Technical Meeting on
Emerging applications of Plasma Science and Technology

Plasma diffusion treatments and coatings for industrial applications

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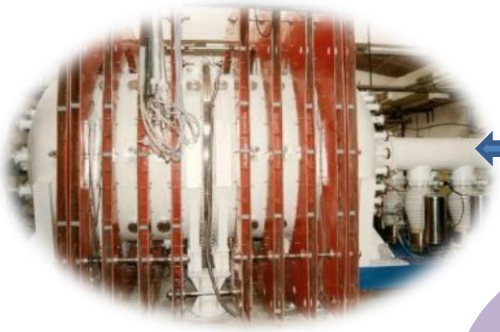
प्लाज़्मा अनुसंधान संस्थान
Institute for **Plasma Research**

ABOUT
INSTITUTE FOR PLASMA RESEARCH (IPR)

ABOUT IPR



Tokamak Systems (Aditya, SST1)

Basic Plasma Sciences

Fusion Technologies

Societal & Industrial Technologies

Robotics & Remote Handling

ITER India (EB)

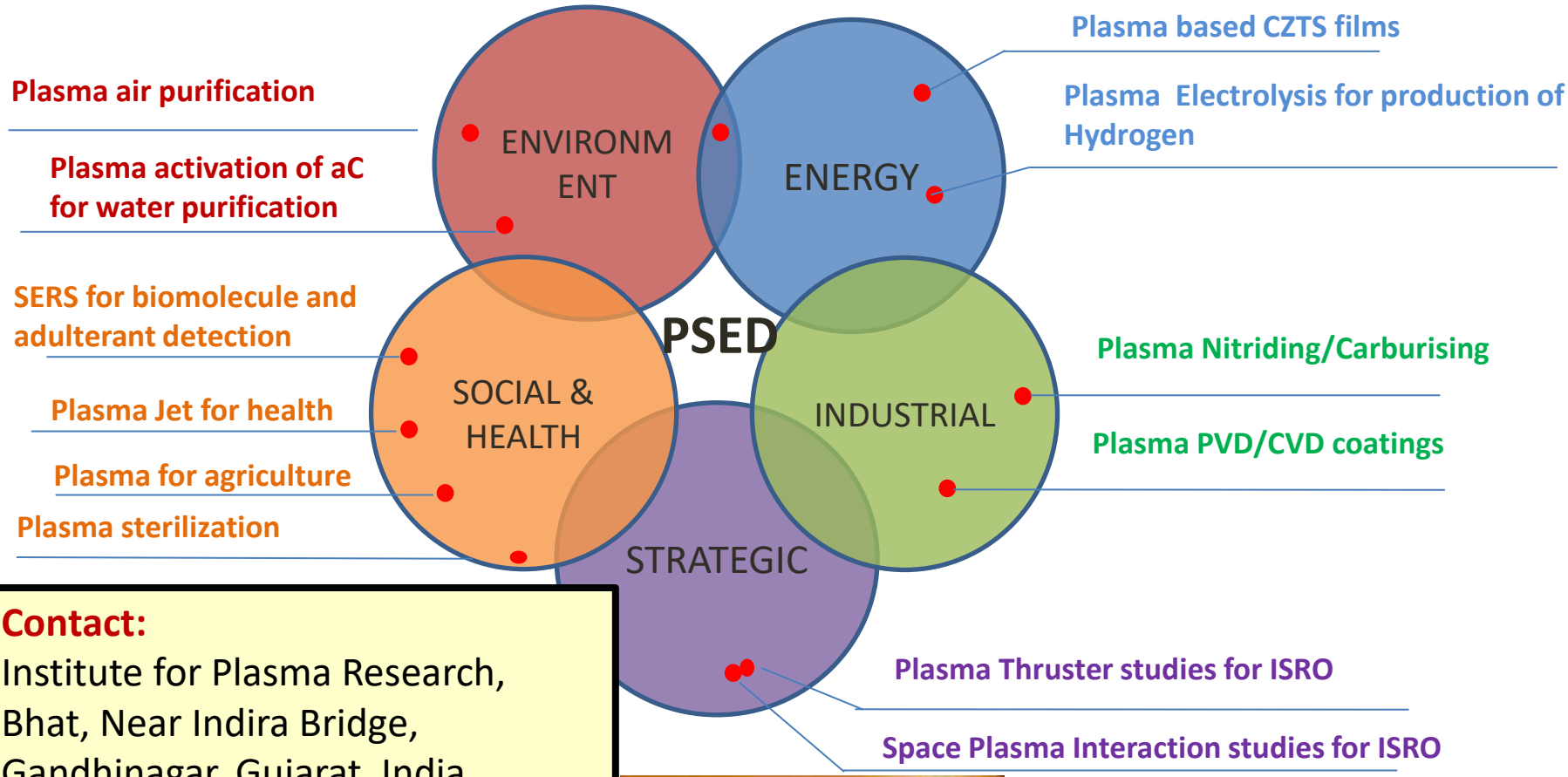
Simulation & Theory

LIGO Activities

Data Acquisition & Control; Diagnostics

Power Supply Technologies

PLASMA SURFACE ENGINEERING DIVISION @ IPR



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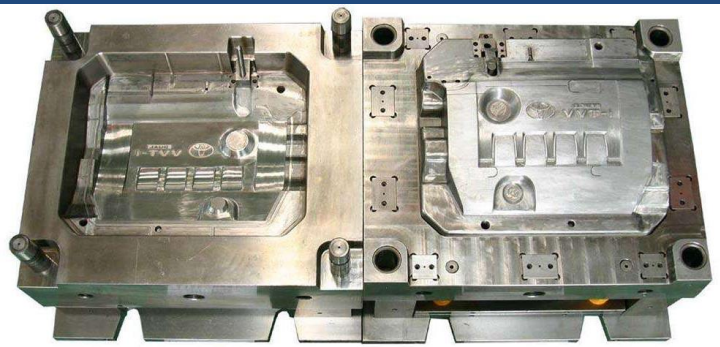
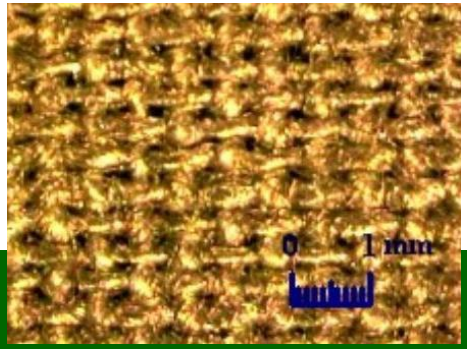
OUTLINE

