



ORIGINAL RESEARCH ARTICLE

Effect of Angular Deposition of Ti Interlayer and Ag-Pd-Cu Final Layer on MEMS Surfaces Adhesion

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The adhesion force is a critical factor in MEMS (micro-electro-mechanical systems) technology, especially in the micro-assembly process using microgrippers. Given the diminutive size and weight of MEMS devices, the contact force between the surfaces of MEMS components can lead to several problematic scenarios during the assembly procedure, such as the undesirable adhesion of the MEMS surface components. This can negatively affect the assembly procedure and result in incorrect positioning of microparts. Therefore, reducing the adhesion force is essential for enhancing micro-assembly. This research aimed to explore the impact of angularization of the interstitial layer and the last layer on surface morphology, surface roughness parameters, and adhesion using the glancing angle deposition method. To achieve this, six samples were simultaneously layered with varying experimental angles of 0, 30, 45, 60, 75, and 85 degrees for the deposition of thin films. These films comprised a titanium interstitial layer and the main layer Ag-Pd (0.9 wt.%)–Cu (1.0 wt.%). Following the glancing deposition, the morphology of the films was examined using SEM, while the layer roughness and adhesion force were determined using AFM. The findings of this study revealed that increasing the angle of the referenced layer resulted in an initial increase and subsequent decrease in both the grain height and surface roughness of the silver alloy. The tests showed that the angle of 85 degrees corresponded to the lowest level of adhesion observed in both experiments. In conclusion, it can be inferred that adjusting the angulation of the Ti interstitial layer in relation to the angulation of the last layer of the silver alloy results in improved grain size distribution, increased grain height, and enhanced regularity in surface adhesion behavior.

Keywords adhesion force, glancing angle deposition method, MEMS, surface roughness

1. Introduction

In recent years, there has been an increasing scholarly focus on investigating the interaction between various objects regarding the control of adhesion forces. This research area holds significant relevance and applicability in micro-electro-mechanical systems (MEMS), particularly in relation to micron clamps employed in micro-assembly processes. Micron clamps play a crucial role in the manipulation and release of micron particles (Ref 1, 2). In situations where two surfaces possess asperities, as opposed to being perfectly flat, there is typically reduced contact between them, resulting in a decrease in surface adhesion. The existence of surface roughness commonly leads to a reduction in the adhesion force between two contacting surfaces.

The surface roughness and subsequent adhesion are also influenced by factors such as angular deposition, coating thickness, and coating type. Therefore, it is of utmost importance to measure the adhesion force after surface decomposition.

Balavana et al. examined the impact of polymer deposition on a silicon sub-layer or micron clamp. While the polymer deposition method employed was straightforward and economically viable, the findings from atomic force microscopy (AFM) analysis revealed that its suitability for MEMS applications is compromised because of non-uniform thickness (Ref 3, 4). In the research conducted by Hamed et al., the spin method was employed for the deposition of polymeric coatings in thin-layer applications. This technique entails the application of a small quantity of liquid resin onto a rotating substrate at a high speed, typically around 3000 rpm. The centrifugal force generated during rotation facilitates the uniform distribution of the resin across the substrate's surface, resulting in the formation of a thin coating layer. The final thickness of the deposited layer and its subsequent properties are influenced by various factors. These include the inherent characteristics of the resin, such as viscosity, drying rate, percentage of dissolved solids, and surface tension. The selection parameters for the rotation process, including rotational speed, acceleration, and rotation time, also play a significant role in determining the thickness and properties of the resulting coating layer (Ref 5). Katanen et al. (Ref 6) examined the impact of various coating parameters, including thickness, surface roughness, and layer density, on a silica substrate coated with titanium oxide. The coating process was carried out utilizing the atomic layer deposition (ALD) method, and the adhesion forces were evaluated through rough surface modeling and atomic force microscopy (AFM). The objective of the investigation was to analyze how these coating parameters influence the adhesion properties of the titanium oxide coating on the silica substrate. Salehi et al. employed a novel technique for the fabrication of a compound sputtering target to analyze surface

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roughness and adhesion force both theoretically and numerically in micro- electro-mechanical systems (MEMS) applications. Their research effectively shows that Ag-Au compound coatings can be produced with precise control over thickness, resulting in notable alterations in adhesion properties. The findings highlight the applicability of the Rabinovich model in predicting adhesion behavior, showing that this innovative method holds significant promise for applications where reduced adhesion is helpful, improving the functionality of microgrippers (Ref 7). Madarakamuma et al. (Ref 8) conducted a comprehensive examination of the surface and optical properties of aluminum thin layers, focusing on the key parameters associated with layer deposition. To investigate the surface characteristics, magnetron sputtering was used to deposit thin aluminum layers onto glass substrates, with varying deposition rates and substrate temperatures. Through the use of atomic force microscopy (AFM) images, the researchers showed that, for layers with a fixed layer pitch and a thickness of 100 nm, the roughness of the layers exhibited an upward trend as the temperature increased up to 450 K. Jong et al. (Ref 9) conducted an experiment whereby silver layers with a thickness of 250 nm were fabricated on a glass substrate using the thermal evaporation method. These layers were subsequently subjected to annealing in ambient air for a duration of 1 hour, within a temperature range of 200 to 400 °C. The process of annealing led to an enhancement in the crystallinity of the layers, coupled with oxidation of the surface when exposed to temperatures exceeding 300 °C. The reflectivity of the silver layers exhibited a decline upon annealing at temperatures higher than 250 °C. Notably, the layer that underwent annealing at 250 °C displayed the highest degree of surface roughness. In a study conducted by Jang (Ref 10), thin silver layers were deposited onto a glass substrate using the magnetron sputtering technique in an argon gas environment. The research aimed to examine the influence of substrate temperature (ranging from room temperature to 250 °C) and layer thickness on the formation mechanism of the texture and preferential orientation of the silver layers. Notably, an increase in layer thickness resulted in grain growth within the layers. Consequently, the grain size and peak intensity exhibited an increment as the layer thickness was augmented.

