High Temperature Characterization of Electroless Tribological Coatings

Mid-semester evaluation of M.Tech project

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Electroless Coatings in Tribological Situations

Advantages

- The ability to plate complicated and irregular shapes [1].
- Ability to deposit on electrically non-conducting substrates [2].
- Improved wear resistance due to a more consistent thickness [3].

Industrial Applications of Electroless Coating

Industries	Applications
Aerospace	Compressor blades, Piston heads, Engine mounts, Bearing journals, etc;
Automotive	Gears & gear assemblies, Fuel injectors, Heat sinks, etc;
Chemical	Valves, Turbine and compressor blades, rotor assemblies, Spray nozzles, Filters and components etc;
Oil and Gas	Tubes, Pump housing, Sucker rods, threaded parts, Packers, etc;
Materials Handling	Hydraulic cylinders and shafts, Extruders, Gears and clutches, etc;
Electronic	Capacitors, Contacts (surface, sliding, pins & sockets), PWB-connectors, PWB-surface mounts, etc;

G.O. Mallory (1990)

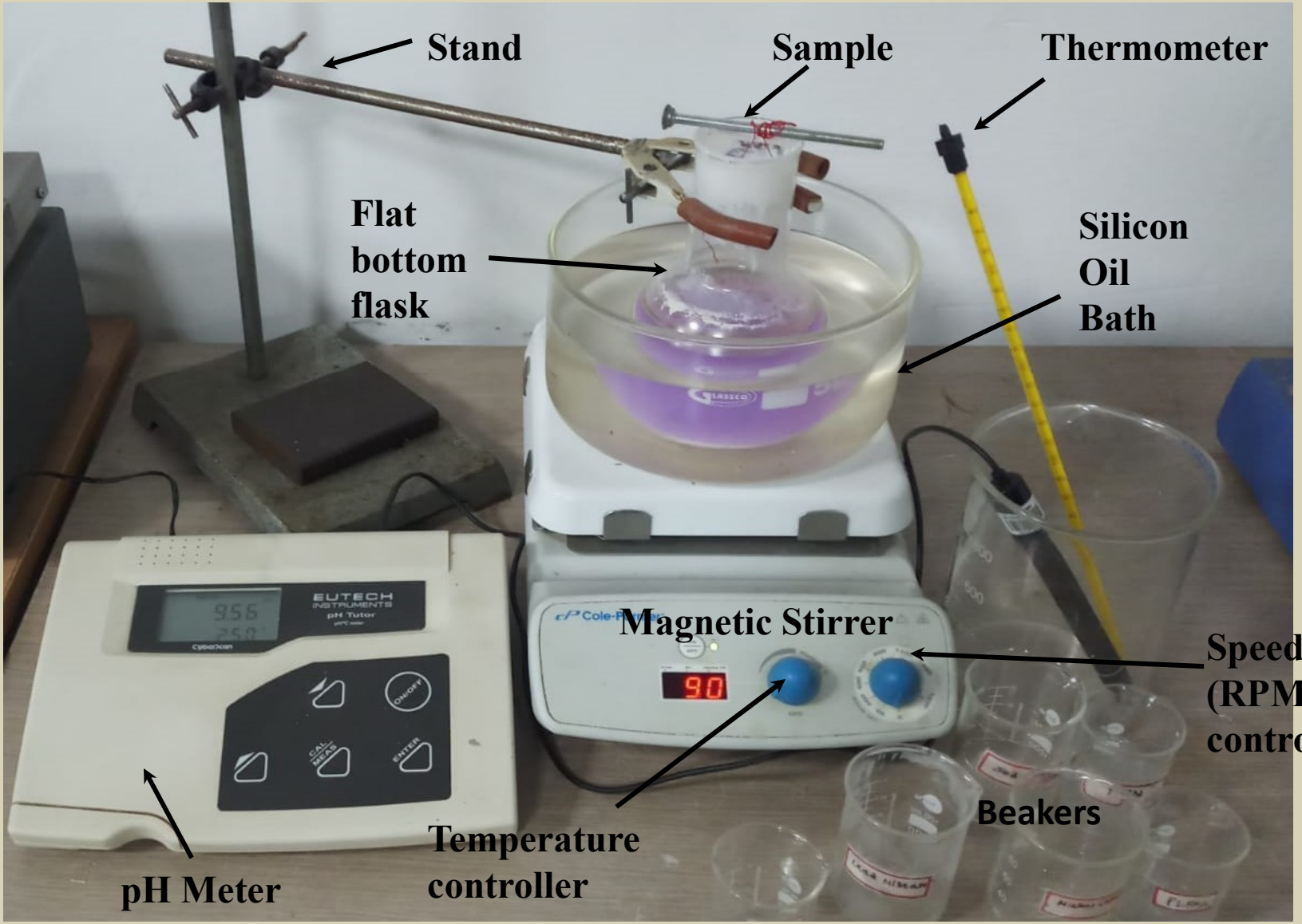
hBN can sustain 1000°C

Incorporation of hBN as solid lubricant can be beneficial in terms of high temperature tribological applications [5]

Objectives

1. To fabricate the coatings.
2. To Incorporate hBN for better mechanical and tribological properties.
3. Characterization of the coatings.
4. To check high temperature suitability of the coatings.

Set up for Deposition of Electroless Coating



Methodology

1. Preparation of substrate (grinding, polishing, degreasing)
2. Measurement of chemicals
3. Preparation of bath
4. Immersion of bath to develop coating
5. Thorough characterization of mechanical and tribological properties.