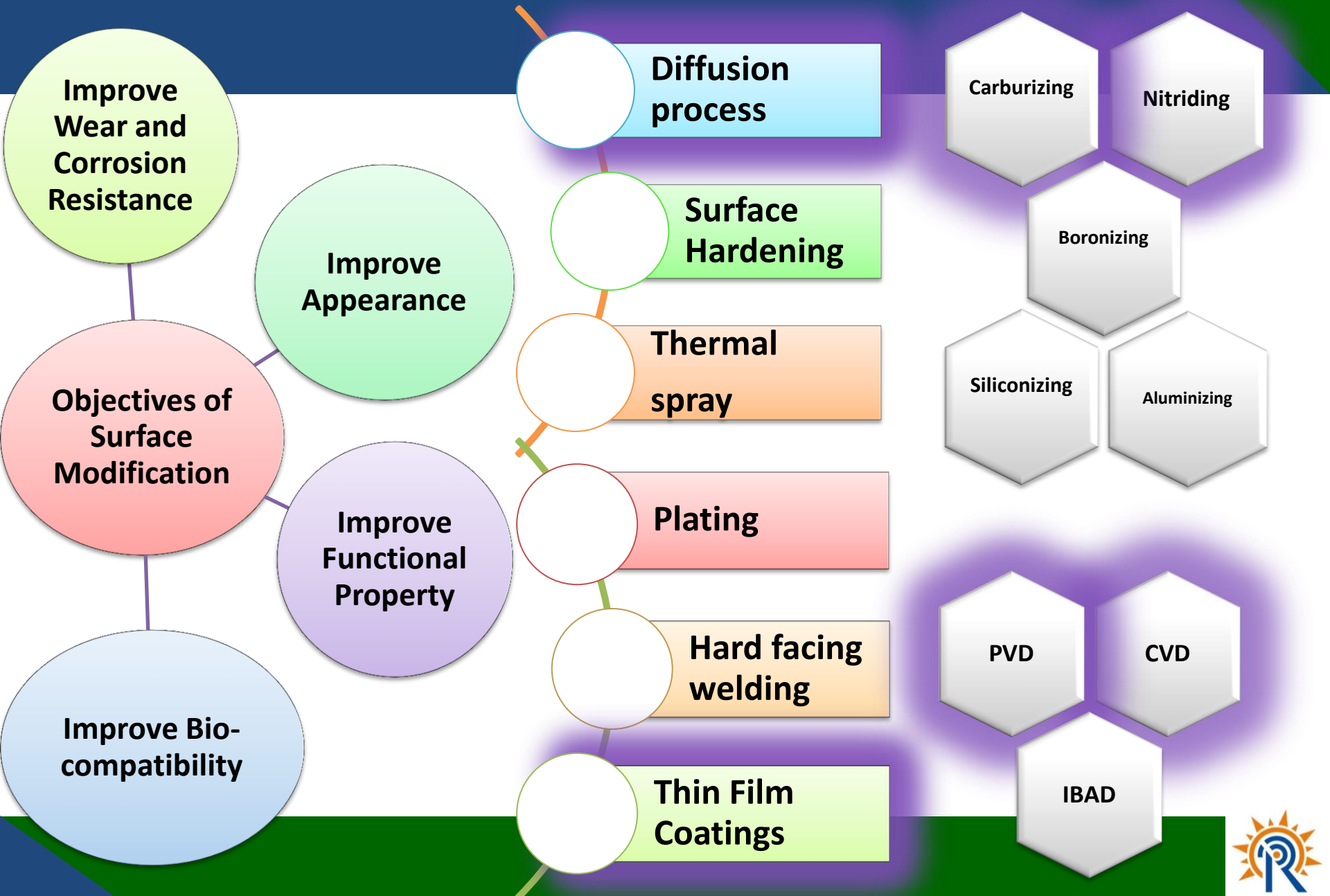
- BACKGROUND
- INTRODUCTION
- DIFFUSION BASED PROCESSES CASE STUDIES
  - Plasma Nitriding / Plasma Nitrocarburizing
  - Plasma Carburizing/ Plasma Carbonitriding
- DEPOSITION PROCESSES
  - Plasma Assisted PVD
  - Plasma Assisted CVD
- Conclusion

Industrial Requirement





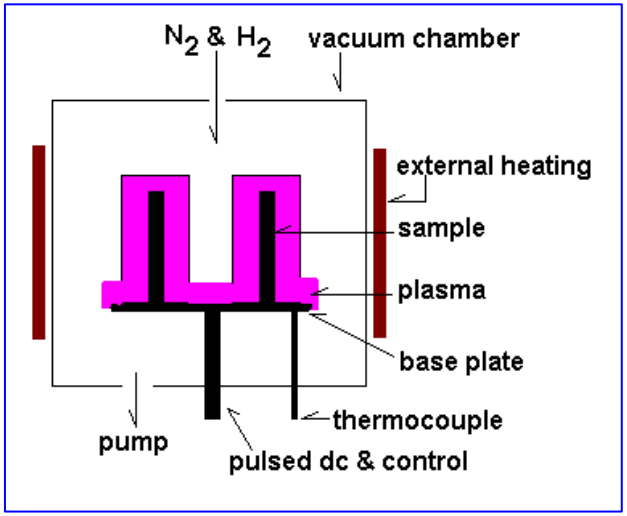
Surface Modification



About Plasma Nitriding

- **Plasma Nitriding –** diffuse nitrogen in the subsurface of steel components (few 100 microns)
- Use high temp (~ 550 C)
- Uniform abnormal glow discharge plasma environment
- Glow created by high negative bias (-600 V)
- p ~ few mbar
- Typical duration: 24 hrs
- **Enhancement of**
  - Surface hardness
  - Wear resistance
  - Corrosion resistance