Liu et al. have conducted a study focusing on exploring the impact of film thickness and annealing temperature on the structural, electrical, and mechanical properties of cobalt-iron-dysprosium (Co₄₀Fe₄₀Dy₂₀) thin films that were deposited on Si(100) substrates. Through x-ray diffraction (XRD) analysis, it was determined that the crystalline microstructure comprised dysprosium oxide (Dy₂O₃) and cobalt oxide (Co₂O₃) phases. A significant finding is that the utilization of annealing treatments led to a notable reduction in surface energy. This reduction can be attributed to the alleviation of residual stress and the enhancement of atomic arrangement, ultimately resulting in a film structure that is more stable. The mechanical characterization showed a decrease in film hardness as thickness increased, suggesting a correlation between thickness and mechanical integrity. The Co₄₀Fe₄₀Dy₂₀ films exhibited outstanding mechanical properties, characterized by improved durability and reduced surface energy. These enhancements, coupled with exceptional magnetic characteristics, demonstrate the films' potential for various applications (Ref 11).

Ghazavi et al. investigated the effect of chromium layer thickness on the thermal stability, adhesion, and agglomeration of Cr/Ag/Cr sandwich layers in micro-mechanical systems. Their research concluded that Cr/Ag/Cr sandwich films play a

crucial role in maintaining thermal stability and preventing the agglomeration of silver layers at annealing temperatures. Specifically, they showed that a chromium thickness of 15 nm provides optimal conditions, resulting in the least agglomeration, the lowest electrical resistance, and the highest thermal stability, which significantly enhances the surface properties (Ref 12). Kapakli et al. (Ref 13) deposited thin layers of Ag on the glass substrate by magnetron sputtering at room temperature. The results showed that the roughness of the silver layers increases as the thickness increases.

Kolahdoozan et al. investigate the effects of silver and gold metal coatings with varying thicknesses on the adhesion force of silicon surfaces. Additionally, the study examines the influence of grain height and radius, as described by the Rabinovich equation, on the adhesion force. Silver and gold coatings of 10, 120, and 500 nm were deposited on silicon <100> substrates using magnetron sputtering. Adhesion forces were determined through Rabinovitch modeling and experimental methods, with atomic force microscopy used to get the experimental data for the modeling. The results show that coatings of both metals, regardless of thickness, reduced the adhesion force compared to the uncoated silicon surface. Importantly, the gold coatings exhibited lower adhesion forces compared to the silver coatings (Ref 14). Purcar et al. employed a novel method for manufacturing a compound sputtering target. Ag-Au compound coatings with thicknesses of 120 and 500 nm were fabricated on the surface of silicon substrates. Single layers of Ag and Au with the same thicknesses were deposited. Adhesion force, surface roughness, and morphology of thin films were measured by Rabinovich, Rumpf, and AFM. The investigations regarding the thickness effect show that increasing the thickness from 120 to 500 leads to a rise in surface roughness and a drop in adhesion force in single and compound films (Ref 15). Soleimani et al. investigated the effects of various coating materials, deposition, and annealing temperatures on the surface roughness and morphology of a micro gripper substrate, to decrease the adhesion force between the micro gripper substrate and MEMS device. The DC sputtering magnetron method was used for coating deposition. The study found that the height and shape of hillocks on the substrate surface directly depend on the deposition and preheating temperature ranges. As the deposition and preheating temperature were increased, more hillocks grew on the surface, and the adhesion force consequently increased (Ref 16).

Jan and his colleagues conducted research focused on the utilization of an angular deposition approach to shape thin layers of chromium using the DC magnetron sputtering method. By varying the angle of incidence from zero to 50 degrees, the researchers aimed to investigate the influence of the inclined structure of the thin chromium layers on the microstructure and surface properties of the resulting coatings (Ref 17). Dravaux et al. investigated the microstructure of Ti layers using the angular deposition and magnetron sputtering techniques. Various types of microstructures were examined as a function of experimental conditions. The influence of the deposition parameters, such as substrate temperature, deposition thickness, gas pressure, and substrate tilt, on the got structures was investigated in this study (Ref 18). The glancing angle deposition (GLAD) method, first reported in 1959 by Hoffman et al. (Ref 19), is a powerful tool for modifying the microstructure and properties of thin films deposited by PVD.

GLAD is a key method for tailoring many of the physico-chemical properties of thin films, as a wide range of morphologies can be tuned simply by changing the direction of the particle flux. The mechanism of glancing angle deposition is shown in Fig. 1. The incidence angle α controls the tilt angle β of the columns. β is the angle between the column axis and the normal to the underlying layer, and it affects the shadowing, which alters the porosity of the films. The incidence angle α and the column angle β are not equal because of shadowing effects (Ref 20). β is always smaller than α , and it increases as α increases. The columns orient themselves toward the vapor source.

Lintymer et al. used the GLAD technique to deposit chromium thin films via DC magnetron sputtering. By adjusting the operating conditions to favor a columnar microstructure and controlling the orientation of the substrate relative to the impinging vapor flux, an inclined columnar film microstructure was produced. The results show that the mechanical and electrical properties of the films depended highly on their morphology. The change in observed behaviors at an included angle (α) between 20 and 30 degrees is attributed to the combined effect of decreasing influence from disoriented column growth and increasing influence from column boundaries. Mechanical and electrical properties were improved for $\alpha = 20$ degrees, but the use of GLAD is limited because of non-homogeneity in film thickness. The thickness homogeneity can be improved by depositing an even number of inclined layers with periodically reversed flux angle, creating a zigzag structure (Ref 21). Hsiao et al. performed GLAD of AlN nanostructures at room temperature using reactive magnetron sputtering in a mixed gas atmosphere of Ar and N₂. The growth behavior of the nanostructures exhibited a strong dependence on the total working pressure and angle of the incoming flux. X-ray diffraction (XRD) and pole figure measurements were used to observe the crystalline orientation of the GLAD-deposited wurtzite AlN nanocolumns. The results revealed *c*-axis $\langle 0001 \rangle$ growth toward the direction of the incoming flux and a transition from fiber-like to biaxial texture as the working pressures were increased. Under normal deposition conditions, the AlN layer morphology changed from $\{0001\}$ to $\{1011\}$ with increasing working pressure. This was attributed

to kinetic energy-driven growth (Ref 20). The effect of glancing angle in a range of 0° to 75° on the growth characteristics, mechanical and electrochemical corrosion behavior of the films were investigated by wei et al. Tetrahedral amorphous carbon (ta-C) films were obliquely deposited on silicon substrate using a custom-made 45-degree double-bent filtered cathodic vacuum arc (FCVA) system. Results showed that increasing the incident angle of carbon ions resulted in a rough surface with agglomerated particles. As the glancing angle changed from 0° to 75°, the residual stress of ta-C film decreased by 28.8%, while the high hardness exceeding 30 GPa was maintained in the films because of the small reduction (13.6%) of *sp*³ hybridized carbon content (Ref 22).