Uncoated Sample

Substrate Material: Steel AISI-1025

Substrate Dimensions: (30 mm × 20 mm × 5 mm)

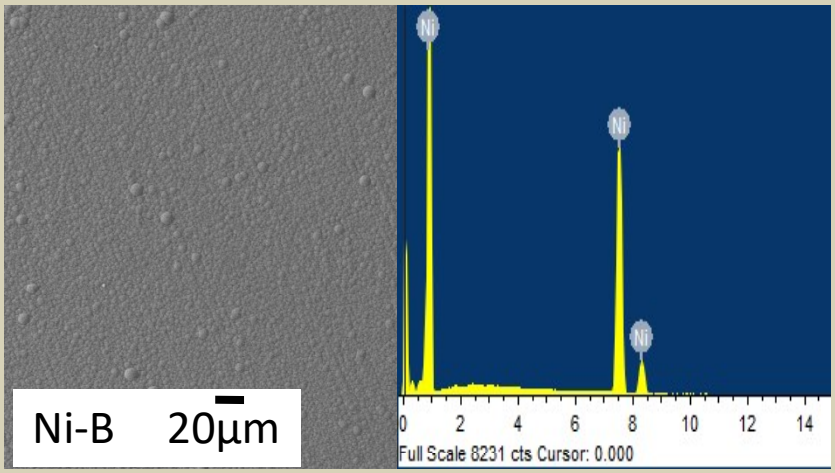
Coatings Fabricated

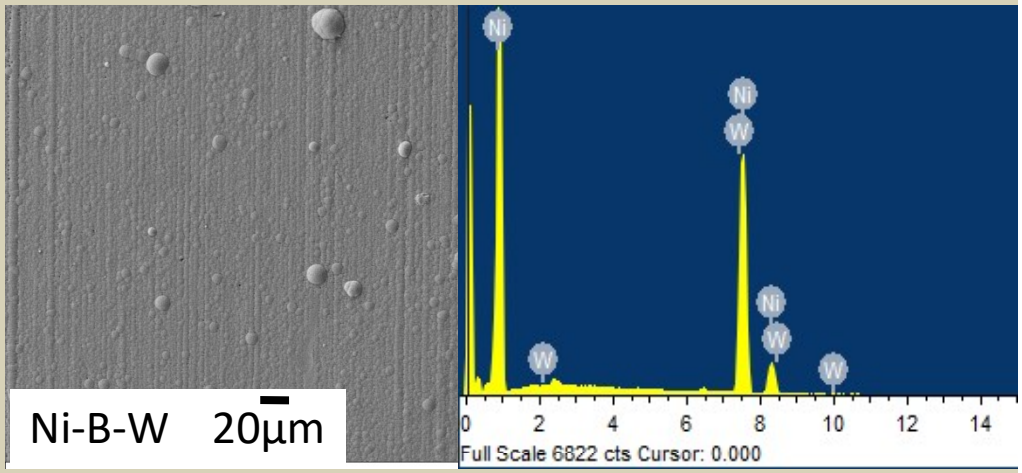
1. **Ni-B** (Alloy Coating)
2. **Ni-B-W** (Alloy Coating)
3. **Ni-B-W-hBN** (Composite Coating)

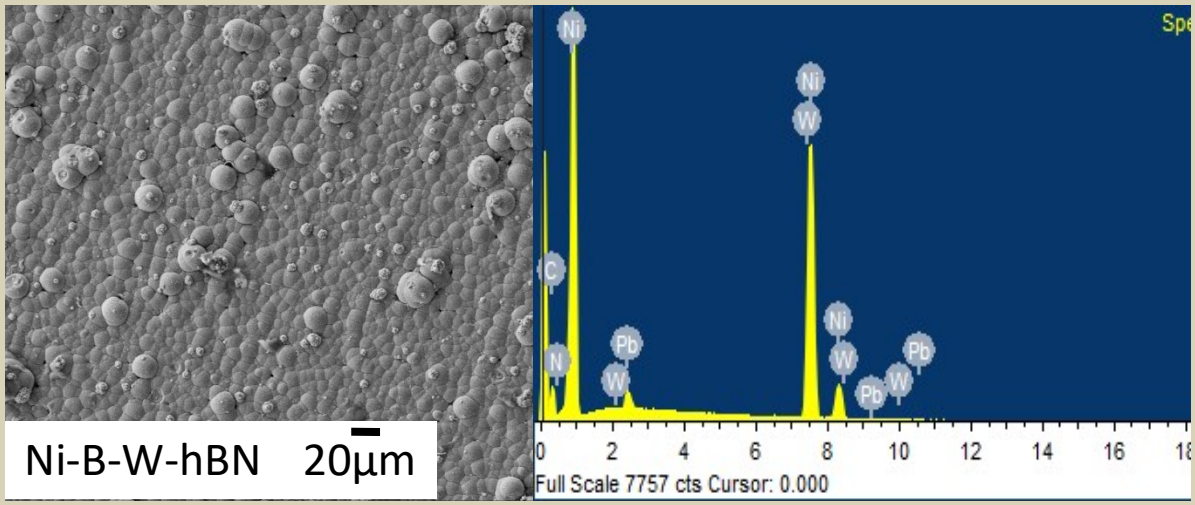
Constituents	Ni-B	Ni-B-W	Ni-B-W-hBN
Nickel Chloride	20 g/l	20 g/l	20 g/l
Sodium Hydroxide	40 g/l	40 g/l	40 g/l
Sodium Borohydride	0.8 g/l	0.8 g/l	0.8 g/l
Sodium Tungstate	–	25 g/l	25 g/l
Ethylenediamine	59 g/l	59 g/l	59 g/l
Lead Nitrate	0.0175 g/l	0.0175 g/l	0.0175 g/l
hBN particles	–	–	1 g/l
Plating Time	3h	3h	3h
Bath pH	13±2	13±2	13±2
Bath Temperature	90±2°C	90±2°C	90±2°C
Tween-20 Surfactant	–	–	0.045ml/l

