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Influences of sputtering process factors on wear resistance of TiN coated on a machine component

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Abstract. In this work, effects of sputtering process factors including DC current, pressure and Ar to N_2 flow rate ratio on wear resistance of TiN coated on cast stainless steel of a fishing net-weaving machine part namely, an upper hook were systematically investigated using 2^k factorial design, analysis of variance (ANOVA) and other statistical tools. Wear testing experiments of the TiN coated upper hooks were carried out on the real fishing net weaving machine. A normal probability of effects and main and interaction effect plots of process factors were constructed in order to identify the statistically significant process factors and to determine an appropriate operating condition of the process factors with the lowest weight loss. The Ar to N_2 flow rate ratio and the interaction between the pressure and the Ar to N_2 flow rate ratio were found to be statistically significant while pressure and sputtering DC current were not statistically important.

1. Introduction

Titanium nitride (TiN) is widely used as a coating to harden and protect cutting and sliding surfaces in abundant tribological applications [1]. It has a cubic structure of NaCl type with modulus of elasticity of 250-450 GPa and high Vickers hardness of 18-21 GPa [2]. Its surface and material properties are considerably dependent on its creation process. The most common techniques for TiN coating are physical vapor depositions (PVD) including sputtering, filtered cathodic arc and electron beam evaporation and chemical vapor deposition (CVD) [3]. Sputtering is the most generally used method because of high quality thin film coating and well-controlled process. Normally, TiN is produced by reactive sputtering, in which sputtered Ti atoms are reacted with nitrogen ions to form TiN molecules [4].

Oblique angle deposition (OAD) technique is a modified deposition method, in which substrate is tilted at an angle greater than 70° with respect to the normal of deposition direction and rotated at a suitable speed [5]. It provides a functional control of surface nanostructures by shadowing effect and surface diffusion [6]. The main advantage of this technique is ability to control the diameter, shape density of nanostructures by varying the deposition conditions including rotation speed, oblique angle, operating pressure, gas mixing ratio and sputtering power. The technique is applied to TiN hard coating to produce high-density nanocolumnar structure, which could have an extreme hardness, low friction coefficient and very good wear resistance [7].

In this work, TiN was coated on cast stainless steel of a fishing net-weaving machine component namely, an upper hook by OAD technique with DC magnetron sputtering. A three factor, two level full factorial design was selected to investigate the effects of the three sputtering process factors on average weight loss and determine an appropriate level of each process factor with minimization of weight loss.

2. Materials and procedure

The cast stainless steel upper hooks were made by the lost wax casting process and were machined by turning and milling operations. Chemical composition of the workpiece material consisted of 62.28% Fe, 21.99% Cr, 6.91% Ni, 3.12% Cu, 2.2% Si, 1.94% Mn and 1.56% Al. A vibratory finishing machine with mixed ball burnishing media in different sizes ranging from 5 to 8 mm was used for surface finishing. The oblique angle coating system included a high vacuum chamber equipped with a 3″ target magnetron gun, 600 W radio frequency generator, 400 W DC power supply and a turbomolecular pump. Figure 1(a) illustrates an oblique angle sputtering schematic diagram. The titanium nitride was deposited on the upper hooks by reactive sputtering of pure titanium target under a mixture of argon (Ar) and nitrogen (N_2) gases. The oblique angle and rotation speed were fixed at 70º and 45 rpm, respectively. Figure 1(b) depicts a typical photograph of the upper hooks coated by TiN. Three process factors including DC current, operating pressure and Ar to N_2 flow rate ratio were simultaneously investigated using the full factorial design method. The ranges of sputtering DC current, pressure and Ar to N_2 flow rate ratio were 0.35 to 0.45 A, 50 to 100 Pa and 0.5 to 1.5, respectively. The TiN coatings were systematically performed according to the 2^3 factorial design as shown in Table 1. The TiN coated upper hooks were then used to carry out the wear testing experiments on the fishing net-weaving machine with direct sliding between the upper hooks and the fishing net (nylon 6). After carrying out the wear testing experiments, each upper hook was cleaned with a dry cloth and then wiped clean with alcohol. Each hook was weighed with 4-digit scale and the weight loss data was recorded. The analysis of variance (ANOVA) was employed to investigate the influences of the three sputtering process factors on the wear resistance according to designed experiments.

