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Pulse Reverse Current Electrodeposited TiO2 Doped Ni-W Coating

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Executive Summary

Problem Statement

Rolling bearings are found in components all over the world. It has been growing substantially more important for these pieces of equipment to endure more wear and corrosion. Electrodepositing coatings is an efficient method for protecting rolling contact components. Combining the Pulse Reverse Current (PRC) method with a Ni-W coating has the potential to be a replacement for other more harmful coatings. Previous work by Timken Engineered Surfaces Laboratory (TESL) showed that Ni-W coatings by the PRC method resulted in uniform and durable coatings that have potential to be improved by adding in dopants such as TiO_2. Testing the efficacy of doping TiO_2 into the Ni-W matrix was done with a series of tribological tests to collect data on wear rate, friction coefficients, and uniformness of coating. The previous Ni-W solution of pH 8 was adjusted with five varying concentrations (3, 5, 10, 20, and 30g/L) of the TiO_2 dopant. The pH of the solution was considered to see how it affected the coating performance by creating another 30g/L TiO_2 solution at a pH of 5. Electrodeposition took place with the solution at 50°C and the original parameters. The optimum concentration and pH were determined based off where TiO_2 does not aggregate/agglomerate, provides enough hardness to the coating, lowers the friction coefficient, and lowers wear volume the most.

Results and Conclusion

Typically, TiO_2 particles can aggregate in solution due to their surface charge, which can be altered from solution pH. Therefore, the pH of the solution was tested for its effect of tribological characteristics. Typically, during the electrodeposition the TiO_2 in the pH 8 solution has a negative charge, while the substrate has a positive charge during the reverse current.

Opposingly, the pH 5 solution (acidic) results in a positive TiO_2 charge on the surface and the substrate has a negative charge during forward current.

Multiple methods of testing concluded that Ni-W doped with TiO_2 shows improved coating characteristics when compared to undoped Ni-W. The pH 5 30 g/L sample exhibited the best wear resistance when compared to all other concentrations and formulations. The 30 g/L samples provided the most uniform wear scars, unlike the smashed and brittle wear scars observed at lower TiO_2 concentrations. The sample with the highest titanium content was the pH 5 30 g/L sample, which also has the highest tungsten and lowest nickel content, however, the samples with the lowest roughness had the highest nickel content. These results indicate that the ratio of the three metals is critical in determining the highest performing composition where TiO_2 is optimally maximized.

Recommendations

It is clearly determined that TiO_2 increases coating performance, however, the process is not fully optimized. Recommendations concluded from this report include performing a study of repeatability to confirm that a pH of 5 and TiO_2 concentration of 30 g/L are optimum solution characteristics. Further optimizations can be made by adjusting the ratio of W and Ni to observe and record its effects on tribological testing. Finally, comparative testing that was not considered for this project, such as rolling contact, should be completed for 30 g/L at pH 5, 30 g/L at pH 8, and Ni-W-SDS coatings.

Broader Implications of Research

From the perspective of an undergraduate student, this project has been an excellent opportunity to expand knowledge into electrochemistry and build confidence in designing and running experiments. Research such as this has the potential to improve leadership, technical skills,

data analysis, and self-motivation in the student while building off previously learned skills and coursework. This research has the potential to have an impact in the rolling bearing industry, and other industries where wear reducing coatings are needed and utilized. The coatings presented in the report have the ability to make these components endure much longer lifespans than previous coatings allowed, which will lead to cost savings and a higher level of safety in some situations.

Introduction

Rolling bearing components have been a mainstay in the mechanical creations of the modern world. As the world of metals has evolved, and bearing technologies began to be more precise, it has never been more important to produce components that will wear less, be resistant to corrosion, and perform well in harsh environments. To achieve this, coatings have been developed to protect the counter parts of tribological systems. Coatings can be applied in a variety of ways, but a common method, electrodeposition, has a lot of potential to revolutionize the world of coatings. Electrodeposition is a method of applying coatings to metal surfaces to improve the objects wear and corrosion resistance. There are various methods of electrodeposition, but this report is focused on the Pulsed Reverse Current (PRC) method to apply Ni-W coatings doped with TiO_2 on 52100 steel. Combining the PRC method with a Ni-W coating has the potential to be a widely used replacement for other Cadmium/Chromium coatings that are restricted under certain laws and industries [1]. The PRC method is beneficial over other methods due to its ability to produce a more uniform distribution of particles on the surface. Previous work completed by Timken Engineered Surfaces Laboratory (TESL) showed that Ni-W coatings by PRC provided promising results and be further improved with the incorporation of dopants such as TiO_2 [2]. This project of continued and successful development of Ni-W materials by the cost-effective PRC electrodeposition technology should provide considerable benefits by improving the wear resistance of rolling bearing applications with fully formulated lubricants. It was hypothesized that incorporating higher concentrations of TiO_2 nanoparticles into the Ni-W matrix will exhibit friction coefficients and wear rates significantly lower than that of the undoped coating. Therefore, the primary focus of the research was to electrodeposit Ni-W coatings doped with various concentrations of TiO_2 and perform tribological testing.

Background

Ni-W coatings have been gaining traction as a possible replacement for existing coatings. The importance of such coatings lay in the fact that they contain no Chromium or Cadmium as well as have less restriction in certain use cases and are more preferred due to environmental concern [1]. Donten states that W alloys improve durability and hardness enough so that they can replace more conventional Cr coatings [3]. Donten continues in stating that out of the amorphous alloys that can be deposited, Ni-W presents the best smoothness and appearance when compared to Fe-W and Co-W. However, the high internal stress of Ni-W can lead to microcracks in the coating surface [3]. Microcracks pose a significant problem as they can cause the coating to have poor corrosion prevention and fail.

The Pulsed Reverse Current (PRC) method, a modification to the Direct Current and Reverse Current methods, can be utilized to minimize the microcracking phenomenon. The PRC method can produce coatings with better coating characteristics when compared to Direct Current plating such as lower porosity and finer grain size [4]. Therefore, PRC deposition can be used to produce a coating with fewer pores and microcracks [4]. The method of PRC allows for the formation of a more uniform, and therefore stronger, coating. To achieve a proper coating, a PRC utilizes both anodic and cathodic currents to repeatedly deposit a layer of coating and then removing a portion of the coating, as shown below in Figure 1 [5]. This is performed by the anodic current density (I_{fwd}) applying a layer of coating for the anodic time period (t_{fwd}) and then the cathodic current density (I_{rev}) removing some of that layer for the cathodic time period (t_{rev}), and this process is repeated for the length of the cycle time (t_{sum}). The average current density is can be calculated by E.1 [5].

$$I_{avg} = \frac{I_{fwd}t_{fwd} - I_{rev}t_{rev}}{t_{fwd} + t_{rev}} \quad \text{(E.1)}$$

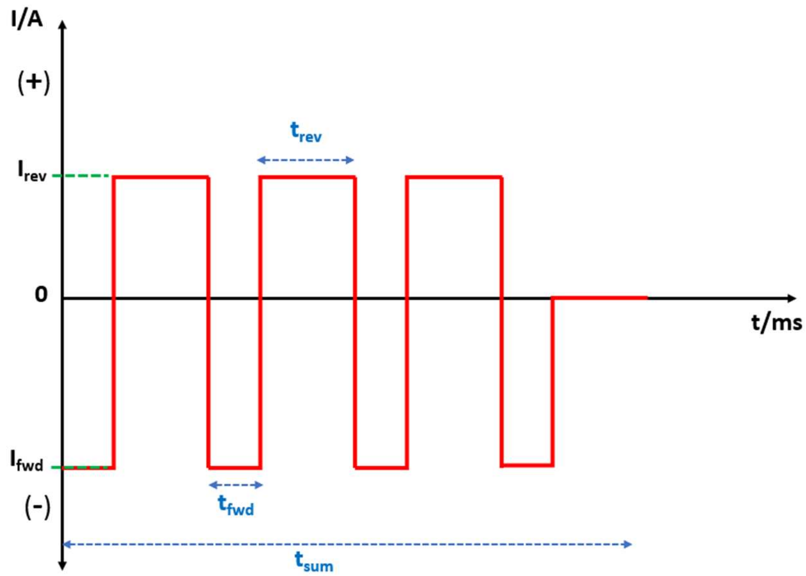

Figure 1: A mode of electrodeposition shown is Pulsed Reverse Current (PRC) deposition. Parameters shown are the anodic time (t_{fwd}), cathodic time (t_{rev}), anodic current density (I_{fwd}), cathodic current density (I_{rev}), average current density (I_{avg}), and the cycle time (t_{sum}).