  Thereby increases component life

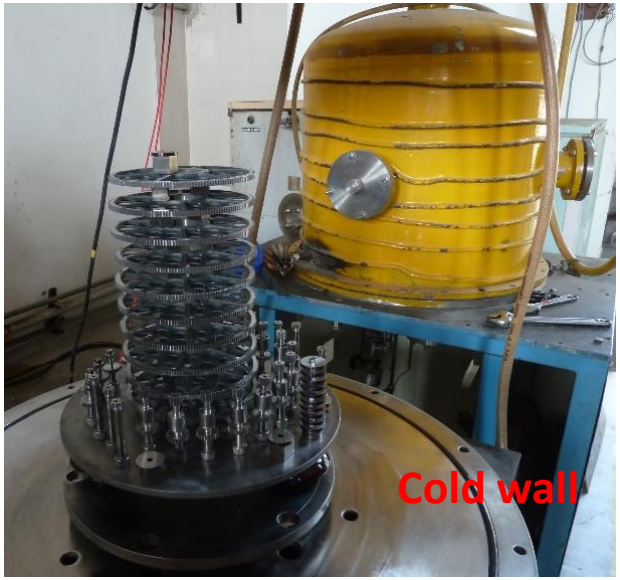


Process Parameters	Typical Range
Voltage	- 400-650 V
Current Density	1-5 mA/cm^2
Frequency	10-30 KHz
Temperature	400-570 ^oC
Pressure	3-5mbar

Plasma glow around components; Note the uniform plasma covering the component

NO HARMFUL EMISSION OF CHEMICALS

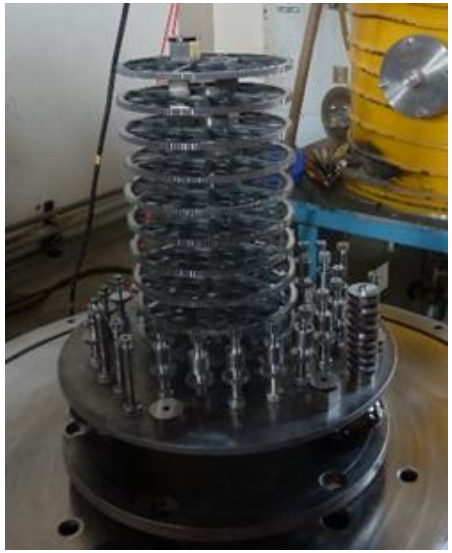
PN Systems @ IPR



- Different configurations (4)
- Scaled up
- Transferred Technologies (4)
- Installations (11)

PN for Space Sector

Gears and Pinion used in DM



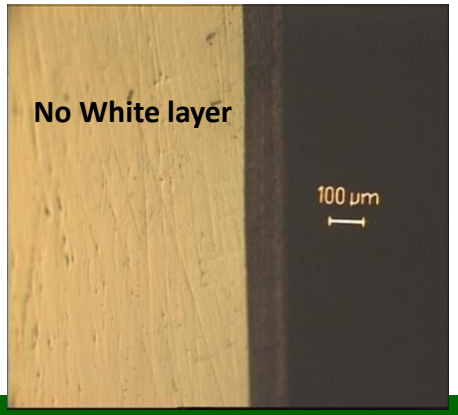
Objective:

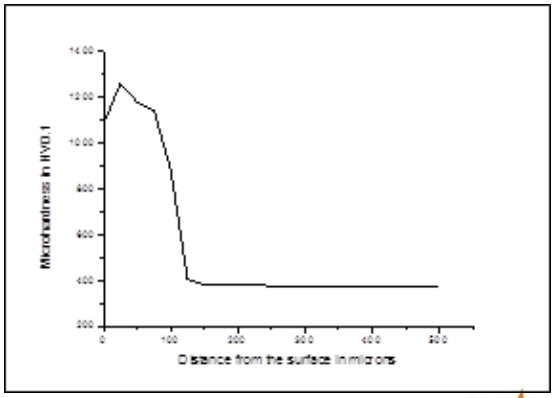
Drive Assembly (DA) is a key component of space crafts sustains and rotate the solar arrays for sunlight acquisition, as well as transfer power and signals from solar array to spacecraft body.

Plasma nitrided gears and pinions (17-4PH material)

Specification	Requirement
Material	17-4PH SS
Surface Hardness	>800HV
Case Depth	100 microns
White layer	Absent

Plasma nitriding of gears and pinions for INSAT satellite

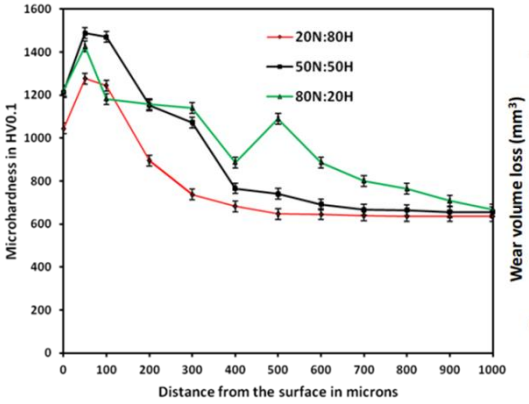


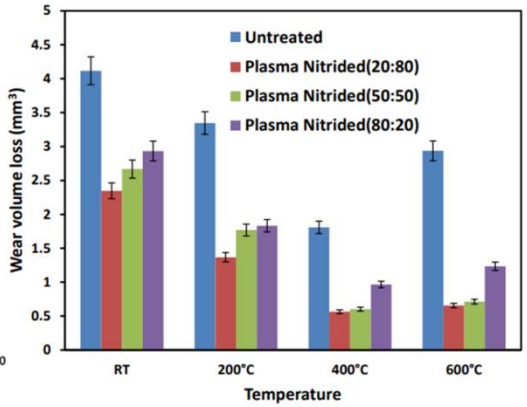


Plasma sciences and the creation of wealth, PI John, 2005, Tata McGraw-Hill Pub. Co.