The research conducted by Chirumamilla et al. investigates the magnetron sputter deposition of nanostructured aluminum nitride (AlN) thin films, which are becoming more prominent in their use for power electronics, sensors, and piezoelectric devices, such as MEMS applications. The authors investigate the effects of using metal seed particles (Ag, Au, and Al) on the growth and morphology of AlN films deposited at both normal and glancing angles. Using various characterization techniques, including electron microscopy and x-ray diffraction, they show that the size and material of the grain particles significantly influence the nanostructure formation and the resulting film properties. Notably, GLAD allows for the creation of tilted nanostructures, enhancing the films' surface area and roughness, which is critical for optimizing piezoelectric performance (Ref 23).

The synthesis of aluminum nitride (AlN) thin films is investigated by Patidar et al. using reactive synchronized high-power impulse magnetron sputtering (HiPIMS) in an oblique-angle deposition geometry. Their main aim is to address the obstacles related to achieving highly oriented films, which play a vital role in the effectiveness of piezoelectric devices and sensors. The study shows that incorporating a moderate substrate bias of -30 V, with HiPIMS, improves the quality and texture of crystals by enhancing adatom mobility and reducing gas-ion incorporation. The characterization results provide confirmation of the improved out-of-plane texture and uniform grain polarization. This suggests that synchronized

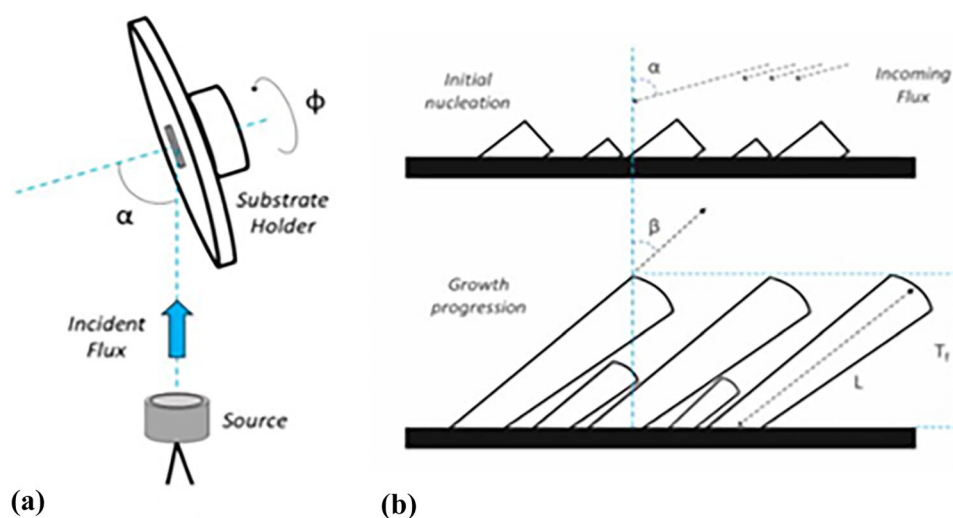


Fig. 1 Angular layering mechanism (GLAD). Reproduced with permission under a Creative Commons 3.0 License, <http://creativecommons.org/licenses/by/3.0/> (Ref 20)

HiPIMS holds promise as a method for producing functional thin films, even in situations with low bias conditions (Ref 24).

Previous research has paid limited attention to the GLAD in surface adhesion in MEMS. Therefore, the primary aim of this study is to examine the effect of angular deposition of a titanium layer and an Ag-Pd-Cu alloy on a silicon substrate. This experimental study involved the deposition of silver alloy and titanium layers on p-type Si <100> substrates using the DC magnetron sputtering method. The silicon tablet was marked with layers from different directions employing a Ti target and an Ag-Pd-Cu silver alloy target. The deposition process was conducted at room temperature. In the first set of experiments, a Ti layer was deposited onto the silicon substrate at a zero-degree angle, followed by the deposition of the silver alloy layer at six different angles: 0, 30, 45, 60, 75, and 85 degrees. In the second set of experiments, a diagonal layer was first applied to the Ti-coated silicon substrate at the same six angles (0, 30, 45, 60, 75, and 85 degrees), and subsequently, the silver alloy layer was deposited at a zero-degree angle on the previously coated samples. The aim of these experiments was to investigate the impact of the angularity of the intermediate layer on the roughness and adhesion properties of the final surface (silver alloy). The adhesion levels of the samples were measured by the apparatus compared to the non-angular deposition mode and the diagonal deposition mode.

2. Material and Experimental Details

2.1 Surfaces and Materials

In the current work, the DC magnetron sputtering method was employed to apply layers of silver alloy and titanium onto p-type Si <100> substrates. The deposition process occurred at room temperature and involved using a 2-inch diameter target. The silver alloy, which contained Ag-Pd (0.9 wt.%)–Cu (1.0 wt.%), originated from a high-purity (99.99%) Ti target. The layers were applied in various directions to mark the substrate. The magnetron cathode was installed at the top of the chamber, and 10 x 5 mm silicon tablet substrates were positioned on a holder, maintaining a 100 mm distance from the cathode. All tests were conducted using a direct current (DC) mode and an argon environment within a steel container, with a flow rate of 12 sccm. To achieve different flux angles of 0, 30, 45, 60, 75, and 85°, a bed fixture with six positions was constructed using stainless steel. The design of the holder was carefully considered preventing any shadowing between the silicon tablets during the layering operation. The deposition rate was predetermined to be 8.8 nm/min, with a pressure of 0.5 Pa and a power of 50 W. The deposition time was adjusted accordingly to achieve a consistent film thickness of 400 nm for both the silver alloy and titanium films in all experimental conditions. In order to eliminate surface contamination, the excised specimens were immersed in a solution of 15% hydrofluoric acid (HF) for a duration of 15 s. Subsequently, they were subjected to cleansing using deionized water, followed by a drying process facilitated by nitrogen gas. The investigation of flux angle impact involved two distinct experiments. In the initial test, a silicon substrate was subjected to titanium (Ti) deposition at a flux angle of zero degrees. Subsequently, a layer of silver alloy was applied onto the titanium layer at six different angles: 0, 30, 45, 60, 75, and 85

