B. TECH. PROJECT REPORT

On

DEVELOPMENT OF ELECTROLESS NIBW AND NIBW-hBN COATINGS AND STUDY OF THEIR TRIBOLOGICAL PROPERTIES.

BY Potnuru Niranjan Prateek Kumar



DISCIPLINE OF MECHANICAL ENGINEERING INDIAN INSTITUTE OF TECHNOLOGY INDORE May 2022

DEVELOPMENT OF ELECTROLESS NIBW AND NIBW-hBN COATINGS AND STUDY OF THEIR TRIBOLOGICAL PROPERTIES.

A PROJECT REPORT

Submitted in partial fulfillment of the requirements for the award of the degrees

of BACHELOR OF TECHNOLOGY in

MECHANICAL ENGINEERING

Submitted by: **POTNURU NIRANJAN & PRATEEK KUMAR** Under The Guidance of: **Dr. Satyajit Chatterjee; Associate Professor**



INDIAN INSTITUTE OF TECHNOLOGY INDORE May 2022

CANDIDATE'S DECLARATION

We hereby declare that the project entitled "DEVELOPMENT OF ELECTROLESS NiBW AND NiBW-hBN COATINGS AND STUDY OF THEIR TRIBOLOGICAL PROPERTIES" submitted in partial fulfillment for the award of the degree of Bachelor of Technology in 'Mechanical Engineering' completed under the supervision of Dr. Satyajit Chatterjee, Associate Professor, Mechanical Engineering IIT Indore is an authentic work.

Further, we declare that we have not submitted this work for the award of any other degree elsewhere.

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CERTIFICATE by B. TECH Project Guide

It is certified that the above statement made by the students is correct to the best of my knowledge.

ft sh

Dr. Satyajit Chatterjee Associate Professor

Signature of BTP Guide(s) with dates and their designation

Thisreporton***DEVELOPMENT**OFELECTROLESSNiBWANDNiBW-hBNCOATINGSAND STUDY OF THEIR TRIBOLOGICAL PROPERTIES" is preparedunder the guidance of Dr. Satyajit Chatterjee.

Through this report, we have tried to give detailed information on the fabrication of ternary NiB based electroless coatings on the steel substrates and checking the feasibility of reinforcement of hBN particles in the alloy matrix to form composite coatings and to check the extent of enhancement its mechanical & tribological properties even at higher temperatures.

We have tried to the best of our abilities and knowledge to explain the content in a pellucid manner. We have added graphs and figures to make it more illustrative.

Potnuru Niranjan Prateek Kumar B.Tech. IV Year Discipline of Mechanical Engineering IIT Indore

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The people with the greatest indirect contribution to this work are my parents, who have been with me constantly encouraged us throughout this work and in life as well

Without their support, this report would not have been possible.

Potnuru Niranjan

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Abstract

The aim of this BTech project is to develop a lubricative coating via an electroless method that can withstand its properties at higher temperatures. Wear and scratching reduce the lifespan of the components significantly. Coating applied on the substrate improves the service life. Electroless plating can create nickel-based protective coatings with excellent mechanical and tribological properties. Among the different compositions explored over the years, borohydride-reduced electroless nickel coatings have shown a lot of promise in a variety of technological applications. Hardness, wear resistance, corrosion resistance, and other physical qualities of binary NiB electroless coatings are improved by adding tungsten (W) to the matrix. Hexagonal-boron nitride (hBN) is a solid lubricant that can sustain lubricity at extremely high temperatures. Our research focuses on the use of hBN in NiB-based electroless tribological coatings. When NiBW-hBN is heat treated, it shows a considerable improvement in sliding wear and scratch resistance when compared to other coatings.

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Chapter 1

Introduction

Various objectives of surface engineering include tailoring surface-dependent properties and improving the function's ability to enhance aesthetic appearance. These can be achieved by various processes that include PVD, CVD, electroplating, and electroless plating. By the process of electroless plating, the coating is deposited through a chemical method that uses an autocatalytic chemical reduction of metal cations in a liquid bath to form metal coatings on diverse substrates. Some of the advantages of electroless coatings include uniform deposit thickness; ability to plate complicated and irregular shapes; good adhesion; does not use of electric power. Some compositions of coatings that are prepared by electroless methods are Ni, Co, Au, Ni-P, NiB, Co-B, etc.,

Among the various compositions of coatings developed through electroless methods, NiB coatings gained high importance due to their mechanical and tribological properties. The microhardness of Ni-P, NiB 500 HV (M. Yan, n.d.), 570 HV (Krishnaveni, et al., n.d.) respectively. Such superior characteristics of NiB matrix can be further enhanced through the addition of elements like W, Mo. etc. Among all, the addition of W shows a marked improvement in tribological and mechanical properties. C.O. F of NiB based titanium coating is in the range of 0.25-0.55 (İlhan Çelik, n.d.). The frictional behavior of such binary NiB and NiBW coatings can be improved with the addition of solid lubricants.

