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Determination of the Best Coating Parameters for Adhesion Strength and Hardness Using Taguchi Method

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Abstract
Because of its unique properties, such as high strength, resistance to corrosion, and biocompatibility, titanium-based alloys are among the metals used in biomedical implants the most frequently. The objective of this work was to cover the surfaces of TiN-coated Ti-51at. %Ni and Ti-27at. %Nb alloys to improve their adhesion strength and surface hardness. Titanium Nitride (TiN) coatings on substrates were produced using the physical vapour deposition magnetron sputtering technique. The deposition parameters, which included temperature, power, nitrogen flow rate and bias voltage, were varied during the process. To keep the experimental runs to nine, the Taguchi orthogonal array standard 9-run matrix L9(34) and Taguchi technique of optimising parameters through experiment design were employed. 370 W, 4.7 sccm, 50 V, and 100 °C were the combination of parameters that produced the best Ti/TiN coating adhesion on Ti-51 at %Ni and the nitrogen flow rate at 4.7sccm added more to the coating's adhesive strength, according to the studies. Ti/TiN coating on Ti-27 at %Nb was investigated using a range of process parameters, such as nitrogen flow rate, temperature, bias voltage, and DC power. Maximum surface hardness and adhesion strength may be achieved at the following ideal values: 370 W, 4.7 sccm, 15 °C, and 100 V, and 6.7 sccm, 150 °C, 100 V, and 300 W, respectively. Using the process parameters of nitrogen flow rate, temperature, bias voltage, and DC power, Ti/TiN was successfully deposited on Ti-25 at %Ta. According to the Taguchi optimisation technique, the best parameter combinations for coating strength and surface hardness are 4 sccm, 150 °C, 75 V, and 370 W, respectively.

Keywords: *Bias voltage, temperature, nitrogen flow rate, power.*

1.0 Introduction

Surface coating in scientific and technological fields has evolved rapidly in the past few decades, becoming a major focus of research. With the increasing value of coatings and the development of new materials for the biomedical and engineering industries, innovative coating processing technologies have risen significantly. There are various coating deposition processes and technologies that are solely based on physical or chemical processes. The most critical coating processes involve the use of gas-phase chemical processes, liquid-phase chemical techniques, glow discharge, and evaporation methods (Miyake, 1997). However, due to the process mechanism overlap and the emergence of hybrid deposition techniques, no single scheme can precisely describe and categorise all coating processes.

Deposition processes may generally be classified as involving droplets such as arc spraying, plasma spraying, wire-explosion spraying and detonating gun coating involving a transfer mode of an atom at a time, for example, evaporation processes for physical vapour deposition (PVD), sputtering processes for ion and plating, chemical vapour deposition (CVD) and electrodeposition. These method's main flaw is the porosity of the final deposit, which has an impact on its properties. Every deposition has several steps in the process. One is synthesising the material to be deposited. These steps include transitioning from the solid or liquid phase to the vapour phase, and for compound depositions, a reaction between the components of the compounds. Some of these reactions can be released into the chamber as a gas. Other steps include transporting vapours between the source and substrate and condensing vapours in the chamber (Boing *et al.*, 2020).

There are several coating processes often used in depositing material on substrates such as Ti-alloys. These procedures are often referred to as post-processing. They have a significant impact on the appearance, function, and longevity of the substrates. These are processes that influence the surface of the substrates or add a thin layer to the substrates overall surface (Adeoye *et al., 2024*). Improved hardness, wear resistance, friction control, and adhesion strength are just some of the essential applications. There are also benefits in terms of lubrication, corrosion resistance, and overall appearance. The current research coating process selected was physical vapour deposition which comprises of coating processes such as, evaporation, ion plating, sputtering and cathodic arc deposition.

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Coatings are often utilized to increase a material's surface qualities while having no detrimental influence on its bulk properties. Coatings can act as an efficient barrier layer to minimize the release of ions that lead to corrosion. (Fauchais and Vardelle, 2012). It may increase hardness while offering high surface finishing, minimizing wear rates and friction (Chen *et al.*, 2013; Chowdhury *et al.*, 2008). It is critical to note that a coating layer functions as intended only if it adheres properly to the metal substrate and is robust enough to transfer all loads. On the other hand, coatings have limited substrate adherence, allowing chemical connections to develop between the layers.

This study seeks to establish the ideal coating parameters for adhesion strength and hardness using the Taguchi technique, as well as assess the impacts of the coating on qualities such as surface hardness and adhesion strength.

The objectives of this work comprise to prepare the experiment sample runs using Taguchi method, coating of the various samples using magnetron sputtering machine, and characterisation of each sample to determine the best coating parameters.

In the research scope, TiN coating was deposited on Ti-51at.%Ni and Ti-27at.%Nb alloys using the DC magnetron sputtering physical vapour deposition (PVD) method. The PVD process was initially studied by adjusting the deposition parameters such as bias voltage, temperature, pressure, and gas flow rate in order to identify optimal parameters for coating production. In addition, an optimization procedure employing the Taguchi technique was used to determine the ideal coating parameters for improved adhesion and hardness.

2.0 Materials and Methods

2.1 Materials

The Ti-based alloys used as substrates in this study were Ti-51at. %Ni and Ti-27at. %N. The compositions of the materials were selected based on the phase diagram and work done by other researchers (de Souza and Robin, 2003; Massalski, *et al.*, 1990; Štěpán and Losertová, 2011), indicating that the alloys have the required properties, such as corrosion resistance, hardness, biocompatibility, shape memory effect and may be used for biomedical applications.

2.2 Methods

2.2.1 *Sample Preparation*

The sample was prepared for the coating process, followed by a mechanical test, characterization, corrosion test and anti-bacterial test, which involved cutting, grinding and polishing. The samples preparation steps follow the standard metallographic method.