Result and Discussion

Surface Morphology and EDS



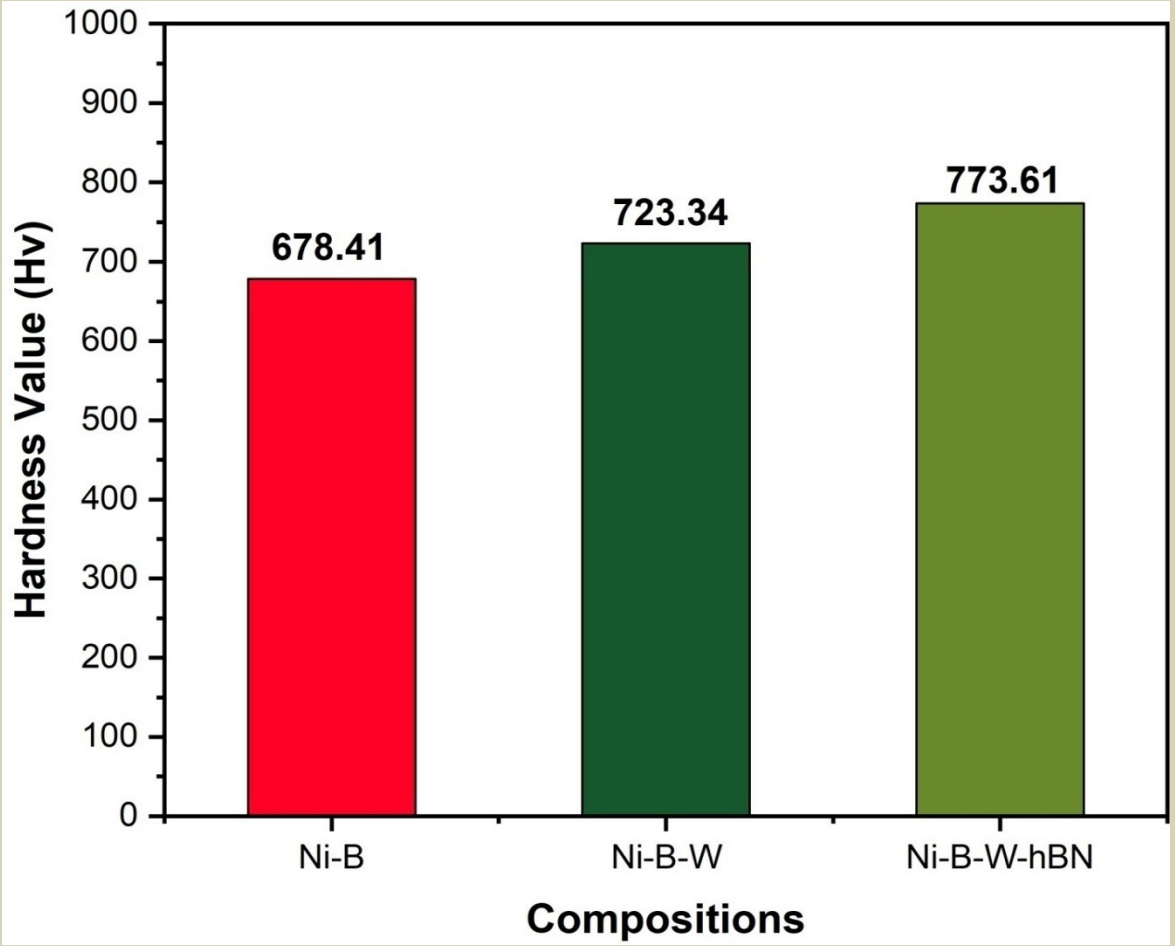




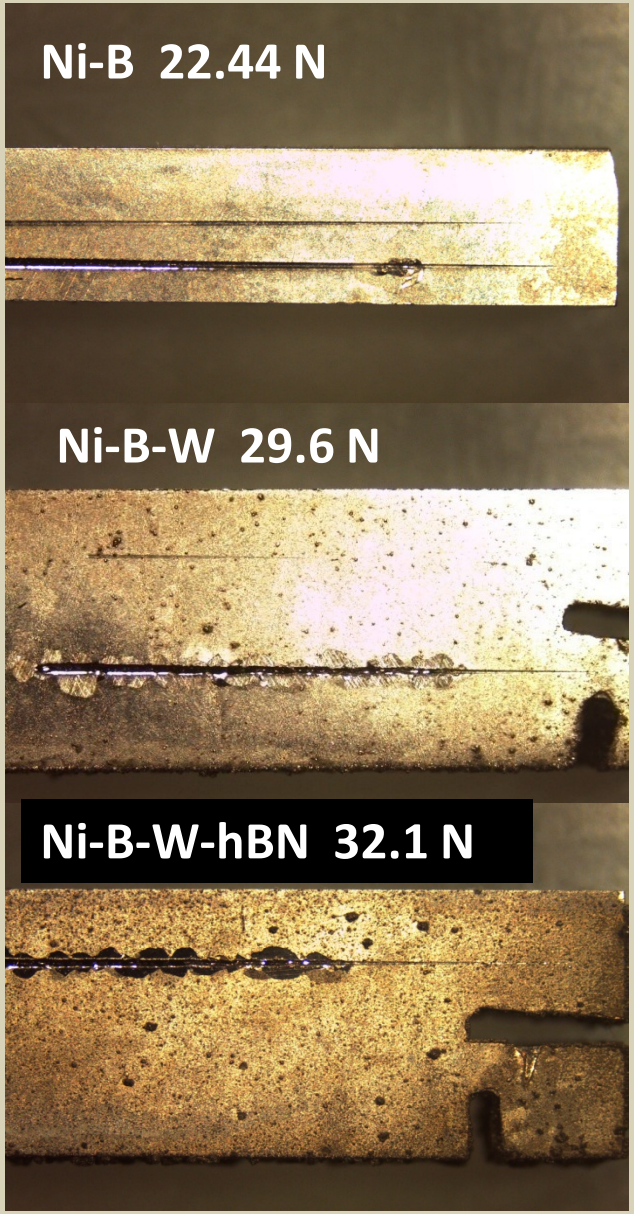
Micro Hardness Comparison

Parameters

Type Vickers hardness
Load 10g
Loading time 10s
Dual time 4s
Unloading time 10s



Scratch Test

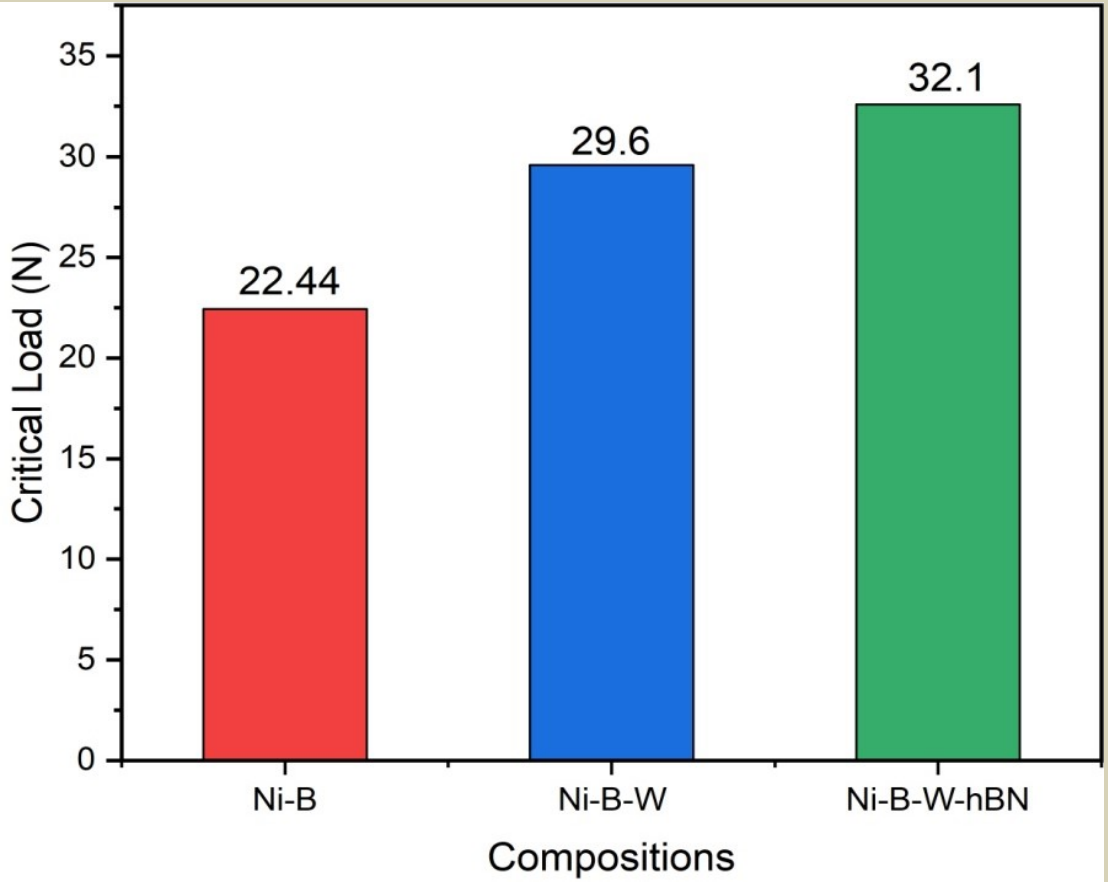


Critical Load Comparison

Parameters

Load 0 to 45N

Loading rate 3N/mm

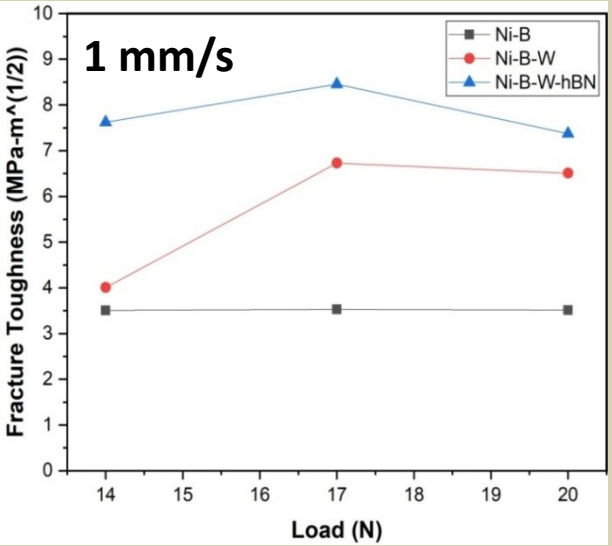


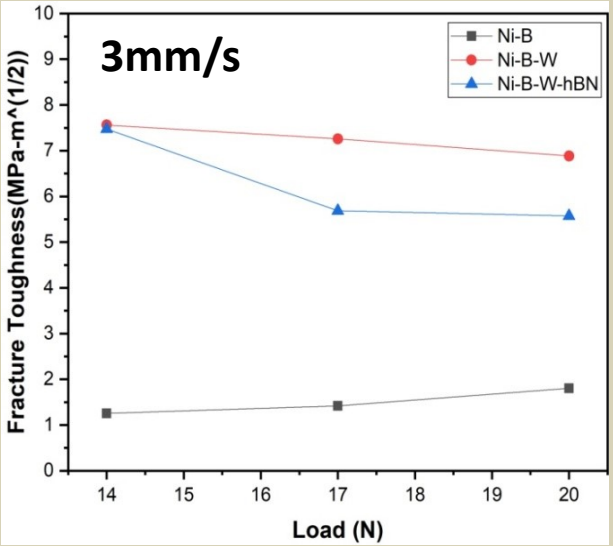
Scratch Hardness and Fracture Toughness

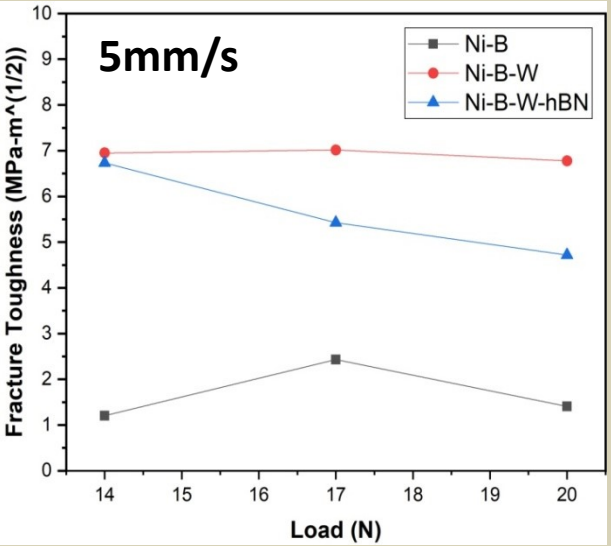
Input Parameter Variables

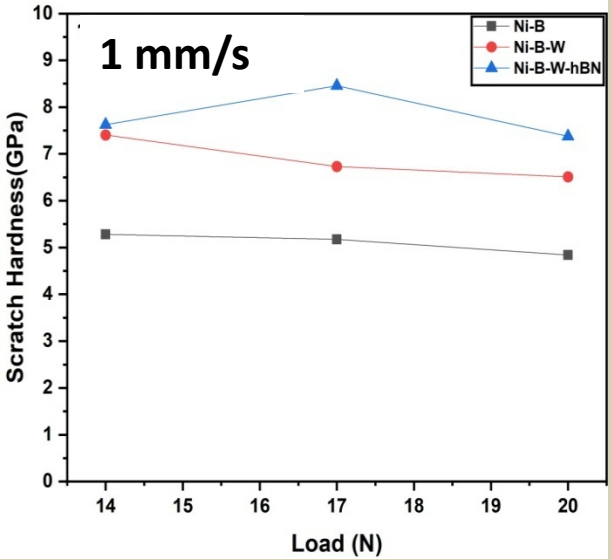