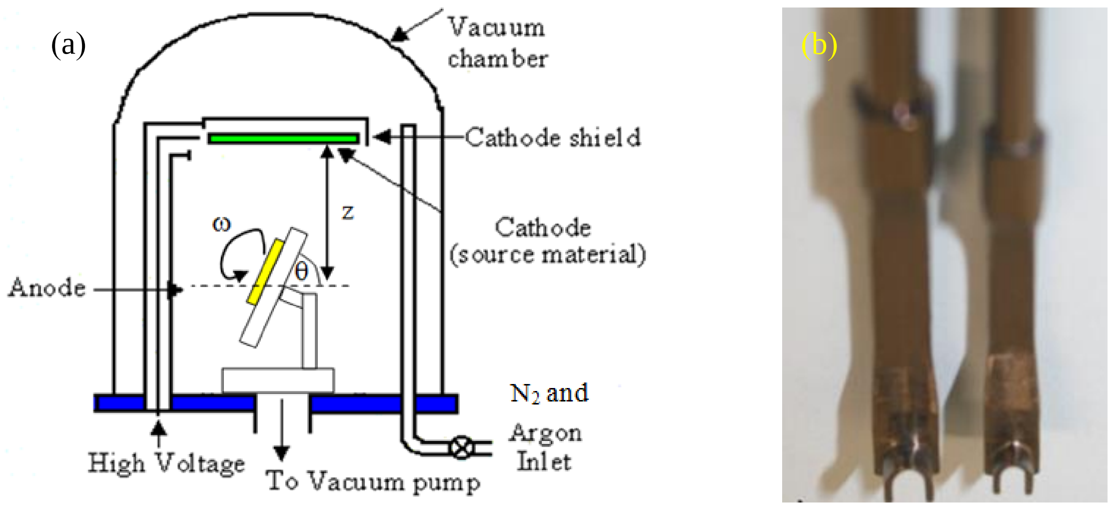

Figure 1. (a) An oblique angle sputtering schematic diagram (b) a typical photograph of upper hooks coated by TiN

Table 1. Factors and their levels in TiN coated on cast stainless steel process

Experimental run	DC current (A)	Pressure (Pa)	Ar /N_2
1	0.35 (-)	50 (-)	0.50 (-)
2	0.45 (+)	50 (-)	0.50 (-)
3	0.35(-)	100 (+)	0.50 (-)
4	0.45 (+)	100 (+)	0.50 (-)
5	0.35 (-)	50 (-)	1.50 (+)
6	0.45 (+)	50 (-)	1.50 (+)

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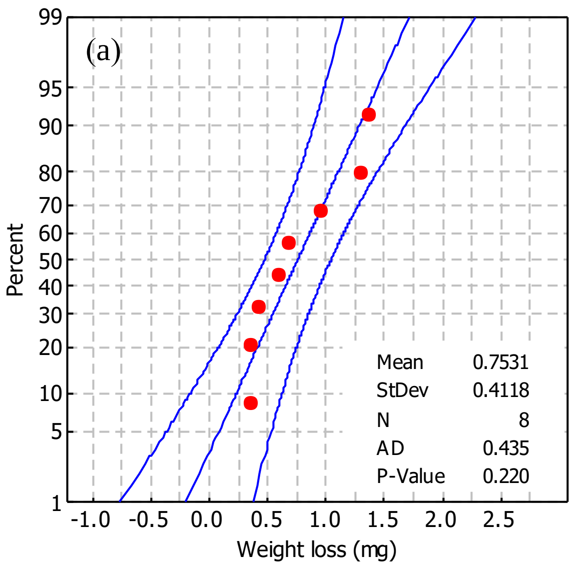
7	0.35 (-)	100 (+)	1.50 (+)
8	0.45 (+)	100 (+)	1.50 (+)

