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Prototype Of Laboratory-Scale Plasma Arc For Thin Film Growth And Its Testing

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Abstract

Thin films, characterized by small-dimensional materials on a substrate formed through sequential deposition of ionic/molecular/atomic materials, have gained significance in various industries. An emerging method, Arc Plasma Deposition (APD), is gaining attention for its effectiveness in minimizing environmental pollution. This research explores the potential of APD, an environmentally friendly technique, in coating materials, particularly copper, at a laboratory scale. The objective is to introduce a cost-effective solution for material deposition, addressing the industrial need for efficient and eco-friendly coating processes. A laboratory-scale prototype for coating materials, specifically utilizing copper metal, is introduced and tested. The APD process is conducted in an atmospheric free-oxygen chamber, where plasma is generated. The deposited copper is analyzed concerning the deposition time, providing insights into the efficiency of the APD technique. The laboratory-scale prototype demonstrates the feasibility of using APD for coating materials. The results indicate the potential of APD in efficiently producing thin films, making it a promising alternative for industrial applications.

Keywords:
Arc plasma deposition, copper deposited, laboratory-scale prototype, thin film coating.

1 Introduction

Thin films refer to small-dimensional materials on a substrate produced through the sequential deposition, one by one, of ionic/molecular/atomic materials with intensification. The thickness of thin films is generally less than a few microns[1]–[4]. One of the important industrial needs is metals/materials coating on its surfaces[5].Surface coating techniques for materials can be carried out in various ways, including chemical vapor deposition, physical vapor deposition, and electrochemical deposition[6].The method of depositing thin film layers on materials has significantly developed, with numerous techniques available. Various surface coating techniques include Physical Vapor Deposition (PVD), implantation, Radio Frequency (RF) sputtering, Chemical Vapor Deposition (CVD), Polymer Supported Electrode (PSE), and electrochemical deposition[7]–[16].PVD is considered a "green engineering" method, generating minimal pollution during its operation, but it comes with a higher cost compared to CVD. Among various thin film deposition techniques, PVD coating requires a vacuum environment to evaporate solid materials and deposit them onto the substrate as a coating, demonstrating effective performance[6], [17]–[20].

APD is an emerging method that stands out as an effective solution for minimizing environmental pollution, making it a powerful and superior technique compared to other methods[6], [21], [22]. This process is conducted in an atmospheric free-oxygen chamber, where plasma is generated from the substrate. Subsequently, this plasma deposits onto the surface it traverses, forming a thin layer. APD proves to be a promising technique with its environmentally friendly approach and the capability to produce thin films efficiently.

One of the applications of APD on machinery is cutting tools, which in the industrial world are essential equipment. It requires coating to enhance the quality of the cutting results, both in terms of the cut's quality and economic efficiency[23], [24]. A significant portion of PVD coatings for industrial cutting tools is contributed by the cathodic arc method[25], [26].The coated cutting tools exhibit superior wear resistance compared to those without coatings[23], [26]. However, research on material deposition using APD on an industrial scale requires a significant amount of cost, hence the need for laboratory-sized equipment to reduce expenses. Therefore, in this research, a laboratory-scale prototype for coating materials is introduced and tested. The coating material is a metal of copper, chosen for its properties of resistance to moderate temperatures and oxidation resistance. We evaluated the performance of equipment for changing the atmosphere chamber from oxygen-containing into an oxygen-free environment. The equipment's ability was shown by its performance during evacuating oxygen into the pressure -20cmHg, and could be filled by argon gas well. Then, we evaluate the effect of deposition time on the substrate. Interestingly, the amount of deposited copper was in line with deposited time.

2 Methods

2.1 Vacuum Chamber

The chamber is constructed by steel pipes with dimensions of Ø250 mm ×300 mm. It features an observation window on the door, allowing the internal part of the chamber to be observed during the process (Fig. 1).

Fig. 1. Vacuum chamber with a glass observation window.

2.2 Plasma Generator

The plasma is generated using a TIG welding generator with a tungsten electrode (WT20), and Pulse Width Modulation (PWM) is incorporated to adjust the plasma arc according to the data acquisition needs. Inside the chamber, there is a holder for the test material, adjustable in height to accommodate the material to be deposited. Other supporting tools to ensure the proper functioning of the equipment include: 1) vacuum pump, 2) pressure gauge, 3) argon gas cylinder, and 4) vacuum gauge.