Solid lubricants provide good lubrication in high humidity, can reduce coefficient of friction under high loads, have great temperature stability, are resistant to fretting corrosion and easily retain lubricity. Some examples of solid lubricants are MoS₂, WS₂, hBN, graphite, CNT, PTFE, etc., compare to other lamellar solid lubricants hBN is chemical stability at elevated temperature; oxidation resistant up to about 700°C (Co, n.d.) in powder form and 980°C (Co, n.d.) in solid form; favorable lubricating properties under dry and wet conditions; ability to stabilize the tribological properties at high temperatures. hBN was reinforced with Ni-P composite coating on AISI 1080 steel and conducted heat treatment tribological tests with 10 N load, 5 mm stroke length, 3 Hz frequency at room

temperature, heat treated (300°C), and in-situ high temperature (300°C), Ni-P-hBN coatings had C.O.F of 0.35, 0.3 and 0.27 respectively. (K.Uday Venkat Kiran, n.d.)

Average friction coefficient of NiP-hBN at room temperature (25°C) is 0.22, while after heat treatment (400°C) it increased to 0.58. (O.A. Leon, n.d.)

There is much more scope to study the effect of hBN reinforcement as a solid lubricant in electroless NiBW coatings. In this study hardness of the obtained coating along with the wear and scratch responses are evaluated.

Chapter 2

Materials and Methods

2.01 Substrate Preparation

The experiments were performed on an <u>AISI 1025</u> steel specimen sectioned into 25*25*5 mm³ dimensions. The substrate is further polished with 80 grade SiC paper for 20 minutes, 10 minutes with 220,400,600 grade SiC papers, and 7 minutes with 800,1000,1200 grade SiC papers. On one of the sides, notches were cut to hold the substrate in place while deposition takes place. The sample is degreased using an ultra-sonification process with acetone for 15 minutes. Followed by ultra-sonification the specimens are immersed in a 10 percent NaOH solution for 15 minutes at a temperature of 50–60 degrees Celsius. The sample surfaces are activated by immersing them for 15–20 seconds in a 37 percent HCl solution. After each phase of the pre-treatment process, the sample is given an intermediate rinse in deionized water for a few seconds. Now the sample is catalytically active without the presence of oxides grease etc, and ready for the development of coating.



Figure 1: Polishing Machine

2.02 Preparation of Electroless Bath

Electroless plating bath was composed of Nickel Chloride which provided Ni^{+2} ions for Ni deposition, sodium borohydride (NaBH₄) as a reducing agent, ethylenediamine (NH₂– CH₂–CH₂–NH₂) as a complexing agent to make complex with excess Ni^{+2} ions and control the reaction, lead nitrate (Pb(NO₃)₂) as a stabilizer by producing the Pb⁺² ions and lead ions gets adsorbed on the activated surface of the substrate and reduces the number of nucleation sites and thus prevents the decomposition of the bath. Sodium tungstate is added to provide tungsten to the coating and an alloy coating of NiBW is developed.

In order to develop a composite coating of hBN in the NiBW matrix, after 20 minutes of a precoat of NiBW on the substrate hBN particles of size 1micron are added to the electroless plating bath. The temperature of the bath was maintained at $90\pm2^{\circ}$ C throughout the deposition process, any increase from this range leads to decomposition of the bath and decrease from this range will not make the coating deposition. NaOH was added to maintain the pH of the bath 13. The precise bath composition is presented in the table below.

Constituents	Concentration
Nickel Chloride	20 g/l
Sodium Hydroxide	40 g/l
Sodium Borohydride	0.8 g/l
Sodium Tungstate	25 g/l
Ethylenediamine	59 g/l
Lead Nitrate	0.0175 g/l
hBN particles	1 g/l