2.2.2 *Coating Process Using Magnetron Sputtering PVD*

The material coated on Ti-51 at %51 and Ti-27 at %Nb substrates were titanium nitride (TiN) which was produced from a reaction between the pure titanium target material and nitrogen gas in the PVD chamber. The substrates were prepared so that a clean metal surface free from any contamination was obtained.

Based on the Taguchi optimization process after the selection of orthogonal array (OA) was to run the experiments based on the OA. The substrates to be coated were prepared for the PVD Magnetron sputtering equipment (SG Control Engineering Pte Ltd, Model: TF450), as seen in Figure 1. The coating was carried out according to the experimental setup illustrated in Figure 1.

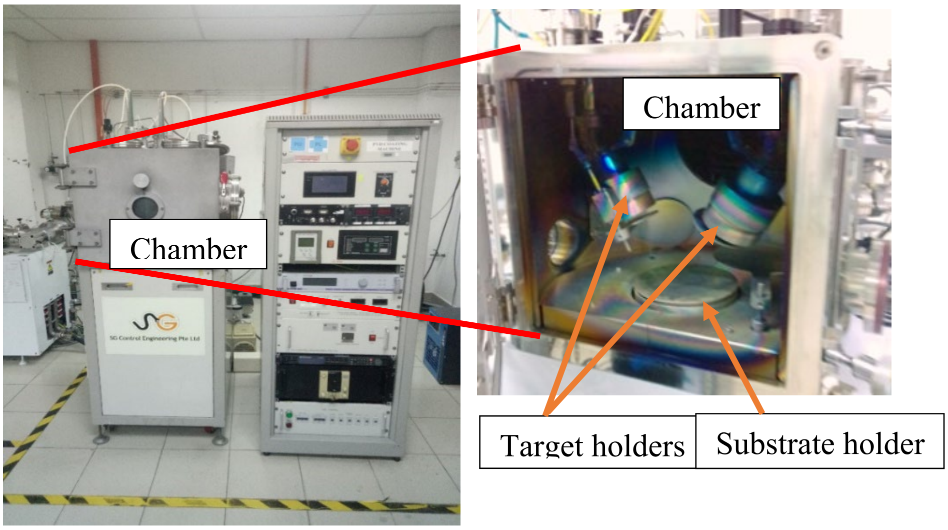

Figure 1: Physical vapour deposition magnetron sputtering machine (SG Control Engineering Pte Ltd, Model: TF450)

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Based on Taguchi method, the number of specimens required were 81 instead of 360 that could achieve the desired results. The process parameters applied in the coating process were power, substrate temperature, bias voltage and nitrogen flow rate. The rotation of the substrate holder was set to 5 r/min throughout the deposition experiments. This was followed by expelling argon gas into the chamber to create the plasma that etched the native oxide layer on the target's surface. The substrates were gradually heated to the desired temperature. TiN was deposited in the PVD chamber by sputtering the Ti target while connected to a DC power source in a 1:5 mixed N2-Ar environment. The substrates were initially coated with Ti interlayer after 60 minutes, followed by a simultaneous sputtering of Ti and N_2 for 240 minutes to generate high surface hardness and adhesion.

A strong negative voltage was given to the sputtering sources in the vacuum chamber depicted in Figure 1. The ensuing electrical gas discharge generates positive argon ions, which are propelled toward and atomize the coating material, the Ti target. In the gaseous phase, the evaporated and atomized material interacted with a gas containing the hard coating's metallic component. As a consequence, a thin, compact coating with the necessary structure and composition is placed onto the substrate. The temperature of the substrate being coated was from 100 – 200 °C. The resultant coating layer produced consists of Ti/TiN

2.3 Materials characterization

Materials characterization and microstructural analysis were performed using standard equipment, namely optical microscope, SEM, EDS and XRD.

3.0 Results and Discussions

Here, the detailed results of the experiments conducted during this research work will be discussed. The results of characterisation and the effects of Magnetron Sputtering Physical Vapour Deposition Magnetron Sputtering (MSPVD) parameters on the properties of Ti-51 at. %Ni and Ti-27 at. %Nb alloys coated with Ti/TiN about their surface morphology, surface hardness, adhesion strength and coating thickness are discussed.

3.1 TiN coating on Ti-51at. %Ni and Ti-27at. %Nb alloys

TiN was effectively deposited on the as-received alloys, Ti-51 at %Ni and Ti-27 at %Nb, using a PVD magnetron sputtering system. To attain the intended findings, the experimental design was robustly prepared before coating. The most crucial stage in the design of the experiment (DOE) is the selection of control factors. This was done to identify non-significant variables. In this regard, the Taguchi technique of experimental design procedure with the smallest number of trials to be carried out to get the important information necessary for optimization through the application of statistical methods as stated in. Arudi (2022).

3.2 Thickness, hardness and adhesion strength results for the coated Ti-51 at. %Ni and Ti-27 at. %Nb

The thickness, adhesion strength and surface hardness results were measured using scratch test machine (Micro Material Nano test, Wrexham, UK) and microhardness tester (SHIMADZU Micro Hardness Tester, HMV-2 Series) respectively to determine the thickness, adhesion strength and surface hardness of the coatings. The scratch method was used to evaluate thin hard coating adhesion and is a useful approach for enhancing coating or quality control (Akhter *et al.*, 2021). The following Tables and Figures show the hardness test and scratch test results for the coated Ti-51. %Ni and Ti-27 at. %Nb respectively.