degrees. In the second experiment, a silicon substrate was subjected to silver (Ag) deposition at a flux angle of zero degrees. Subsequently, a layer of titanium was applied onto the silver layer at six different angles: 0, 30, 45, 60, 75, and 85 degrees. Scanning electron microscope (SEM) observations were then conducted on the cross section of the film to analyze the resulting morphological characteristics. The effect of interlayer angularity on surface roughness and adhesion properties on the final silver alloy surface was examined. The adhesion levels of the samples were compared between the non-angular lamination mode and the diagonal lamination mode.

2.2 AFM Method

In order to analyze nanoscale thin film characteristics, the Nano wizard II model (JPK Instruments, Germany) was employed in the atomic microscope laboratory. This instrument facilitated the extraction of various details, including topographic images, surface roughness measurements, and adhesion force calculations. The research activities were conducted under controlled experimental conditions, with a temperature of 296 K maintained throughout the analysis. The scanning frequency used for the measurements was set at 0.74 Hz. For the analysis, the HYDRA V-Shaped Probe Series (Hydra 6V-100NG) was selected and employed during the testing. According to the manufacturer's specifications, this probe possesses dimensions of 100 μm in length, 18 μm in width, and 0.6 μm in thickness. The spring constant factor is determined to be 0.292 N/m, and the probe exhibits a response frequency of 66 kHz. It is important to note that the cantilever of the probe has a V-shaped profile and is composed of a low silicon nitride material. The tip of the probe is made of silicon material, featuring a tetrahedral shape and a height ranging from 4 to 6 μm , with a radius of curvature (ROC) less than 10 nm.

3. Results and Discussion

3.1 Influence of Flux Angle on Grain Size and Column Formation

The deposition angle, or flux angle, at which thin films are grown is a critical parameter that strongly influences the resulting microstructure, including the grain size and the tendency for columnar structures to form. Petrov et al. provide a comprehensive overview of how the deposition angle (flux angle) can influence the microstructural evolution of thin films, leading to the formation of columnar structures and a decrease in grain size as the angle deviates from normal incidence, and conclude that oblique deposition angles can lead to the formation of columnar microstructures in thin films. This is because of the shadowing effect, where arriving atoms/molecules are prevented from reaching certain areas on the substrate, resulting in the growth of columnar structures (Ref 25).

In this research, the influence of the flux angle α on the grain size and film surface height of the tested samples in the angular index layer of a silver alloy was investigated using atomic force microscopy (AFM), as depicted in Fig. 2. The AFM images clearly illustrate the impact of the flux angle on both the granulation size and height of the samples. Specifically, Fig. 2 reveals that as the flux angle increases from zero to 60 degrees in the angular layering of the silver alloy, there is a gradual

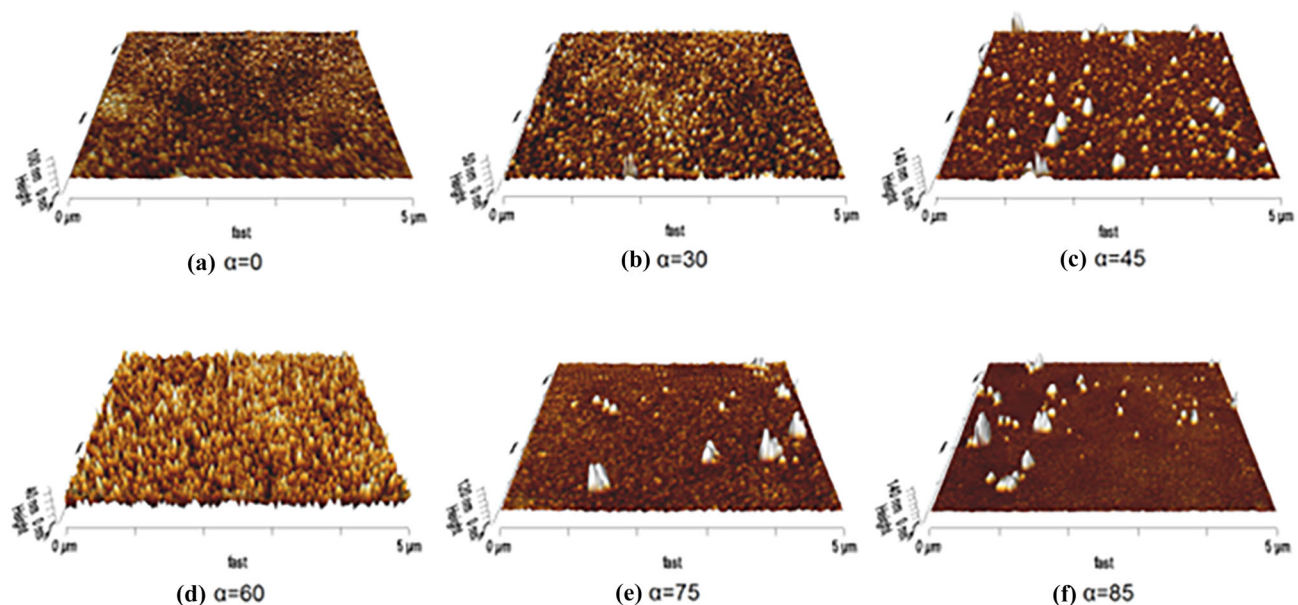


Fig. 2 Surface characterization of titanium-coated surface without angle application and silver alloy deposition at different angles: (a) $\alpha = 0$, (b) $\alpha = 30$, (c) $\alpha = 45$, (d) $\alpha = 60$, (e) $\alpha = 75$, and (f) $\alpha = 85$