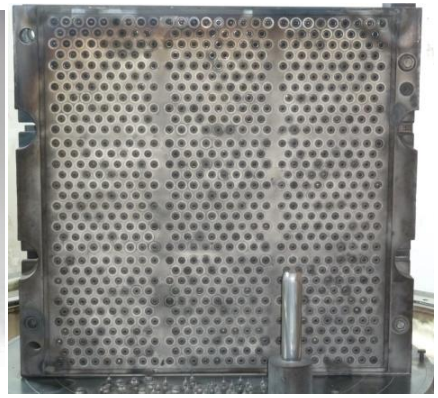
PN of Dies

Dies





Hardness and wear loss of AISI H13 material



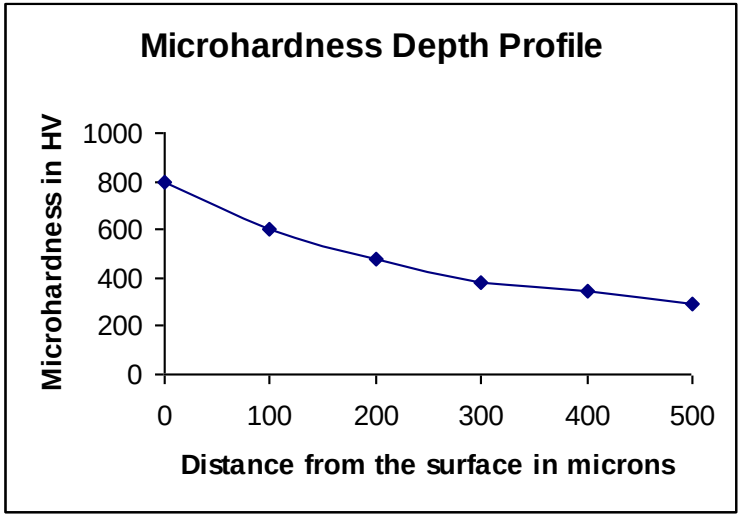
Plasma nitrided die for manufacturing bottle top lid (D2 material), extrusion dies (H13) , Wash basin (Inconel 600)

Specification	Requirement
Material	D2, H13, Inconel 600
Surface Hardness	>1000HV
Case Depth	300 microns
White layer	Few microns

A. Kumar, et al. , **High-temperature tribological** studies of plasma-nitrided tool steels, Surface Engineering, 2017, 1-14

Plasma Nitriding for Automobile and machine tools sector

Specification	Requirement
Material	AISI 4140, En41B
Surface Hardness	>800 HV, >1000HV
Case Depth	300 microns



A WIDE VARIETY OF AUTOCOMPONENTS ARE ROUTINELY PLASMA NITRIDED IN THE INDUSTRY

Mukherjee S., **Industrial Applications of Plasma Nitriding**, Transactions of the Indian Institute of Metals. Vol. 58, no. 5. Oct. 2005.

PN of CTC roller in Tea Industry

Specification	Requirement
Material	AISI 302
Surface Hardness	>800 HV,
Case Depth	20 microns

Life increased by a factor of 2

Plasma Nitriding System Installations

Gujarat

Ludhiana

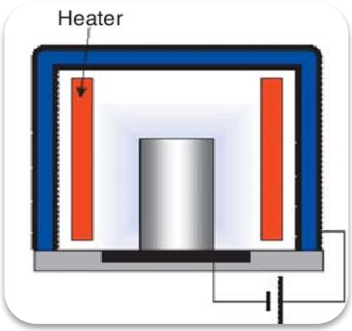
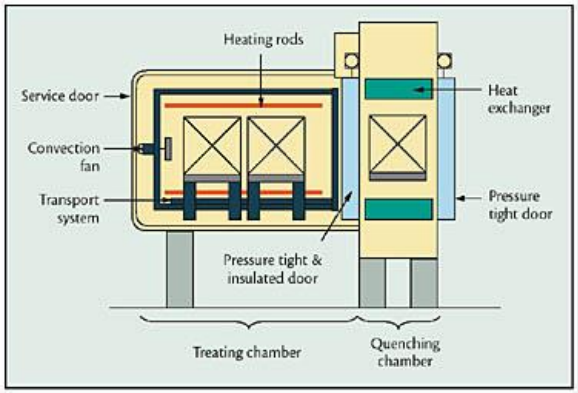
Kerala


Chennai

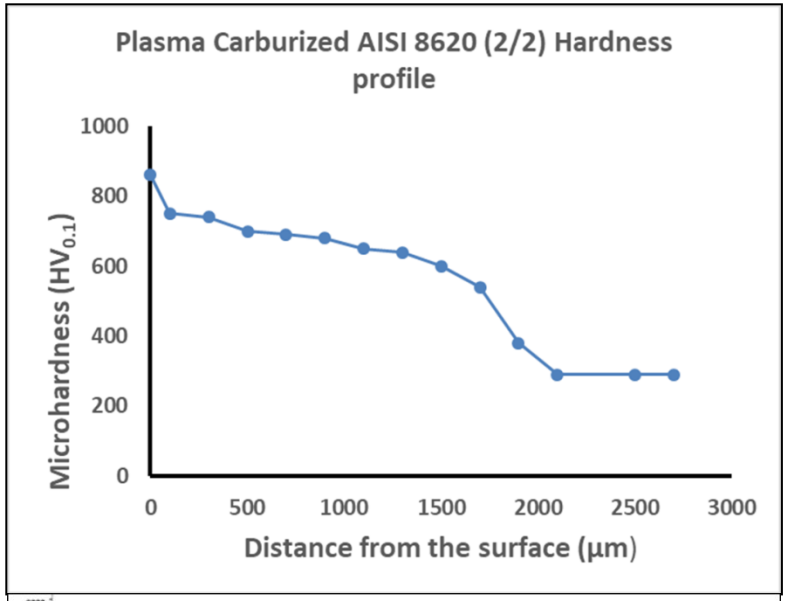

Nagaland

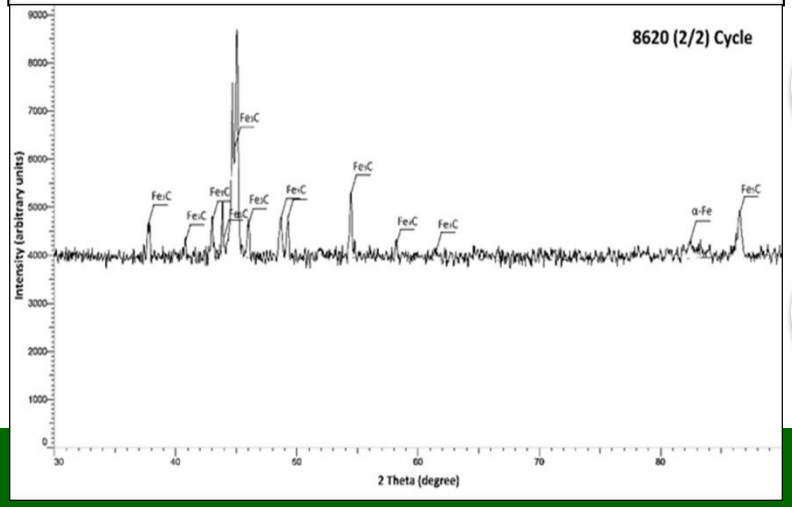

Kerala

Plasma Nitriding and Carburizing (PC)