Table 1: Bath Composition of Sodium Borohydride reduced electroless Ni Bath

2.03 Synthesis of Electroless NiB Coatings

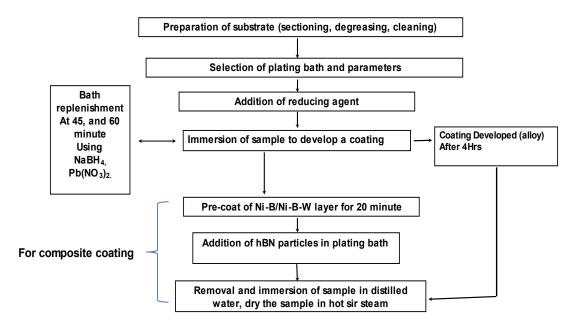


Figure 2: Flow Chart of the Electroless Deposition

Electroless nickel-boron coatings are made by the electroless plating method. In this type of coating, there is no need for an external power source as in electrolysis, the heating of the electroless bath provides energy for deposition. Metal salts present in the bath are the source of metal ions in the bath. Reducing agent reduces the metal ions on the substrate surface. In the initial stage, Ni atoms get attached to the active sites of the substrate and there will be the formation of large nuclei on the surface. With time the growth of the crystallites in the lateral direction gets confined by the adjacent crystallites due to mutual contact. Thus, during deposition, the crystallites grow faster in the vertical direction than that in the horizontal direction till a stage is reached where lateral growth is being hindered, only vertical growth takes place. This leads to the generation of a typical cauliflower-like surface and an alloy coating will be developed on the substrate. If hBN particles are added to the bath after the precoat of NiB/NiBW, they will get reinforced in the NiB/NiBW matrix and a composite coating will be developed.

2.04 Sectioning

After the deposition, the substrate is taken out from the bath and submerged into 50°C DM water for pre-treatment, and further cleaned by ultra-sonification with acetone. An increase in weight of the sample is observed after coating and the sample is now sectioned into smaller pieces for the purpose of heat treatment, observing the coating thickness and various tests. A small section of the sample is mounted and polished till diamond polishing to impart mirror finish for the purpose of hardness testing and observation of coating thickness.



Figure 3 : Hot Mounting Machine

2.05 Heat Treatment

Heat treatment of the coated samples is carried out at 450°C in argon environment by using a tube furnace (VB Ceramic Consultants 1400°C, India). The metallic substrate is protected from high temperature by packing it with a ceramic material. The temperature of the chamber has been raised with a rate of 5°C/min. The samples are kept at a set elevated

temperature for 1 hour and are allowed to undergo furnace cooling till the room temperature is reached.



Figure 4 : Tubular Furnace

Samples Prepared:

As-Deposited	Heat-Treated
NiB	NiB
NiBW	NiBW
NiBW-hBN	NiBW-hBN

2.06 Microhardness Test

Vickers microhardness test is one of the most commonly used techniques for the measurement of microhardness of coated samples. In this technique, indentation is performed by a square-based, highly polished, and pointed pyramidal diamond indenter having a face angle of 136°

Vickers microhardness value is typically expressed in kgf/mm² without mentioning the unit. Of late, the literature on coating has expressed Vickers microhardness in GPa. In the current research work, the cross-sectional hardness of the coatings has been measured by using a Mitutoyo HM-210A microhardness tester. An average of at least 10 readings has been reported for a specific coating.



Figure 5 : Micro Hardness Machine

2.07 Optical Microscope

Samples are observed at various instances under the optical microscope (before and after tests) to get an insight into changes on the coating surface being after subjected to different tests. Coating thickness is calculated by observing the polished cross section of the sample under optical microscope. The width of the wear track and scratch track on the coating are measured by using the optical Microscope (Fig. 6).



Figure 6: Optical Microscope

2.08 Field Emission Scanning Electron Microscopy (FESEM) Analysis

To get the further insight into the coating, the samples were observed under FESEM. FESEM offers high magnification and higher quality of images than a optical microscope. The samples were thoroughly polished until diamond polishing with SELYVT cloth. The coating cross-section is observed to know the father insight into coatings homogeneity and the thickness.



Figure 7: FESEM Machine

With the increase in sophistication of investigation, instruments having superior depth of focus or spatial resolution is required. So, for the purpose of detailed material characterization Scanning Electron Microscope is used. The resolution of SEM can approach a few nm and it can operate at wide range of magnifications.

2.09 X-ray Diffraction (XRD) Analysis

X-ray Diffraction (XRD) is a powerful technique for identifying and measuring the structural properties of crystalline phases present in materials. XRD can also be used to determine the thickness of thin films and multilayers, as well as the atomic arrangements in amorphous materials (including at interfaces). Thin-film XRD can precisely determine the presence and composition of phases.

X-ray diffraction analyses play an important role in the identification of phases present in the coating as the properties of the coatings are closely related to the phases present in it.