In a series of previously Timken sponsored projects, Ni-W coating for improved wear and corrosion resistance was developed using a PRC electrodeposition method. One of the first projects in this line of testing was completed by Shreeram et al. where tribological properties of two identified Ni-W coatings, T7 & T16, were compared. The coating known as T7 was found to be a superior coating as it had a lower surface roughness and higher W content [4]. It was also concluded that better tribological performance was correlated to a smaller grain size and a higher hardness, both properties exhibited by T7 [4]. One weakness in the T7 coating was that it exhibited pores in the surface coating, despite the PRC method, which can lead to decreased corrosion resistance [4].

Then, it was hypothesized that the addition of dopants, such as TiO_2, could help increase the corrosion protection and reduce the presence of pores and microcracks [2]. This hypothesis inspired the next project which tested the efficacy of doping a Ni-W coating with TiO_2 nanoparticles to produce a coating with high tribological performance and corrosion resistance [2].

It was determined that TiO2-doped Ni-W exhibited significantly less wear than undoped Ni-W [2]. The project discussed in this report takes the previous work mentioned and makes improvements by discovering how the doping of the Ni-W coating at various TiO_2 concentrations impacted the performance of the coating.

The TiO_2 dopant used has a particle size of less than 100 nm [6]. It is important to know the concentration in solution since the distribution of the particles in the coating affects the corrosion and mechanical properties of the coating due to the formation of aggregates/agglomerates. Amount of aggregation/agglomeration of the solution is very important and is tested with a Zeta Potential Analyzer. Aggregates/agglomerates may cause a rough surface and uneven distribution in the coating which causes a higher coefficient of friction and a less protective surface. Formation of aggregates/agglomerates is dependent on the zeta potential and is related to particle size. The dispersion of particles within solution is dependent on surface charge, which changes with the pH and therefore affects the aggregation/agglomeration of particles on the sample surface [7].

Experimental Methods

Solution Development/Analytics

An experimental matrix was developed to perform a design of experiment to optimize the wear and corrosion resistance of a TiO_2-doped Ni-W coating. Ni-W solutions were produced with TiO_2 concentrations 3, 5, 10, 20 and 30 g/L at pH of 8 and 30g/L at pH of 5 following the recipe found in the Appendix Table A.1. Small amounts of each sample tested with the Mobius zeta potential analyzer for electrophoretic and zeta potential measurements to determine surface charge, aggregation/agglomeration, and particle size.

Electrodeposition

Deposition solution that contained the nickel, tungsten and TiO_2 nanoparticles (see Appendix Table A.1 for base solution formulation) was stirred and heated to 50°C prior to starting experimentation. The disk samples were prepared prior to the depositions by sonicating in a degreaser solution for 5 minutes, rinsing with deionized (DI) water, etching in 30% HCl for 20 seconds, and rinsing with DI water once more to thoroughly clean and prepare the surface for coating. The disk was then attached to a magnetic rotating rod set to 30 rpm. Nickel acted as an anode and a 52100 disk was used as a cathode. The experimental setup is shown in Figure 2.

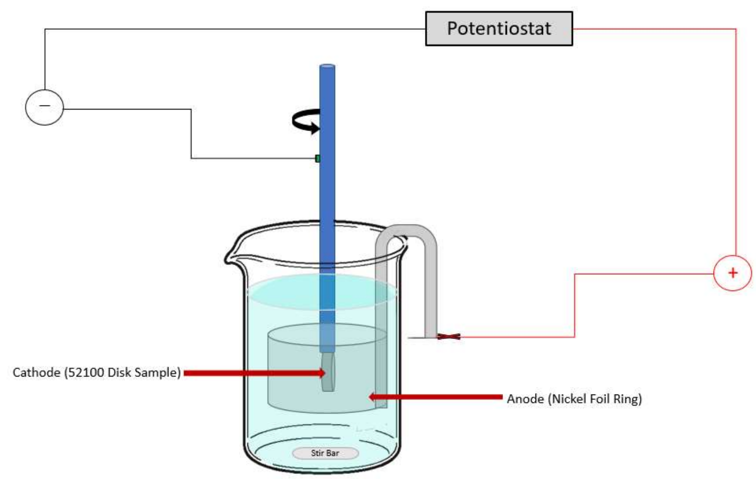

Figure 2: Model of set-up for electrodeposition. 52100 steel disk sample attached to rotating magnet and placed in middle of cylindrical nickel anode. There is a magnetic stir bar rotating at the bottom.

The forward current density was set to 40 mA/cm^2 and the reverse current density was set to 12 mA/cm^2. With a disk surface area of ~0.35cm^2, the I_{fwd} was -0.113A with t_{fwd} of 0.16s and the I_{rev} was 0.034A with t_{rev} of 0.16s. The deposition time was 30 minutes. After the deposition was completed, the sample was removed and rinsed with DI water. The samples were examined with the X-ray fluorescence (XRF) to determine the composition of the coating.

Tribological Experiments

Deposited samples were passed on for tribological experiments with the High Frequency Reciprocating Rig (HFRR) performed with ISO 68 mineral oil. The parameters of the test were a 10N load, 1200μm stroke length, 20 Hz frequency, 60-minute period, and this was repeated at three temperatures (40°C, 75°C, and 100°C). For each test, 1mL of lubricant was used. After tribological testing, the samples were examined with the ZYGO Optical Interferometer Microscope for profilometry and wear analysis. X-ray diffraction (XRD), energy dispersive spectroscopy (EDS), scanning electron microscopy (SEM), and optical microscopy were used to analyze the samples with respect to their composition and elemental makeup.

Results and Discussion

Particle size of TiO_2 was tested with the Mobius for relation to aggregation/agglomeration. The Mobius data of TiO_2 particles size showed to have three polydisperse peaks. It was determined that TiO_2 has a multimodal size distribution, which can be seen when comparing the three peaks in Figure 3 to the multimodal size distribution in the Appendix Figure A.1. The multimodal distribution indicates that TiO2 particles will aggregate.

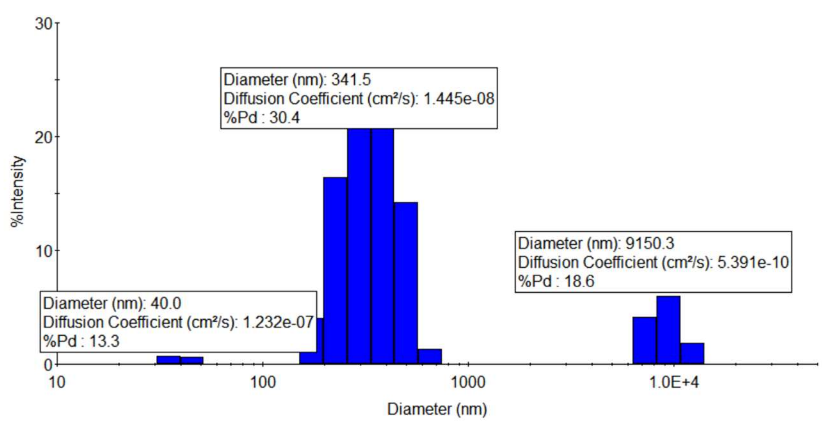

Figure 3: Results from Wyatt Technology Mobius displaying TiO_2 particle size peaks. Three broad peaks indicate a multimodal size distribution for pH 8 solution.

Table 1: TiO_2 Mobius Peak information from Figure 3.

Peak	Diameter (nm)	Mw-R (kDa)	%Pd
Peak 1	40	3734	13.3
Peak 2	342	562741	30.4
Peak 3	9150	1235355975	18.6

The Zeta potential of TiO_2 Ni-W solutions were tested on the Mobius at concentrations of 0.2, 3, 5, 10 and 20 g/L and pH 8. The slope is related to particle mobility and the direction of the V-graph indicates the sign of the particle charge. As seen in Figure 4, the direction of the V-graphs for all concentrations at pH 8 shows that each resulted in a negative charge with no discernable trend with concentration. The surface charge is dependent on pH, therefore with a pH of 8 (basic) as tested here, this results in a negative surface charge from deprotonation [7].

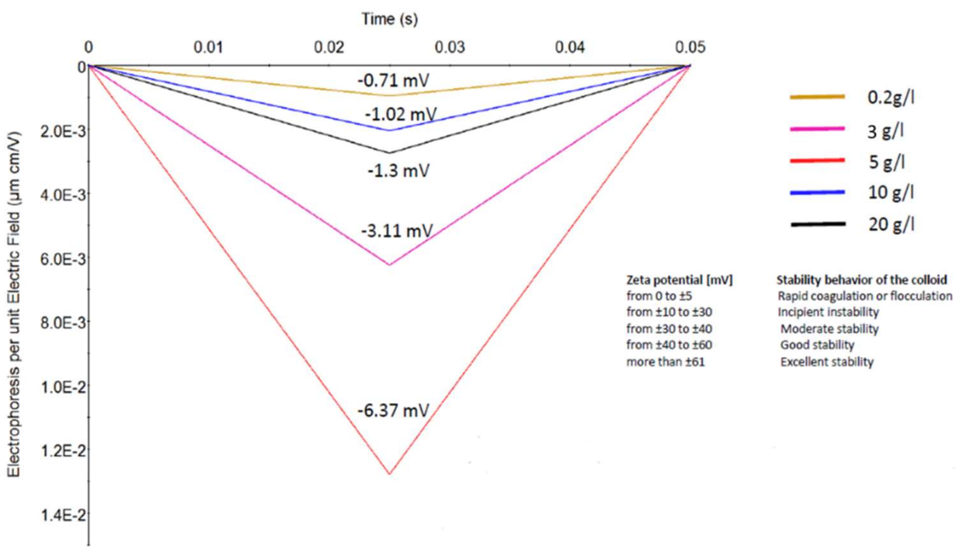

Figure 4: The Mobius electrophoretic mobility graph of the model Ni-W solution with varying concentrations of TiO_2. The TiO_2 at pH 8 results in negative charge in the solution. The slope is related to the particle mobility and the direction of the V-graph indicates the sign of the particle charge.

