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Analysis of Wear Using the Taguchi Method in TiSiNOS-Coated and Uncoated H13 Tool Steel

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Abstract: Titanium–silicon oxynitride sulfite (TiSiNOS) is a coating material that is deposited on H13 tool steel using the scalable pulsed power plasma (S3P) technique, where the coating deposition is a hybrid process consisting of a mix of sputtering and arc evaporation. The maximum hardness and coating thickness measured on TiSINOS-coated H13 tool steel are 38 GPa and 3.1 µm, respectively. After implementing the L_9 orthogonal array, nine samples were coated with TiSiNOS, which consists of the same properties. The nine coated and uncoated samples were tested separately based on the L_9 pattern to achieve accurate results. The experimental results indicate that the wear loss can be reduced by minimizing the load at 25 N even if the temperature rises to 250 °C. SEM analysis reveals that the uncoated sample has higher wear loss when compared with the coated samples, and material pullout is visible from the uncoated sample. Based on these results, it can be concluded that TiSINOS coating in H13 tool steel helps in improving the tool life during the drilling process. Taguchi was used in this research to evaluate the wear behavior. The data observed from the experiment were analyzed using the Minitab tool. The most crucial factor is to determine the effects of process parameters. A higher temperature influenced the wear behavior of the tool.

Keywords: H13 tool steel; TiSINOS coating; pin-on-disc; surface morphology; Taguchi

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1. Introduction

The usage of materials by the manufacturing industry is increasing day by day. Manufacturers constantly view the materials used and seek alternate materials that extend tool life. In connection with this search, various coatings in materials have been observed to enhance the life of tools for improving the production in industries. Researchers have constantly extended their support to find all possible materials that enhance the life of materials. The term coating has recently been widely used in the manufacturing sector. The trend focuses on finding the best coating, where researchers aim to provide a suitable solution. Coatings like titanium nitride (TiN), aluminum–chromium (AlCr), diamond-like carbon (DLC), liquid nitriding and plasma nitriding are commonly used [1,2]. These coatings have also been the focus of developing research to find better coatings for more extendable tool life.

A recent search found that coating materials can improve hardness and wear resistance. It is known from some studies that depositing coatings onto a material can reduce wear even when temperature rises [3,4]. The key factors of depositing coatings are durability, heat

resistance, strength and corrosion resistance [5]. Deformation in grain boundaries plays a vital role in the material, which may enhance bonding properties [6,7]. Generally, the sliding contact method is used to analyze wear properties. Frictional heat and stresses on the material are the key factors of this method [8,9]. In coating research, minimization of wear is a crucial concern. Tribological properties, such as the coefficient of friction, frictional force and wear rate between two surfaces in contact, are measured using a tribometer [10,11]. In another study, it was observed that thin hard coatings could offer longer tool life and better resistance to wear [12,13]. Chromium nitride (CrN) PVD coating has shown encouraging results, especially when paired with nitriding of duplex layers. Studies on hot-working of tool steels revealed a strong link between microstructure and stability at high temperatures, which similarly soften during tempering and high-temperature fatigue [14,15]. It is essential to analyze wear and friction coefficients.

AISI H13 tool steel is a hot-working tool with chromium, molybdenum and vanadium. It is frequently used to make dies, extrusion mandrels, plastic molds, cores, die holder blocks, tools and hot work punches [16,17]. It has also been proposed that increasing the quenching temperature may increase the toughness of steels. In addition, it has also been proved that AISI H13 tool steel can be used for mass production [18,19]. Continuous mechanical and thermal loadings are involved in materials during production. The hot working condition damages a tool's surface and affects properties [20,21]. The chemical composition, microstructure, stress level and surface qualities of materials play an important role in the wear mechanisms of H13 tool steel [22,23]. Stresses caused by cyclic loading lead to tool failure due to abrasion and adhesion. Plastic deformation, thermal fatigue and mechanical fatigue necessitate an analysis of a tool's lifespan. Due to friction between the die and workpiece, wear was recorded as one of the common failure patterns. For high strength, outstanding impact toughness and resistance to thermal cracking, AISI H13 hot work tool steel has been widely utilized as a high-pressure die-casting tool [24,25]. Despite these advantages, failures occur in H13 tool steel under cyclic thermal and mechanical load along with tribological conditions [26,27]. The surface of mechanical components is the most vulnerable since it can experience corrosion, oxidation, abrasion, adhesion, etc. A widely utilized method in this sector is to develop a coating with high-quality material while tailoring the surface qualities to the service requirements [28]. A composite coating has recently been highlighted based on observed mechanical properties. Combining titanium nitride (TiN) and silicon oxide (SiO_2) strengthens the material. In a study, during coating deposition, temperatures were varied to observe hardness. Based on the survey, it was observed that the grain size grows based on temperature. As the temperature increases, the hardness also increases, thereby making the bonding more suitable for the material [29]. The Taguchi 2^9 sequence has been used in this research for evaluating the wear behavior. The data observed from the experiment were analyzed using the Minitab tool. The most crucial factor is to determine the effects of process parameters. An analysis of variance (ANOVA) was used to determine these effects [30,31].