Figure 8 X-ray Diffraction machine

2.10 Wear Tests

Wear is the loss of material from the surface by the action of another surface in contact with it and wear resistance is the ability to resist that loss. Wear can be classified in 3 ways

- 1. observation and study of the visual aspects of the wear scar e.g., pitting, scratch, fretting, scuffing etc.
- 2. by considering the physical mechanisms of material removal and damage like adhesion abrasion, delamination etc.
- 3. by studying the wear environment e.g., lubricated or unlubricated wear, sliding wear, rolling wear etc.

In this study, a coating has been developed on the substrate with a view to impart high wear resistance and to decrease the coefficient of friction of the surface.

Sliding wear test (reciprocating ball-on-disk type sliding wear test) was performed on the samples using a hard spherical (WC with 6mm diameter) counter body in contact under load. Various wear tests were carried out on NiBW and NiBW-hBN samples with different amplitude of reciprocation, and frequency at various loads. The test parameters were:

Load (N)	Amplitude (mm)	Frequency (Hz)	Time (Minutes)	
	0.5	50		
3	3	10		
	6	10		
	0.5	50		
10	3	10		
	6	10	15	
	0.5	50		
12.5	3	10		
	6	10		
	0.5	50		
20	3	10		
	6	10		

Table 3: Wear Test Parameters

After the tests were performed, optical images of the wear track were observed under inverted microscope and wear volume was calculated by measuring the width of the wear track. Variation in coefficient of friction with respect to the amplitude and frequency at various load was also plotted in graph and comparison was made between NiBW alloy coating and NiBW-hBN composite coating for both as-deposited and heat-treated case.

In-situ high temperature wear test at 450°C were also performed for NiBW-hBN for 3 mm amplitude and 10 Hz frequency under 10 N load.



Figure 9 Ball on Disc Tribometer

2.10 Scratch Tests

In scratch testing, a diamond ball indenter of 200µm radius is drawn across the surface of coated samples (as-deposited and heat-treated samples) over a defined distance with a predefined load. the load can be constant or progressively increasing. The constant load tests were performed with 8,10,12,14 NEWTON loads, to see the coatings variation of friction coefficient when exposed to low loads, to check the scratch response of the coating, a high load progressive scratch test

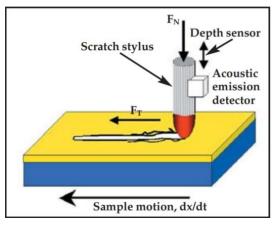


Figure 10: Schematic of scratch testing

is conducted, where the maximum load reached is 150N. scratch hardness and scratch volume loss are also calculated from the tests performed.

 Table 4: Parameters of scratch test

Test type	Load (N)	Scratch speed (mm/s)	Loading rate (N/s)	Scratch length (mm)
	8	0.3 -		
Constant load	10			3
	12		5	
	14			
Progressive load	0-150	1	15	10

Samples Tested: NiBW (as deposited and heat treated)

NiBW-hBN (as deposited and heat treated)



Figure 11 High Load Scratch Tester



Figure 12 : Constant Load Scratch Tester

Chapter 3

Results and Discussions

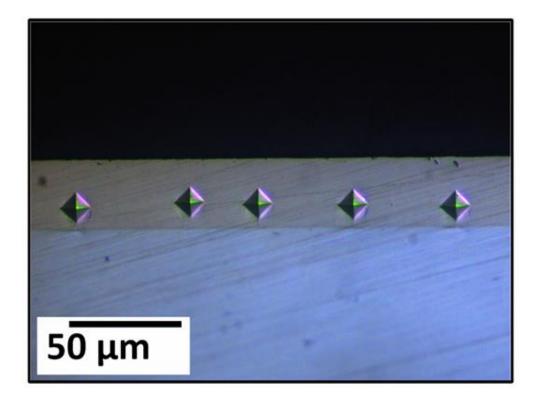


Figure 13 Indentation on the cross section of coatings (NiBW)

Vickers hardness tests were performed 10 times on the coated samples and the results are plotted in the bar graph shown below. the hardness of the heat-treated samples is more than those of the as deposited samples, the hBN reinforced samples shown slight improvement in hardness than the NiBW samples this may be due to formation harder NiB intermetallic as identified by the XRD data or the change in grain size of the crystals formed due to presence of hBN.

