtering process [1]

2.2. Magnetron Sputtering Settings

Magnetron sputtering is an evolution of DC discharge, incorporating magnetic fields to confine electrons near the target surface. This confinement increases plasma density and, consequently, the sputtering rate [4].

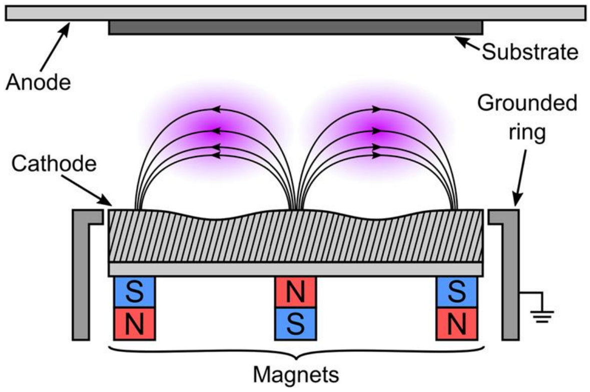

Figure 3: Schematic drawing of the PVD process, cathodic sputtering [4]

- **Balanced Magnetrons:** In this configuration, the magnetic fluxes through the outer pole faces and the inner pole face are equal. The plasma is confined predominantly in front of the target, resulting in low ion incidence on the substrate. This is advantageous for deposition on heat-sensitive substrates [4].
- **Unbalanced Magnetrons (UM):** Designed to increase the ion flux to the substrate. This is achieved by strengthening or weakening the magnetic flux through one of the poles, which causes the magnetic field lines to extend toward the substrate [4]. This significantly increases the ionic current density in the vicinity of the substrate, allowing the energy of the ions bombarding the substrate during film growth to be adjusted through substrate bias[6] UM systems are popular for enhanced ionization.
- **Rotatable Magnetrons:** These cylindrical targets can rotate during sputtering, which leads to more uniform target erosion (up to 90% target utilization) and extended target life [7]. They are essential for large-area coating [4].

3. Methodology: Stages of the Sputtering Process

- Obtaining high-quality thin films by sputtering involves a set of well-defined steps:

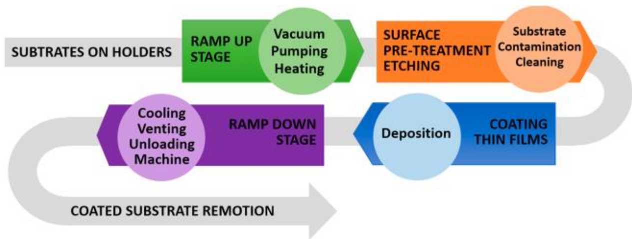

Figure 4: Phases of the PVD process [8]

3.1. Substrate Surface Preparation

Thorough substrate surface preparation is essential to ensure good film adhesion. This typically includes:

- **Mechanical pretreatment:** Sanding and polishing to remove burrs and level the surface [3]. In non-homogeneous materials, such as tool steels, irregularities such as craters and protuberances may appear in harder or softer inclusions [9].
- **Chemical wet cleaning:** Samples are chemically degreased (e.g., in sodium hydroxide and sodium cyanide solution),

washed, and dried (e.g., in a centrifuge and oven at 90°C) [3]. Demagnetization of steel substrates and the use of ultrasonic cleaning are crucial to reduce foreign particles [4].

3.2. Ion Etching

After wet cleaning, the parts are transferred to the vacuum chamber where ion blasting takes place. This step is crucial for removing impurities and native oxides and improving the bond strength between the coating and the substrate [8].

- Ion blasting can, however, induce irregularities on the substrate surface if the material is composed of different phases with different blasting rates [9]
- There is a risk of **backscattering** and **redeposition** of sprayed material back to the surface, especially at high pressures.
- **Cross-contamination** of the substrate by batch material during ion blasting can be avoided by covering the target with movable shutters [9].

3.3. Coating Deposition by Sputtering

Deposition occurs in a vacuum chamber, where specific parameters are controlled to form the film:

- **Process Parameters:** The type of target (e.g., Chromium, Copper), its purity (e.g., 99.99% Cr, 99.9% Cu) [10], the target-substrate distance (e.g., 150 mm), voltage (e.g., 1000V), amperage (e.g., 25A), the working gas (typically argon), its flow rate (e.g., 80 sccm), and the pressure (e.g., 6x10^-4 mbar) are critical [3].
- **Substrate Temperature:** The deposition temperature in sputtering is generally lower (350–600 °C) than in CVD (750–1150 °C), which is an advantage for heat-sensitive materials [8]. Substrate temperature is an important parameter affecting the structure and properties of the film [5,6].
- **Deposition Rate:** Although traditionally lower than evaporation, the deposition rate can be optimized through adjustments in gas flow, bias voltage, and external magnetic fields [8].
- **Adhesion:** Film adhesion in sputtering is generally high compared to evaporation [8]. Adhesion strength is influenced by the deposition technique and temperature; higher temperatures can promote the transfer and diffusion of atoms at the interface, improving adhesion [1].
- **Vacuum and Pressure:** PVD processes operate in a vacuum to minimize unwanted reactions with free space and to easily control film composition. Lower pressures lead to fewer collisions and less efficient energy transfer, while higher pressures result in more frequent collisions and more uniform temperatures. In reactive sputtering, pressure significantly affects deposition

rate, discharge voltage, and film composition [2].

- **Target Material and Substrate Conditions:**
  - The choice of target material and its properties (e.g., purity) influence the sputter yield and potential for defect formation.
  - Substrate surface energy, nucleation sites, and atomic structure play a crucial role in coating deposition and growth [2].
  - Substrate irregularities (scratches, pits, foreign particles) act as "seeds" for growth defects due to the shadowing effect inherent in line-of-sight deposition
- **Gas Type and Flow:** Inert gases like argon are typically used to generate plasma and bombard the target. Reactive gases (e.g., nitrogen, oxygen) are introduced to form compounds in the film (e.g., nitrides, oxides), significantly influencing microstructure and mechanical properties. Gas flow control is crucial in reactive sputtering to prevent target poisoning [2].

4. Comments and Analysis

The properties of thin metal films deposited by sputtering are highly influenced by process conditions, resulting in unique characteristics for various applications.

4.1. Film Properties and Microstructure

- **Stress and Microstructure:** Residual stress in films is a critical factor, affecting the design, processing, and service life of advanced materials [11]. Energetic particle bombardment during film growth can strongly modify stress [11]. Films sputtered at low pressures tend to exhibit compressive stress, while at higher pressures, due to the thermalization of sputtered atoms, the stress can become tensile [6]. Compressive stress and film density increase with increasing ion bombardment [6,11]. Techniques such as X-ray diffraction (XRD) and the focused ion beam method (FIB-FID) are used to measure stress [11]. Films in compression tend to be compact and shiny, with microstructures of dense small crystallites, while films in tension are porous and opaque.
- **Corrosion Resistance:** Corrosion resistance is strongly influenced by the deposition conditions and microstructure of the coating [8]. Sputtered chrome coatings can exhibit electrochemical performance equivalent to that obtained by conventional electrodeposition, representing a sustainable alternative [3]. The porosity of metal films is often a limiting factor in the use of sputtering for

anti-corrosion applications. The application of basecoats and topcoats, especially thermally cured ones, has been shown to significantly increase corrosion resistance by acting as a physical barrier [3]. For example, an acrylic urethane-based varnish catalyzed with aliphatic isocyanate (thermal curing) exhibited better corrosion resistance and lower porosity than a UV-cured varnish.

- **Wettability:** Materials with a greater hydrophobic character are typically more resistant to corrosion in aqueous environments [3].

4.2. Growth Defects

Growth defects are microscopic imperfections in the coating microstructure that can degrade quality and even cause catastrophic failure [9].

- **Origin:** They can be formed by the coating of topographic imperfections (pits, asperities) on the substrate surface or by foreign particles of various origins (dust, debris, scale) [9]. Foreign particles can be residues from the cleaning process or generated during ion blasting and deposition. In magnetron sputtering, the formation of specific particles (scale) can be caused by flaking of cones formed on the target track, flaking of redeposited nodules from the target surface, or by arcing [9].
- **Impact:** They affect the quality of optical coatings and thin layers for semiconductor devices, as well as their resistance to wear, corrosion, and oxidation. They can facilitate crack initiation, leading to premature coating failure [9] localized oxidation can occur in pores or detached defects.
- **Minimization:** High-quality wet cleaning procedures, a clean processing environment, and proper handling are essential to reduce the density of harmful particles. Ion beam sputter deposition produces the lowest defect density of all PVD techniques [9].

4.3. Process Advances and Optimization

Continuous research has led to the development of advanced techniques to improve sputtering:

- **High Power Impulse Magnetron Sputtering (HiPIMS):** Uses high peak power density at a low duty cycle to generate a highly ionized stream of sputtered material. This results in significant improvements in coating structure, properties, and adhesion compared to conventional MS. [8] Increases in deposition rate have also been observed, especially with the application of external magnetic fields.
- **Deep Oscillation Magnetron Sputtering (DOMS):** A variant of HiPIMS that allows the deposition of dense, compact films without the need for high-energy particle bombardment, reducing the atomic shadowing effect [8].