Table 1 shows the results of all nine experimental runs. The hardness measurement for each experiment was measured three times and the average was recorded as shown in Table 1. Figures 3 and 4 show the images of the (a)lowest and (b) highest hardness measurements of the alloys. The hardness and adhesion tests for Ti-51 at. %Ni shows that Experiment number 5 with the coating parameters of 370 W, 150 ^{O}C, 100 V and 4.7sccm gave 395 HV and 3998mN as the highest hardness and the highest adhesion strengths respectively, while Experiment number 7 having coating parameters of 440 W, 100 ^{O}C, 100 V and 5.7sccm showed 278 HV and 1674mN as the lowest hardness and lowest adhesion strengths respectively.

Table 1: The hardness test, scratch test and their corresponding calculated S/N ratio results for Ti-51 at. %Ni

No.	Average Measured Surface hardness (HV)	Scratch Force (mN)	Thickness (nm)	Calculated S/N Ratio	
				Hardness S/N(dB)	Adhesion S/N(dB)
S1	339	2989	2100	50.5754	69.5105
S2	371	1694	1400	51.2214	64.5783
S3	316	1947	1600	49.7499	65.7873

S4	370	3693	2500	51.2617	71.3476
S5	**395**	**3998**	**2390**	**51.8489**	**72.0369**
S6	315	3319	3340	49.8436	70.4201
S7	278	1674	1330	48.7898	64.4751
S8	289	2648	1260	49.1663	68.4584
S9	318	2417	2200	50.0471	67.6655
Average S/N	332	2709	2014	50.2782	68.2533

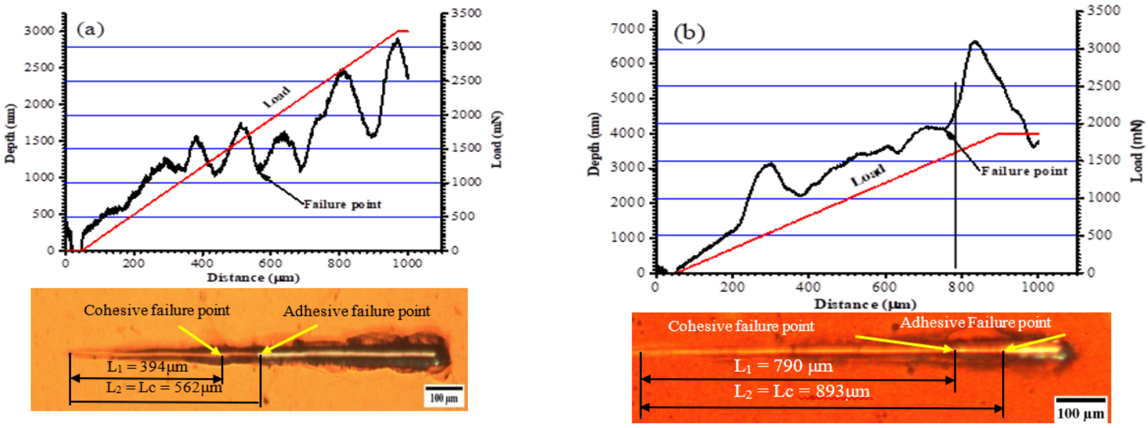

Figure 2: Scratch test for the coated Ti-51at%Ni (a) Lower adhesion: exp.no. 7, (b) Higher adhesion strength: exp. no. 5

In Ti-27 at. %Nb coating, the hardness and adhesion test results revealed that the coating parameters 370 W, 150 ^{o}C, 100 V and 4.7sccm in exp. no. 5 showed the highest hardness value of 358, while exp. no 3 with 300 W, 200 ^{o}C 100 V and 6.7sccm coating parameters showed the highest adhesion strength of 2110mN and the lowest hardness of 264. However, exp.no. 6 with 370W, 200^{o}C, 50V and 5.7sccm deposition parameters gave the lowest adhesion strength of 1061mN.

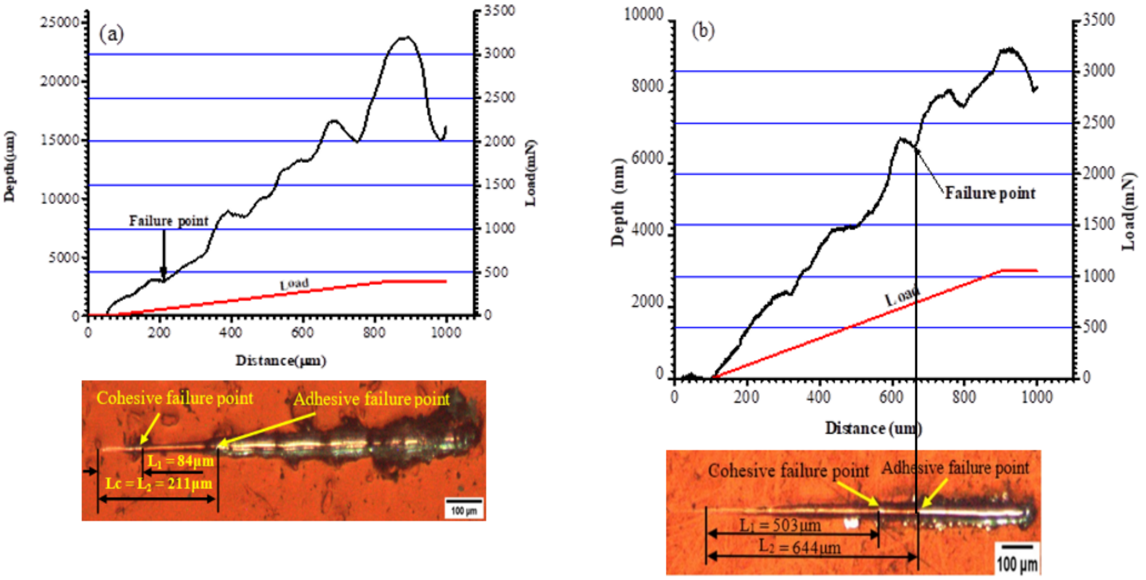

Figure 3: Scratch test for the coated Ti-27at%Nb (a) Lower adhesion: exp.no. 6, (b) Higher adhesion strength: exp. no. 3