This study aims to improve tool life and find an appropriate tool material with lesser cost, wherein effective coating can be adopted. This will reduce the production cost during the machining process. Based on this study, a new layer has been identified to enhance tool life. Titanium–silicon oxynitride sulfite (TiSiNOS) coating material is coated onto H13 tool steel. The layer is determined based on high-temperature applications. TiSiNOS coating is unique for its smoothness, ability to withstand thermal stress, complex machining, tool precision and wear resistance.

2. Materials and Method

2.1. Tool Material

In the current study, H13 tool steel is used as it has unique features like hot hardness, resistance to wear, etc., when compared with other tool materials like tungsten carbide and high-speed steel. The superior properties of H13 tool steel are due to the availability of chromium and molybdenum in the metal matrix. H13 tool steel can quickly adapt

to both cold and hot working applications. The unique features of H13 tool steel are its higher toughness and hardenability. This material can resist thermal cracking wear and can withstand higher temperatures. Due to the lower hardness, researchers are focused on finding a coating that can be adopted for better tool life for H13 tool steels. EN31 material is used as the disk material in this study with a diameter of 50 mm and thickness of 10 mm. It is composed of nickel, chromium and molybdenum, giving it high tensile steel strength with good ductility and wear resistance. Tables 1 and 2 show the chemical composition of H13 tool steel and EN31 steel, respectively.

Table 1. Individual elemental composition of H13 tool steel.

Alloy	The Concentration of Elements in wt%							
	Cr	**Mo**	**Si**	**V**	**Co**	**C**	**Mn**	**Fe**
H13	5.0	1.4	1.0	1.0	0.50	0.39	0.35	Rest

Table 2. Individual elemental composition of EN31 steel.

Alloy	The Concentration of Elements in wt%						
	C	**Si**	**Mn**	**Cr**	**Co**	**S**	**P**
EN31	5.0	1.4	1.0	1.0	0.50	0.39	0.35

The constituent of chromium in H13 tool steel makes the material more oxidation-resistant, and the molybdenum content ensures the steel is corrosion-resistant. Due to the higher vanadium percentage of 0.95, the wear resistance will improve. Further improvement in the wear resistance of H13 tool steel requires attention to case hardening, either by coating or nitriding. The silicon content helps in removing impurities from the material.

Process parameters play a significant role in the analysis of wear factors. Tool wear depends upon three conditions: load, speed and temperature. The wear resistance changes accordingly upon increasing or decreasing these parameters [32,33]. In this research, the characteristics of H13 tool steel are viewed. Experiments were conducted using the L9 orthogonal array method [34,35]. The process parameters used in this study are shown in Table 3. A few parameters like load, speed and temperature are directly correlated with surface quality; it is mandatory to choose an appropriate parameter for avoiding difficulties [36]. This research selects process parameters based on a survey of research articles [4,13].

Table 3. Process parameters.

Control Factor	Unit	Level 1	Level 2	Level 3
Load	N	25	35	45
Speed (angular)	rpm	600	700	800
Speed (linear)	m/s	1.5708	1.8326	2.0944
Temperature	°C	30	150	250

2.2. Coating Materials and Method

The H13 tool steel sample was coated with titanium–silicon oxynitride sulfite (TiSiNOS) coating using the scalable pulsed power plasma (S3P) technique, where the layer is a hybrid process consisting of a mix of sputtering and arc evaporation. INGENIA was used to coat TiSiNOS onto the H13 tool steel sample. The processing temperature of INGENIA was a maximum of 1000 °C. The coating was carried out under 500 °C.

2.3. The Taguchi Method

Response surface methodology (RSM) was used for optimizing the process parameters. The conditions used were load, spindle speed and temperature. With the help of this method, a clear relationship between the factors and response can be obtained. Taguchi is a well-defined method that is suitable for minimizing the experiment. Based on this, an L_9 orthogonal array was chosen for the investigation. To obtain an accurate result, nine samples were chosen and coated with the same conditions. A wear test was performed separately in this sequence to reduce the number of experiments (Taguchi L_9 orthogonal array 3 factor, 3 levels). Minimum wear tests were conducted based on the factors and levels. The Taguchi method is framed based on the principles of design of experiments (DoE), optimization and the signal-to-noise ratio (S/N). DoE is framed for analyzing the process and method. In addition, to minimize the experiments, the orthogonal array was used. The desired and undesired outputs were analyzed based on the S/N ratio. The optimal level based on the process was analyzed using the S/N ratio.

2.4. Experimental Method

The wear property is an important factor in manufacturing because most materials undergo different machining processes at different parameters. In this research, the weight loss of the coating in grams was investigated with the support of process parameters. In addition, to quantify the frictional force between objects, the coefficient of friction is used. The wear rate, as well as the coefficient of friction, is comparatively low [37].

In the present study, a pin-on-disc tribometer was used to evaluate the wear loss and coefficient of friction. H13 tool steel material was used to prepare the pin and EN31 steel was used to prepare the disc material. Initially, the hardness of the pin and disk material was low, it but improved with the tempering process. The pin and disk material results were 47 HRC and 63 HRC, respectively. After the deposition of TiSiNOS coating into the tool material, experiments were carried out based on the process parameters in a pin-on-disc tribometer. For the analysis, scanning electron microscopy (SEM) was used.