3. Results and discussion

Before performing ANOVA, the data set of weight loss values should be evaluated whether it distributes as normality. The probability plot was used to illustrate the data points, fitted line of the data points and associated confidence intervals (C.I.) based on parameters estimated from the data set along with an Anderson-Darling (AD) goodness-of-fit statistic and associated *p*-value [8]. Figure 2(a) illustares probability plot of weight loss value. The probability plot shows that the plotted points of the complete data set of weight loss roughly form a straight line and fall within the 95% confidence interval. Moreover, AD statistic of the data set was small with high *p*-value compared to the level of significance of 0.05. This confirmed that normal probability distribution fitted the data set of the weight loss values capably. Furthermore, the adequacy of the underlying model should be checked using a primary diagnostic tool, residual analysis. Figure 2(b) displays a normal probability plot of residuals. If the underlying error distribution is normal, this plot will resemble a straight line [8]. Obviously, the normal probability plot of residuals for weight loss did not indicate anything particularly troublesome problem of normality. Hence the residual analysis was satisfactory.

The results from the 2^3 full factorial experiment were analyzed by a half normal probability plot of effects using the Design Expert software package [9]. A half normal probability plot of effects is a plot of the absolute value of the effect estimates against their cumulative normal probabilities [8]. The significant effects with nonzero means will not lie along the straight line while the effects that are imperceptible are normaly distributed, with mean zero and variance σ^2 and will contribute to fall along a straight line on this plot. The straight line on the half normal plot always passes through the origin and passes close to the fiftieth percentile data value. Figure 3(a) presents the half normal plot of the effects for weight loss value. The important effects for the weight loss were factor *C* (Ar to N_2 flow rate ratio) and interaction between factor *B* (pressure) and factor *C*. Factor *B* was included to preserve hieraarchy in the model. This implied that if the *BC* interaction was in the model, both the main effects *B* and *C* should be included. Pareto chart of effects is another tool used to evaluate the effects of the process factors**.** Figure 3**(**b**)** illustrates the Pareto chart of effects for this study**.** The lower horizon line indicates Student**'**s *t*-test value for the minimum statistically significant effect magnitude for a level of significance of 0**.**05**.** Only factor *B* and *BC* interaction were higher than the threshold. Like the half normal plot of the effects, factor *B* was included to preserve hieraarchy in the model. Thus, only pressure, Ar to N_2 flow rate ratio and the interaction between pressure and Ar to N_2 flow rate ratio were statistically significant at the level of significance of 0.05.



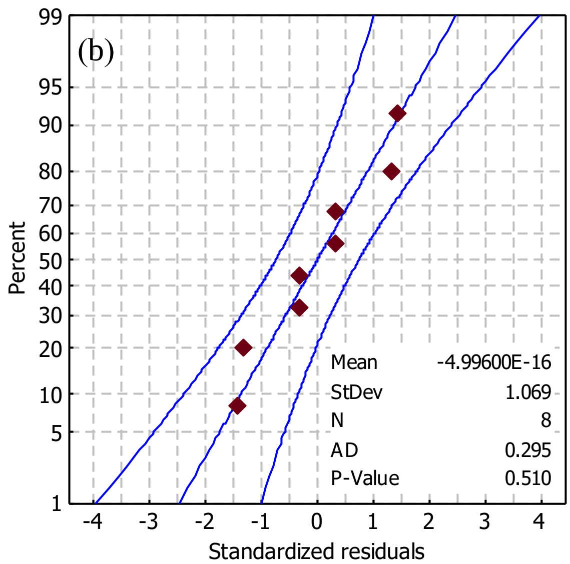


Figure 2. (a) Probability plot of weight loss value and **(b)** normal probability residual plot for weight loss