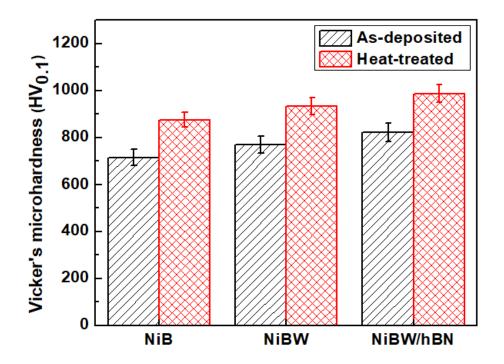


Figure 14: Bar graph comparison of coatings hardness

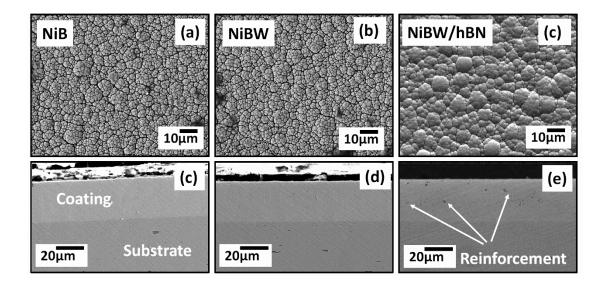


Figure 15: top surface and cross-section images of the coating of NiB, NiBW, NiBWhBN

Typical cauliflower-like structure is observed in the FESEM analysis of the coated surface of all the three NiB, NiBW, and NiBW-hBN developed coatings. Cross section of 3 coatings is also observed, in case of NiB and NiBW coatings the coatings cross section looks homogeneous whereas in NiBW-hBN composite coatings, reinforcement of particles is observed and looking like a heterogeneous coating.

In X-ray diffraction analysis of the as-deposited samples, the peak 45degree in all the 3 coatings represent Ni and the peak in the XRD pattern of NiBW-hBN at around 27 degrees confirms the presence of hBN in the coating. After heat treatment, the structure of the coating changes from amorphous to crystalline, and new harder phases of nib crystallites such as Ni_2B and Ni_3B have formed.

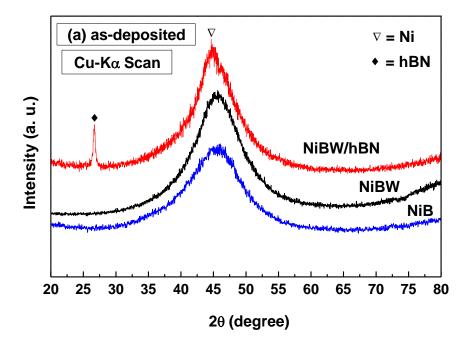


Figure 16: XRD patterns of as deposited samples

After heat treatment, the structure of the coating changes from amorphous to crystalline, and new harder phases of nib crystallites such as Ni₂B and Ni₃B have formed

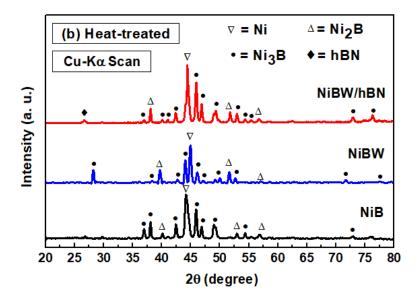


Figure 17 : XRD Analysis of the Hear-Treated samples

From the FESEM and XRD analysis of the developed coatings it can be concluded that hBN has been successfully reinforced into the NiBW metal matrix and heat treatment was also successfully performed.

The mechanical properties and elastoplastic behavior of the interacting surfaces, as well as the test parameters, govern the interaction between the counter bodies (e.g., Load, sliding speed, and sliding distance). The frictional force is ideally dependent on the shear strength and the contact area in the ball-on-flat test performed over coated samples in sliding mode. A tougher covering reduces the contact area, which affects friction and wear. The superposition of static and dynamic forces at a greater frequency causes a reduction in effective normal force at the contact surface, resulting in a reduction in effective normal force at the contact surface.