As a result of implementing the L_9 orthogonal array, nine samples were coated with TiSiNOS coating, and an uncoated tool was tested separately to achieve accurate results.

3. Results and Discussion

Based on the standards of the L_9 orthogonal array [38,39], several trials were carried out. The current study was planned based on preparing tools for friction drilling, which works in dry conditions. This research aims to identify a new coating for friction drilling tools, where friction drilling works around 500–600 °C. So, it is essential to identify a coating that can resist high temperatures, and the wear rate should be low [40].

3.1. Effect of Wear with the Influence on Process Parameters

The present study evaluates the wear behavior of TiSiNOS coating for H13 tool steel. For the tribological study, the pin was prepared with a length of 10 mm and a diameter of 6.5 mm. The wear loss observed at different conditions for all nine samples is shown in Figure 1.

The results show the influence of temperature and load, with minimal wear loss when the load is kept constant despite increasing temperature. However, when the load and temperature are increased, samples 5 and 7 reach a peak wear loss of 0.0631 g and 0.0617 g, respectively. From the experiment, it is observed that the wear loss is minimal for lower temperatures. If the temperature rises, the load needs to be less, as observed in samples 4, 6 and 9. The material loss reduces gradually, as observed in sample 8. The wear loss for the uncoated H13 sample is 0.05 at ambient temperature. Based on the experimental output, it is understood that wear loss is not fully dependent on spindle speed. Still, by varying the load and even temperature rises, wear loss can be controlled. The uncoated tool wear loss is very high compared with the other coated tools.

The coefficient of friction (COF) on wear is mechanical and molecular. The presence of uneven surfaces and adhesive components on the wear surface, along with the interaction between atomic molecular and intermolecular surfaces, results in the formation of a higher contact area, which leads to deformation [41,42]. Figure 2 shows a COF comparison among the nine samples. By looking at the trend, it is noticeable that the rise in COF depends on the load and temperature. With an increase in the load and temperature, material loss also increases simultaneously. Thus, the load must be maintained to improve the tool life even if the temperature increases.

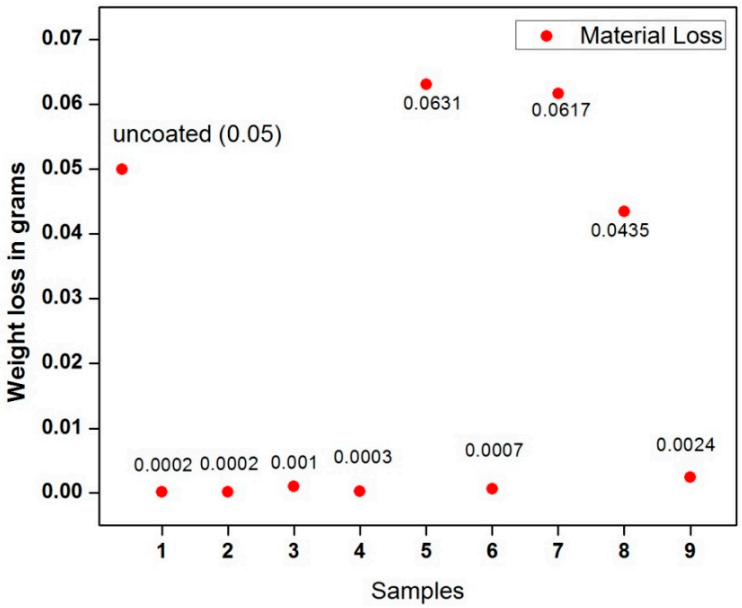


Figure 1. Graph comparing the nine samples and wear loss.

3.2. Experimental Results and Taguchi

The regression equation obtained is given below. The regression equation is used in this study to minimize the predicted errors with the help of the samples.

$$Y = 0.0085 + 0.00177 \text{ Load} - 0.000097 \text{ Speed} + 0.000115 \text{ Temperature} \quad (1)$$

Equation (1) shows the wear rate observed for the coated material, where Y is the wear rate in the TiSiNOS-coated H13 tool; the regression equation is based on a smaller-to-larger ratio. The signal-to-noise (S/N) ratio observed based on the processing parameters is shown in the table. A response table of means is shown in Table 4, where the temperature has more weightage. Table 3 shows the process parameters used, with three input parameters at a constant time of 10 min for the experiment. Wear tests were conducted based on the process parameters. The hardening and tempering process was carried out to make the tool and the disk harden.

Initially, the tempering process was carried out to improve the hardness of the materials. Preheating the samples was carried out in three different stages: the first preheat at 650 °C, the second at 850 °C and austenizing at 1030 °C, followed by quenching with high-pressure N_2 gas. After the completion of the hardening cycle, a tempering cycle occurred, followed by three temperature cycles at 550 °C, 590 °C and 550 °C. For the initial H13 tool steel, the hardness is 35 HRC. After undergoing the hardening mentioned above and the tempering cycle, the hardness of the sample was increased to 46-47 HRC.