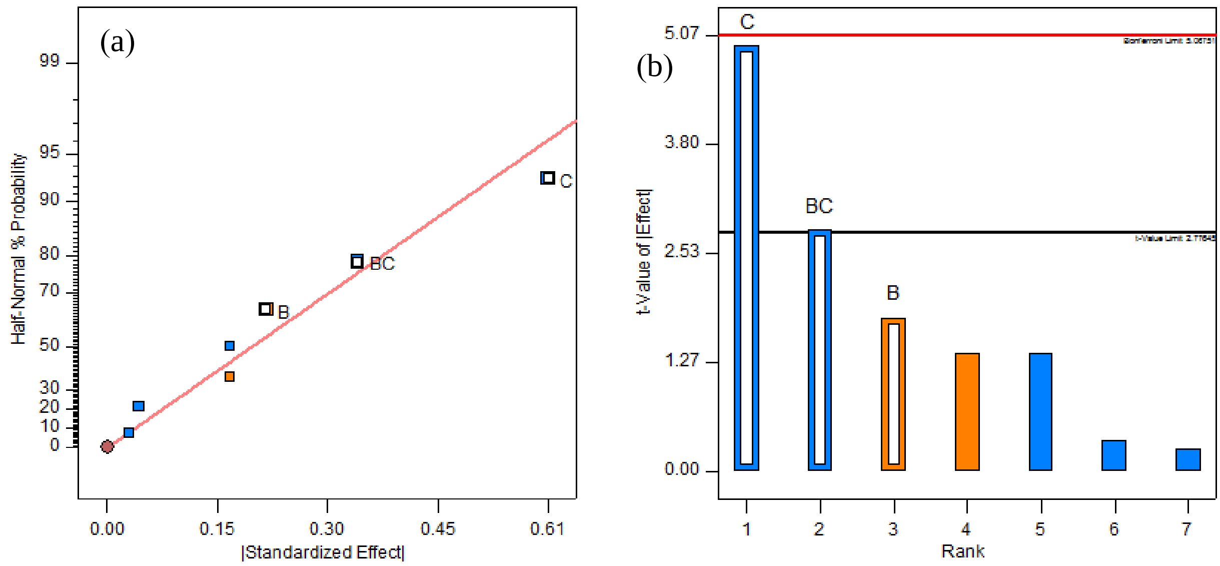


Figure 3. (a) Half normal probability plot of effects and **(b)** Pareto chart of effects for weight loss value

Table 2 shows the effect estimates, sum of squares and percent contribution of the three sputtering factors on the weight loss of the TiN coated upper hooks. Based on variance component estimates, 61.94% of the total variation in weight loss value was due to the difference between the Ar to N_2 flow rate ratio levels while 19.91% resulted from the differences between pressure levels and Ar to N_2 flow rate ratio levels. Hence Ar to N_2 flow rate ratio was approximately three times as influencial as the interaction between the pressure and the Ar to N_2 flow rate ratio.

Table 2. Effect estimates, sum of squares and percent contribution

Model term	Effect estimate	Sum of squares	Percent contribution
A-DC current	0.17	0.057	4.80
B-Pressure	0.22	0.096	8.06
C-Ar/N_2	-0.61	0.740	61.94
AB	-0.17	0.057	4.80
AC	-0.044	0.004	0.32
BC	-0.34	0.240	19.91
ABC	-0.031	0.002	0.16

The effects of the statistical significance of the three sputtering factors on the weight loss of the TiN coated upper hooks were evaluted by ANOVA as shown in Table 3. The results in Table 3 were analyzed with ANOVA used for investigating the influence of the three sputtering factors on response weight loss. Table 3 reveals that the model F-value of 11.89 with a p-value of 0.0184 was adequate. A coefficient of determination (R^2) of 0.8992 defined as the ratio of the explained variation to the total variation and was a measure of the degree of fit. The adjusted R^2 of 0.8235 was in reasonable agreement. This concluded that the Ar to N_2 flow rate ratio and the interaction between the pressure and Ar to N_2 flow rate ratio significantly affected the mean weight loss at the level of significance of 0.05.