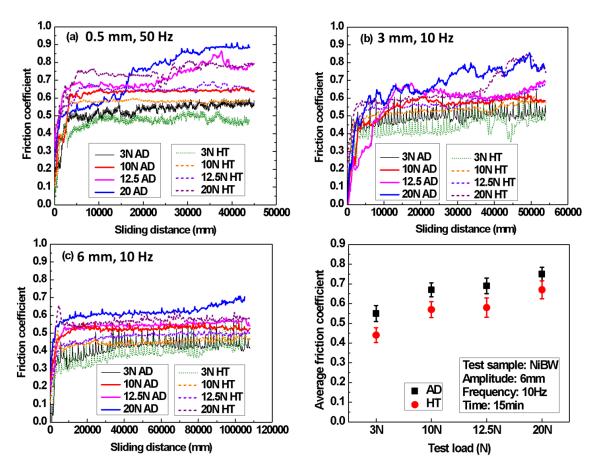


Figure 18 Plot of friction coefficient v/s sliding distance for various loads

(a) 0.5mm amplitude,50 Hz

- (b) 3mm amplitude, 10Hz
- (c) 6mm amplitude, 10 Hz

(d) 6mm amplitude, 10 Hz for all samples

Variation in C.O.F w.r.t. amplitude of reciprocation and frequency at various loads can be seen in the above plots. Solid line represents the as-deposited samples and the dotted curve represent the heat-treated coatings. Tests done at all 3 parameters show increase in coefficient of friction with increase in load for both as-deposited and heat-treated samples. After heat treatment slight reduction in coefficient of friction of the coatings is observed, this may be due to strengthening of coating matrix and presence of harder NiB crystallites

which resist the deformation of coating due to external load which lead to reduction in area of contact between the ball and the coating.

Wear tests done at 6mm and 3mm, 10Hz showed lower C.O.F as compared to 0.5mm, 50Hz, In case of higher amplitude the wear debris stays in the wear track and acts as a separation layer between the coating surface and counter body and hBN being weak in shear helps the counter body to slide over it resulting in lower C.O.F, for 0.5 mm amplitude and 50 Hz the wear debris come out of the wear track and every time fresh coating material gets in contact with the ball, resulting in higher C.O.F.

C.O.F for NiBW-hBN coating is reported to be lower than NiBW alloy coating for wear test done at same parameters of 6 mm and 10 Hz at various loads, this may be due to the hardening of coating and enhancement in the lubrication after addition of hBN.

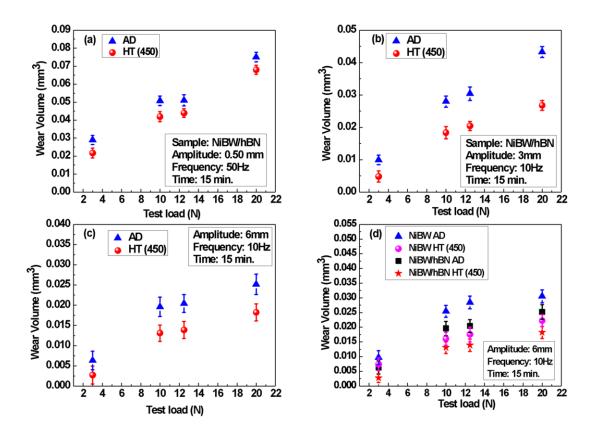


Figure 19 Plot of wear volume v/s test load of as deposited and heat-treated samples

- (a) 0.50 mm amplitude, 50Hz
- (b) 3mm amplitude, 10Hz
- (c) 6mm amplitude,10 Hz

(d) 6mm amplitude,10 Hz

In the above plots wear volume at different testing parameters is shown. It can be observed that with the increase in load the wear loss increase, due to increase in contact area. After heat treatment lower wear loss is reported due to strengthening of the coating.

The wear rate also lowers slightly as the reciprocating amplitude increases; this phenomenon could be explained by the fact that the interaction time between the ball and the surface decreases as the reciprocating amplitude increases at a fixed load and frequency. Contact occurs between the asperities of the counter bodies during the earliest stages of sliding. The asperities distort, break, or rupture during this first break-in period. This increases the contact area and establishes the conformity of the surfaces in contact. This occurs to varying degrees at various test-load values, and various forms of wear result in material volumetric loss during the course of the test.

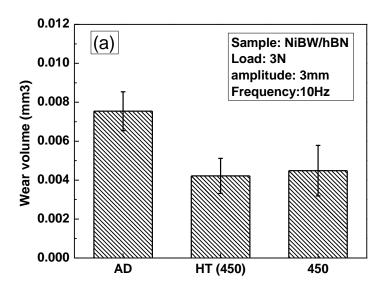


Figure 20 : Wear Volume comparison of NiB-hBN samples under various conditions

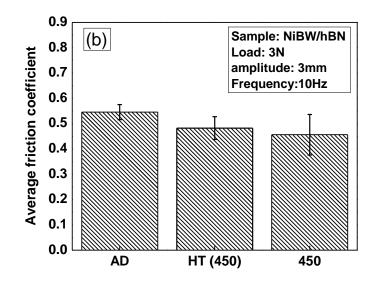


Figure 21 : Frictional Coefficient comparison of NiB-hBN samples under various conditions

Above bar chart represents the comparison between the results of wear test (3mm, 10 Hz, 3N) done on NiBW-hBN as-deposited, Heat Treated (450°C) and in-situ high temperature (450°C). For both the case of wear volume and avg. friction coefficient there has been improvement after heat treatment as well as in-situ high temp test. This may be due to formation of some harder phases of NiB during high temperature testing.

