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Modeling of TiN Coating Thickness Using RSM Approach

Abdul Syukor Mohamad Jaya[1,2,a], Siti Zaiton Mohd Hashim[2,b], Habibollah Haron[2,c], Muhd. Razali Muhamad[3,d], Abd Samad Hasan Basari[1,e], Md. Nizam Abd. Rahman[3,f]

[1] Centre of Advanced Computing Tech., Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia

[2] Soft Computing Research Group (SCRG), Faculty of Computing, Universiti Teknologi Malaysia, 81300 UTM, Skudai, Johor, Malaysia

[3] Faculty of Manufacturing Eng., Universiti Teknikal Malaysia Melaka, Hang Tuah Jaya, 76100 Durian Tunggal, Melaka, Malaysia

[a]syukor@utem.edu.my, [b]sitizaiton@utm.my, [c]habib@utm.my, [d]muhdrazali@utem.edu.my, [e]abdsamad@utem.edu.my, [f]mdnizam@utem.edu.my

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Abstract. In this paper, modeling of Titanium Nitrite (TiN) coating thickness using Response Surface Method (RSM) is implemented. Insert cutting tools were coated with TiN using Physical Vapor Deposition (PVD) sputtering process. N_2 pressure, Argon pressure and turntable speed were selected as process variables while the coating thickness as output response. The coating thickness as an important coating characteristic was measured using surface profilometer equipment. Analysis of variance (ANOVA) was used to determine the significant factors influencing TiN coating thickness. Then, a polynomial linear model represented the process variables and coating thickness was developed. The result indicated that the actual validation data fell within the 90% prediction interval (PI) and the percentage of the residual errors were low. Findings from this study suggested that Argon pressure, N_2 pressure and turntable speed influenced the TiN coating thickness.

Introduction

During a machining process, high temperature on the cutting tool tip could exceed 800^{o}C. This condition reduces cutting tool performance and increases cutting tool wear. Therefore, a cutting tool with high resistance to wear is very important to deal with the condition. This performance could be improved by applying thin film coating on the cutting tool. The thin film could enhance the surface properties while maintaining its bulks properties. It was proven that the coated tool has forty times better in tool wear resistance compared to the uncoated tool [1].

Two main techniques in depositing coating on cutting tool are physical vapor deposition (PVD) and chemical vapor deposition (CVD). The main different between the both processes is the vapour source. In the PVD coating process, the sputtered particle from harder material embedded on the cutting tool in presence of reactive gas. In PVD coating process, many factors are reported have significant influence to coating characteristics especially coating thickness [2, 3]. Determination of sufficient thickness in coating is very important to avoid substrate penetration during machining process. A coating thickness has been reported influenced the other coating characteristics. It has reported that higher coating thickness increased the grain size and the roughness of the coating [4]. Some of the studies shown that N_2 pressure, Argon pressure and turntable speed could have significant effect on the deposited coating and surface morphology [5-7].

Modeling is a sufficient method to address the magnetron sputtering process issues such as cost and customization. Some modeling approach like Taguchi is difficult to detect the interaction effect of nonlinear process [8], while full factorial approach is only suitable for optimization [9]. In this study, the application of RSM to model the TiN coating thickness has been discussed. The model is used to predict the thickness and indicates the effect of process factors to the TiN coating thickness.

Experiment

Material and Method. The experiment was run in unbalanced PVD magnetron sputtering system made by VACTEC Korean model VTC PVD 1000. The coating chamber was fixed with a vertical titanium (Ti) target. The surface of tungsten carbide inserts was cleaned with alcohol bath in an ultrasonic cleaner for 20 minutes. The tungsten carbide inserts were loaded in the rotating substrate holder inside the coating chamber. To produce the electron in the coating chamber for sputtering purpose, an inert gas called Argon was used. The tungsten carbide inserts were coated with the Ti in presence of nitrogen gas. Details of the process is indicated in Table 1.