The statistical model of weight loss as the function of pressure, Ar to N_2 flow rate ratio and the interaction between the pressure and the Ar to N_2 flow rate ratio was expressed in Eq**.** (1)**:**

$$weight\,loss = 0.75 + 0.11B - 0.3C - 0.17BC \tag{1}$$

where *weight loss* represented the response variable, *B* denoted pressure and *C* was Ar to N_2 flow rate ratio. The equation was in terms of coded factors on the -1, +1 scale**.**

Table 3. ANOVA table for weight loss

Source	Df	SS	MS	*F*-value	*p*-value
Model	3	1.07	0.36	11.89	0.0184
B-Pressure	1	0.09	0.09	3.20	0.1482
C-Ar/N_2	1	0.74	0.74	24.57	0.0077
BC	1	0.24	0.24	7.90	0.0483
Error	4	0.12	0.03		
Total	7	1.19			
	PRESS = 0.48	R^2 = 0.8992	Adj. R^2 = 0.8235		

Figure 4(a) presents a plot of actual weight loss versus the predicted weight loss values. This plot shows the prediction capability of the model. The relationship between the actual value and the predicted value of weight loss was expressed in Eq. (2):

$$\hat{P} = 0.076 + 0.8992\,weight\,loss \tag{2}$$

where $\hat{P}$ represented the predicted weight loss whereas *weight loss* denoted the actual value. The R^2 was 0.899 indicating the model in Eq. (2) accounted for 89.9% of the variability in the data. The intercept of the linear model was close to zero and the slope was about 1. The plot indicated that model could satisfactorily be used in predicting the weight loss response. Similarly, Figure 4(b) also shows the pair difference between the actual value and the predicted value of weight loss in each experimental run. This plot clearly indicated that there was a small difference between the actual value and the predicted value of weight loss in each experimental run. This also confirmed that the model in Eq. (1) was adequate.