Description	Plasma Nitriding / Nitrocarburizing	Plasma Carburizing / Carbonitriding
Atomic size of interstitial	Nitrogen : 0.075 nm	Carbon: 0.077 nm
Schematic		
Environment	Nitrogen, hydrogen, hydro carbon gas mixture	Carbon, nitrogen, hydrocarbon gas mixture
Operation temperature	400-550 °C max	> 930 °C
Operating pressure	3-5 mbar	5-10 mbar
Types of steel	Low and High alloyed steels	Low carbon steels
Application	Gears, crankshafts, camshafts, cam followers, valve parts, extruder screws, pressure-die-casting tools, forging dies, cold forming tools, injectors and plastic-mould tools, long shafts, axis, clutch and engine parts	Bearings, gears and pinion shafts

Plasma carburizing @ IPR





Different configurations (2)

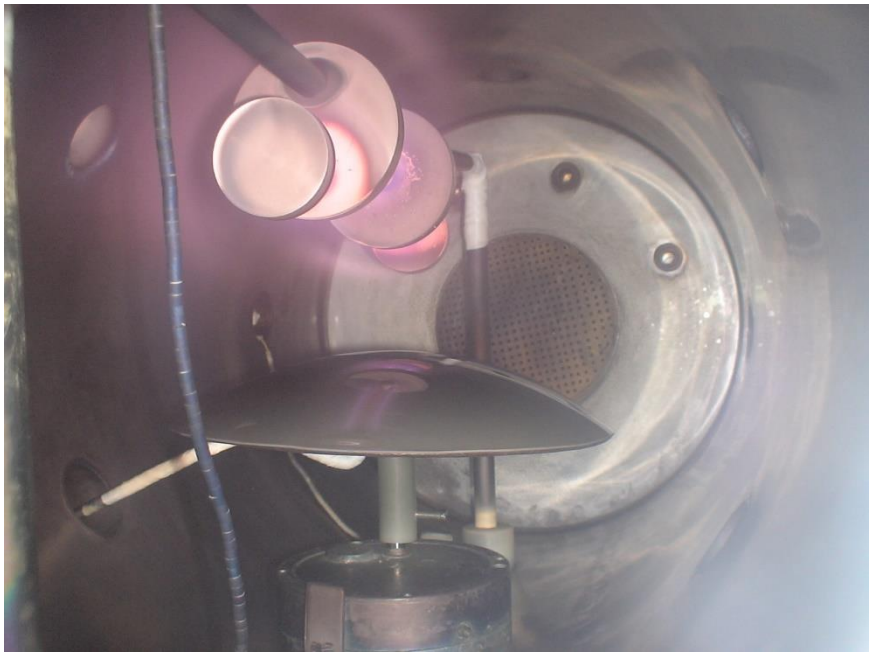
Scaled up

Plasma Assisted Physical Vapour Deposition (PAPVD)

PVD system in IPR

- Different <u>control parameters</u> to develop special coatings.
- Ability to coat <u>heat sensitive</u> substrates.
- Circular, Rectangular or cylindrical Magnetrons available.
- <u>Up scaling</u> for large area coatings is possible

PAPVD coating for space applications



Physical Vapour Dep.
Sputter = Copper
P = 10 microbar
Gas = Argon
Sputter bias = -800 V
Substrate bias = pulsed – 1 kV

Lexan **Cu coating**

Copper coating

Thickness : 8 micron

Application : Low weight antenna for Microwave reflectivity.

Need of Surface Engineering for Implant material

Performance of Implantable device :

- **Bulk material (mechanical properties)**
- **Surface properties of used biomaterial (wear and biocompatibility)**

Metals are prone to corrosion and wear

wear of the articulating components – primary reason for failure.

Released Cr and Ni ions from the SS or Co-Cr metal can cause allergic reactions

Ceramics are prone to crack under heavy load even though wear rate is less

One of the approach to improve surface characteristics is

Deposition of biocompatible, wear resistant and corrosion resistant coatings

Coating materials : Titanium Nitride (TiN), TiNbN,

Diamond like coating (DLC) etc.

TiN coating for Hip Implant

Titanium Nitride (TiN) is interested choice

- high chemical stability
- high hardness
- excellent wear properties
- Good bio-Compatibility
- Good wettability by body fluids
- Approved by FDA

TiN can be deposited on Stainless Steel (SS) Titanium Alloy or Co-Cr alloys

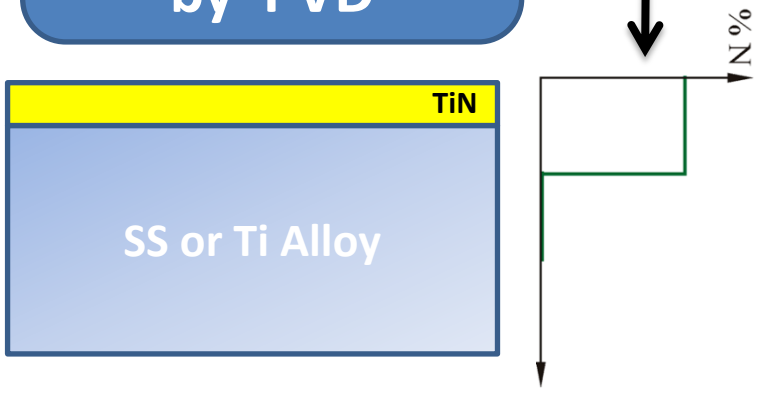
Adhesion of the coating (TiN) with the Substrate (SS) is important factor for long term Performance of the implant.