Exp. no 6 revealed that the coating carried out using deposition parameters, 370W, 200 ^{o}C, 50V and 5.7sccm showed the highest hardness of 450HV and the highest adhesion strength of 3422mN. The lowest hardness of 230 was achieved in exp.no. 7 with deposition parameters of 440W, 100 ^{o}C, 100V and 5.7sccm, and exp. no. 2 with the deposition parameters of 300W, 150 ^{o}C, 75V and 5.7sccm produced the lowest adhesion strength of 1386(mN).

The summary of the influence of process parameters on the hardness, adhesion and the thickness of all the TiN coated samples before the optimization process are as tabulated in Table 2.

Table 2: Influence of Process Parameters on TiN Coating

Samples	Exp. No	Properties	Value	Power (W)	Temp. (^{O}C)	Bias (V)	N_2 (sccm)
Ti-51at. %Ni	5	Hardness	Highest	370	150	100	4.7
		Adhesion	Highest	370	150	100	4.7
	7	Hardness	Lowest	440	100	100	5.7
		Adhesion	Lowest	440	100	100	5.7
	6	Thickness	Highest	370	200	50	5.7
	8	Thickness	Lowest	440	150	100	6.7
Ti-27at. %Nb	5	Hardness	Highest	370	150	100	4.7
	3	Adhesion	Highest	300	200	100	6.7
		Hardness	Lowest	300	200	100	6.7
	6	Adhesion	lowest	370	200	50	5.7
	6	Thickness	Highest	370	200	50	5.7
	8	Thickness	Lowest	440	150	100	6.7

3.3 Data analysis and optimization process

The optimization technique utilized Signal-to-Noise ratio (S/N) response analysis to analyze data, optimize parameters, and identify statistically relevant process parameters.

3.3.1 Signal-to-Noise ratio (S/N) response analysis

Following the experimental runs, data analysis is performed using the signal-to-noise ratio (S/N) to optimize the parameters and determine which process parameters are relevant to the optimization process. Table 3 displays the computed S/N ratios for hardness and adhesion strength of TiN coatings on Ti-51 at %Ni and Ti-27 at %Nb substrates.

Table 3. Calculated S/N ratio for hardness and adhesion strength of TiN coating on Ti-51at. %Ni and Ti-27at. %Nb.

Experimental No.	(a) Calculated S/N ratio for Ti-51at. %Ni S/N Ratio (dB)		(b) Calculated S/N ratio for Ti-27at. %Nb S/N Ratio (dB)	
	Hardness	Adhesion	Hardness	Adhesion
1	50.5754	69.5105	48.557	60.9377
2	51.2214	64.5783	48.1	65.168
3	49.7499	65.7873	48.377	66.4857
4	51.2617	71.3476	50.361	65.1295
5	51.8489	72.0369	51.033	66.4195
6	49.8436	70.4201	49.855	60.5143
7	48.7898	64.4751	49.223	62.0349
8	49.1663	68.4584	49.032	65.6615
9	50.0471	67.6655	49.083	62.709
Average calculated S/N	50.2782	68.2533	49.291	63.8956

The S/N response value of each of the level measured data for adhesion strength and surface hardness of Ti-51at. %Ni and Ti-27at. %Nb is shown in Table 4 with their corresponding plotting Figures below each respectively as shown. The data recorded were plotted, as shown in Figures 4 (a) and (b). The difference between the S/N ratio of the highest and lowest of specific levels is ranked and regarded as a delta. The highest delta value in the S/N response is ranked as 1, while the next second, third, and fourth delta are ranked as 2, 3, and 4 respectively.

3.3.1.1 Signal-to-Noise ratio (S/N) response analysis for hardness and adhesion strengths of TiN coated on Ti-51at. %Ni

In Table 4(a), DC power assigned to factor (A) has the highest delta value, and it is ranked as number 1, and temperature designated to factor (B) with the lowest delta is ranked as number 4. The further the graphs

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deviate from the horizontal axis, as seen in Fig. 4(a), the higher their rankings. DC power (factor A) is more deflected than other factors, therefore it is considered the most important, followed by N2 flow rate (factor D), bias voltage (factor C), and temperature (factor B). As a result, the process parameters with the highest S/N on each factorial level, such as DC power (A2), N2 flow rate (D1), bias voltage (C1), and temperature (B1), are chosen as the optimal parameters to improve adhesion strength and are denoted as A2D1C1BI (370W, 4.7sccm, 50V, and 100°C).