Coating features that were tested on the samples were the coating composition, roughness of surface, and microhardness. The XRF determined the percent composition of nickel, tungsten, and titanium on the samples surface and is displayed in Figure 5. The titanium content in the

coatings significantly started increasing above the 5 g/L TiO_2 in solution. As the TiO_2 concentration increases from 5 to 20 g/L, the tungsten content decreases, and nickel content remains fairly stable. However, above the 20 g/L concentration in the solution the tungsten content on the surface significantly increases while the nickel content decreases. Later results that test performance reveal that raising the TiO_2 content had a beneficial effect on the coating.

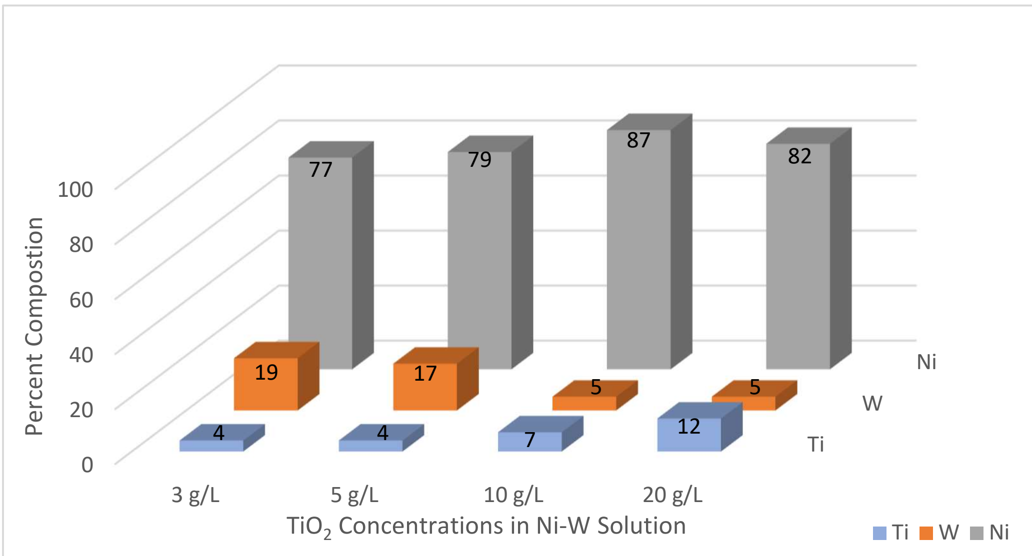

Figure 5: The percent composition of Ti, Ni, and W for the deposited samples from various TiO_2 concentrations. Tested using the XRF.

The ZYGO microscope and SEM were used to observe the surface roughness (Ra) of the deposited samples. The lowest Ra values were the TiO_2 concentrations of 10 g/L and 20 g/L, with roughness of 14 nm and 19 nm, respectively (Figure 6). The increased surface roughness of the other samples is correlated to the nickel and tungsten ratio in the coating. SEM images of the sample surfaces can be seen in Figure 7. An increasing TiO_2 concentration also resulted directly in increasing microhardness as seen in Figure 8.

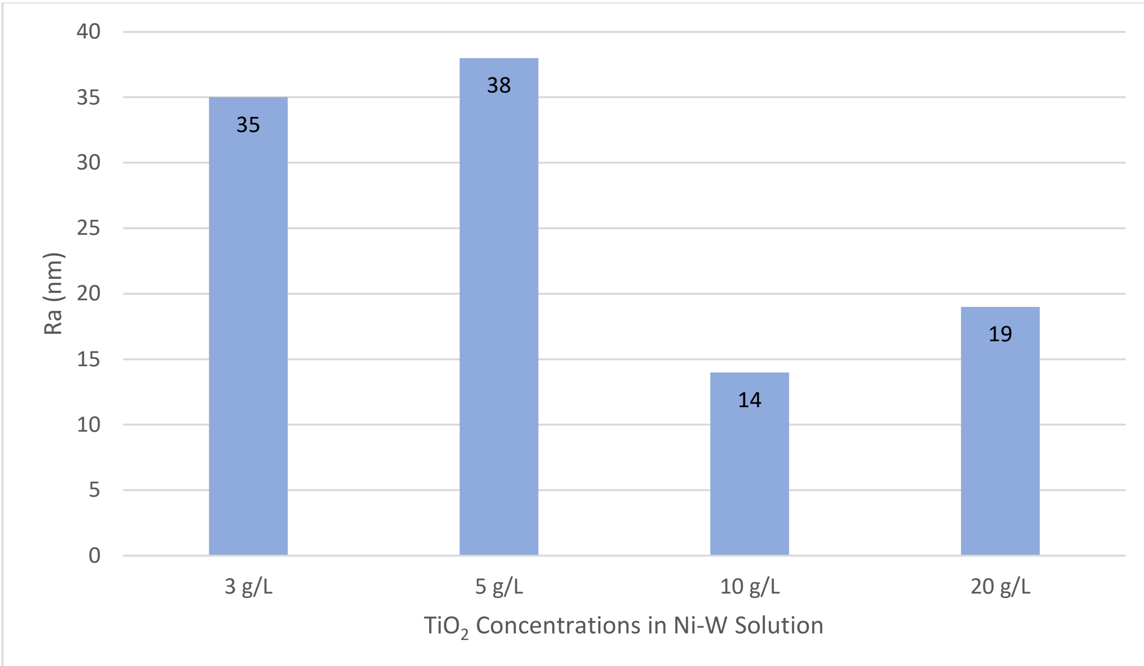

Figure 6: Surface roughness (Ra) in nm of coatings from Ni-W solutions with 3, 5, 10, & 20 g/L TiO_2 at pH 8.

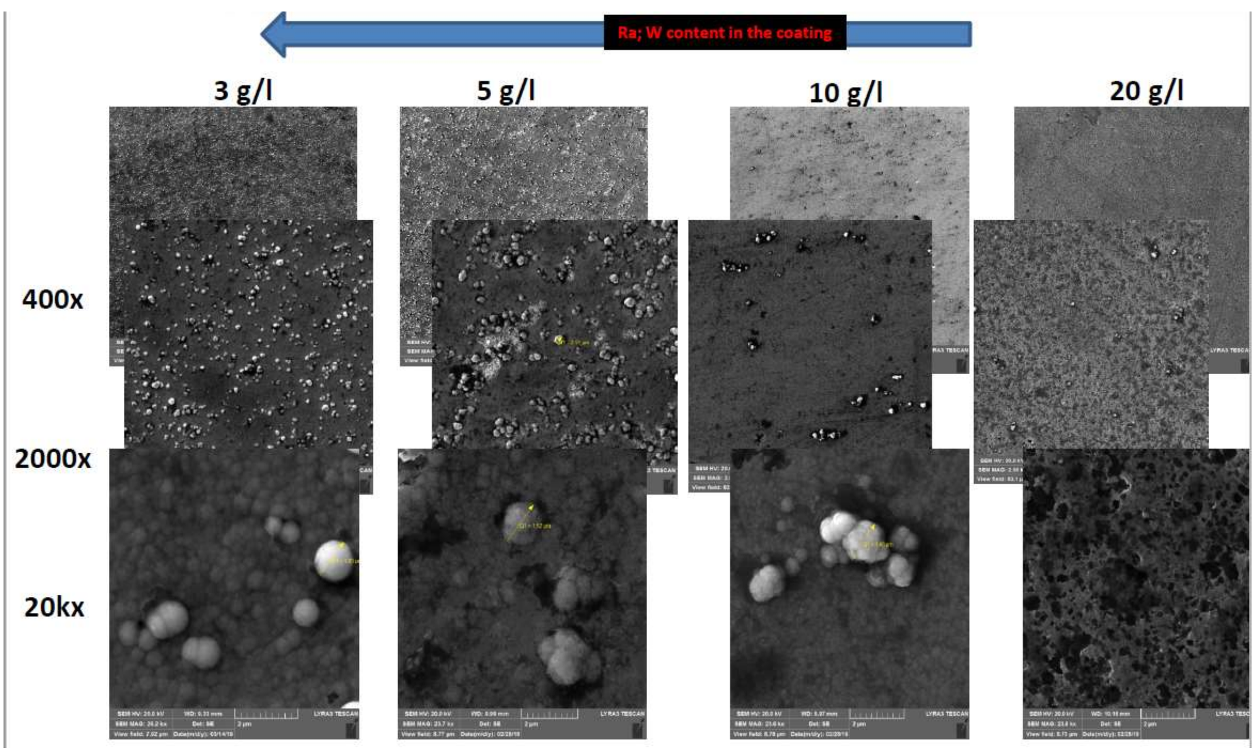

Figure 7: SEM plan-view images of 3, 5, 10, & 20 g/L TiO_2 in Ni-W solutions at pH 8. As the TiO_2 concentration increases, the Ra and tungsten content in coating decrease.