2.3 Prototype Testing

The prototype APD configuration was based on the schematic in Fig. 2 and Fig. 3. The test procedure includes the preparation of equipment set-up, and substrate, and performing the experiment. The details of the test procedure are explained as:

1. Equipment set-up. This set-up involves installing the material, copper (Cu) with a purity level of 99.99% is used in the test to be deposited. It has dimensions of 50mm × 45mm × 4mm.

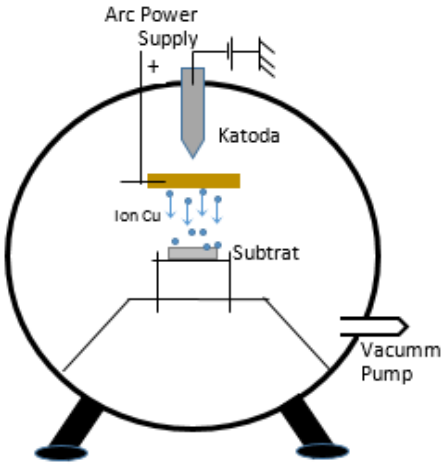


Fig. 2. Instrumentation on a 2-dimensional scheme.

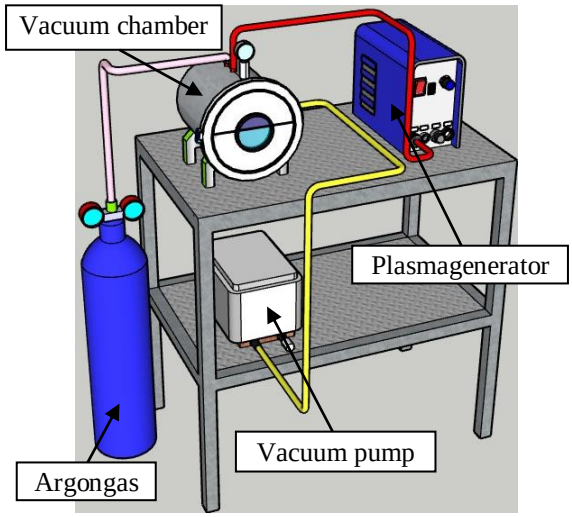


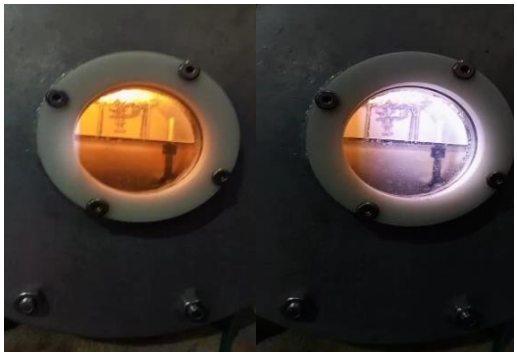
Fig. 3. Instrumentation on a 3-dimensional scheme.

2. Substrate preparation. The substrate is an HSS ¼" × 22 mm milling cutter. Before the process, the substrate is cleaned with 96% ethanol, followed by an ultrasonic cleaner (BRANSONIC/ultrasonic cleaner 42 kHz) for 10 minutes (please see Fig. 4), then it is dried. Ensuring the cleanliness of the workpiece is a crucial factor in the coating growth process [8].

(a) (b)

Fig. 4. (a) Substrate cleaning by an ultrasonic cleaner, (b) substrate retrieving by a tweezer and placing it on fiber-free tissue.

3. The specimen/substrate is placed beneath the material to be deposited. The distance is 10 mm [9]. Then, changing the chamber atmosphere from an oxygen-containing into one oxygen-free environment. The pressure of the vacuum process is carried out up to -20cmHg, and then filled with argon gas until the pressure returns to 0cmHg. This process is carried out 2 (two) times (Fig. 5).