Samples haven't lost its lubrication property which obtained on the reinforcement of hBN even after subjected to high temperature rather it has improved.

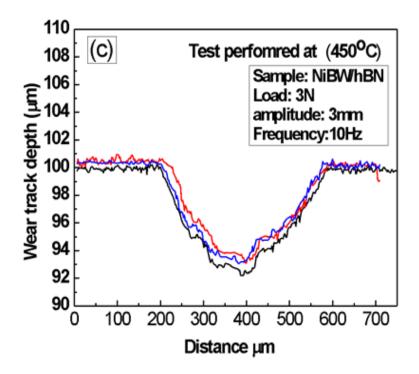


Figure 22 : Plot of wear distance v/s distance at in-situ conditions

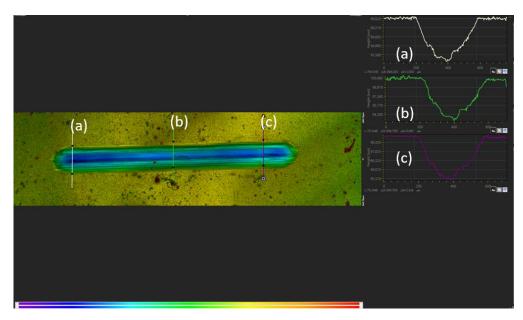


Figure 23 Optical profilometry of wear track (450°C)

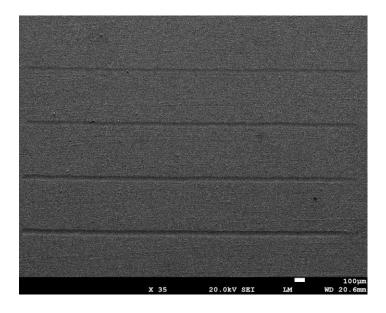


Figure 24 : FESEM image of scratch test under constant load

The results of the scratch testing are shown below in the form of graphs, the friction coefficient is plotted against scratch length (3mm for constant load). The friction coefficient is observed to be increasing with the increase in external load, the heat-treated samples showed lower friction compared to as-deposited samples at all loads, the hBN reinforced samples showed a lower friction coefficient than the as-deposited one in both the cases, overall, the hBN reinforced heat-treated sample shown lowest friction among all samples.

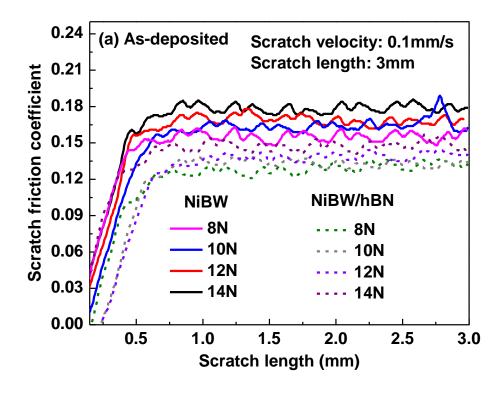


Figure 25 : Plot of friction coefficient v/s scratch length of the as deposited samples under constant load

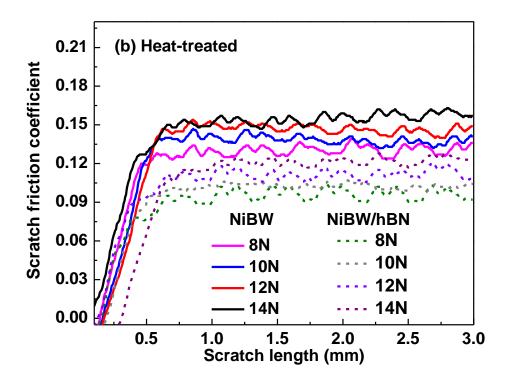


Figure 26 : Plot of Friction coefficient v/s Scratch length of the Heat-Treated samples

Scratch hardness is observed to follow the same trend as Vickers hardness test, the heat treated hBN reinforced sample shown the highest scratch hardness compared to other samples. the scratch volume loss is found to be lowest in the heat treated hBN reinforced samples.