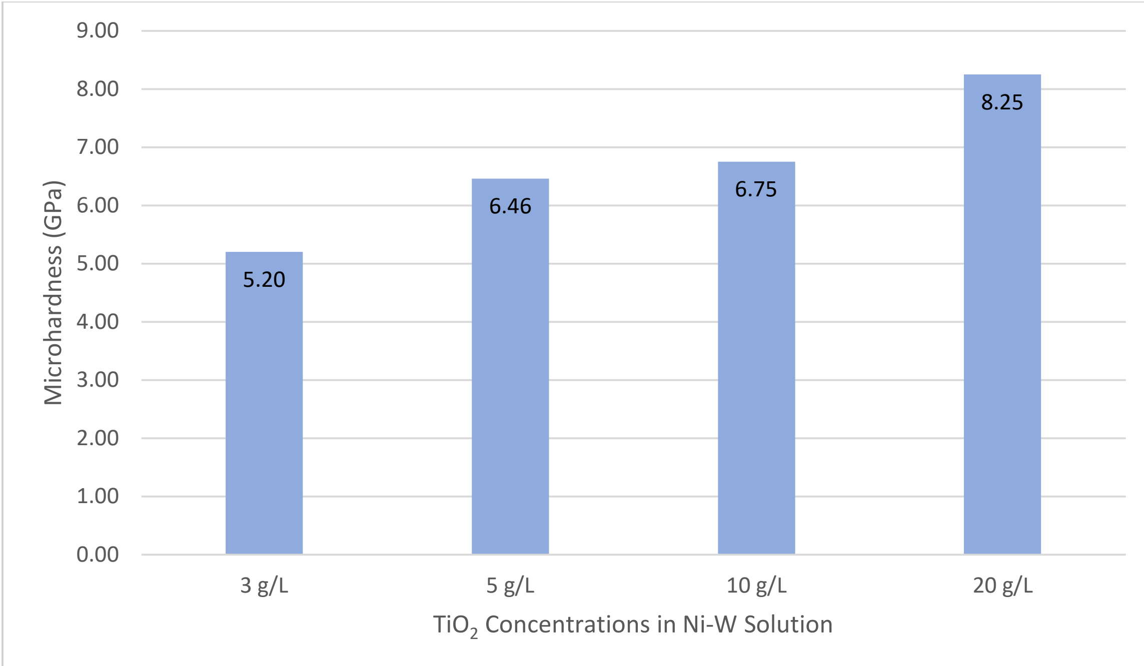

Figure 8: Microhardness (GPa) graph for the deposited samples from various TiO_2 concentrations.

Coated samples were tested on the HFRR with ISO 68 mineral oil each at three varying temperatures. Figure 9 depicts the decrease in the coefficient of friction with the increase in TiO_2 concentration. Decreased coefficient of friction implies that there will be less wear on the material. This was supported from the corresponding decrease of absolute wear volumes on the disk and ball in Figure 10 and Figure 11. In Figure 12 it's seen that as the TiO_2 concentration increases, the nature of the wear scar alters. Lower concentrations resulted in a smashed surface while higher concentrations resulted in a brittle surface, indicating that the lower concentrations have less surface hardness than the higher concentrations. These conclusions are corroborated by the hardness results in Figure 8. For this test, temperature seemed to have little effect.

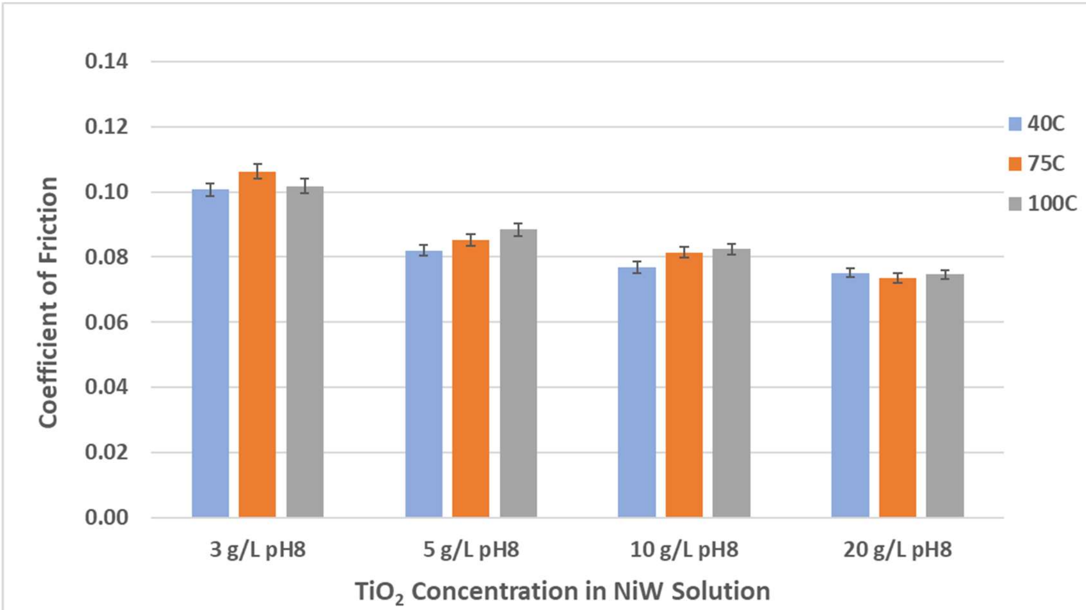

Figure 9: HFRR coefficient of friction data for the deposited 52100 samples from various TiO_2 concentrations. Tested with ISO 68 mineral oil at three temperatures (40, 75, & 100°C) and steel ball.

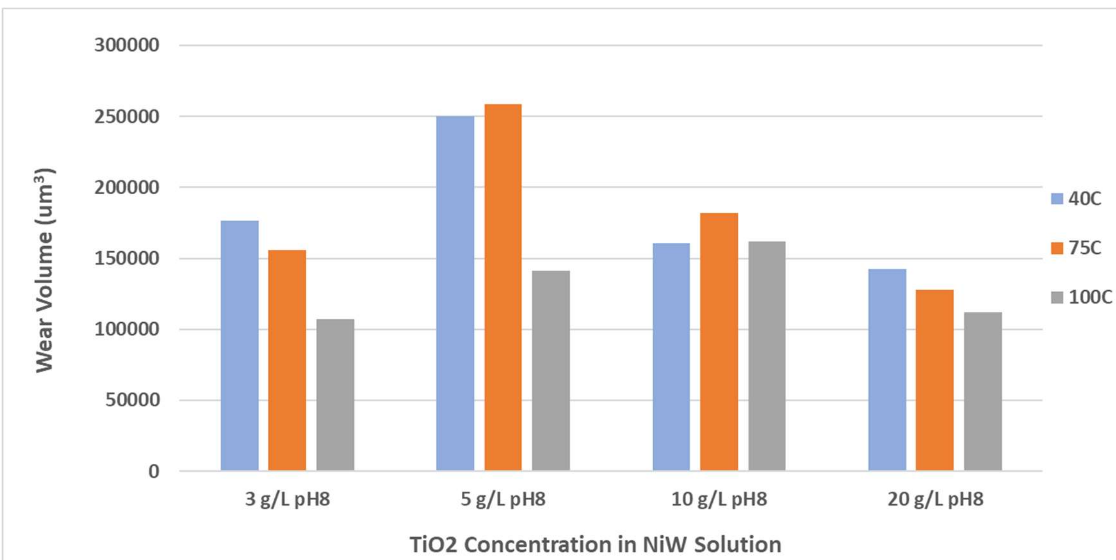

Figure 10: 52100 disk wear volume for the deposited samples from various TiO_2 concentrations after running on the HFRR with steel ball. HFRR tests with ISO 68 mineral oil at three temperatures (40, 75, & 100°C).