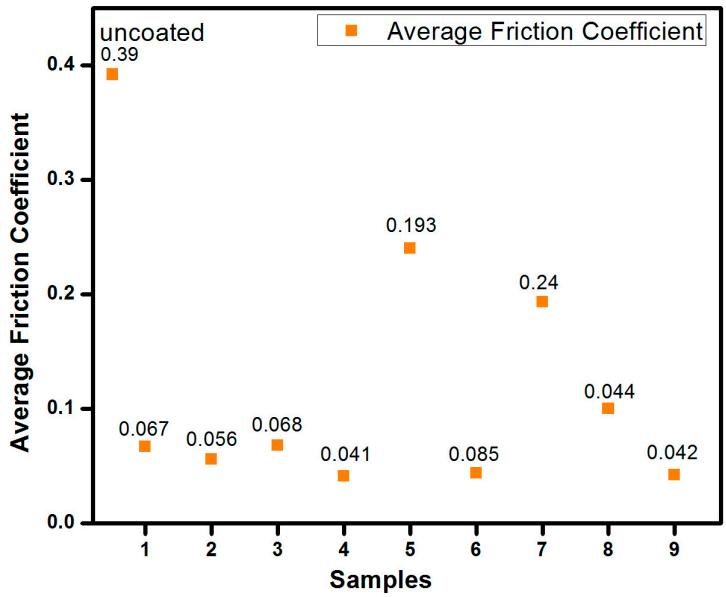


Figure 2. Coefficient of friction.

Table 4. Response table of means.

Level	Load	Speed	Temperature
1	0.000467	0.020733	0.014800
2	0.021367	0.035600	0.000967
3	0.035867	0.001367	0.041933
Delta	0.035400	0.034233	0.040967
Rank	2	3	1

The reason for choosing TiSiNOS coating is due to its unique vital features like obtaining a smooth surface, the precision of the tool and improving its hardness [43]. TiSiNOS coating was chosen to coat H13 tool steel material using the scalable pulsed power plasma (S3P) technique with a coating based on AlTiSiN. The processing temperature of INGENIA was a maximum of 1000 °C and coated at <500 °C. As a result, the maximum hardness measured is 38 GPa and the coating thickness measured is 3.1 µm for TiSINOS-coated H13 tool steel. Before directly manufacturing tools, samples are analyzed to identify their properties. Pin-on-disk is a tribological method used to evaluate the wear tracks for the prepared samples [44,45]. In the experiment, factors like load, speed and temperature are varied to identify the less worn samples that can be recommended for higher production.

A response table of the mean values observed based on the processing parameters is shown in Table 4. The response for smaller means is better, and those values are reported, where the temperature is the decision factor in reducing the wear rate of the material.

The signal-to-noise (S/N) ratios observed based on the processing parameters are shown in Table 5. The response for smaller signal-to-noise ratios (S/N) is better, and those values are shown, where the temperature is the decision factor in reducing the wear rate of the material.

Table 5. Response table for signal-to-noise ratios: smaller is better.

Level	Load	Speed	Temperature
1	69.32	56.21	54.77
2	52.52	41.74	65.61
3	34.61	58.50	36.06
Delta	34.71	16.76	29.55
Rank	2	3	1

Based on the experiment conducted and the values observed, the effect of the S/N ratio and mean is shown in Figure 3. From the figure, it can be understood that as the temperature increases, the wear rate also increases. Moreover, the load and speed of the spindle prove that it can control the wear even if the temperature increases. The least material loss observed is 0.0002 g when the process parameters load is 25N, the spindle speed is 600 rpm and the temperature is 30 °C.