Table 1. Substrate preparation and deposition process setting

Variables	**Unit**	**Processes**			
		Step 1	**Step 2**	**Step 3**	**Step 4**
		Alcohol Bath	**Ion cleaning**	**TiN deposition**	**Cooling**
• Equipment	-	Ultrasonic bath cleaner	PVD magnetron sputtering machine		
• Sputtering power	kW	-	-	4.0	-
• Substrate temperature	℃	-	300	400	400-60
• Ion source power	kV/A	-	0.24/0.4	0.24/ 0.4	0.24/ 0.4
• Substrate bias voltage	V	-	-200	-200	-200
• N_2 pressure	$\times 10^{-3}$ mbar	-	-	0.16-1.84	-
• Argon pressure	$\times 10^{-3}$ mbar	-	-	3.66-4.34	4.0
• Turntable speed	rpm	-	4.0	4.0-9.0	4.0
• Duration	min	20	30	150	60

Experimental design. In this study, the experimental matrix was based on RSM centre cubic design, using Design Expert version 8.0 software. It was designed based on 8 factorial points, 6 axial points and 3 central points. In the matrix, the extreme points (operating window) as the +/- Alpha value was designed. Based on the defined extreme point values, the software then dispensed the high and low settings for the factorial points. This is to ensure the characterization could be performed by covering the widest range of operating window.

Surface Profiler. Surface profilometer KLA Tencor model was used to measure the TiN coating thickness. The measurement were taken in three times on three different point. Average of the points was taken as thickness value.

Response Surface Method (RSM). RSM is a collection of mathematical and statistical techniques to model and analyze problems in which responses are influenced by several input variables [10]. The relationship between the input parameters and output responses is defined using regression analysis in form of polynomial equation. In this work, the regression coefficients such as the coefficients of the model variables including the intercept or constant terms were calculated. The model was tested for statistical significance using the analysis of variance approach (ANOVA). The tests for significance of the regression model, significance of individual model coefficient, and lack of fit were calculated.

Result and Discussion

Seventeen experimental runs including output response data are indicated in Table 2. In this study, the analysis of variance (ANOVA) is used to determine the significant factors influencing the TiN coating thickness and the present of interactions affecting the characteristic. As shown in Table 3, the ANOVA analysis indicates that the Argon pressure, N_2 pressure and turntable speed are the significant influencing factors of the TiN coating thickness. From the ANOVA analysis, a linear polynomial equation model for TiN coating thickness is generated as shown in Eq. 1.

Argon pressure. As shown in Fig. 1, as Argon pressure increases from 3.80×10^{-3} mbar to 4.20×10^{-3} mbar, the TiN coating thickness decreases from 0.159 μm to 0.094 μm. This happen when the deposition in the process decreases due to increase of the atoms collision after the mean free path in the coating chamber reduced with the present of many Argon atoms.

N_2 pressure. As N_2 pressure increases from 0.5×10^{-3} mbar to 1.5×10^{-3} mbar, the coating thickness decreases from 0.156 μm to 0.097 μm. This behavior is showed in Fig. 2. Huang et. al [11] in his study explained that the increased in N_2 flow rate resulted in decrease of TiN coating thickness. This behavior happen when the increase of N_2 gas atom in coating chamber decreases the mean free path in chamber and disturbs the deposition process. By that, the coating thickness become thinner.

Turntable speed. As shown in Fig. 3, the coating thickness decreases from 0.157 μm to 0.096 μm as turntable speed increases from 5.0 rpm to 8.0 rpm, respectively. Chang et al. [12] in study on the microstructure and performances of TiAlN/CrN multi-layer coatings has reported that the thickness of thin films and bi-layer of the TiAlN/CrN coatings decreased when the substrate holder rotation speed increased from 1.5 rpm to 12.0 rpm. By moving faster, the number of sputtered atom that deposited on a the substrate surface also reduced.