Different PVD methods are used to produce these ceramic coatings on metal substrate.

Plasma based PVD method is used to deposit these coatings on Implant

Our Approach for Surface Modification

Conventional TiN deposition by PVD



Scope for Improvement :

1) Improving Corrosion resistance of TiN
2) Improving Adhesion of hard TiN layer with soft metal

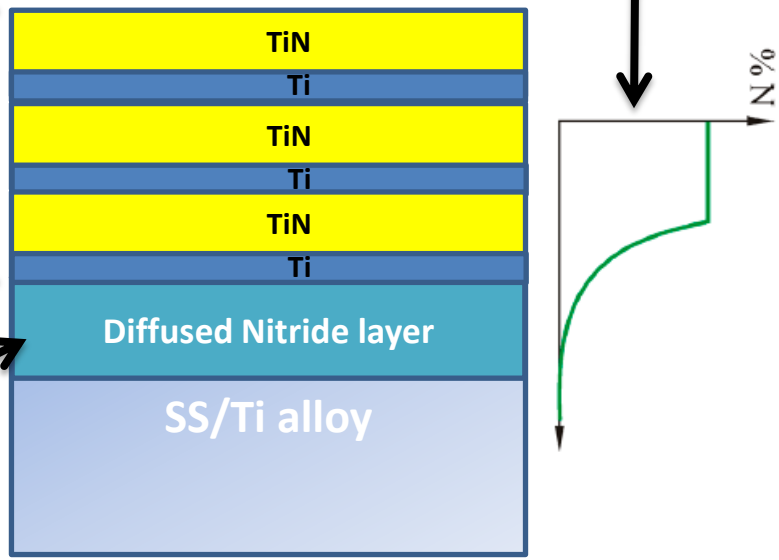
Our Approach for TiN deposition by PVD

Multi-layering Improves Corrosion resistance.
Plasma nitriding + multi-laying Improves adhesion

Diffusion + overlay coating

Ti-TiN Multilayer above Surface by sputtering

Hard layer in the surface by Plasma nitriding

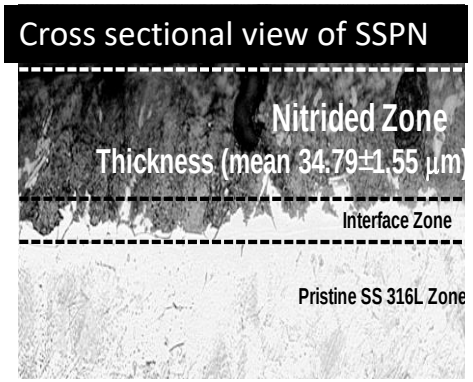


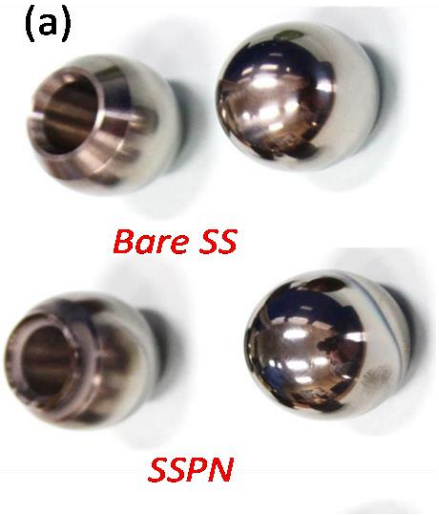
Plasma Nitriding of SS femoral head

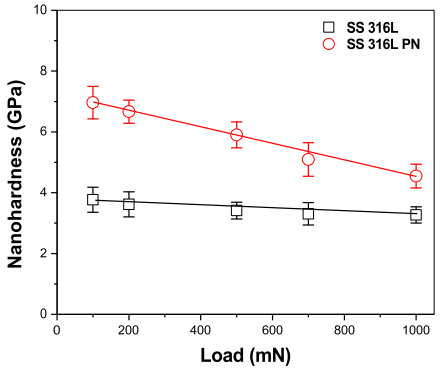
SSPN : Plasma nitrided Steel

Material : AISI 316L
Temperature : 350 °C- 550 °C
Pressure : 5 mbar (80% (H_2) +20%(N_2))
Pulse Voltage : - 550V
Discharge Current : 1A
Duty cycle : 63%
Treatment time : 4-8 hrs.

Glow around the sample



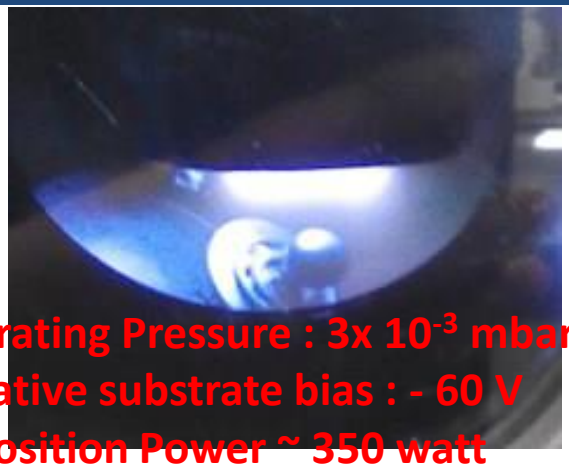




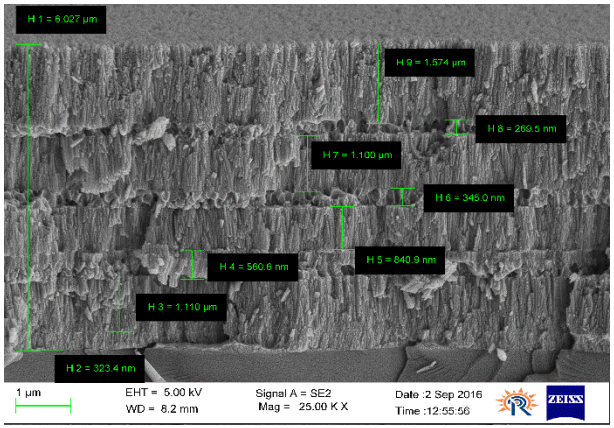
Hardness at different load

- **Uniform Treatment all over the surface**
- **Hardness increases after plasma nitriding**
- **hardening up to few micron inside the sample**