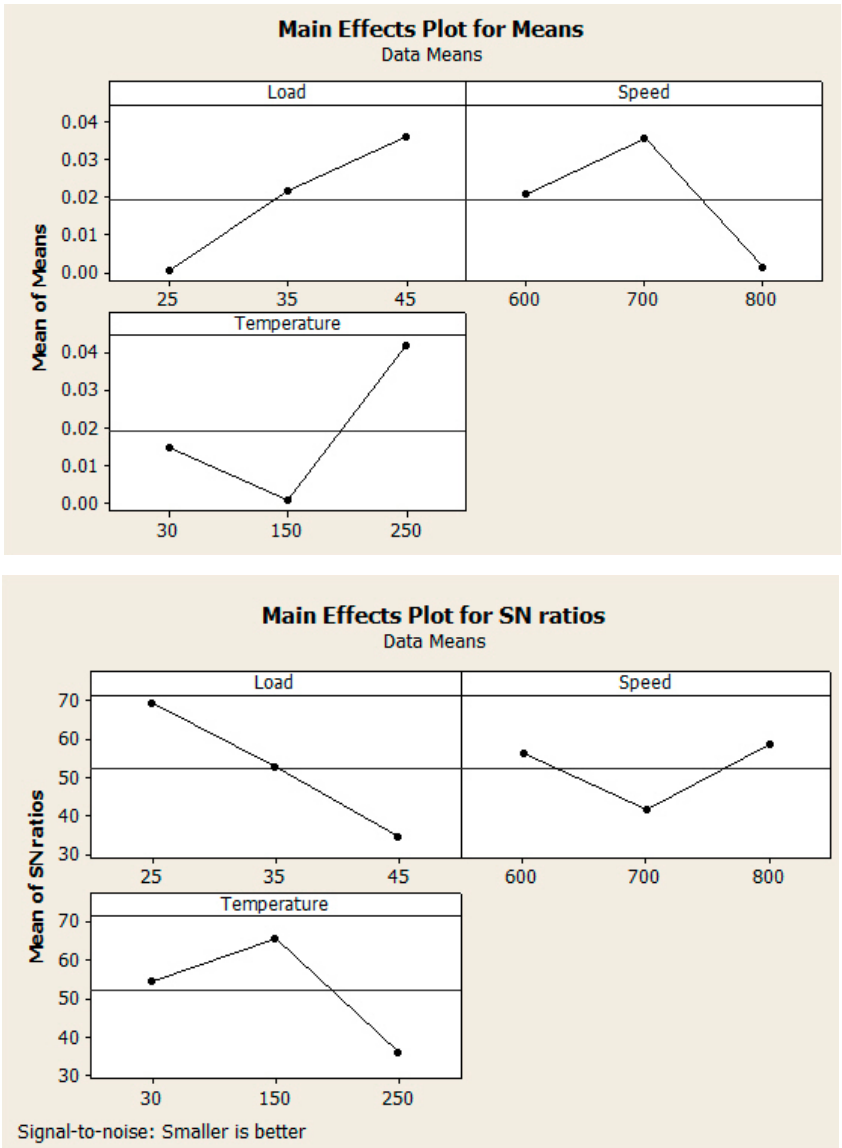


Figure 3. Effect of process parameters on wear (signal-to-noise: smaller is better).

The ANOVA results are based on a confidence level of 95% and a significance of 5%. The results obtained using ANOVA are shown in Table 6. The table also indicates that the residual (R^2) is 91.56%, which is close to 1. The result is more accurate if the residual (R^2) value is close to 1.

Table 6. Analysis of variance (ANOVA).

Variance Source	Degree of Freedom (df)	Sum of Squares (SS)	Mean Square (MS)	F	P
Regression	3	0.0034130	0.0011377	1.93	0.243
Residual Error	5	0.0029459	0.0005892		
Total	8	0.0063589			
S = 0.024 R^2 = 89.62 R^2 (adj) = 91.56					

A detailed discussion of the ANOVA results is shown in Figure 4. The actual versus the predicted value is shown. The diagonal line shows the mean line. In theory, the predicted values are kept from the input that it is given. The experimental results were compared with the expected. It is understood that the predicted and actual values are most probably equal by looking at the graph plotted in Figure 4.

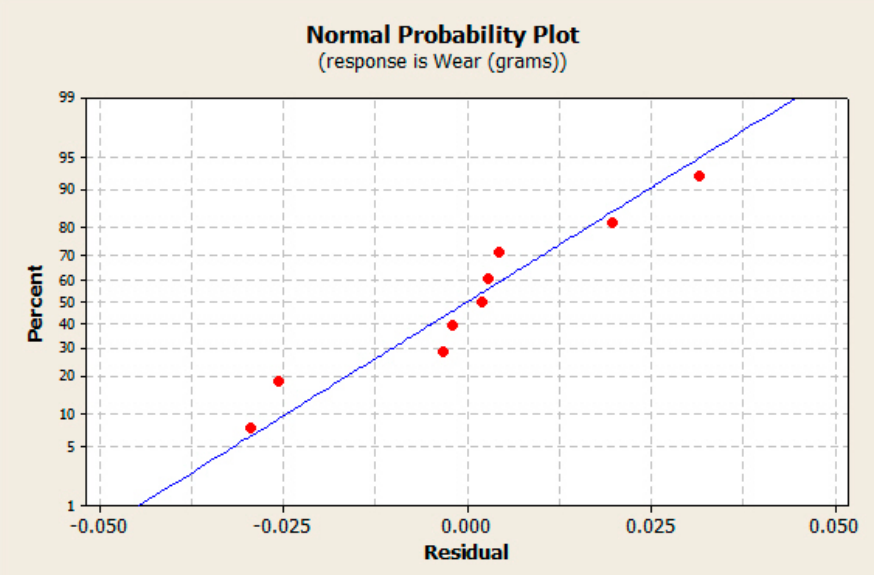
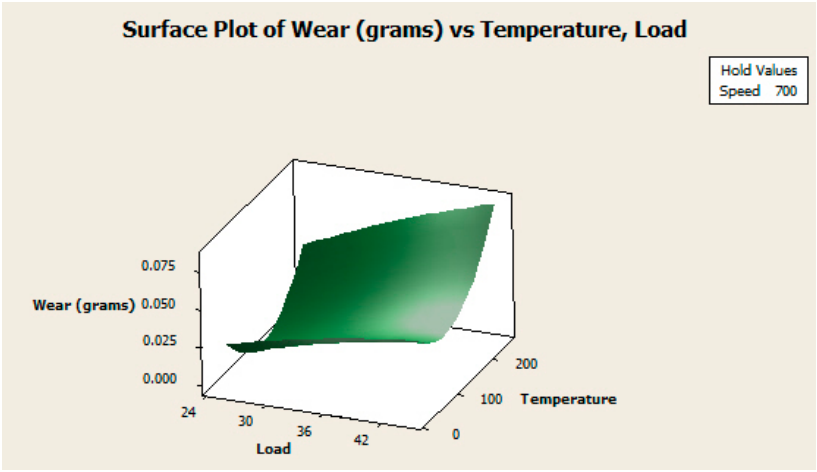
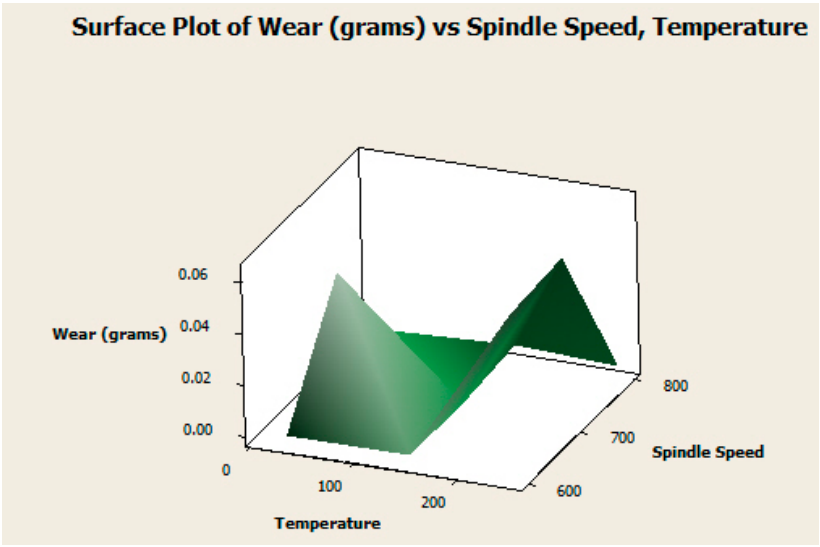