Table 2. Experimental run and result of TiN coating thickness

Run	Factor 1 A:N_2 pressure [$\times10^{-3}$ mbar]	Factor 2 B:Argon pressure [$\times10^{-3}$ mbar]	Factor 3 C: Turntable speed [r.p.m]	Response Thickness [μm]
1	1.84	4.00	6.50	0.181
2	1.00	3.66	6.50	0.204
3	1.00	4.34	6.50	0.139
4	0.16	4.00	6.50	0.189
5	1.50	3.80	5.00	0.088
6	0.50	3.80	5.00	0.241
7	0.50	4.20	5.00	0.152
8	0.50	4.20	8.00	0.032
9	1.50	4.20	5.00	0.061
10	1.00	4.00	9.02	0.055
11	1.50	3.80	8.00	0.068
12	0.50	3.80	8.00	0.208
13	1.50	4.20	8.00	0.026
14	1.00	4.00	3.98	0.180
15	1.00	4.00	6.50	0.043
16	1.00	4.00	6.50	0.085
17	1.00	4.00	6.50	0.193

Table 3. ANOVA analysis for TiN coating thickness

Source	Sum of Squares	df	Mean Square	F Value	p-value Prob > F	
Model	0.0392	3	0.0131	3.90	0.0346	significant
A-N2	0.0119	1	0.0119	3.56	0.0817	
B-Ar	0.0144	1	0.0144	4.31	0.0583	
C-Turntable	0.0128	1	0.0128	3.82	0.0725	
Residual	0.0435	13	0.0033			
Lack of Fit	0.0317	11	0.0029	0.49	0.8252	not significant
Pure Error	0.0118	2	0.0059			
Cor Total	0.0827	16				

$$\textbf{\textit{Coating Thickness}} = +\,0.96823 - 0.059109\,p_{N_2} - 0.16259\,p_{Ar} - 0.020406\,\omega_{TT} \quad (1)$$

where p_{N2} is N_2 pressure, p_{Ar} is Argon pressure and ω_{TT} is turntable speed.