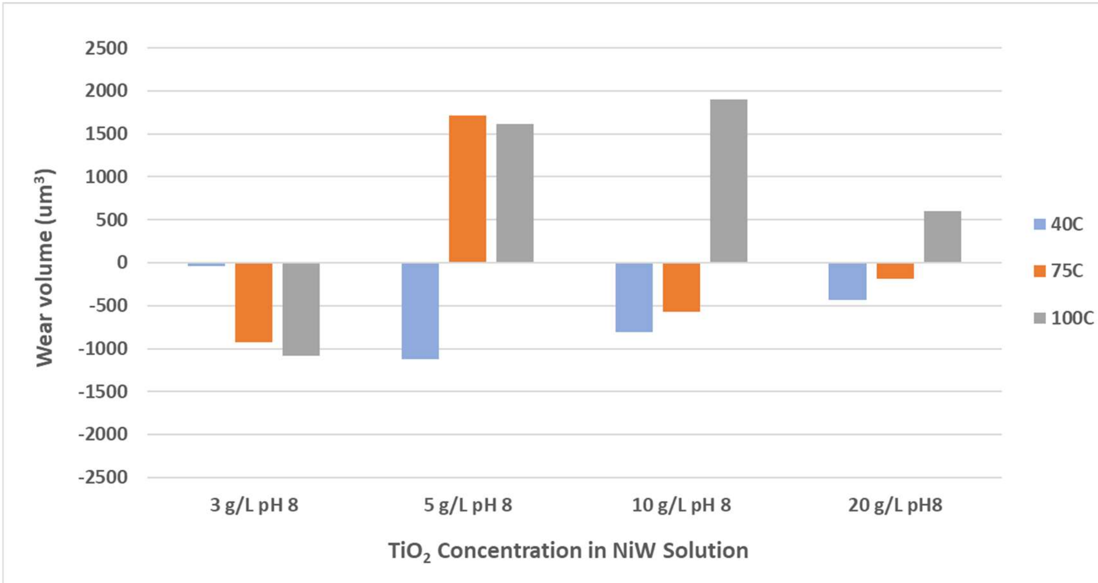

Figure 11: Steel ball wear volume for the deposited 52100 disk samples from various TiO_2 concentrations after running on the HFRR. HFRR tests with ISO 68 mineral oil at three temperatures (40, 75, & 100°C).

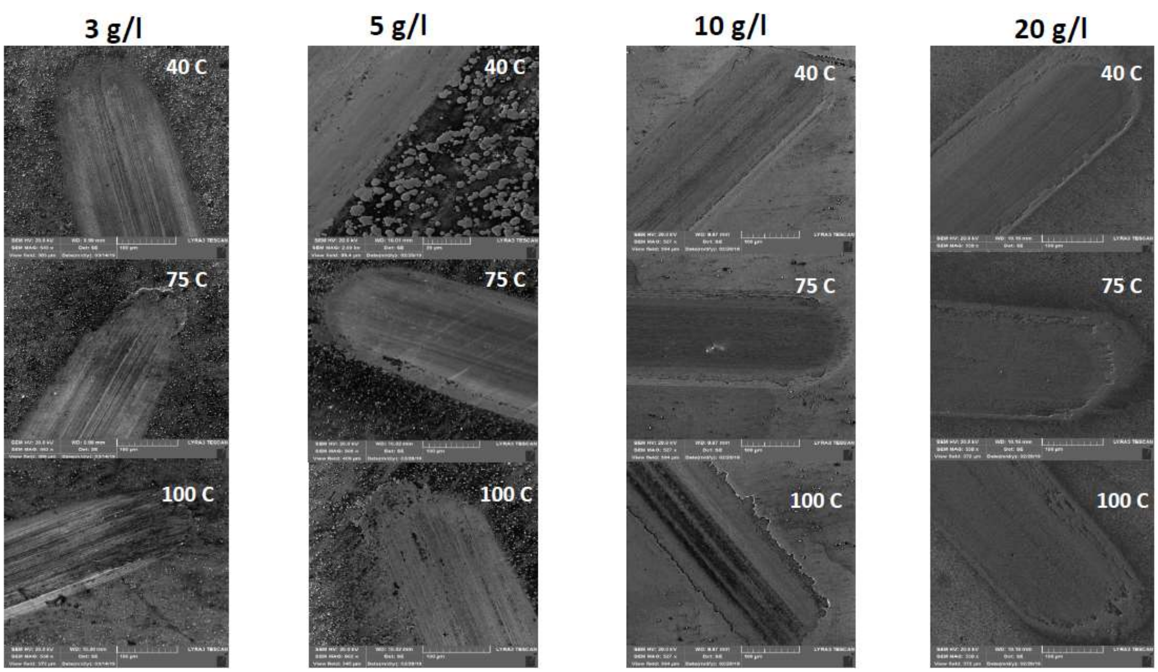

Figure 12: Disk wear scars on the deposited samples from various TiO_2 concentrations after running on the HFRR. HFRR tests with ISO 68 mineral oil at three temperatures (40, 75, & 100°C).

From the results of the 3 to 20 g/L TiO_2 solutions, it was determined that higher TiO_2 concentrations should be tested in addition to a lower pH solution. The particles of pH 8 (basic) solutions were negatively charged as previously mentioned, whereas the pH 5 (acidic) solution particles were positively charged. Higher TiO_2 concentration increased the titanium and tungsten

content comparatively to all other concentrations at the cost of the nickel content, which is displayed in Figure 13. Decreasing the pH from 8 to 5 for the 30 g/L TiO_2 in solution continued to increase the titanium and tungsten content while decreasing the nickel content.

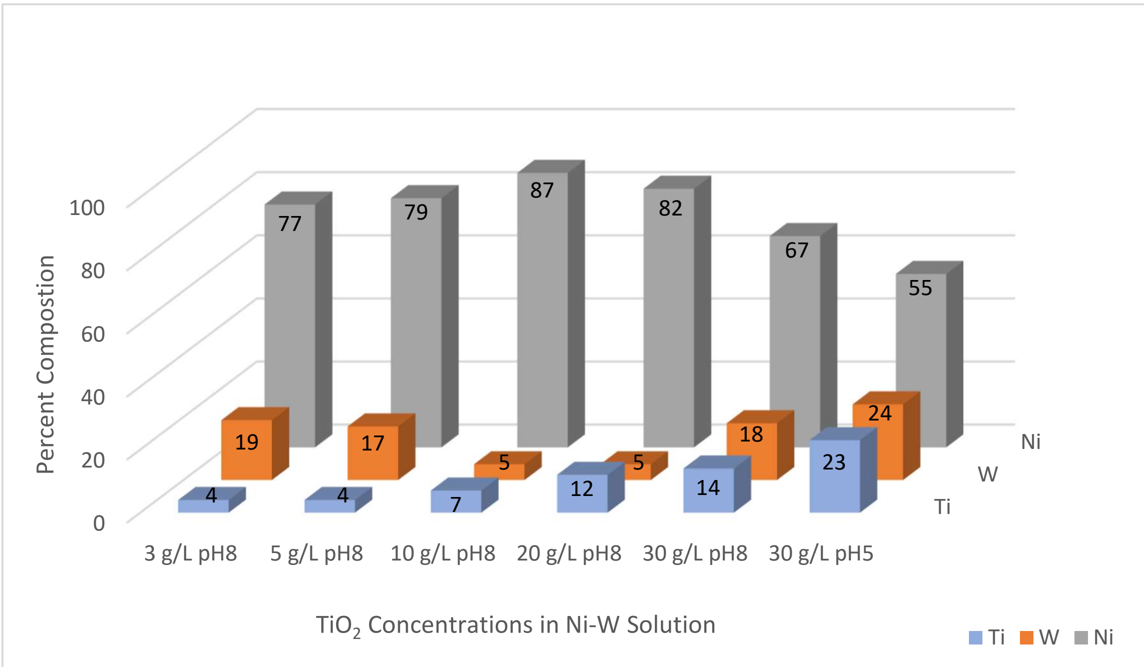

Figure 13: The percent composition of Ti, Ni, and W for the deposited samples from various TiO_2 concentrations from *Figure 5* with the addition of 30 g/L at pH8 and pH5 samples. Tested using the XRF.

Compared to results of lower TiO_2 concentrations, the 30 g/L at the same pH had a relatively higher surface roughness of 84 nm. When pH was decreased to pH 5 the roughness decreased to 46 nm, shown in Figure 14. It can be seen from Figure 15 that the surface roughness of the pH 8 coating is much higher compared to the surface roughness of the pH 5 coating in Figure 16. The microhardness as previously discussed increases with increasing TiO_2 concentration, however, shown in Figure 17 at 30 g/L pH 8 and pH 5 the microhardness of the coating drops 68% and 61% from the 20 g/L sample, respectively.