Figure 4. Predicted vs. actual values.

The surface plots of three different observations are given in Figure 5. These show the comparison between material loss and process parameters. The surface plot observed is shown in Figure 5a–c. Figure 5a shows a surface plot of wear loss vs. temperature and load. Figure 5b shows a surface plot of wear loss vs. temperature and spindle speed. Figure 5c shows the surface plot of wear loss vs. spindle speed and load. The pattern shows the wear loss in the coated tool and how the parameters influence it. To be clear, the given load is a crucial concern even as the temperature varies.



(**a**)



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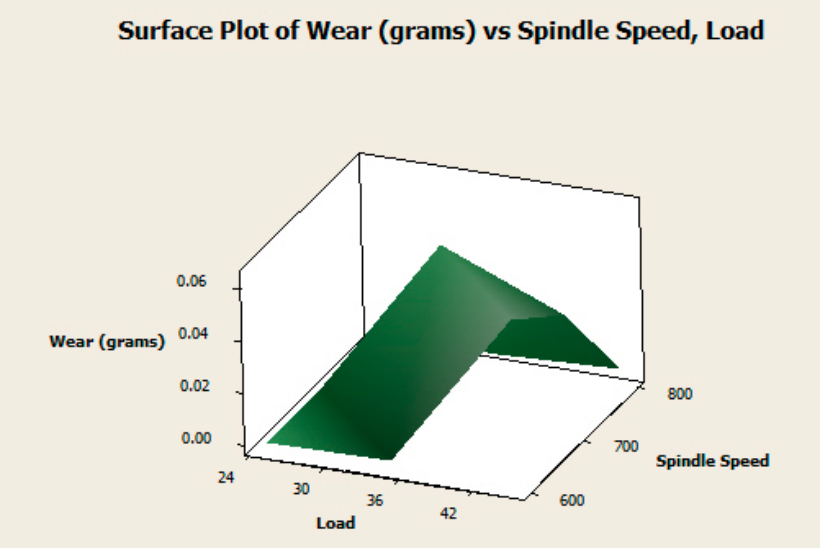


(**c**)

Figure 5. Surface plot of wear in coated tool. (**a**) Wear loss, load vs. temperature, (**b**) wear loss, temperature vs. spindle speed and (**c**) wear loss, load vs. spindle speed.

3.3. Surface Morphology

The surface morphology was analyzed using a scanning electron microscope (SEM) for all the TiSiNOS-coated and uncoated samples after the wear study, as shown in Figure 6. The microstructure of the materials after wear analysis was analyzed using a scanning electron microscope (SEM).