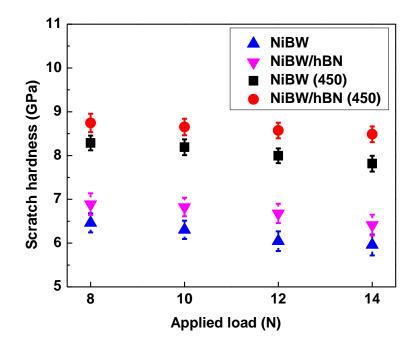


Figure 27 : Comparison of Scratch Hardness of all tested samples

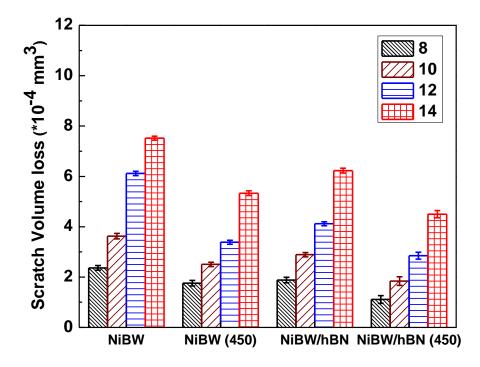
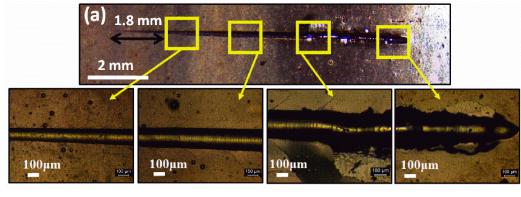


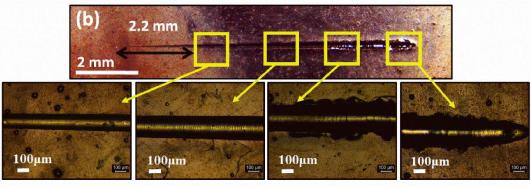
Figure 28 : Comparison of Scratch Volume loss of all tested samples



High Load progressive scratch test observations under optical microscope:

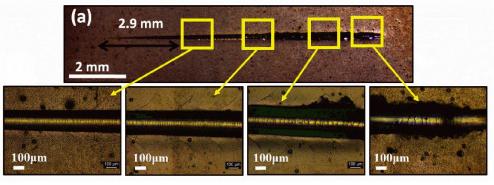


Figure 29 : Wear track of NiBW sample under progressive load scratch testing



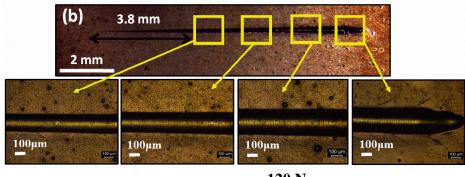
105 N

Figure 30 : Wear track of Heat-Treated NiBW sample under progressive load scratch testing



85 N

Figure 31 : Wear track of NiBW-hBN sample under progressive load scratch testing



120 N

Figure 32 : Wear track of Heat-Treated NiBW-hBN sample under progressive load scratch testing

Spalling of the coating can be observed over the edges of the scratch track in the case of all samples. it was around 70N in the case of the NiBW sample, which was improved to 105 N after heat treatment (significant improvement after heat treatment). and in the case of hBN reinforced samples, it was around 82N for the as-deposited case. detachment of the coating(s) material in fewer zones within scratch track around 120 N is observed for the heat-treated hBN sample.

The length marked in the figures (1.8 mm; 2.2 mm; 2.9 mm; 3.8 mm) signifies the initiation of radial cracks. The length is more in the heat treated NiBW-hBN sample, which signifies its improved scratch resistance.

The reasons for this observation could be the nickel boron intermetallic formed after treatment and the decrease in grain size of crystal structure because of the presence of hBN.

Chapter 4

Conclusions and Future Scope

4.1 Conclusions

- Electroless NiB, NiBW, NiBW-hBN composite coatings are successfully deposited on AISI 1020 mild steel substrates.
- Typical cauliflower like structure is observed under FESEM for both the alloys and composite coatings.
- Amorphous structure of the as deposited coatings is confirmed by dome shaped XRD pattern. Coatings when annealed at 450°C that revealed different peaks of Ni₃B, Ni₂B thus confirming its crystalline nature post heat treatment.
- Formation of harder intermetallic phases after annealing improves mechanical properties such as hardness, and scratch behavior.
- Lower scratch friction coefficient is shown by heat treated NiBW-hBN samples.
- Presence of hBN in the NiBW alloy matrix significantly improves the tribological properties such as friction coefficient and wear. Heat treated NiBW-hBN composite coatings showed superior tribological aspects compare to as deposited one.

4.2 Future Scope

- In-situ high temperature scratch test.
- Incorporation of hard phase along with hBN to improve the hardness and wear.
- Modification of bath composition such as complexing agent, stabilizer, surfactant to optimize deposition parameters for better deposition rate and thicker coating.

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