increase in both the grain size and height. The analysis of the images got through uniform scanning that is shown in Fig. 2 reveals an intriguing trend at the 60-degree angle of incidence. While the grain height continues to increase, the grain size decreases. This observation suggests that the grain density is notably higher at the 60-degree orientation, as evidenced by the 3D morphological features. The formation and growth of columnar structures appear to exhibit superior uniformity at the 60-degree configuration compared to other angular configurations examined. This phenomenon can be attributed to several factors influencing the thin film deposition process. First, the 60-degree angle may promote a specific crystallographic orientation or texture in the thin film, leading to a higher density of grains in that preferred orientation. Second, the angle of incidence can influence the adatom mobility and surface diffusion processes during film growth, with the 60-degree angle potentially facilitating more efficient surface diffusion and aggregation of atoms, resulting in a higher grain density. The shadowing effects from previously deposited grains can lead to preferential growth in certain locations, further increasing the local grain density at the 60-degree angle. In conclusion, the higher grain density and superior columnar structure uniformity observed at the 60-degree angle of incidence in the thin film deposition process can be attributed to a combination of crystallographic and kinetics factors.

The influence of different angles, specifically those occurring before and after the 60-degree mark, on the formation of columns and their subsequent characteristics in the silver alloy film were examined. This study focused on the position of the substrate relative to the target, which resulted in shading effects impacting the column formation. The columns formed exhibited a thicker structure and island-like texture, with larger diameters observed at angles of 75 and 85 degrees compared to the 60-degree angle. The assessment revealed that the angle of 60 degrees displayed superior column formation and uniform growth, with a significantly more porous structure. This observation suggests that the angle of 60 degrees promotes favorable conditions for the formation and growth of columns

in the silver alloy film. Figure 3 illustrates the cross-sectional view of the diagonal columnar microstructure observed in the silver alloy layer, featuring an angle of $\alpha = 60$, on the non-angled titanium layer deposited on a Si $\langle 100 \rangle$ substrate. Notably, the primary axes of the titanium columns do not exhibit any inclination. However, with the application of a 60-degree angle to the silver alloy layer, the slope of the columns along their primary axes is changed. An increase in the flux angle enhances the diagonalization of the columns, as expected. However, it is important to note that the flux angle and column angle may not be identical due to shadow effects. Nevertheless, there is a strong correlation between the two.

Figure 4 illustrates the impact of the flux angle α on the grain size and surface height of the silver alloy film within the angular index layer of the titanium interstitial layer. The results were got using atomic force microscopy (AFM) analysis. The investigation of the angular layering of the titanium interface layer revealed superior formation and uniformity of columns on the final film surface compared to the angular layering of the silver alloy. Additionally, the structure formed at a 45-degree angle displayed greater uniformity and porosity in the titanium layers. The completion of the cross-sectioned line in the 2D images of the 25 regions was accomplished using JPK software, serving as proof. These images were obtained from AFM data. As a result, it became feasible to estimate both the maximum and minimum elevations of surface prominence in the vertical direction. Table 1 and 2 presents the peak-to-valley values of surface roughness, which confirm the consistency of the obtained results with previous observations.

The investigation into angular layering showcased that the titanium interface layer demonstrated superior formation and uniformity of columnar structures on the final film surface, as opposed to the silver alloy layer. Significantly, the 45-degree angle displayed the greatest granulation height and column uniformity on the final surface, as shown in Fig. 4 and 10, resulting in a substantially more porous structure.

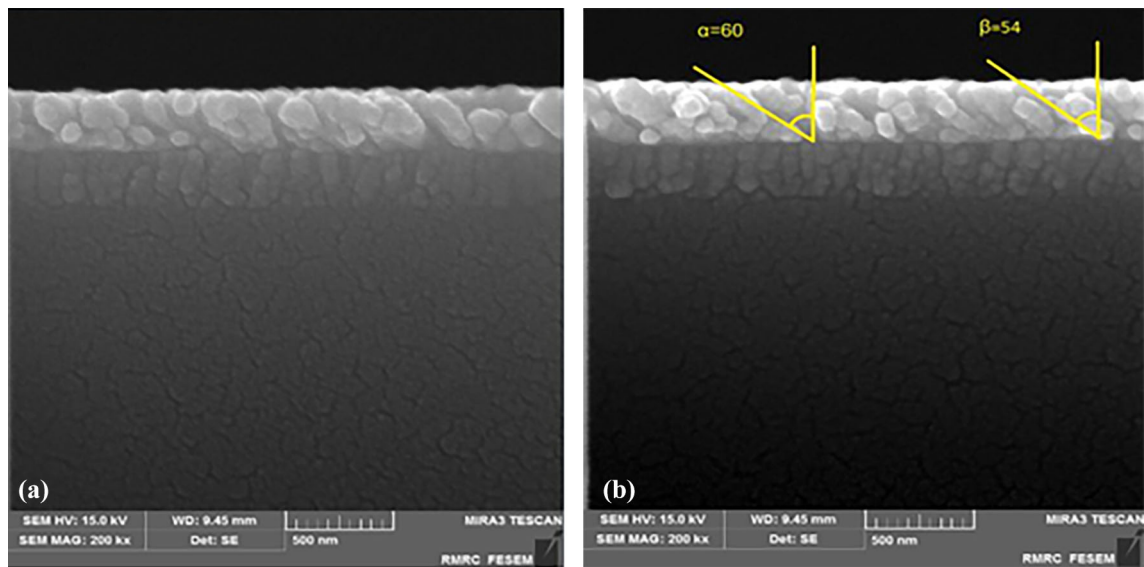


Fig. 3 Cross-sectional scanning electron microscopy results of a 60-degree angle deposition of a coated layer film on a silver alloy