Compositions	Load	Scratch Speed
Ni-B	14N	1mm/s
Ni-B-W	17N	3mm/s
Ni-B-W-hBN	20N	5mm/s

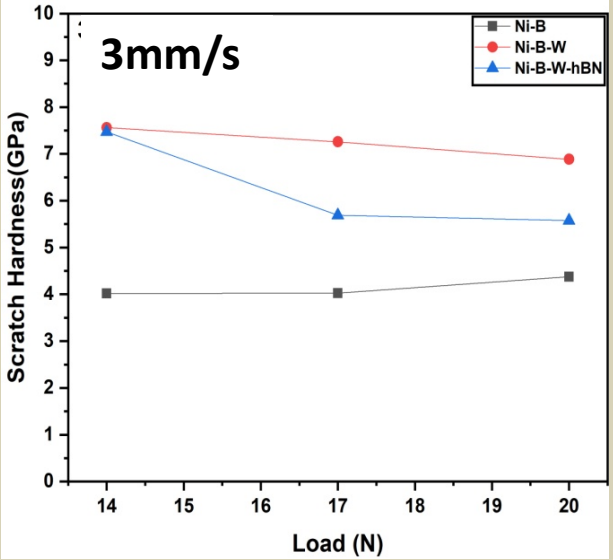
Scratch Hardness and Fracture Toughness Comparison

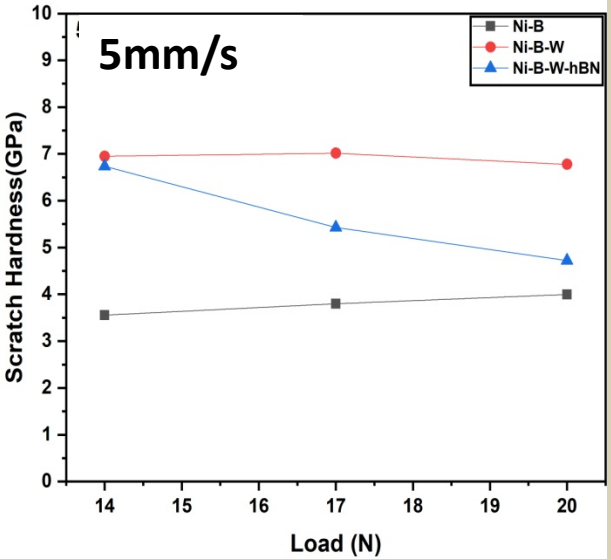












Conclusions

1. Addition of tungsten causes remarkable increment in Various mechanical and tribological properties such as hardness , critical load and fracture toughness of the coating.
2. And addition of hBN further improves the hardness , critical load and fracture toughness of the coating.

Future Plan

Now my future plan is to study the effectiveness of this composite coating, at higher temperature in two ways one by Ex-situ heat treatment and other by in-situ heat treatment testing and I will do comparison at both situation.

Phase 1	Phase 2	Phase 3	Phase 4
✓Literature Review	✓Coating Fabrication (incorporation of hBN)	Tribological and Mechanical Properties Testing and Analysis (Ex situ Heat Treatment)	Comparison And Conclusion
✓Substrate Preparation	✓Tribological and Mechanical Properties Testing Analysis (As it is)	Tribological and Mechanical Properties Testing and Analysis (In situ Heat Treatment)	

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Thank You

Engineering Application at High Temperature

Examples:

1. Metal Cutting.
2. Hot rolling.
3. Internal combustion engine etc.

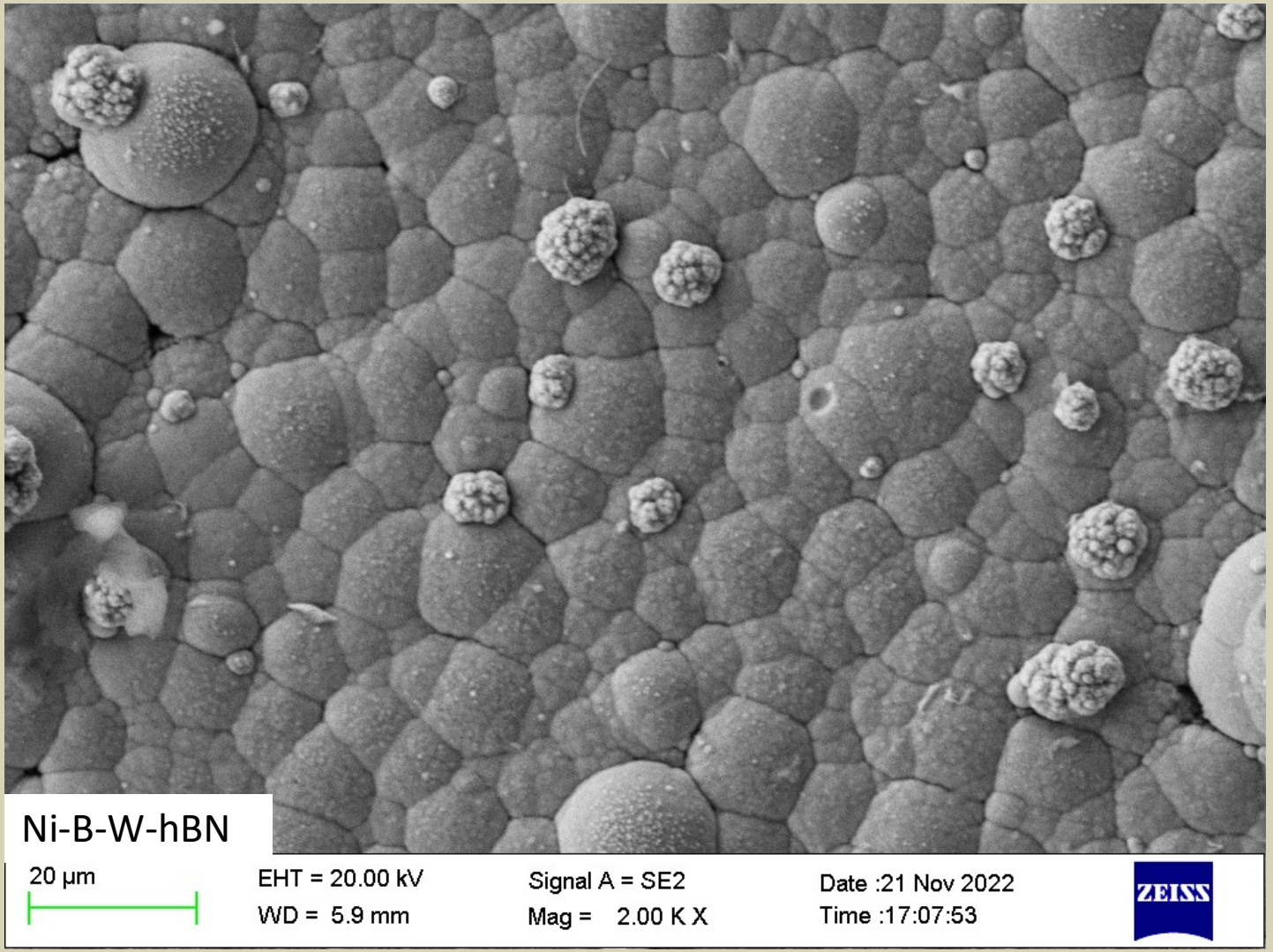
Application of *coatings*

Protection from wear/friction

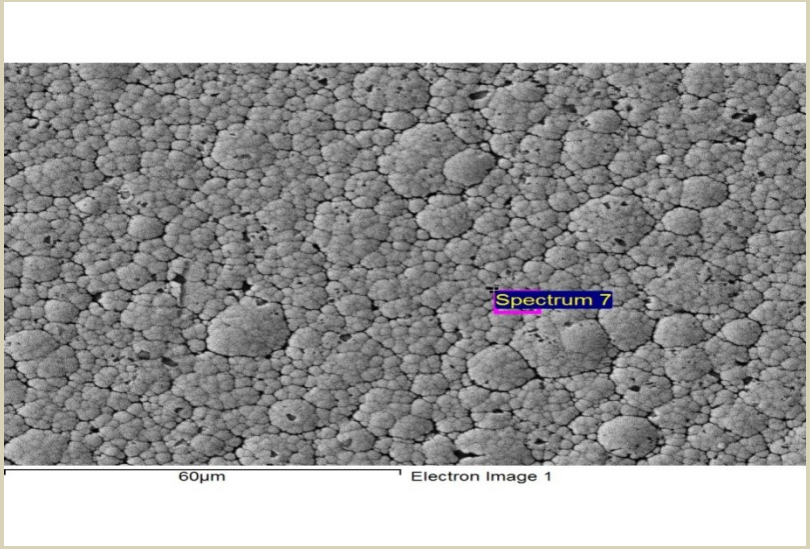
Protection from wear at higher temperature

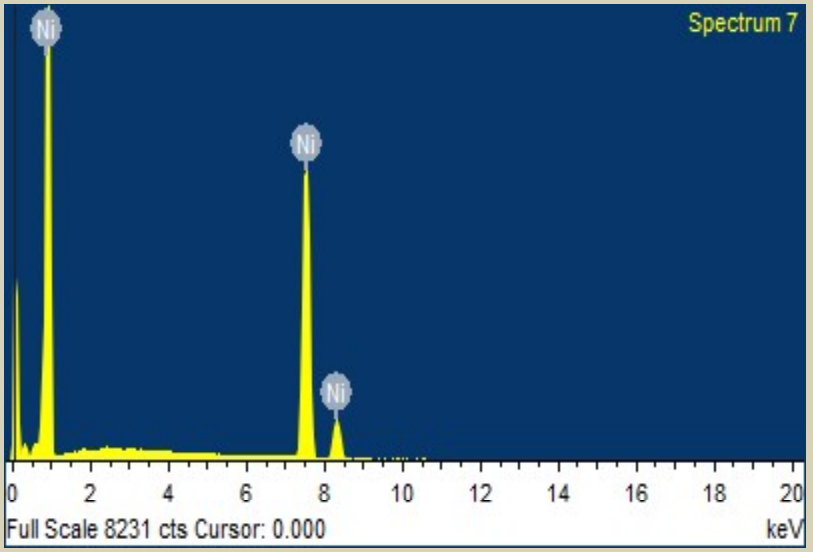
- *Thermal barrier coating* for rollers in hot rolling process
- *TiN coatings* on cutting tools in conventional machining