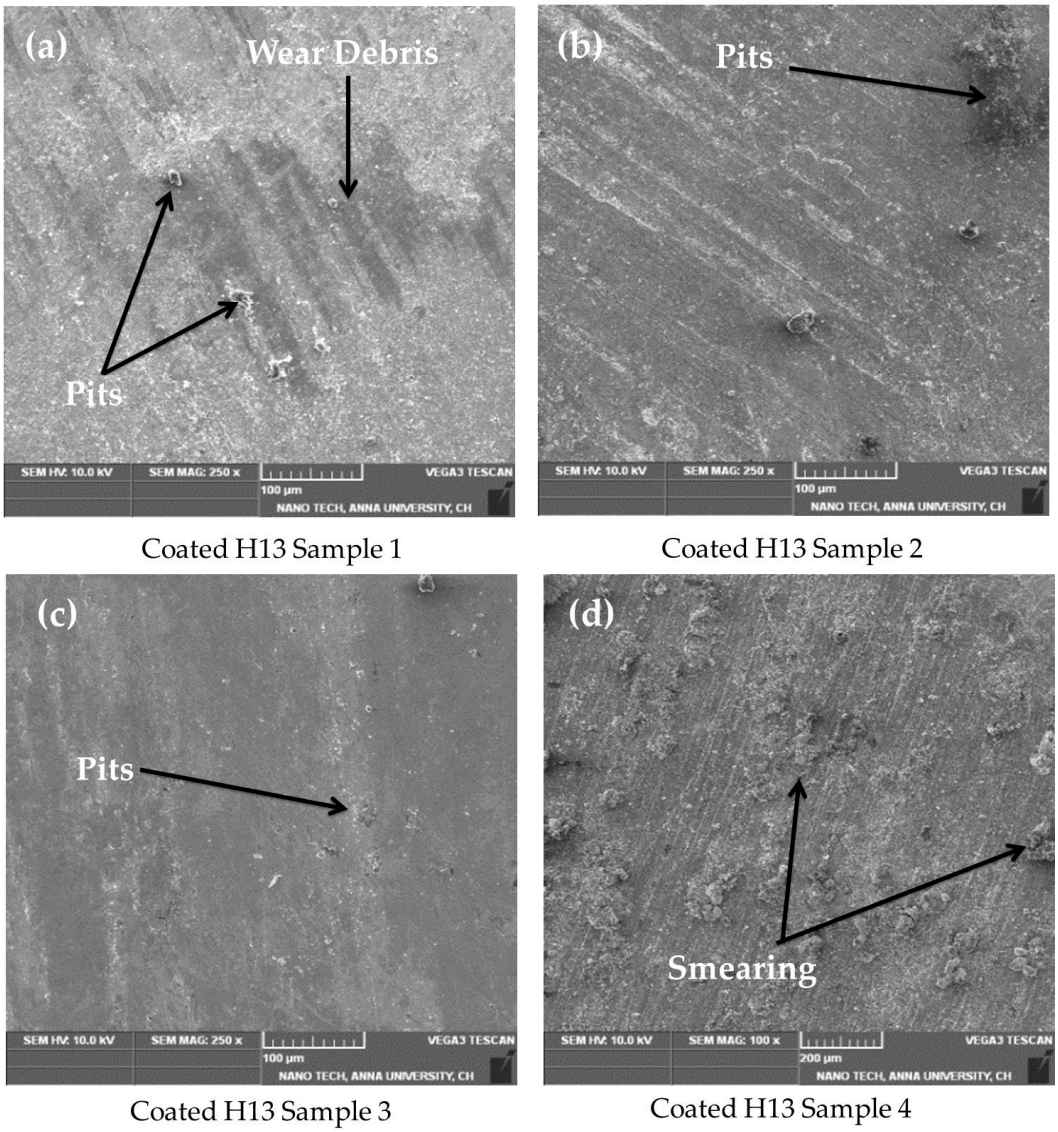
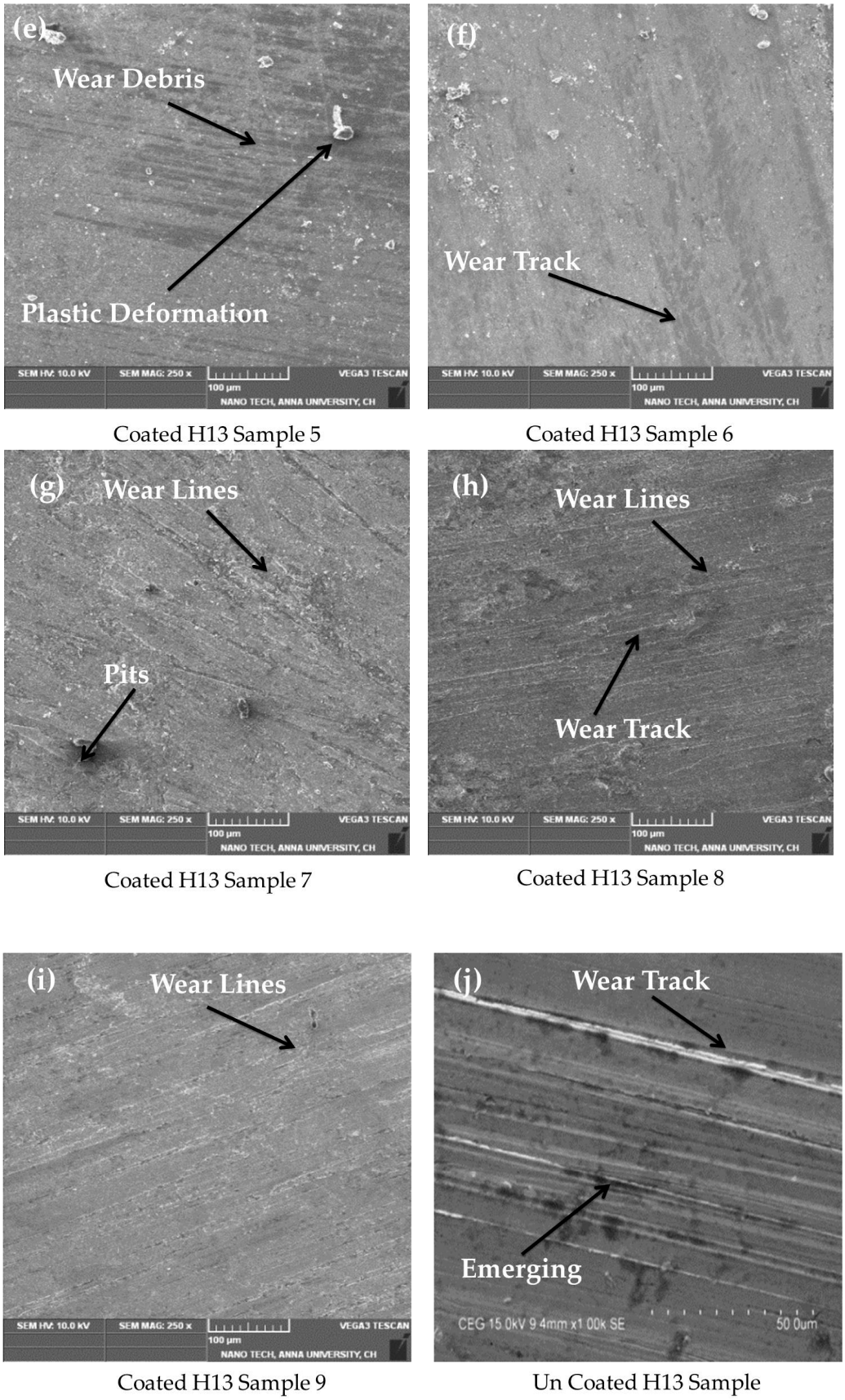