Table 4. Signal-to-noise ratio(S/N) response values for adhesion strength and surface hardness of TiN coated on Ti-51at. %Ni

Level	**(a) Signal-to-noise ratio(S/N) response value for adhesion strength**				**(b) Surface hardness signal-to-noise ratio(S/N) response value**			
	Power (A)	**Temp. (B)**	**Bias (C)**	**N_2 (D)**	**Power (A)**	**Temp. (B)**	**Bias (C)**	**N_2 (D)**
1	66.63	**68.44**	**69.46**	**69.74**	50.67	50.28	49.93	**50.86**
2	**71.27**	68.36	67.86	66.49	**51.08**	**50.85**	**50.94**	50.07
3	66.87	67.96	67.43	68.53	49.39	50.01	50.27	50.2
Delta	4.64	0.48	2.03	3.25	1.69	0.84	1	0.79
Rank	1	4	3	2	1	3	2	4
Average	**68.25**				**50.38**			

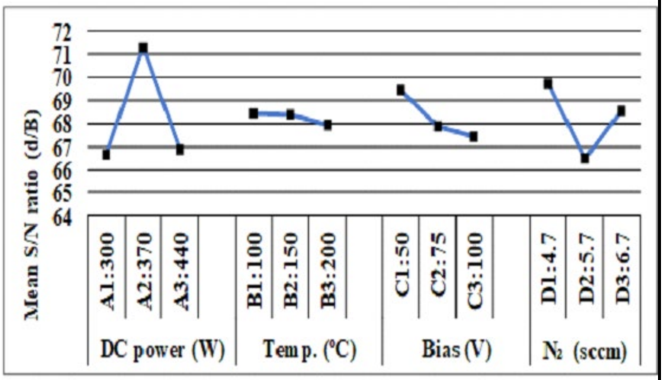

Figure 4(a): Response graphs showing adhesion strength in terms of S/N ratio versus parameters

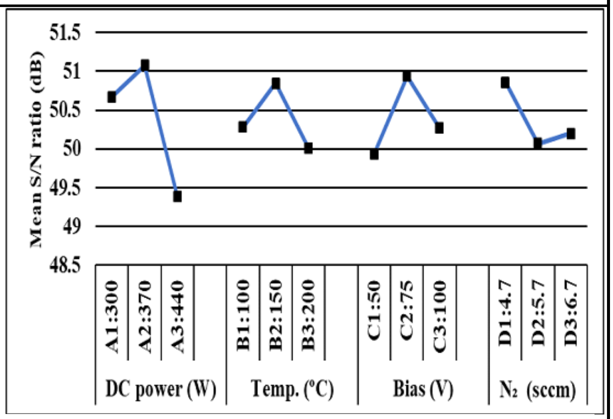

Figure 4(b): Response graphs showing surface hardness in terms of S/N ratio versus parameters

In a similar analysis for surface hardness, the highest S/N ratio assigned to each factorial level, as shown in Table 4(b), and in order of their deflection from the axis of the horizontal, as shown in Figure 4(b), shows that DC power (factor A) is ranked first as the most significant parameter, followed by bias voltage (factor C), N2 flow rate (factor D), and temperature (factor B). The optimal combination of process parameters to accomplish the requisite high surface hardness are therefore DC power (A2), bias voltage (C2), nitrogen flow rate (D1), and temperature (B2), which are denoted as A2C2D1B2 (370w, 75v, 4.7sccm, and 150°C).

A confirmation test is required to validate the results of optimal combinations of parameters obtained. Table 5 shows the optimum parameters to be used for the confirmation test. The validity of the confirmation test will be determined by using the reduced predicted value of the S/N ratio equation (Bushroa, *et al.*, 2011b).

$$\bar{y} = \bar{y}_m + \sum_{i=1}^{n} (\bar{y}_i = \bar{y}_m) \qquad 1$$

Where:
$\bar{y}$ is the predicted S/N ratio;
$\bar{y}_m$ *is the total average of the S/N ratio*
$\bar{y}_i$ *is the average S/N ratio of the optimal level ,*

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Table 5: Optimum parameters for confirmation test

Parameter		Power(W)	Temp. (^{O}C)	Bias (V)	N_2 (sccm)
		A	B	C	D
Hardness					
Ti -51at%Ni	S/N	**51.08**	**50.85**	**50.94**	**50.86**
	VP	370	150	75	4.7
	Adhesion				
	S/N	**71.27**	**68.44**	**69.46**	**69.74**
	VP	370	100	50	4.7

3.3.1.2 Effect of process parameters on the hardness and adhesion strength of Ti/TiN coated on Ti-51at. %Ni

In the current work, the Taguchi optimization approach was utilized to determine the optimal parameter combinations for coating adhesion strength and surface hardness. The process factors under investigation were DC power, substrate temperature, bias voltage, and nitrogen flow rate. The impact of Ti/TiN coating on Ti-51at %Ni was investigated under various conditions, with parameter combinations A2D1C1BI (370W, 4.7sccm, 50V, and 100^{o}C) and A2C2D1B2 (370W, 75V, 4.7sccm, 150^{o}C) offering the best adhesion strength and surface hardness, respectively.

The combination of parameters A2D1C1BI (370W, 4.7sccm, 50V, and 100^{o}C) illustrated in Figure 4(a) resulted in the best Ti/TiN coating adhesion on Ti-51at %Ni. The results revealed that applying DC power in the 300-370 W range increases coating adherence; however, increasing DC power to 440W reduced adhesion strength. This phenomenon might be explained by the increased energy available to the expanding film. As a result, high-energy atoms have higher mobility to seek low-energy surface sites, hence improving adhesion properties. Increasing DC power at steady pressure increased ion density, which increased the sputtering rate. If power is raised further, the sputtering rate will decrease owing to back diffusion. (Zalnezhad *et al*., 2013b). Back diffusion occurred at higher DC power (440 W) in this study because the ionized and sputtered particles became more energetic and collided with each other.