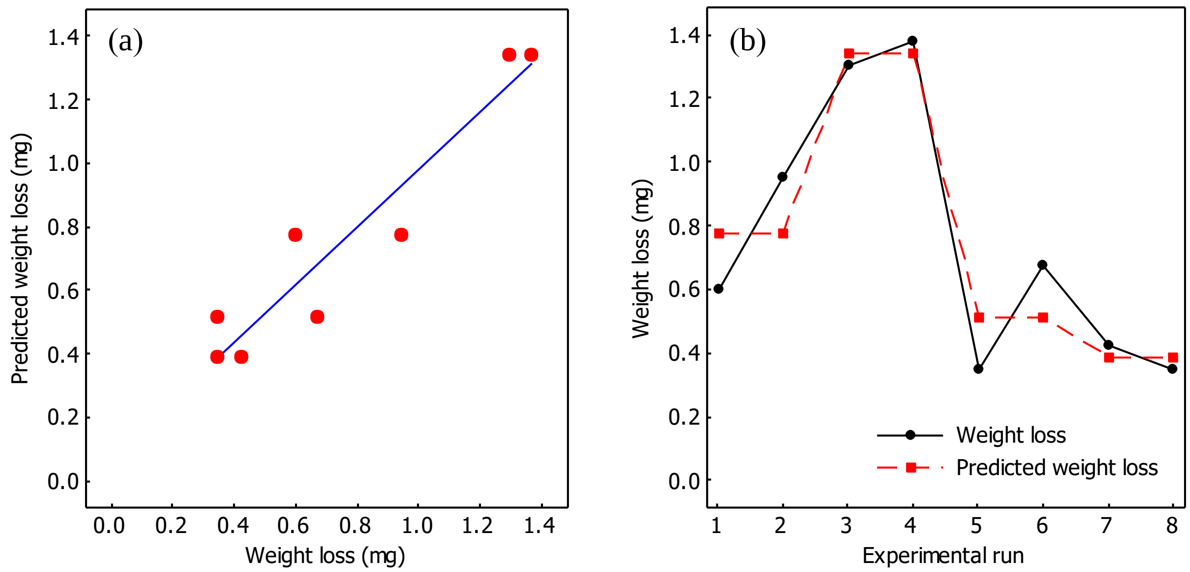
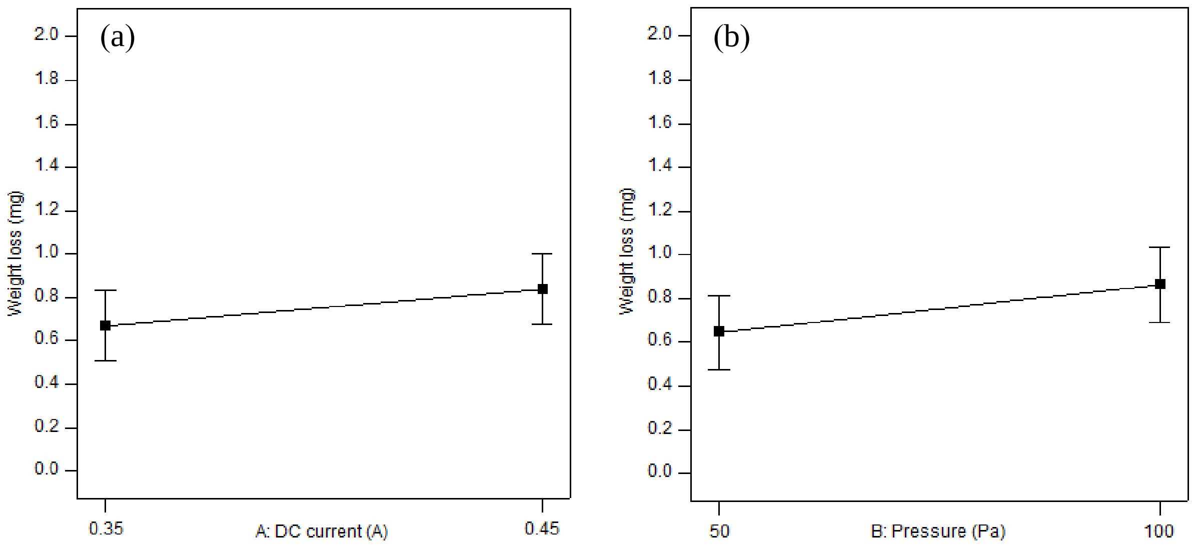


Figure 4. (a) Scatter plot between actual weight loss and predicted weight loss and (b) the pair difference between the actual value and the predicted value in each experimental run

The objective of this study was to minimize weight loss of the TiN coated upper hooks after carrying out the wear testing experiments**.** To assist in accomplishing this objective, graphical methods can be effectively used**.** A main effect plot is a graph of the averages at each level of a factor [8]. A main effect occurs when the average response changes across the levels of a factor. The main effect plot is employed to compare the relative strength of the effect of the factor. Although this plot is useful to compare main effect, the ANOVA table is a primary tool used to significantly assess the influence of the factor. The main effect plots displayed in Figures 5(a) and 5(b) prove that there were no statistically significant differences of the averages weight loss values between the two levels of the factor DC current and the factor pressure. On the other hand, there was a statistically significant difference between the two levels of the factor Ar to N_2 flow rate ratio as shown in Figure 5(c). The main effects do not have much meaning when they are involved in statistically significant interactions [8]. Hence it is necessary to evaluate any interractions that are important. In order to interpret interaction effectively, an interaction plot was constructed as illustrated in Figure 5(d), (Pressure and Ar/N_2 interaction). The non-parallel lines revealed that there was a strong interaction between pressure and Ar to N_2 flow rate ratio. The interaction indicated that Ar to N_2 flow rate ratio had little effect at high (+) Ar to N_2 flow rate ratio but a high positive effect at low (-) Ar to N_2 flow rate ratio. The pressure effect was very small when the Ar to N_2 flow rate ratio was at the high level and very large when the Ar to N_2 flow rate ratio was at the low level. However, the effect of Ar to N_2 flow rate ratio was not different at low level of pressure. Therefore, the minimum weight loss value was obtained when pressure and Ar to N_2 flow rate ratio were at low levels (i.e., pressure at 50 Pa and Ar to N_2 flow rate ratio at 0.5). This would allow the reduction of the Ar to a lower production cost. The previous studies have found that as a flow rate of Ar decreases, the grain size of TiN coating decreases resulting higher hardness as well as wear resistance improvement [1,10,11]. In addition, the effect of DC current was not different. Hence weight loss value was prefered for lower level of DC current.