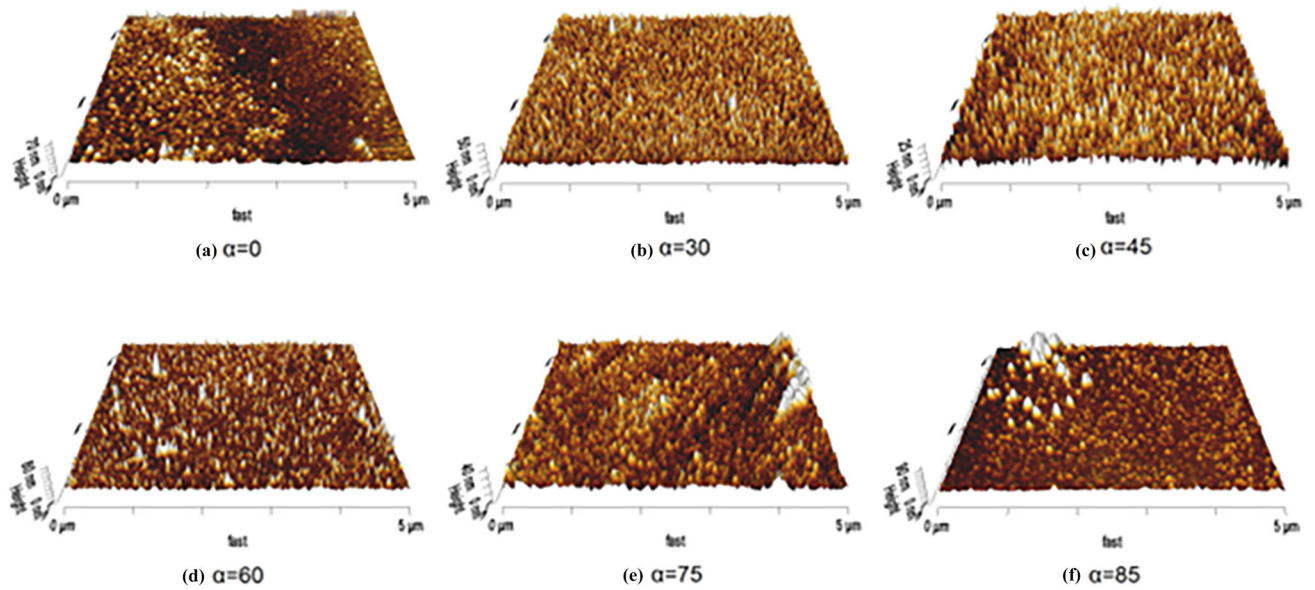


Fig. 4 Surface characterization of the silver alloy film on tested samples in the titanium coating, considering different flux angles: $\alpha = 0$ (a), $\alpha = 30$ (b), $\alpha = 45$ (c), $\alpha = 60$ (d), $\alpha = 75$ (e), and $\alpha = 85$ (f)

Table 1 Peak-to-valley roughness data from cross-sectioned lines in 25 regions got from AFM data analyzed using JPK software for silver alloy angular layering at various angles

Row	First layer(Interstitial)Titanium	Second layerSilver alloy	Minimum in Z range, nm	Maximum in Z range, nm	Peak to valley roughness, nm
1	$\alpha = 0$	$\alpha = 0$	- 4.1	6.4	10.5
2	$\alpha = 0$	$\alpha = 30$	- 7	16.1	23.3
3	$\alpha = 0$	$\alpha = 45$	- 7.7	17.3	25
4	$\alpha = 0$	$\alpha = 60$	- 13.2	18.2	31.4
5	$\alpha = 0$	$\alpha = 75$	- 8.7	20.5	29.2
6	$\alpha = 0$	$\alpha = 85$	- 6.4	12.6	19

Table 2 Peak-to-valley roughness data from cross-sectioned lines in 25 regions got from AFM data analyzed using JPK software for titanium angular layering at various angles

Row	First layer(Interstitial)Titanium	Second layerSilver alloy	Minimum in Z range, nm	Maximum in Z range, nm	Peak to valley roughness, nm
1	$\alpha = 0$	$\alpha = 0$	- 6.4	10.2	16.6
2	$\alpha = 30$	$\alpha = 0$	- 8.9	20.5	29.4
3	$\alpha = 45$	$\alpha = 0$	- 10.2	22.0	32.2
4	$\alpha = 60$	$\alpha = 0$	- 6.5	22.5	29.0
5	$\alpha = 75$	$\alpha = 0$	- 9.2	21.3	30.5
6	$\alpha = 85$	$\alpha = 0$	- 8.6	18.4	27.0

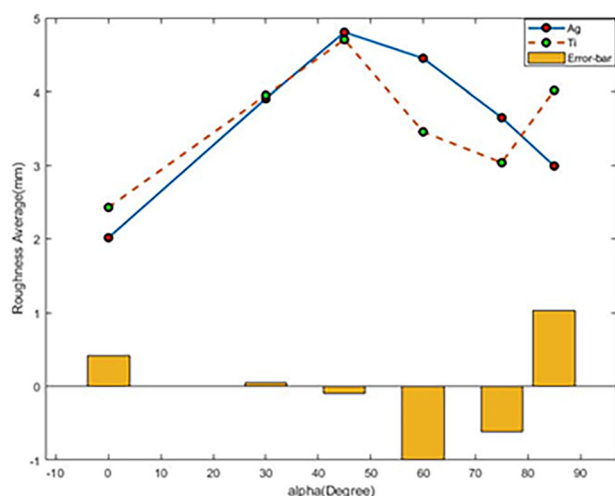


Fig. 5 Comparing the results of the roughness (Ra) of surface obtained from AFM of the tested samples in the angular layering of silver alloy and the angular layering of titanium

3.2 The Effect of Angular Layer of the Silver Alloy Coating and Titanium Interstitial Layer on the Roughness, Adhesion, Grain Size, and Grain Height

The deposition of a silver alloy coating, influenced by the angle at which it is applied, along with including a titanium interstitial layer, can have a substantial effect on the surface roughness, adhesion, and grain size properties of the resultant thin film structure. The results of the surface roughness and adhesion force, grain size and grain height got from AFM, which were derived from the averaged adhesion and morphology plots of the tested samples using JPK software in a $5 \times 5 \mu\text{m}$ scan size, are presented in Fig. 5, 8, 9 and 10, respectively. The solid line displays the results of the first test, where titanium was deposited at a zero-degree angle on a silicon substrate, followed by the application of a silver alloy at six different angles on the previously deposited titanium. The dashed line presents the results of the second experiment, where a layer of titanium was deposited on a silicon substrate at angles of 0, 30, 45, 60, 75, and 85 degrees. Subsequently, an alloy of silver was deposited at a zero-degree angle on the same six samples.