The optimal coating adhesion strength was likewise attained at a critical load of 2989 mN, and temperature was shown to influence the substrate's adhesion strength. The ideal temperature for excellent surface adhesion was discovered to be 100 degrees Celsius (B1).; this shows the likelihood of an increase in atomic energy of the substrate surface at 100°C where a void is formed due to movement of the atom at the optimal temperature, the target ions infiltrate the substrate's surface void, Protecting the substrate's surface. The N2 atoms helped create a denser substrate surface. As a result, the released Ar ions created a strong adhesion between the substrate surface and the coating layer. (Huynh *et al*., 2019). Meanwhile, the adhesion strength decreased when the temperature was increased to 150 ^{O}C and 200 ^{O}C, as shown in Figure 4(a). This happened as the increase in temperature caused the rapid movement of atoms that eventually resulted in the collision of the atoms with one and as a result, the ions produced by the target reduced the possibility of adequate penetration through the substrate's surface. As a result, at higher temperatures, the connection between the substrate surface atoms and the released ions was less than at 100°C.

The bias voltage is a significant process parameter that affects the adhesion between the substrate and the thin film coating; it is required in the TiN coating process to achieve the appropriate adhesion strength and surface hardness. A2D1C1B1 is the optimal parameter combination for TiN coating adhesion on Ti-51at. %Ni, as illustrated in Figure 4(a). It demonstrates that applying a bias voltage of 50V enhances coating adherence strength; this means that the optimal parameter for adhesion strength applied to the surface was obtained at D1. However, coating adherence reduces when bias voltage increases from 50V to 75V and 100V due to flaws in the growing film generated by high-energy bombardment. (Gangopadhyay *et al*., 2010).

Nitrogen flow rate is another important parameter that affects the adhesion of TiN coating. The sputtering chamber pressure is linked to the nitrogen flow rate and sputtering power (Kelly and Arnell, 2000). A greater nitrogen flow rate raises chamber pressure, which increases the sputtering rate as ionized atoms grow in the plasma (Reichelt and Jiang, 1990). On the contrary, the high concentration of gas atoms enhances the possibility of collision between nitrogen atoms and sputtered particles on their route to the substrate (Frey and Kienel, 1987b). In that situation, the collision hinders the free passage of nitrogen gas, resulting in a poor deposition rate. However, the low nitrogen flow rate produced a rapid deposition rate due to the drop in chamber pressure, which allowed for the free mobility of atoms and sputtered particles to be deposited on the substrate. Figure 4(a) shows that the nitrogen flow rate at 4.7sccm contributes more to the coatings adhesion strength while further increase to 5.7sccm and 6.7sccm will not enhance adhesion strength.

Figure 4(b) displays response graphs indicating the surface hardness in terms of S/N ratio versus parameters. The DC power plays a critical role in surface hardness as it is ranked number one in the S/N

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ratio response shown in Table 4(b). The best value for the optimum surface hardness of Ti/TiN coated Ti-51at%Ni shown in the graph is 370W.

As the power rose from 300 W to 370 W, the electrically charged ionized and sputtered particles received more energy, increasing the sputtering rate and resulting in a denser surface. The best surface hardness was, therefore, achieved at 370 W(A2) as indicated in Figure 4(b). The ion energy was not the only factor in this process, regardless of the thin layer material; the mass of ions, charge, and size also had an impact on the film layer coating process (Vallée *et al.*, 2020). As a result, increasing the power from 370 W to 440 W enhanced the collision rate between chamber particles and sputtered particles. As a result of high-power sputtering, the sputtering rate lowers dramatically, which does not enhance surface hardness but rather reduces it somewhat.

The substrate temperature is another important element in PVDMS that determines the quality and mechanical qualities of Ti/TiN coatings. Increasing the substrate temperature from 100 ºC to 150 ºC enhanced surface hardness. However, increasing the temperature to 200 ºC lowered the hardness of the coated surfaces. This is because the influence of gas pressure is reduced when the substrate temperature rises. The substrate temperature is thought to have had a key role in controlling atom mobility during Ti/TiN development. (Thornton, 1977).

The optimal procedure parameter for improving surface hardness was 150 ºC (B2). Furthermore, there was a correlation between coating hardness and bias voltage. The TiN coating's hardness rose with an increase in bias voltage from 50V to 75V and reduced when bias voltage was raised from 75V to 100V due to the re-sputtering of weakly bound atoms (Thompson, 2000). The parameter A2C2D1B2 is a combination of key characteristics that increase surface hardness. Bias voltage at 75V, denoted as C2 is the second in rank to achieve an improved surface hardness. The other very important process parameter in surface hardness is nitrogen flow rate. An improved surface hardness was achieved at 4.7sccm as shown in Figure 4(b). The hardness decreased as the nitrogen flow rate increased from 4.7sccm to 5.7sccm; this is because the deposition rate at a higher nitrogen flow rate is low due to an increase in chamber pressure, which hinders the free movement of nitrogen gas as a result of nitrogen atom collisions.

3.3.2 Signal-to-Noise ratio (S/N) response analysis for hardness and adhesion strength of TiN coated on Ti 27at. %Nb

The S/N ratio delta value ranking and parameter designation as indicated in Table 6 are explained previously. Therefore, the combination of process parameters with the greatest S/N on each factorial level, such as DC power (A2), N2 flow rate (D1), temperature (B2), and bias voltage (C3), are considered the ideal parameters to get better adhesion strength and are indicated as A2D1B2C3 (370W, 4.7sccm, 150ºC, and 100V). Meanwhile, the combination of process parameters taken as optimal parameters to obtain improved surface hardness as shown in Table 6(b) are N2 flow rate (D3), temperature (B2), bias voltage (C3), and DC power (A1) and are denoted D3B2C3A1(6.7sccm, 150 ºC, 100V, and 300W).

Table 6: Signal-to-noise ratio(S/N) response values for adhesion strength and surface hardness of TiN coated on Ti-27at. %Nb.