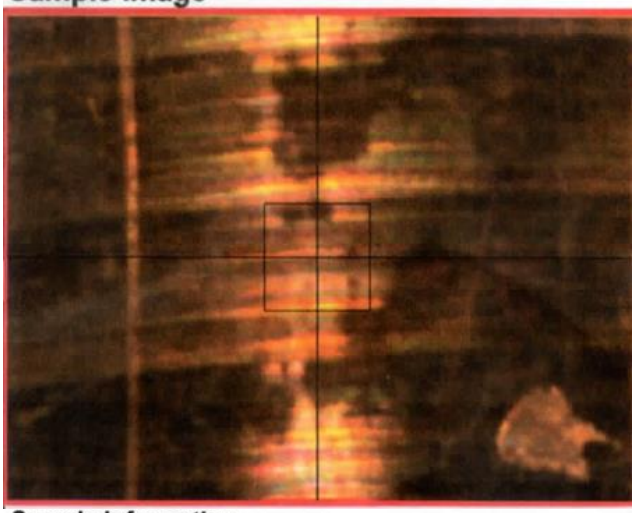
(a) (b)

Fig. 5. The color of plasma during the deposition process produces a white glow (a) and a reddish glow (b) inspected by a glass observation window.

4. The next process is running the plasma arc for copper deposition into the substrate. The current of the plasma generator is 60 A, 10 Hz in frequency, and a duty cycle is 10%. During deposition, argon gas flow rate is maintained at 3-4 L/minute, and the deposition time is varied for 10, 25, and 35 minutes (Fig. 6).

(a)

Sample image



(b)

Sample Information

Measurement Date	11/11/2022 14:37
Recipe	HSM6000
Sample Name	Copper
Sample No.	
Lot No.	
Operator	
Note	

Measurement Conditions

	1st	2nd	3rd	4th
Measurement time(sec)	100	100	100	
Dead time(%)	14	2	4	
X-ray tube voltage(kV)	50	50	15	
X-ray tube current(uA)	1000	1000	1000	
Primary Filter	for Pb	UNI	for Cr	
Chamber Environment	Air	Air	Air	
Inspection Equipment	EA6000VX(Rh) ID6743 (Ver. 11.08.3.0)			
Collimator	1.2x1.2mm			
Measurement method	Bulk C:\XRAY\ANA\HSM6000.bfp			
Note	PeakingTime=1.0usec			

Elements	Cu	Co	Ag	
Conc. (wt%)	99.99	0.01	0.00	

Fig. 6. Pure copper analysis result, (a) sample image, and (b) measurement condition and result.

5. The last step is performing composition analysis by XRF Analyzer Hitachi EA 6000VX (Fig. 7).

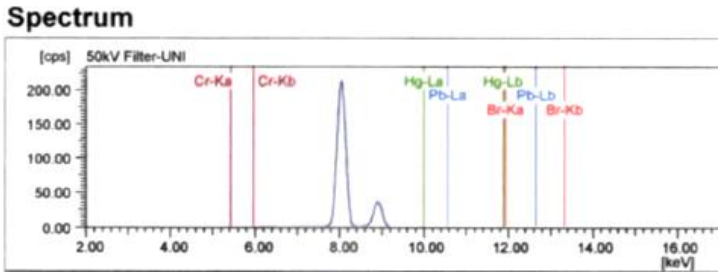
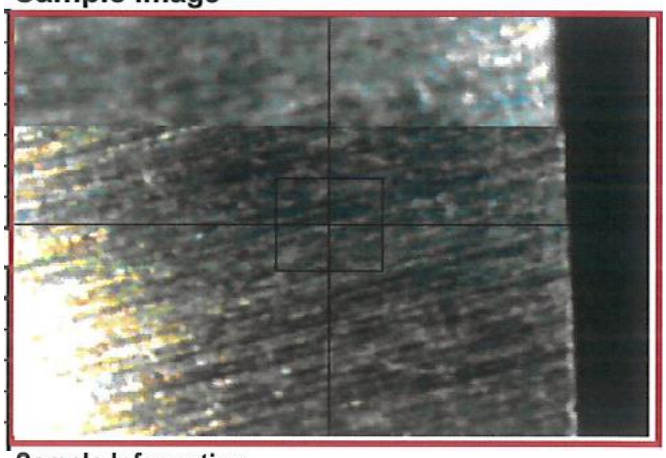


Fig. 7. Pure copper analysis diffractogram.