3.2.1 The Effect of the Layer Angle on the Roughness Value (Ra). The surface roughness characteristics got using atomic force microscopy (AFM) are presented in Fig. 5. The

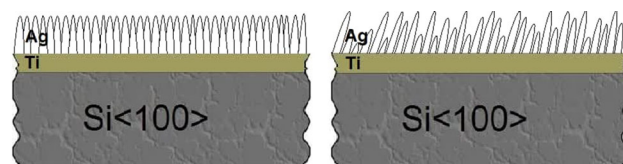


Fig. 6 Serration of the surface as a result of the angled layer on the surface

focus is on the surface morphology of the tested samples that underwent angular layering of a silver alloy and titanium. The impact of varying the layering angle on the roughness value is clearly observed. As demonstrated in Fig. 5, there is an initial rise in the roughness value, which is then followed by a decline as the layering angles increase in both test series. Comparing the results to the coating conditions without angle application, it is notable that layering angles of 45 degrees yield the highest levels of roughness in the diagonal coating test series. Specifically, an increase of 138% was observed for silver and 93% for the titanium.

The explanation for these findings can be attributed to the consideration of the angular indexing layer of the silver alloy, as shown in Fig. 6. With the indexing angle increasing up to 45 degrees, owing to the diagonalization of the silver alloy columns, the surface adopts a serration mode. However, as depicted in Fig. 7(a), when the layer angle increases from 45° to 85°, most columns experience a reduction in height protruding from the surface of the silver alloy. The growth of the grains and its direction should be vertical or oblique on the contact surface, which affects the effective interaction between the two surfaces, and therefore influences the adhesion, and leads to a decrease in roughness. The way grain growth is oriented significantly impacts the contact area and interaction dynamics between two surfaces. When grains grow in a vertical or angled orientation to the contact surface, they increase their effective surface area compared to grains that grow parallel to the surface. The enhanced surface area facilitates stronger intermolecular forces, such as van der Waals interactions and hydrogen bonding, between the surfaces. As a result, this leads to better adhesion. In contrast, if the grains grow parallel to the contact surface, the effective area for interactions is smaller, resulting in weaker adhesion. In summary, the way grains grow, either vertically or at an angle, increases the surface area available for interactions between molecules. This has a significant impact on the strength of adhesion between surfaces that come into contact, which is not ideal for micro-assembling in MEMS applications.

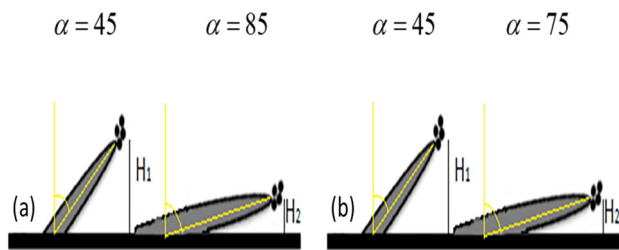


Fig. 7 Reducing the height of the layered columns by increasing the layering angle (a) from 45 degrees to 85 degrees for silver (b) from 45 degrees to 75 degrees for titanium

Additionally, the reasoning behind these findings regarding the angular arrangement of the titanium interface layer can be expounded upon: As the layering angle is elevated to 45 degrees through the diagonalization of titanium columns, the surface shows an enhanced jagged state. However, Fig. 7(b) reveals that an increase in angle from 45 to 75 degrees leads to a reduction in height protrusion from the titanium surface for most columns within the indexing layer. Therefore, the roughness of the final layer of silver alloy, when applied to this indexed layer, effectively mimics the properties of the underlying titanium surface. As a result, the rough behavior resembles that of the interface layer's surface. Figure 5 is derived from two-dimensional data generated using JPK software, based on the three-dimensional data presented in Fig. 4. This figure illustrates that the increase in roughness within the angle range of 75° to 85° can be attributed to enhanced angular layering, which facilitates the formation of a greater number of surface peaks. As a result, there is a rise in roughness levels within this range of angles.

3.2.2 The Effect of Angular Layering on Adhesion Levels. Investigating the role of surface topography and roughness in mediating the relationship between layering angle and adhesion strength could provide valuable insights. Delplanque et al. concluded that the orientation and contact area of deposited particles on a surface are critical parameters that determine the overall adhesion and bonding between the particles and the surface (Ref 26).

Figure 8 presents the adhesion values got from the angular stacking test of the silver alloy and titanium. In each layering configuration, the adhesion value of the final surface of the silver alloy exhibits an increasing trend with the rise of the layering angle, reaching its peak at 60 degrees, followed by a subsequent decline. With the layering angle progressively increasing from 0 degrees (perpendicular) to 60 degrees, there is a corresponding augmentation in the effective surface area of contact, fostering closer molecular-level interactions, such as van der Waals forces and hydrogen bonding, between the surfaces. This enhanced interfacial interaction leads to a rise in the overall adhesion strength, reaching a peak at the 60-degree layering angle. However, once the layering angle goes beyond 60 degrees, the effective contact area between the deposited layers decreases. This results from the grains becoming more inclined and oriented away from the surface normally, reducing the area for effective surface interactions. The reduction in contact area and the weakening of interfacial interactions lead to a decrease in the overall adhesive strength. Notably, the lowest level of adhesion is observed at an angle of 85 degrees in both the angular layering of the silver alloy and the angular layering of titanium because of The reason is effective contact area between the deposited layers is minimized because of

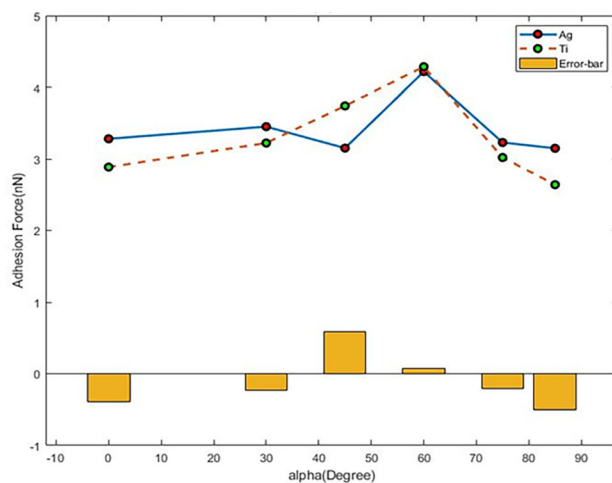


Fig. 8 Comparing the results of the surface adhesion force obtained from AFM of the tested samples in the angular layering of silver alloy and the angular layering of titanium

the grains being oriented almost parallel to the surface, reducing the area for intermolecular interactions. This angle results in approximately a 4% reduction in adhesion for the angular layering of the silver alloy and an approximately 8% reduction in adhesion for the angular layering of titanium, compared to the layering mode without the utilization of an angle.