Level	(a) Signal-to-noise ratio(S/N) response value for adhesion strength				(b) Surface hardness signal-to-noise ratio (S/N) response value			
	Power (A)	**Temp. (B)**	**Bias (C)**	**N_2 (D)**	**Power (A)**	**Temp. (B)**	**Bias (C)**	**N_2 (D)**
1	48.61	49.50	49.29	**49.70**	**64.2**	62.7	62.37	63.36
2	**50.46**	**49.59**	49.38	49.22	64.02	**65.75**	64.34	62.57
3	49.17	49.16	**49.58**	49.32	63.47	63.24	**64.98**	**65.76**
Delta	1.85	0.42	0.29	0.48	0.73	3.05	2.61	3.19
Rank	1	3	4	2	4	2	3	1
Average	**49.415**				**63.90**			

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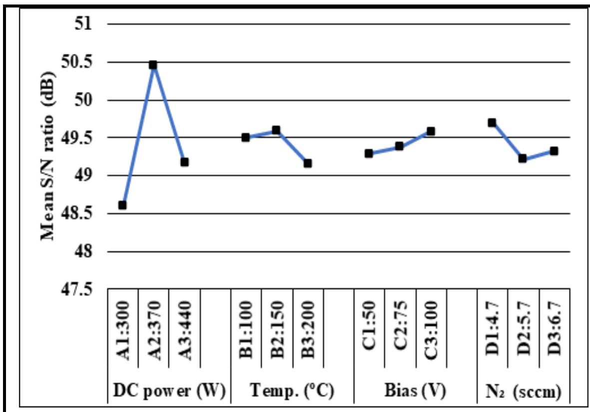

Figure 5(a): Response graphs indicating the adhesion strength in terms of S/N ratio versus parameters

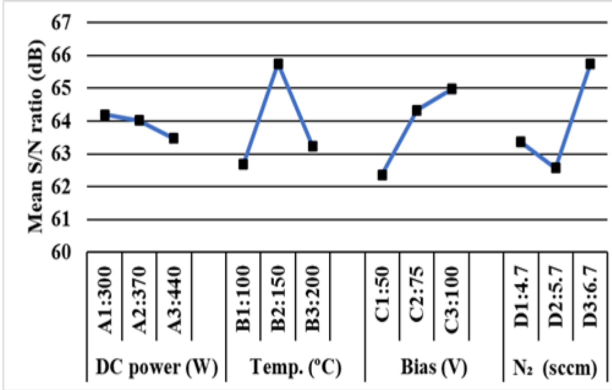

Figure 5(b): Response graphs indicating the surface hardness in terms of S/N ratio versus parameters

The total average S/N ratio value, of overall hardness, as indicated in Table 6(a) is 49.415 dB, and the predicted S/N ratio is 51.085 dB as calculated based on the reduced predicted value of the S/N ratio equation (2). However, the average result of the hardness would be obtained from the confirmation test.

$$\overline{\gamma} = \overline{\gamma_{A2}} + \overline{\gamma_{B2}} + \overline{\gamma_{C3}} + \overline{\gamma_{D1}} - 3\overline{\gamma}_m \qquad 2$$

The total average S/N ratio value $\overline{\gamma}_m$, of overall adhesion strength as indicated in Table 6(b) is 63.90 dB and the predicted S/N ratio is 68.99 dB as calculated based on the reduced predicted value of the S/N ratio equation (4.3);

$$\overline{\gamma} = \overline{\gamma_{A1}} + \overline{\gamma_{B2}} + \overline{\gamma_{C3}} + \overline{\gamma_{D3}} - 3\overline{\gamma}_m \qquad 3$$

Meanwhile, the average result of the adhesion strength would be obtained from the confirmation test. Table 7 shows the combinations of optimum parameters for the confirmation test and their S/N ratio value.

Table 7: Optimum parameters for confirmation test

Parameter		Power(W)	Temp. (°C)	Bias (V)	N_2 (sccm)
		A	B	C	D
		Hardness			
Ti - 27at%Nb	S/N	**50.46**	**49.59**	**49.58**	**49.70**
	VP	370	150	100	4.7
		Adhesion			
	S/N	**64.20**	**65.75**	**64.98**	**65.76**
	VP	300	150	100	6.7

3.3.2.1 Effect of process parameters on the hardness and adhesion strength of Ti/TiN coated on Ti-27at. %Nb

The impact of Ti/TiN coating on Ti-27 at %Nb was studied utilizing a variety of process factors, including DC power, temperature, bias voltage, and nitrogen flow rate. The optimal values determined to produce the best surface hardness and adhesion strength are A2D1B2C3 (370W, 4.7sccm, 150°C, and 100V) and D3B2C3A1 (6.7sccm, 150°C, 100V, and 300W), respectively. Figure 5(a) depicts response graphs of surface hardness as a function of S/N ratio versus parameters. The S/N ratio response in Table 6(a) shows that DC power is very important in surface hardness. The best value in the graph for the optimum surface hardness of Ti/TiN coated Ti-27at. %Nb is 370W.

The electrically charged ionized and sputtered particles gained more energy as the power increased from 300W to 370W, which increased the sputtering rate and thus created a denser surface. The best surface hardness was, therefore, achieved at 370 W(A2), as indicated in Figure 5(a). Ion energy was not the only factor in this process; ion mass, charge, and size affected the film layer coating process, regardless of the thin layer material (Vallée, *et al.*, 2020). Therefore, when the power was raised from 370 W to 440 W, an increase in collision between chamber particles and sputtered particles was observed. As a result of the high-power sputtering, the sputtering rate drops significantly, which does not increase surface hardness but does reduce it slightly.