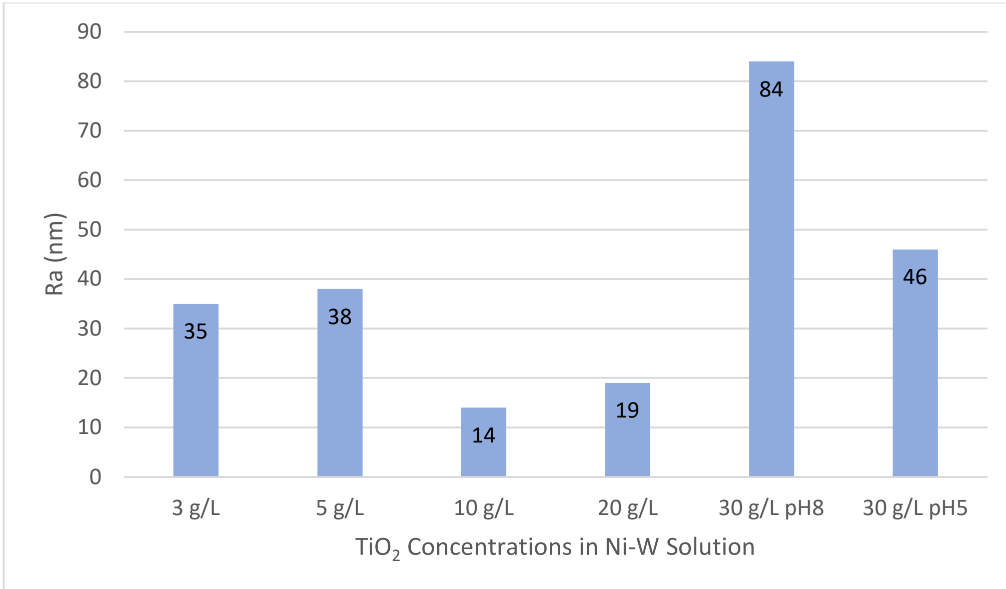

Figure 14: Surface roughness (Ra) in nm of coatings from Ni-W solutions with results from *Figure 6* with added results for 30 g/L TiO_2 at pH 8 and 30 g/L TiO_2 at pH 5.

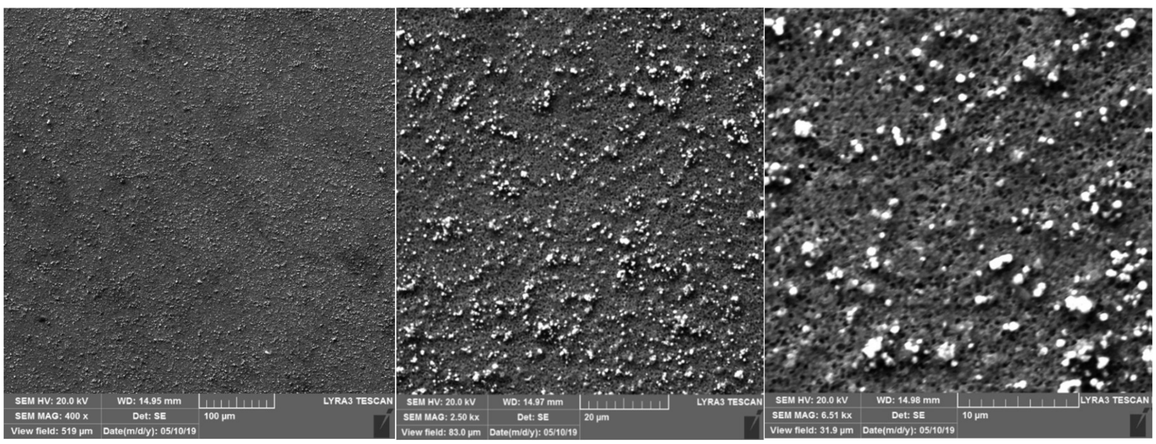

Figure 15: SEM images for the 30 g/L TiO_2 pH8 sample surface. The images are in order from left to right as 400x,2600x, and 6500x. The surface has an Ra=84nm.

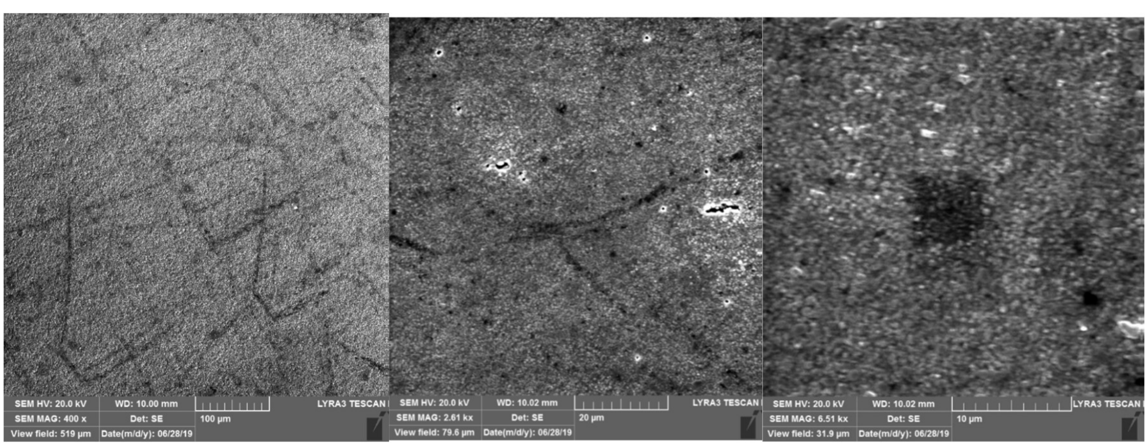

Figure 16: SEM images for the 30 g/L TiO_2 pH5 sample surface. The images are in order from left to right as 400x,2600x, and 6500x. The surface has an Ra=46nm.

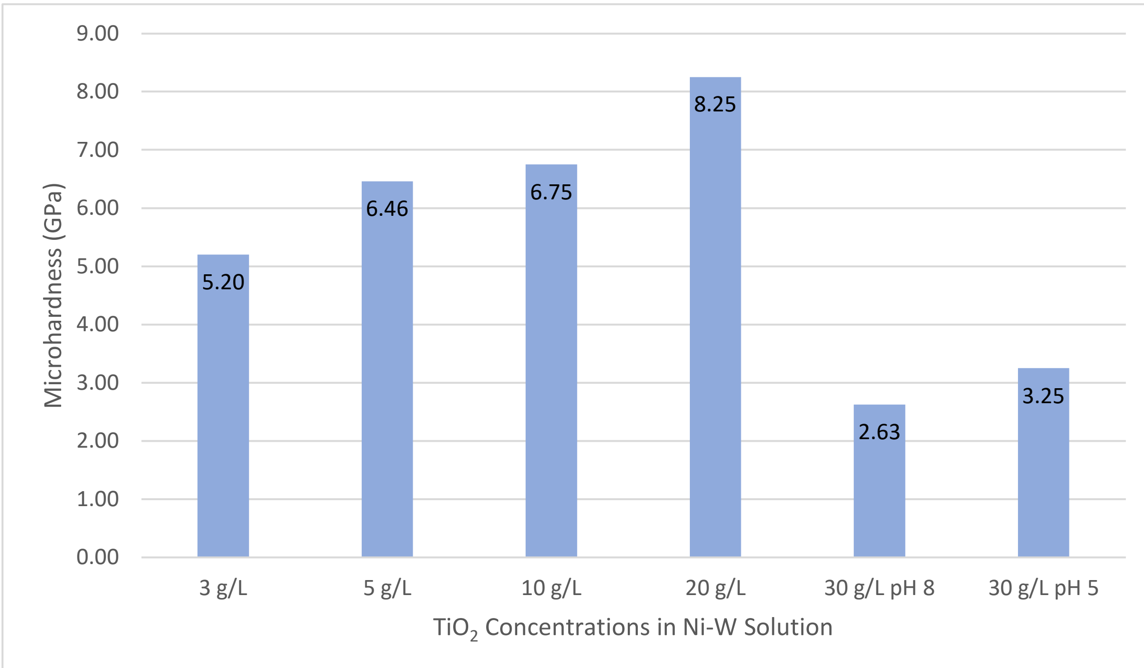

Figure 17: Microhardness (GPa) graph for the deposited samples from various TiO_2 concentrations.

Figure 18 depicts data showing that the 30 g/L samples at both pH's have higher coefficient of frictions for all temperatures. Figure 19 shows data from the resultant wear analysis where both 30 g/L samples had decreased disk wear, more noticeably for the pH 5 sample, and increased ball wear. This can be accounted to that the coating on the disk is more durable than prior coatings.

In Figure 20, Figure 21, and Figure 22 it's seen that the 30 g/L coating with the highest TiO_2 content of 14% has the most uniform wear scar compared to the smashed and brittle wear scars at lower concentrations. These results imply that an increased content of TiO_2 in the coating results in a better wear scar, and therefore a stronger coating that is not too soft or brittle. Comparing the surfaces of the 30 g/L concentrations at pH 5 and pH 8, there is mild pitting and a much higher surface roughness of pH 8 than the pH 5 which is shown in Figure 23. This is due to the different surface charges affecting the deposition.