3 Discussion

In this research, the initial materials used were analyzed by XRF to validate their composition. There are 2 (two) initial composition tests: copper as the material will be deposited and HSS cutting tool as a substrate (Fig. 8).

(a)



(b)

Sample Information

Measurement Date	17/05/2023 10:23
Recipe	HSM6000
Sample Name	Blanko Spesimen
Sample No.	-
Lot No.	-
Operator	Arifin Suryo
Note	-

Measurement Conditions

	1st	2nd	3rd	4th
Measurement time(sec)	100	100	100	
Dead time(%)	13	1	6	
X-ray tube voltage(kV)	50	50	15	
X-ray tube current(uA)	1000	1000	1000	
Primary Filter	for Pb	UNI	for Cr	
Chamber Environment	Air	Air	Air	
Inspection Equipment	EA6000VX(Rh) ID6743 (Ver. 11.08.3.0)			
Collimator	1.2x1.2mm			
Measurement method	Bulk C:\XRAY\ANA\HSM6000.bfp			
Note	PeakingTime=1.0usec			

Elements	Fe	W	Mn	Ni	Cu	Pd
Conc. (wt%)	93.86	0.68	0.56	0.08	0.07	0.00

Fig. 8. Results of the blank specimen/initial substrate.(a) Sample image, and (b) measurement condition and result.

3.1 Composition of Copper (Cu)

It is important to validate the copper composition which is used as deposited material. Fig. 6 confirms that the composition of the copper is 99.99%, and only 0.01% is cobalt. Below are the test results for the purity of the copper (Cu) material to be grown. The XRF diffractogram was shown in Fig. 7 which shows only 2 (two) peaks; the high one (exhibiting a very high peak exceeding 200 counts per second) at 8 keV identified as copper, and the low intensity at 8.9 keV identified as cobalt.

3.2 Composition of Blank Specimen/Initial Substrate(HSSCutting Tool)

Fig. 8(a) shows the image of the substrate and the examination point under XRF. Fig. 8(b) confirms that the composition of the initial substrate. The initial substrate composition is shown in detail in Fig.8(b), and the spectrum is shown in Fig. 9.

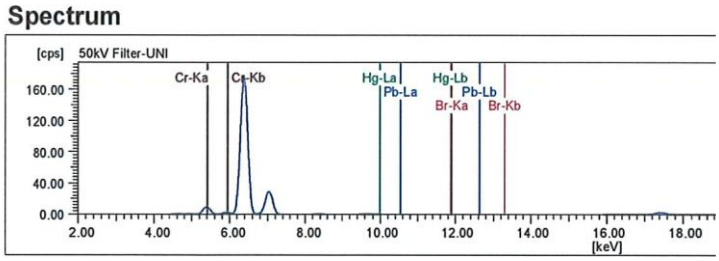


Fig. 9. Spectrum of the blank specimen/initial substrate.

Fig. 9 shows there is a high peak with a value higher than 160 cps at 6.4 keV, which corresponds to the Fe (iron), and it also be observed that the initial substrate (lathe tool bit) has a very low copper (Cu), specifically at concentration 0.07wt%, while the iron (Fe) content is 93.86wt%.

3.3 Equipment Test

Prior to copper deposition under plasma arc, the inside chamber atmosphere was changing from an oxygen-containing into an oxygen-free environment. It was performed by evacuating the oxygen gas inside the chamber and fulfilled by argon gas. The evacuating of oxygen was performed by vacuuming the chamber into the pressure -20cmHg. The argon gas was introduced into the vacuum chamber slowly. The process of vacuuming and fulfilling was conducted 2 times to ensure the oxygen inside the chamber was completely removed. During the process, the equipment could deliver the process well. No structural defect and no leakage was observed during this process. It shows that the equipment which has a laboratory scale was running well.

3.4 Copper Deposition under Plasma Arc

The experiment was evaluated to understand the effect of deposition time on the amount of deposited copper, i.e.: 10, 25, and 35 minutes. Fig. 10 shows the result of deposited copper on the substrate for 10 minutes. The amount of copper was 0.14wt%, increased 2 times compared to that before deposition (0.7wt%).