Protection from *Friction* at High Temperature


Ni-B-W-hBN
20 µm
EHT = 20.00 kV
WD = 5.9 mm
Signal A = SE2
Mag = 2.00 K X
Date :21 Nov 2022
Time :17:07:53
ZEISS

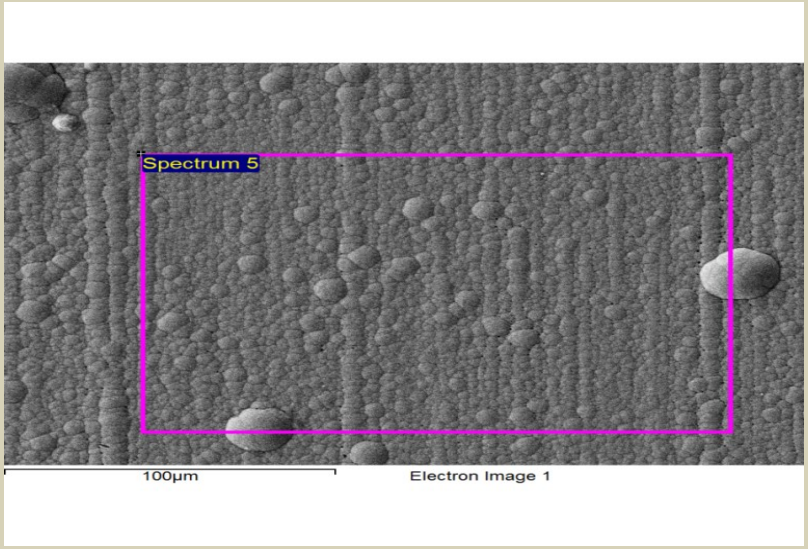
EDS Analysis : Ni-B

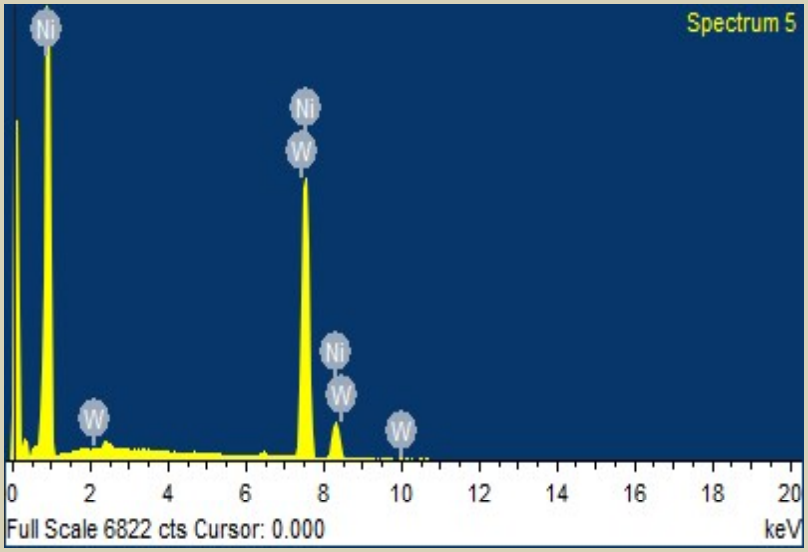




Element	Weight %	Atomic%
Ni K	100.00	100.00
Totals	100.00	

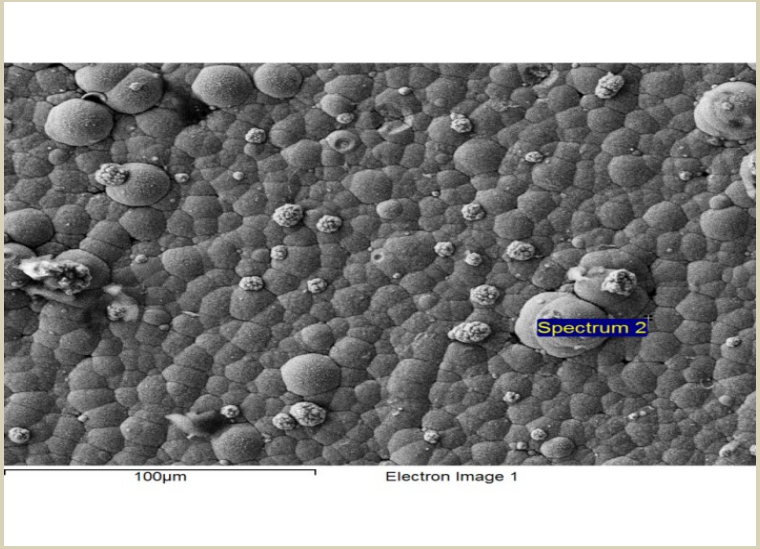
EDS Analysis : Ni-B-W

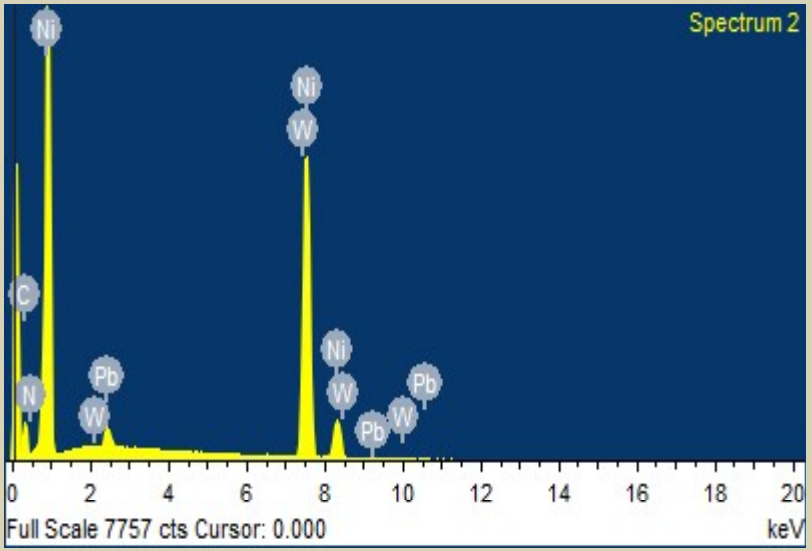




Element	Weight%	Atomic%
Ni K	99.39	99.80
W M	0.61	0.20
Totals	100.00	

EDS Analysis : Ni-B-W-hBN





Element	Weight%	Atomic%
C K	23.54	58.62
N K	2.16	4.61
Ni K	71.34	36.34
W M	0.13	0.02
Pb M	2.83	0.41
Total	100	

Possible Scratch Combinations

Sr.No	Compositions	Load(N)	Sratch Speed(mm/s)
1	Ni B	14	1
2	Ni B	17	1
3	Ni B	20	1
4	Ni B	14	3
5	Ni B	17	3
6	Ni B	20	3
7	Ni B	14	5
8	Ni B	17	5
9	Ni B	20	5
10	Ni-B-W	14	1
11	Ni-B-W	17	1
12	Ni-B-W	20	1
13	Ni-B-W	14	3

Possible Scratch Combinations

Contd from previous page…

13	Ni-B-W	14	3
14	Ni-B-W	17	3
15	Ni-B-W	20	3
16	Ni-B-W	14	5
17	Ni-B-W	17	5
18	Ni-B-W	20	5
19	Ni-B-W-hBN	14	1
20	Ni-B-W-hBN	17	1
21	Ni-B-W-hBN	20	1
22	Ni-B-W-hBN	14	3
23	Ni-B-W-hBN	17	3
24	Ni-B-W-hBN	20	3
25	Ni-B-W-hBN	14	5
26	Ni-B-W-hBN	17	5
27	Ni-B-W-hBN	20	5

Lubricants:

- Liquid Lubricants
- Solid Lubricants

Coatings for High Temperature Lubrication

Coatings	Method of coating	Lubricant Phases	Provide lubrication upto	Ref
CrN-Ag coating	Magnetron sputtering	Ag	600°C	[1]
CrN /Mo_2N multilayer	Magnetron sputtering	Due to oxidation of MoO_3	400-600°C	[2]
Yttria-stabilized zirconia (YSZ) nanocomposite coatings	Magnetron sputtering and pulsed laser hybrid mode	Ag	700°C	[3]
TiN Coating	Magnetron sputtering	Magneli phases of Ti oxides	700°C	[4]
VN coating	Magnetron sputtering	Magneli phases of vanadium oxides	700°C	[4]
In625-Cr_2O_3-Ag coatings	Cold spraying	Ag	1000°C	[5]

Contd…

Coatings for High Temperature Lubrication

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Coatings	Method of coating	Lubricant Phases	Provide lubrication upto	Ref
Cu-Mo coating	Ion beam deposited	Oxide phases $CuMoO_4$ and MoO_3	530°C	[6]