Figure 6. *Cont.*



Coated H13 Sample 5 Coated H13 Sample 6

Coated H13 Sample 7 Coated H13 Sample 8

Coated H13 Sample 9 Un Coated H13 Sample

Figure 6. Surface morphology: 6 (**a**–**i**) coated samples (1–9) and one (**j**) uncoated sample.

Due to the direct contact of the pin and disc without lubrication, the possibility of abrasive wear is high, which results in the emergence of pits throughout the sample. The evidence of abrasive wear is when the material in the pin starts to transfer to the disc, as the disc material is more complex than the pin material. Figure 6j shows the uncoated H13 sample, where material loss is more significant than in the other samples. It is concluded that coated H13 tool steel will have better tool life improvement. In Figure 6a, it can be observed that there are significantly fewer cracks and deformations in the material. Wear debris was observed in the sample because of a lower load and spindle speed. In Figure 6b, pits formed in the coating due to the rise in temperature as the load remains low, and a few small cracks are also visible at a few places in the sample. It is expected that the coating is still visible in Figure 6c for sample 3, even in high temperatures, because of a lower load and higher rpm. The material becomes soft upon heating, but the load given to the materials helps to protect the layer. Considerable smearing was only observed in Figure 6d even though the temperature rose to 150 °C because of the lower rpm. However, when the load and temperature increase simultaneously by 35 N and 250 °C, plastic deformation is visible in Figure 6e. Due to the temperature difference, material loss is observed in the wear samples, as shown in Figure 6e,g. Figure 6f shows that the coating can be protected even at higher speeds by keeping the temperature low. The material deformation and loss of material are noted in a few places in the sample. Nevertheless, from the observation of Figure 6h, cracks were visible on the surface. Even though the temperature is low, the rpm and load are higher. Figure 6i shows wear lines along the material's surface, although the material loss is much less than in the other samples. The wear loss from all the surface morphological images is minimal, so the TiSiNOS coating still exits and is visible in the surface study. This coating has higher bonding and can be used for industrial applications.

4. Conclusions

This study, based on a pin-on-disc test, was used to evaluate the wear behavior of TiSiNOS-coated H13 samples. The samples were tested at different load, speed and temperature conditions. The coated samples were compared with an uncoated sample to validate wear evaluation. The Taguchi methodology was also used to observe the samples. Based on this study, the following conclusions were noted.

- SEM analysis reveals the presence of a TiSiNOS coating of 3.1 µm on the sample. The TiSiNOS coating is best for this study because the layer still exists for the process parameters; it can be best utilized for machining applications.
- When compared with an uncoated sample, in the coated sample, wear loss was very minimal; this is because of the coating that was used.
- With the increase in temperature, the wear behavior of the material changes even when there is a change in speed and load. However, by maintaining a lower speed, the material losses can be controlled.
- The loss of coating thickness can be controlled by controlling the process parameters.
- Wear losses can be reduced by increasing the load applied and maintaining wear studies at a lower temperature during the course of work.
- For better wear results, even if the temperature is raised to 250 °C, the load must be kept at a minimum of 25 N.
- The observed residual R^2 value is 91.56%, which is close to one. Thus, the samples are significant.
- The predicted and actual values are close, so the samples suggested are significant.
- The critical factor is that the wear loss is much less than the materials. TiSiNOS coating can be suggested for higher production applications.

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