The substrate temperature is another important PVDMS parameter that affects the mechanical characteristics and quality of Ti/TiN coatings. As the substrate temperature rose from 100 to 150 degrees Celsius, the hardness of the coated surface increased, and as the temperature rose to 200 degrees Celsius, it

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reduced. This is because the impact of gas pressure is reduced as the substrate temperature rises. The substrate temperature is thought to have played an essential role in controlling atom mobility during Ti/TiN growth (Mahieu and Depla, 2009). The best process parameter to achieve improved surface hardness was obtained at 150ºC (B2). Furthermore, there was a relationship between coating hardness and bias voltage. The Ti/TiN coatings hardness increased with an increase in bias voltage from 50V to 75V and further increased when bias voltage was increased from 75V to 100V. When the substrate bias voltage rises, the Ti/TiN films get harder. (Chun, 2010). As shown in Figure 5(b), A2D1B2C3 is the combination of adequate parameters to achieve improved surface hardness. Bias voltage at 100V, denoted as C3 is the fourth in rank to achieve an improved surface hardness. Nitrogen flow rate is another critical process parameter in surface hardness. The hardness decreased as the nitrogen flow rate increased from 4.7sccm to 5.7sccm; this is because the deposition rate is low at a higher nitrogen flowrate due to increased chamber pressure, which hinders the free movement of nitrogen gas as a result of nitrogen atom collisions in the chamber (Shah *et al.*, 2010).

The combination of parameters D3B2C3A1 (6.7sccm, 150ºC, 100V, and 300W) as shown in Figure 5(b), is the condition where the best coating adhesion of Ti/TiN on Ti-27at. %Nb was achieved. The results showed that DC power plays the least role in adhesion strength among the combination of process parameters obtained. The observed improvement in adhesion strength cannot be ascribed to the increase in DC power. When the DC power was increased from 300 to 370 W, a decline in the adhesion strength was noticed and a further increase from 370 to 440 W did not improve the coating adhesion strength either; instead, the adhesion strength decreased noticeably. This could be attributed to the collision of energetic ionized and sputtered particles caused by an increase in DC power at constant pressure, resulting in a low sputtering rate (Tan and Miao, 2009).

At a critical load (L_2) of 2110mN, when temperature was also discovered to have an impact on the substrate's adhesive strength, the ideal coating adhesion strength was also reached. 150 °C was shown to be the ideal temperature for maximum surface adhesion (B2); this shows the possibility of an increase in atomic energy of the substrates surface at 150°C, where void is formed due to atom movement. At the optimal temperature, the target ions permeate the substrate surface void, acting as a barrier on the substrate surface. The atoms helped the substrate surface to become denser. As a result, the substrate surface and the coating layer developed a high degree of adhesion due to the released ions. (Huynh et al., 2019). Meanwhile, as shown in Figure 5(b), the adhesion strength decreased when the temperature was raised to 200 ºC; this happened as the increase in temperature made it possible for atoms to move more rapidly, resulting in the atom's collision with one another. As a result, the target's released ions reduced the possibility of sufficient penetration into the substrate's surface. As a result, compared to 150 °C, the link between the ions released and the substrate surface atoms was weaker at 200 OC.

To obtain the appropriate adhesion strength and surface hardness in the TiN coating process, the bias voltage is one of the crucial process factors that affect the adhesion between the substrate and the thin film coating. As seen in Figure 5(b), D3B2C3A1 represents the optimal combination of parameters for TiN coating adherence on Ti-27at. %Ni, with the best adhesion being attained at 100V (C3). It showed that the application of bias voltage from 50V to 100 V improved the coating adhesion properties; this implies that the best parameter for adhesion strength was achieved at C3. The TiN coating process depends on the bias voltage to provide the required adhesion strength and surface hardness. It is one of the crucial process factors that affect the adhesion between the substrate and the thin film coating. Figure 5(b) illustrates the optimal parameter combinations for TiN coating adherence on Ti-27at. %Ni. The highest adhesion was attained at 100V (C3) in D3B2C3A1. (Lee *et al.*, 2006).

Nitrogen flow rate as earlier reported is another very important parameter that affects the adhesion of TiN coating. The nitrogen flow rate influences the pressure in the sputtering chamber (Kelly and Arnell, 2000). According to Reichelt and Jiang (1990), an increase in nitrogen flow rate generates a rise in chamber pressure, which in turn causes an increase in the sputtering rate as the number of ionized atoms in the plasma grows. Conversely, because there are so many gas atoms present, there is a greater chance that nitrogen atoms may collide with spat particles as they approach the substrate (Frey and Kienel, 1987). In such instances, the collision impedes the free flow of nitrogen gas, which ultimately results in a poor deposition rate. However, because the chamber pressure dropped as a result of the reduced nitrogen flow rate, atoms were able to travel freely and spewed particles were able to deposit on the substrate at a rapid rate. Figure 4(a) shows that the nitrogen flow rate at 4.7sccm showed initial adhesion strength while a further increase to 5.7sccm led to a decrease in adhesion strength. The further increase of nitrogen flow rate to 6.7sccm triggered more bombardment with sputtered Ti atoms that eventually formed TiN. The highest S/N ratio response value of 65.76 dB for adhesion strength as shown in Table 6(b) was achieved at 6.7 sccm (D3). This could be due to high nitrogen activity, which induces more bombardment and consequently increases the number of TiN molecules deposited on the substrate surface, thereby increasing adhesion strength at the same time (Bushroa, *et al.*, 2011b)

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4.0 Conclusion and Recommendation

In this research, the effects of Ti/TiN coating deposited by physical vapor deposition method on the properties of Ti-51at. %Ni and Ti-27at. %Nb was investigated. Further investigation is required to determine the corrosion resistance, biocompatibility, and effect of the alloy production methods (casting or powder metallurgy) on the microstructure and bonding capacity of the alloys to the coating material.

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