aromatic thermosetting polyester (ATSP) coating	electrostatic spray deposition (ESD)	PTFE	300°C	[7]
nano-Cu/h-BN/Ni60	Laser cladding	hBN	600	[8]

Working Temperature of Solid Lubricants

Solid Lubricants	Working Temperature Range	Ref.
Graphite	500°C	[9]
Molybdenum Disulfide (MoS_2)	300 °C	[10]
Tungsten Disulfide (WS_2)	540°C	[11]
Hexagonal Boron Nitride (hBN)	1000°C	[12]

Decomposition of solid lubricants

Application of hBN in Tribological Coatings

Coating	Solid Lubricants	Process of Coating Fabrication	Ref.
Nano -Cu/h-BN/Ni60	hBN	Laser Cladding	[13]
TiB2–TiN–SiC-hBN		Laser Assisted Processing	[14]
Ni-B- hBN		Electro Deposition	[15]
Ni-P- hBN		Electroless Depositon	[16]

Popular Electroless Tribological Coatings

Ni [19], Ni-P [20], Ni-B [21] etc.

Electroless Ni-B based coatings are superior in terms of:

- Hardness
- Wear resistance [21]

Strengthening by Alloy and Composite Formation

- Ni-B [21]
- Ni-B-W [22]
- Ni-B-Mo [23]
- Ni-B-Co [24] etc.

- Ni-B-W [22]
- Ni-B-SiC [25]
- Ni-B-W-SiC [26]
- Ni-B-Al_2O_3 [27] etc.

Ni-B-hBN **Ni-P-hBN**

High temperature tribological characterization of Electroless Ni-B-hBN

Ni-B-hBN

Ni-B-W-hBN

Now I am in position to judge the effectiveness of this composite coating, at higher temperature in two ways one by Ex-situ heat treatment and other by in-situ heat treatment testing and I will do comparison at both situation.

Constituents	Concentration
Nickel Chloride (Source of nickel)	20 g/l
Sodium Hydroxide (Buffering Agent)	40 g/l
Sodium Borohydride (Reducing Agent)	0.8 g/l
Sodium Tungstate (source of W)	25 g/l
Ethylenediamine (Complexing Agent)	59 g/l
Lead Nitrate (Stablizer)	0.0175 g/l
hBN particles	1 g/l

Component of Bath	Function
Metal ions	Source of metal
Reducing agents	Supply electrons to reduce the metal ions
Complexing agents	Prevent excess of free metal ions concentration
Accelerators	Accelerate the reducing agent and increase the deposition
Stabilizers	Stabilize the bath from decomposition
Buffers	Sustain the pH for long time
pH regulators	pH adjustment
Temperature	Energy for deposition
Surfactant	Lower the surface tension of a liquid and promotes the coating deposition reaction