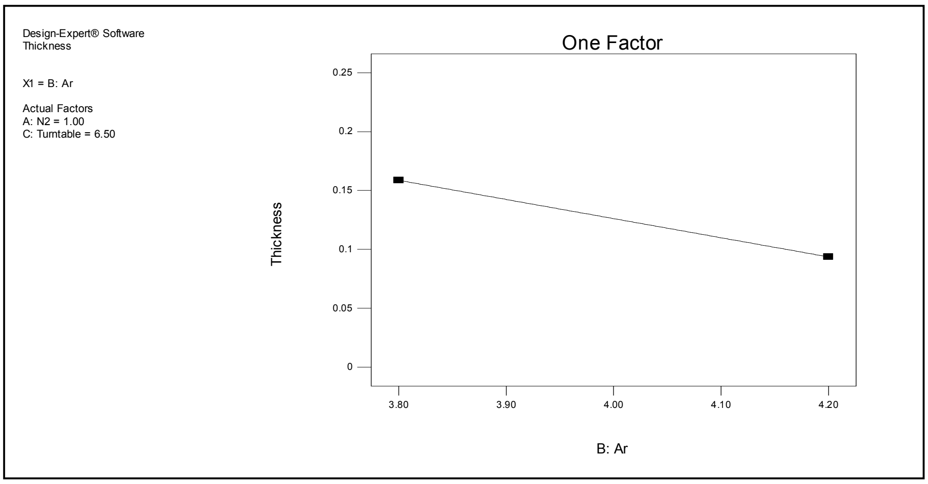

Figure 1. Behaviour of TiN coating thickness in response of Argon pressure

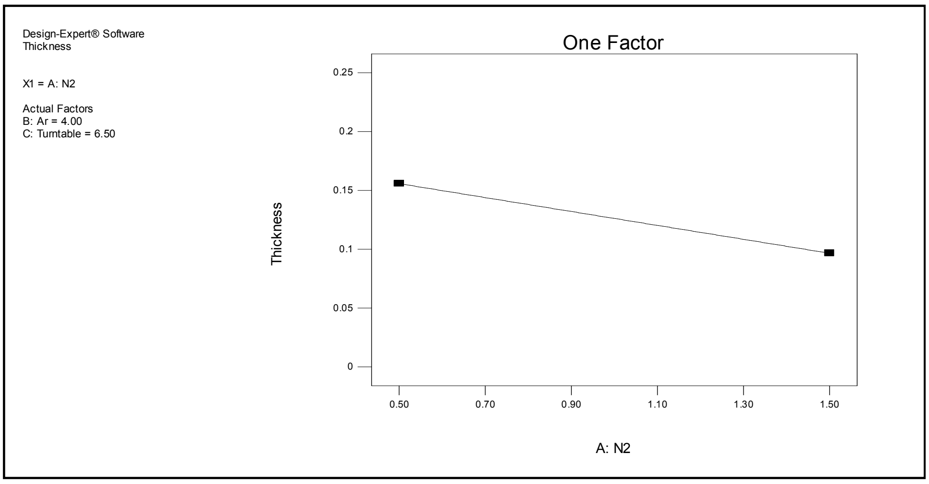

Figure 2. Behaviour of TiN coating thickness in response of N_2 pressure

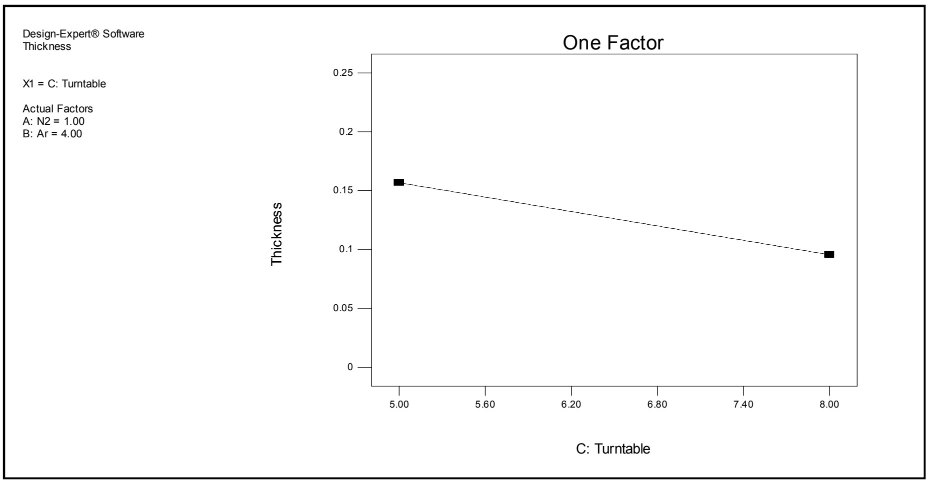

Figure 3. Behaviour of TiN coating thickness in response of turntable speed

Model validation. Three set of data were collected in other experiment to validate the model. As shown in Table 4, the actual coating thickness for validation data fall within the 95% prediction interval (PI). The residual errors which is difference between actual and predicted value for each point as shown in Eq. 2, are ranging between 0.102 to 0.160 µm in absolute value which percentage residual errors were very low. This indicates that the model is accurate to predict the coating thickness.

$$e_{residual} = \frac{v_p - v_a}{v_p} \tag{2}$$

where v_a is actual value and v_p is predicted value.

Table 4. Summary of validation run for TiN coating thickness

Input parameters				**Coating Thickness**					
A:N_2 pressure [$\times10^{-3}$ mbar]	**B:Argon pressure [$\times10^{-3}$ mbar]**	**C: Turntable speed [rpm]**		**Predict (nm)**	**95% PI low (nm)**	**95% PI high (nm)**	**Actual (nm)**	**Error (nm)**	**Error (%)**
0.7	3.85	5.6	0.133	0.187	0.077	0.30	0.160	-0.027	20.3
1.1	3.95	7.4	0.100	0.110	0.003	0.22	0.102	-0.008	2.0
0.9	4.05	6.2	0.130	0.130	0.024	0.24	0.117	-0.013	10.0

Conclusion

TiN coatings were deposited using PVD magnetron sputtering process at different levels of N_2 gas pressure, Argon gas pressure and turntable speed. In this study, the modelling works were done based on RSM technique. The findings of this study have indicated that Argon pressure, N_2 pressure and turntable speed were the significant parameters that influence the deposited TiN coating thickness. Increase in Argon pressure from 3.80×10^{-3} mbar to 4.20×10^{-3} mbar resulted in decrease of the coating thickness. The increase of N2 pressure from 0.5×10^{-3} mbar to 1.5×10^{-3} mbar also resulted in decrease of the TiN coating thickness. Then, as increases of turntable speed from 5.0 rpm to 8.0 rpm resulted in decreases of coating thickness from 0.157 µm to 0.096 µm. Finally, the linear polynomial model was validated and showed accurate result in predicting coating thickness with less residual error.

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