3.2.3 The Influence of the Layering Angle on the Grain Size. Recent studies, such as those referenced in Ref 13, 14), have investigated grain size of deposited silver on the substrate using the magnetron sputtering method. These studies show that variations in deposition conditions significantly impact surface geometric parameters, including grain radius, grain height, and overall grain size. Hence, based on the Rabinovich equation, it can be concluded that surface asperities have a critical influence on adhesion forces. The JPK software was used to quantify grain size in this investigation, using data got from AFM. To be more precise, the measurement procedure entailed traversing the effective asperities, where the longitudinal distance was defined as the grain size. The value was subsequently averaged over 75 measured points and presented as the grain size for various angular layering of a silver alloy and titanium. The results got from the angular layering of the silver alloy in Fig. 9 show that, as the angle increases, the grain size initially shows an upward trend, followed by a subsequent decrease. Notably, the largest grain size is observed in the silver alloy angle coating at a 60-degree angle, showcasing a 17% increase compared to the coating condition without angle application. In addition, the titanium coating applied at an 85-degree angle has the largest grain size. It shows a 1.2% increase compared to the coating without an angle application.

3.2.4 The Effect of Angular Layering on Grain Height. Figure 10 presents the measured grain height values got from the test involving angular layering of the silver alloy and titanium. The results show a significant 168% increase in the titanium angular index layer at a 45-degree angle, showing notable enhancements. Similarly, the silver alloy angular layering exhibits the greatest grain height, demonstrating a substantial 107% increase at a 75-degree angle, compared to the condition without angle application. As depicted in Fig. 10, when the angle is set to 0 degree, the grain height in both layers

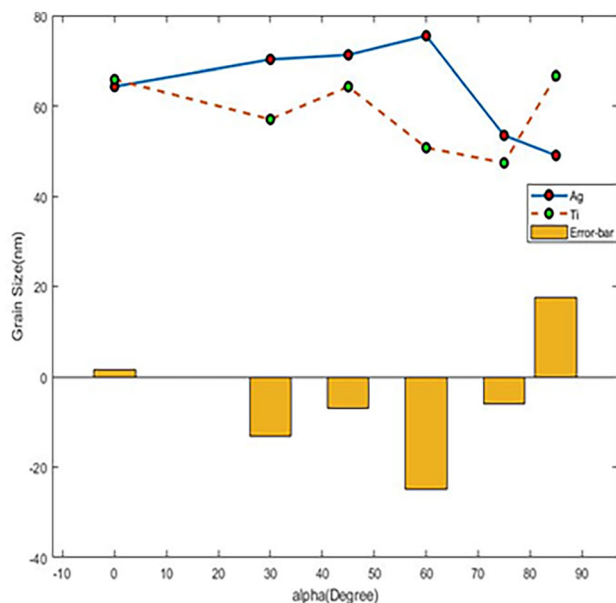


Fig. 9 Comparison of the size of the grains of the tested samples in angular layering of silver alloy and the angular layering of titanium

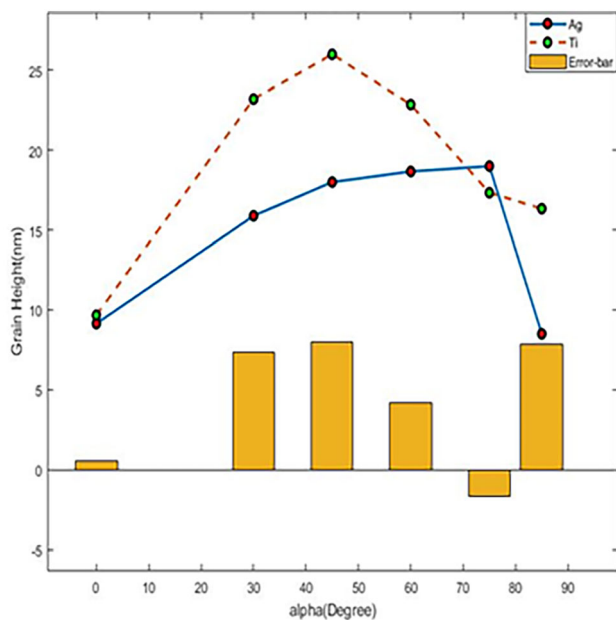


Fig. 10 Comparing the height of grains in the tested samples in the angular layering of silver alloy and titanium

exhibits close values. Grain height follows a consistent pattern in all cases: It initially increases and then decreases with the layering angle increment. Comparing Fig. 5 and 10, changes in grain height and surface roughness behave similarly.

4. Conclusion

This study examined the effects of altering the angular layering of coatings on surface adhesion force and roughness in

micro-electr-omechanical systems. Adhesion force was affected by the angular deposition process, which altered the effective contact area because of changes in surface roughness. The results showed that the silver alloy diagonal coating had a 138% higher surface roughness compared to the non-angled condition. Similarly, the titanium diagonal coating showed a 93% increase in roughness at a 45-degree angle. In terms of adhesion, the silver alloy and titanium diagonal coatings showed 4 and 8% reductions, respectively, relative to the non-angled applications. The process of angular layering impacted both grain size and height, initially causing an increase, which was then followed by a decrease. These findings have practical implications for reducing adhesion effects in micro-assembly applications, such as coating surfaces in micron clamps.

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Conflict of interest

The authors declare that they have no conflict of interest.

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