Ti/TiN Coating : Hip Implant SS femoral head

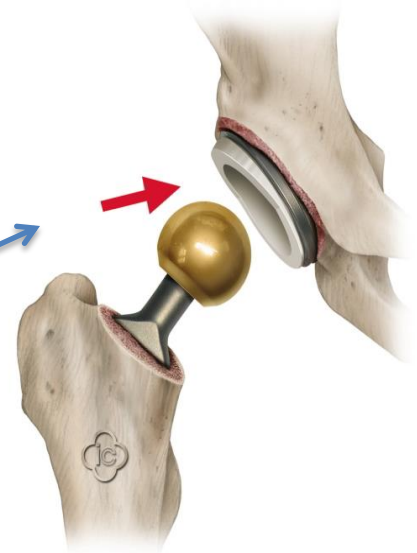


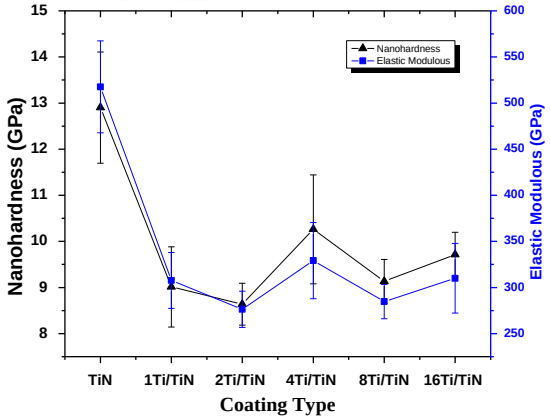
Operating Pressure : 3x 10^{-3} mbar
Negative substrate bias : - 60 V
Deposition Power ~ 350 watt
Deposition Time : 60 minute



Thickness : 6 micron (8 Layers, 4- Ti and 4- TiN layers

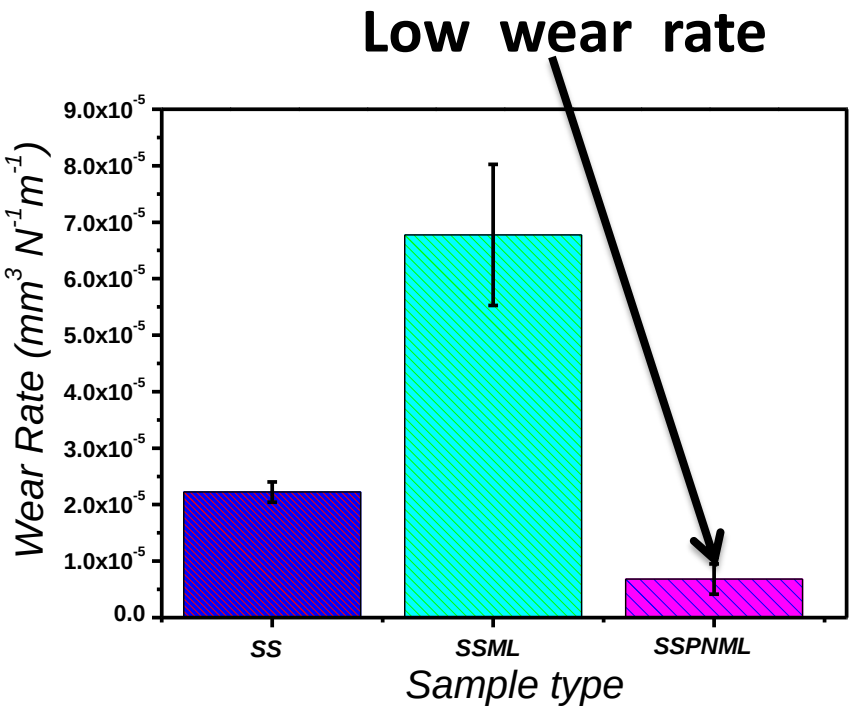
Hardness : 10-12 GPa





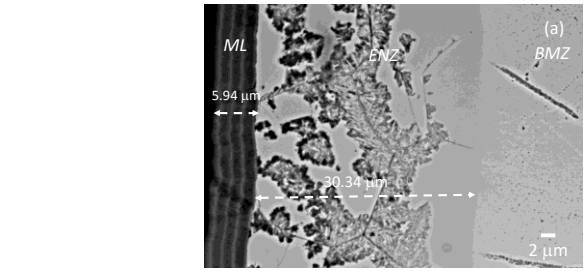
A. Samata et al. Journal of the Mechanical Behavior of Biomedical Materials 77 (2017) 267-294.

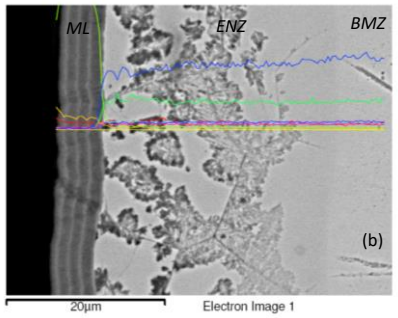
Nitriding + Coating : Duplex Surface Engineering

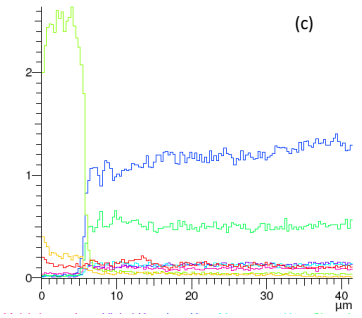


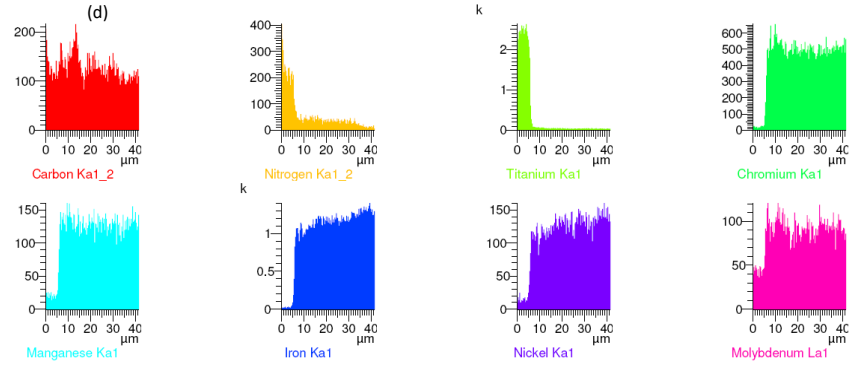
Wear rate in Simulated Body Fluid (SBF)