Elements	Fe	W	Mn	Cu	Ni	Ge
Conc. (wt%)	94.30	0.74	0.48	0.14	0.10	0.00

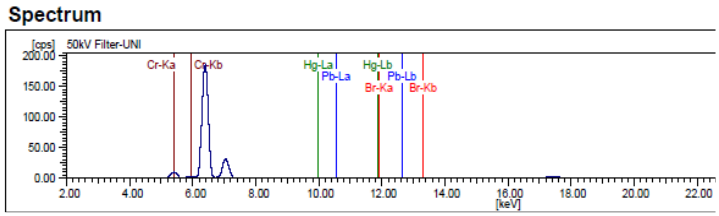


Fig. 10. Copper deposited spectrum on the substrate for 10 min.

The second specimen was tested by extending the coating process duration from the initial 10 minutes to 25 minutes. The test results indicate an increase in the Cu value, where the concentration value changed from 0.14% for a 10-minute coating duration to 0.53% for a 25-minute coating process (Fig. 11).

The third sample test, with an extended coating process duration of 35 minutes, resulted in a significant increase in the Cu value. While the second specimen test showed a Cu concentration of 0.53%, the third specimen test increased to 11.56%. This is likely due to continuous exposure of the Cu material used as a coating material to heat at the same point, making it easier for the material to deposit (Fig. 12).

Elements	Fe	W	Mn	Cu	Ni	Pd
Conc. (wt%)	93.32	0.79	0.56	0.53	0.08	0.00

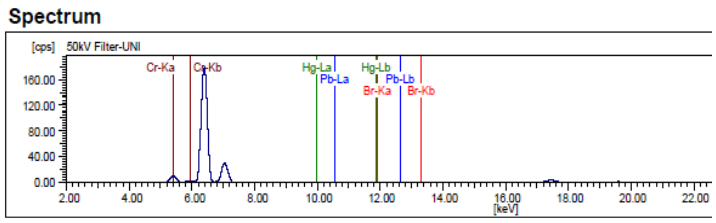


Fig. 11. Copper deposited spectrum on the substrate for 25 min.

Elements	Fe	W	Cu	Mn	Sb	Bi	Ge
Conc. (wt%)	71.28	13.18	11.56	0.47	0.00	0.00	0.00

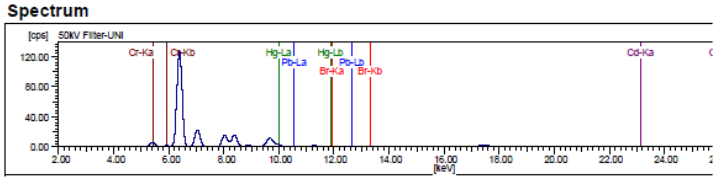


Fig. 12. Third copper deposited spectrum (time for deposited copper is 35 min).

As a suggestion, it is recommended to replace the Cu material for each coating process or allow cooling of the Cu material to be deposited before proceeding with the second coating process. Additionally, shifting the plasma focal point on the Cu material from the previous focal point might be considered.

From the Table 1 test results, it is evident that there is an increase in the Cu concentration in the specimen before and after the coating process. The duration of the deposition/coating significantly influences the resulting Cu value.

Table 1. Comparison of Cu deposited

Deposition condition	
The current of the plasma generator	60 A
Distance of Cu to substrate	10 mm
Argon flow rate	3-4 L/minute
Comparison of test result	
Spesiment	**Deposition time (minutes)** / **Conc. Cu (wt %)**
Blanko	- / 0.07
Test 1	10 / 0.14
Test 2	25 / 0.53
Test 3	35 / 11.56

4 Conclusion

The growth of thin metal layers can be achieved using a TIG welding generator as a plasma source, with a current setting of 60A and a distance of 10mm between the specimen and the coating material. Copper (Cu) material can be grown onto the surface of another material. The test results on the specimens conclude that there is an increase in the concentration value of the coating metal (Cu) with the duration of the coating process.

5 Suggestion

Conducting coating processes with variations in parameters such as specimen distance, current magnitude (Ampere), or the type of coating material can provide valuable insights into how these changes affect the coating outcomes. This helps in understanding the characteristics and optimizing the coating process, such as the influence on coating thickness, adhesion, or even the properties of the resulting coated material. The variation process is useful for finding the most effective coating conditions that align with specific requirements.

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