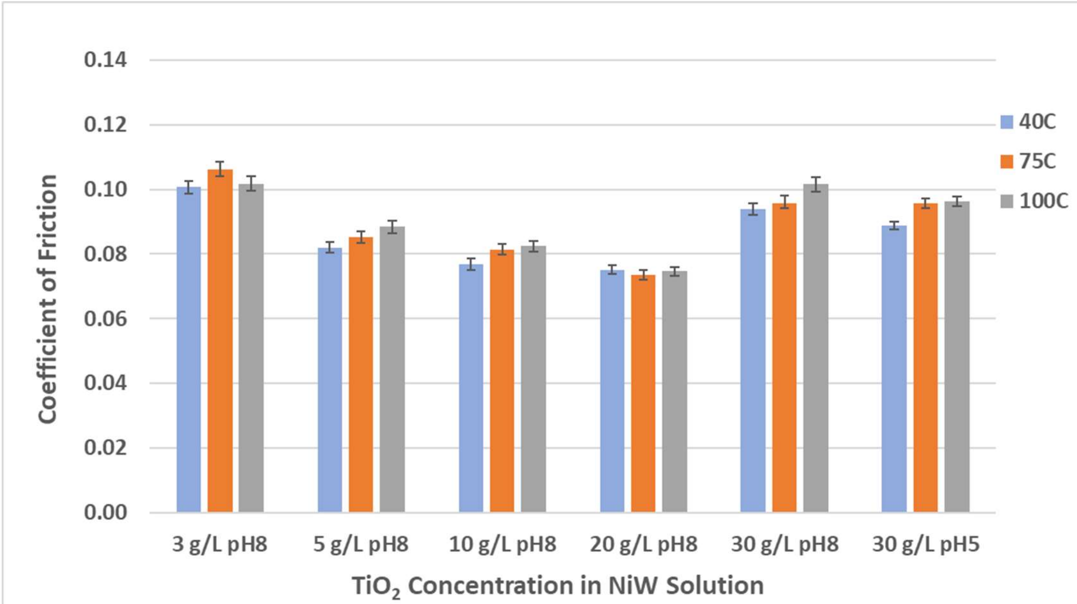

Figure 18: HFRR coefficient of friction data for the deposited 52100 disk samples from various TiO_2 concentrations from *Figure 9* with the addition of 30 g/L at pH8 and pH5 samples. Tested with ISO 68 mineral oil at three temperatures (40, 75, & 100°C) and steel ball.

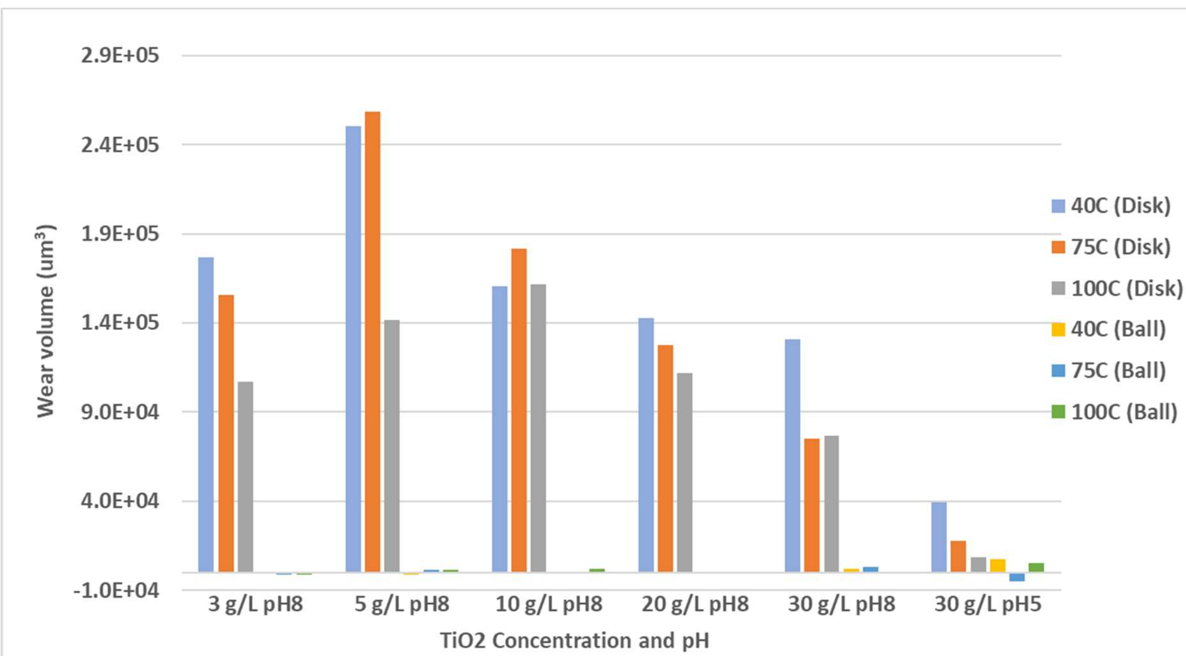

Figure 19: 52100 disk and steel ball wear volume for the deposited samples from various TiO_2 concentrations from *Figure 10* and *Figure 11* with the addition of 30 g/L at pH8 and pH5 samples. HFRR tests with ISO 68 mineral oil at three temperatures (40, 75, & 100°C).

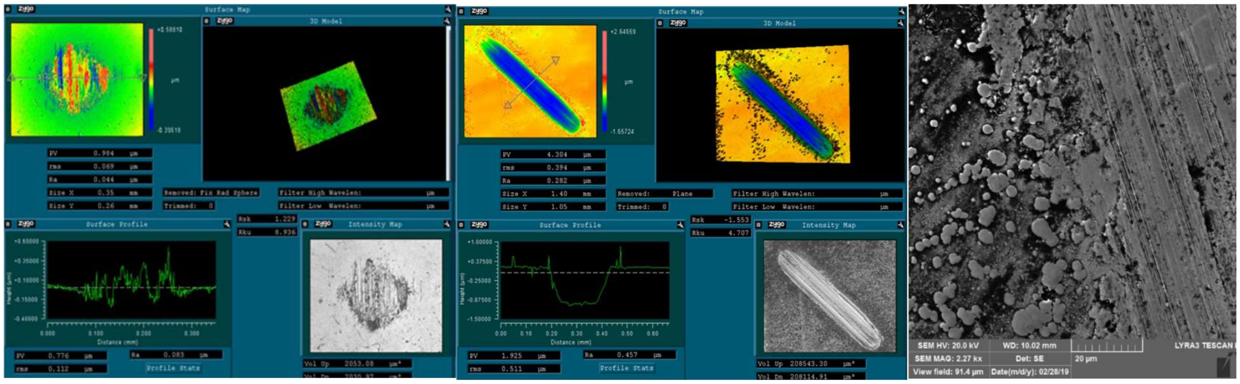

Figure 20: ZYGO images and SEM image for the 5 g/L TiO_2 sample wear scar that was ran at 100°C on the HFRR. The TiO_2 content in coating is 4%. The wear scar characteristic is smashed.

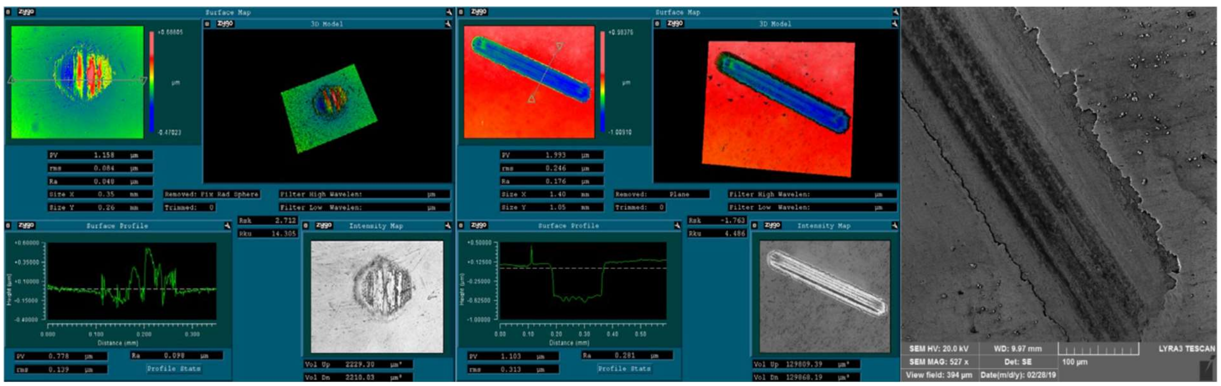

Figure 21: ZYGO images and SEM image for the 10 g/L TiO_2 sample wear scar that was ran at 100°C on the HFRR. The TiO_2 content in coating is 9%. The wear scar characteristic is brittle.