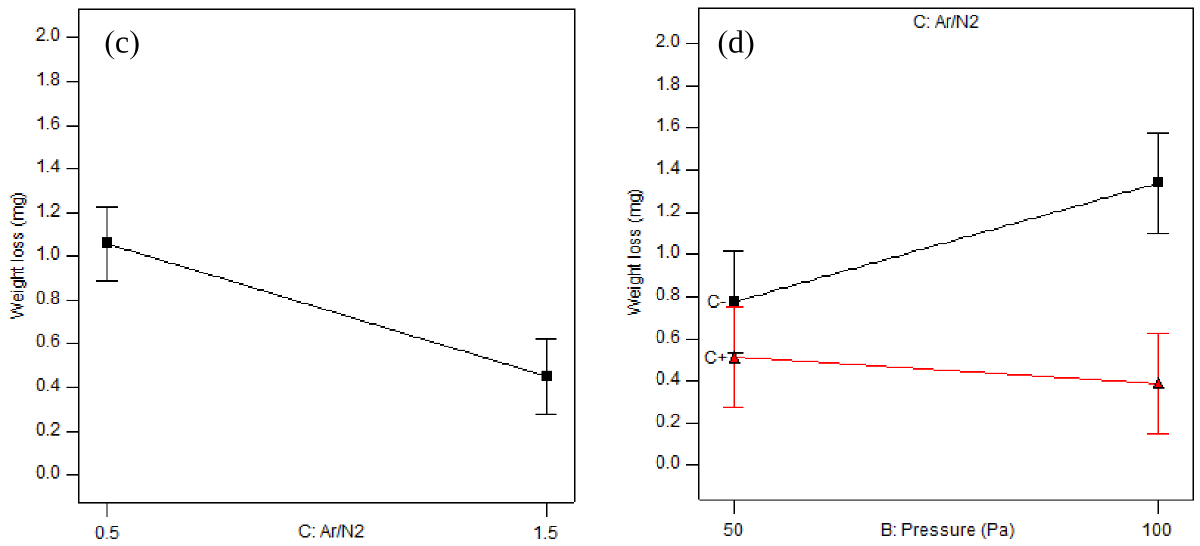


Figure 5. Effect graphs for factors (a) DC current **(b)** pressure **(c)** Ar to N_2 flow rate ratio and **(d)** interaction between pressure and Ar to N_2 flow rate ratio

4. Conclusion

This study had shown how statistically designed experiments combined with other statistical techniques could be used for investigating the influences of sputtering process factors affecting weight loss of TiN coated fishing net weaving machine component during carrying out the wear testing experiments. A normal probability of main and interaction effects of process factors was plotted against cumulative probability in order to identify the statistically significant process factors. The Ar to N_2 flow rate ratio and the interaction between the pressure and the Ar to N_2 flow rate ratio were found to be statistically significant at the level of significance of 0.05. An appropriate operating condition of the process factors was at DC current of 0.35 A, pressure of 50 Pa and Ar to N_2 flow rate ratio of 0.5.

Acknowledgements

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