- **Hybrid Systems:** Combining sputtering with other techniques, such as cathodic arc evaporation (Arc-PVD/Arc-MS) or ECR (Electron Cyclotron Resonance) plasma, can produce good quality films with improved adhesion and reduced defects [7]. HiPIMS can also be combined with cathodic arc plasma deposition and ion plating [8].

4.4. Industrial Considerations and Sustainability

- **Energy Efficiency:** The PVD process (specifically MS) consumes less total energy compared to CVD (primarily due to lower operating temperatures), although the coating step itself is more energy-intensive for PVD [8]. Waste heat recovery and recycling of target materials are ways to reduce energy consumption.
- **Cost Optimization:** The length of the cleaning process and machine downtime are disadvantages for the industry. Optimizing process parameters is necessary to reduce production times and costs. [8]
- **CFD Simulation:** Computational Fluid Dynamics (CFD) modeling has been used to analyze gas flow and its mixing behavior within the reactor chamber. This helps optimize reactor geometry and process parameters, contributing to more homogeneous coating thickness and reduced production time [8].

5. Conclusion

PVD sputtering has established itself as a fundamental and constantly evolving technology for the deposition of thin metal films, offering a wide range of applications due to its flexibility and control over material properties. Advanced magnetron sputtering techniques, such as HiPIMS and DOMS, along with hybrid systems, continue to advance the ability to produce films with improved microstructures and properties, including superior adhesion, increased corrosion resistance, and precise control over residual stress.

Despite the challenges of minimizing growth defects and optimizing energy efficiency, advances in understanding plasma phenomena and the increasing use of numerical simulations (CFD and FEM) are paving the way for even more efficient, sustainable, and economically viable coating processes. PVD sputtering is not only a promising alternative to traditional methods, but also a technologically superior solution that will continue to be an area of intense research and development, driving innovation across various industries.

6. References

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1. Fundamentals of PVD Sputtering

The basic principle of PVD involves the vaporization and subsequent condensation of a coating material on a surface to be coated. In the sputtering method, the solid coating material (target) is evaporated by ion bombardment1. The atoms are removed from the target by energetic means and diffuse within a vacuum chamber until they reach a solid surface, where condensation is likely2. At the same time, a reactive gas can be introduced, forming a compound with the mixed metal vapor of the target components present in the chamber, and deposited on the substrate as a thin film with a highly adherent coating1. **Fundamentals of PVD Sputtering**

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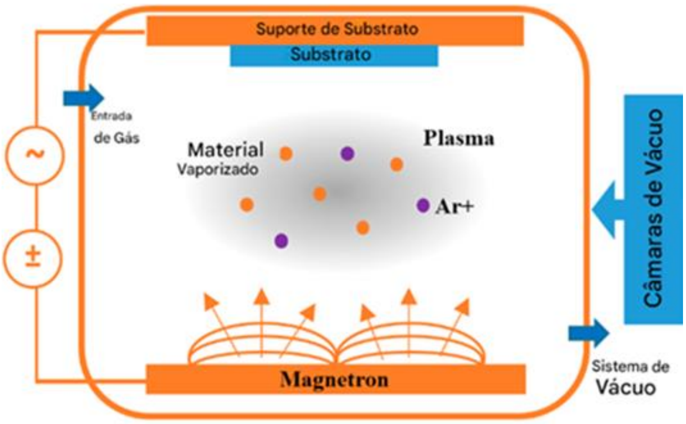

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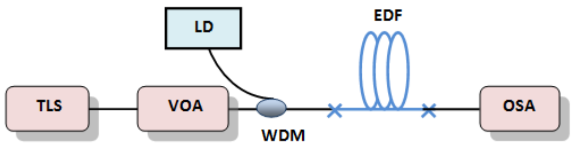
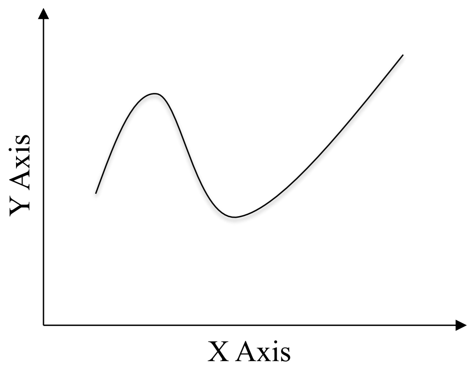


LD, lase diode. OSA, optical spectrum analyzer TLS, tunable laser source. VOA, Abbreviations or legends should be separated by a dot.

Figure 2. Biochip Reader integrated circuit (details).

Graphic 1. Diagram example.



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Acknowledgement

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