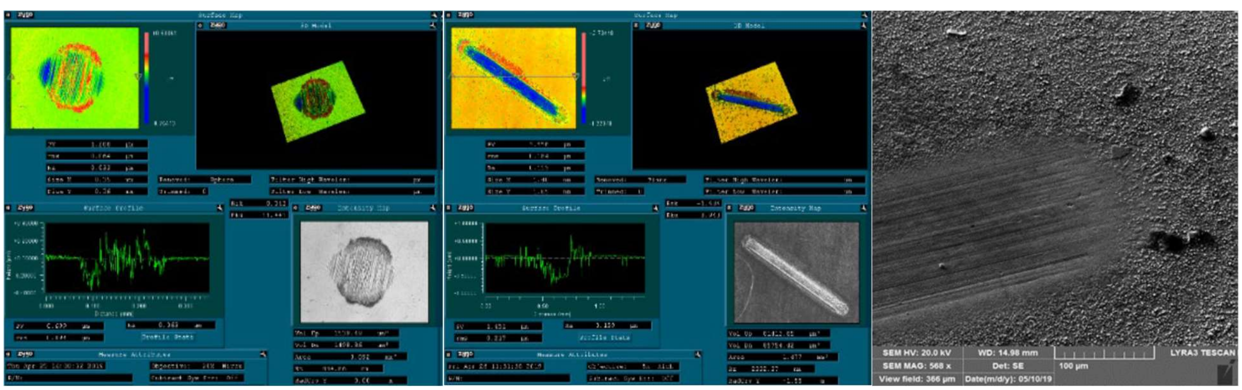

Figure 22: ZYGO images and SEM image for the 30 g/L TiO_2 sample wear scar that was ran at 100°C on the HFRR. The TiO_2 content in coating is 14%. The wear scar characteristic is uniform.

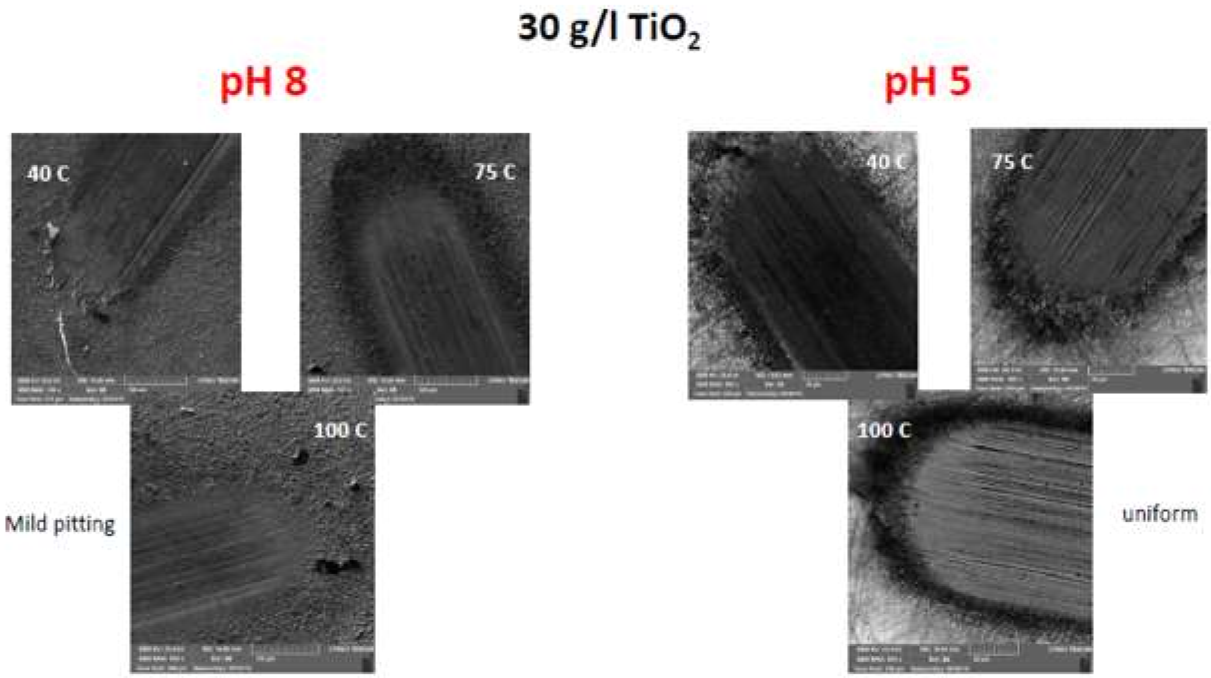

Figure 23: SEM images for the 30 g/L TiO_2 pH8 and pH5 sample wear scars being compared. Ran at 40, 75, and 100°C on the HFRR. The wear scar characteristic did not change significantly by lowering the pH.

Conclusions and Recommendations

TiO_2 solutions have a multimodal size distribution meaning the TiO_2 particles tend to aggregate in solution. The charge of the particles in solution are dependent on the solutions pH, which alters the way the particles are deposited on the surface of the sample material. Typically, during the electrodeposition the TiO_2 in the pH 8 solution (basic) has a negative charge, while the substrate has a positive charge during the I_{rev}. Opposingly, the pH 5 solution (acidic) results in a positive TiO_2 charge on the surface and the substrate has a negative charge during I_{fwd}. The concentration and the pH of the solution used has impacts in the results of the testing.

Ni-W doped with TiO_2 shows promise as a better performing coating than undoped Ni-W. This conclusion can be made based from the multiple methods of testing performance. The sample with the highest titanium content was the pH 5 30 g/L sample, which also has the highest tungsten content and lowest nickel content. The samples with the lowest roughness had the highest nickel content. It was determined that the ratio of the three metals in the coating is critical to determining

the characteristics of the coating. TiO_2 coatings at higher concentrations directly saw better wear resistance. The pH5 30 g/L sample exhibited the best wear resistance when compared to all other concentrations and formulations. Both 30 g/L samples provided the most uniform wear scars, unlike the smashed and brittle wear scars observed at lower TiO_2 concentrations.

Recommended further research of the optimization for the TiO_2 coating includes confirming that a pH of 5 and concentration of 30 g/L are optimum solution characteristics. Another coating property that should be investigated is the optimum ratio of Ti, W, and Ni coating content for tribological testing and how to repeatedly obtain such coatings. Further testing includes evaluation and comparison of 30 g/L TiO_2 at pH 5, 30 g/L TiO_2 at pH 8, and Ni-W-SDS coatings in rolling contact.

Literature Cited

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Appendix

Table A.1: Recipe for Ni-W+SDS electrodeposition solution, this does not include the various concentrations of TiO2 that was added in for the research of this report.

NiW+SDS Solution	MW (g/mol)	Molarity (mol/L)	Amount Used (g/L)
Nickel Sulphate	154.75	0.10	15
Sodium Tungstate	293.82	0.16	46
Ammounium Chloride	53.491	0.50	27
Sodium Bromide	102.894	0.16	16
Trisodium Citrate	258.06	0.57	147
Sodium Dodecyl Sulfate	288.372	0.01	2.88

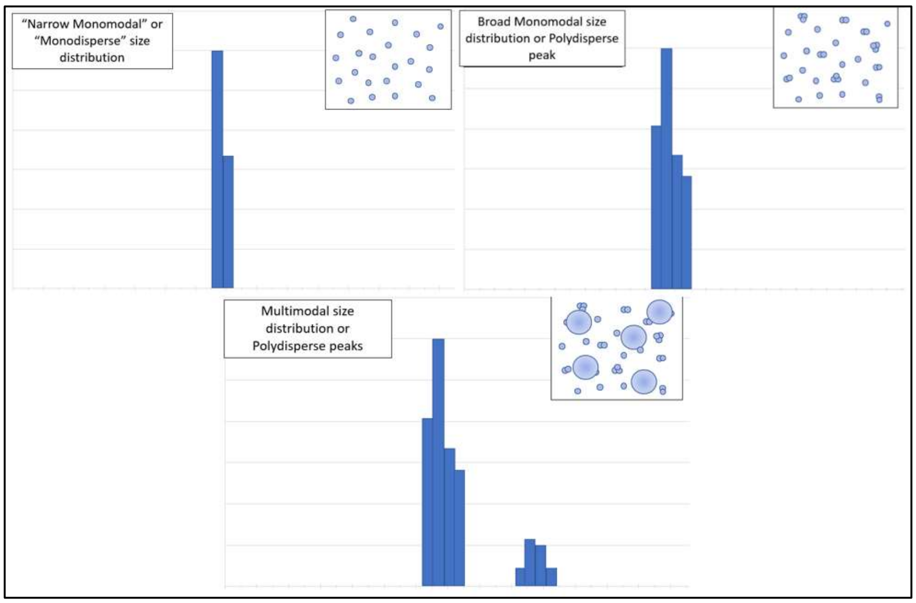

Figure A.1: Various particle size distributions of narrow monomodal, broad monomodal, and multimodal.