SS : Without any Plasma treatment
SSML : Multilayer coating on SS
SSPNML : Nitriding + Multilayer coatings





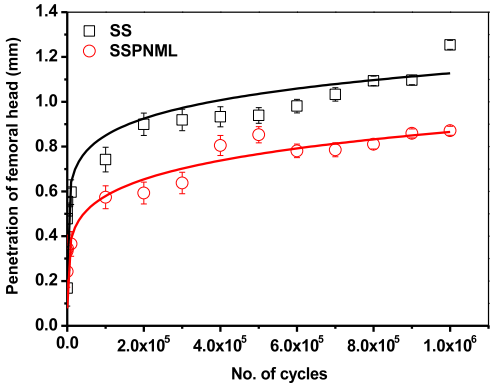


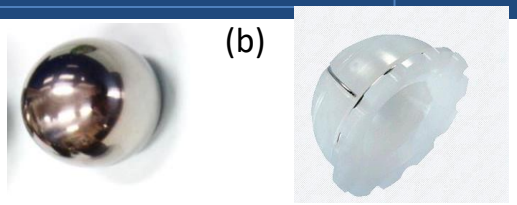


Aniruddha et al. Applied Surface Science 507:145009.

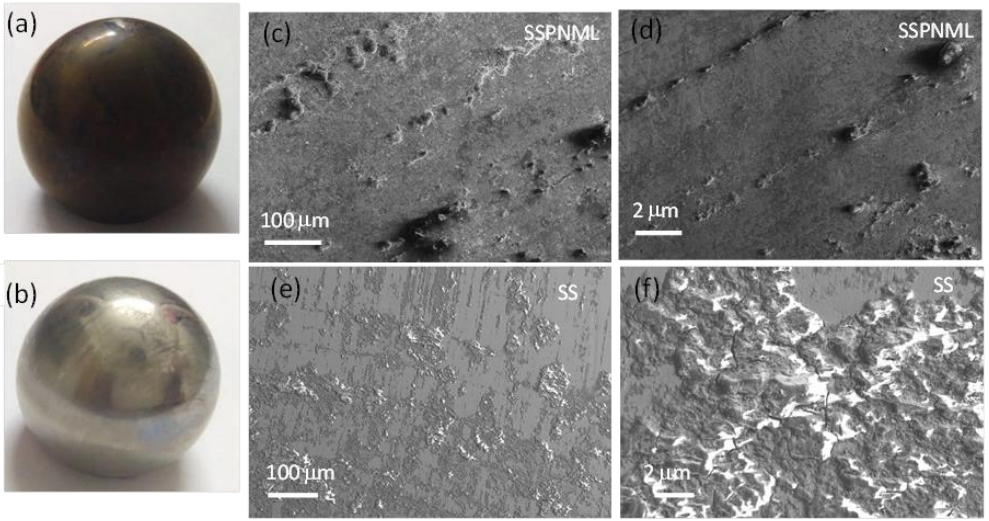
Preclinical Evaluation of wear using Hip-Joint Simulator machine

Frequency of load cycle : 1 Hz
Testing up to 10^6 cycles
Fluid : SBF
Load : identical to that experienced by the normal hip of a person with a body weight of about 100 kg.





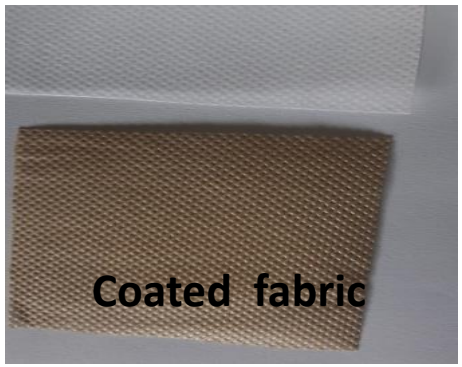
Volumetric wear rate of UHMWPE cup For SS : 120-130 mm^3/million cycle For SSPNML : 40-50 mm^3/million cycle

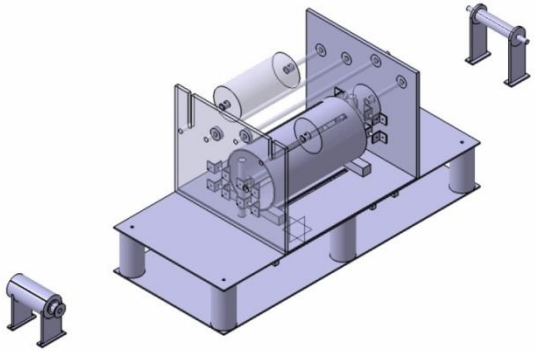


Antimicrobial coatings on yarn and fabric

Major Achievements

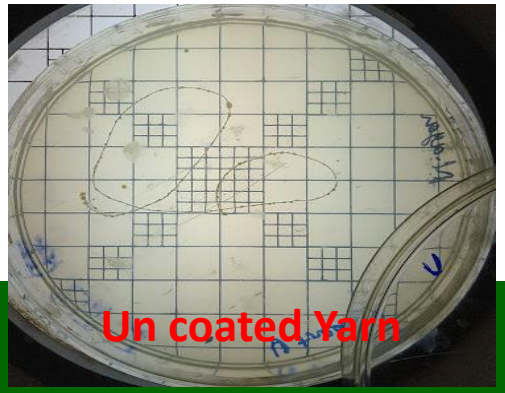
- Cylindrical magnetron sputtering for copper oxide Coating.
- Antibacterial/antiviral testing done
- Uniform coating on yarn
- Existing Speed of the coating for yarn : 5 mtr/min

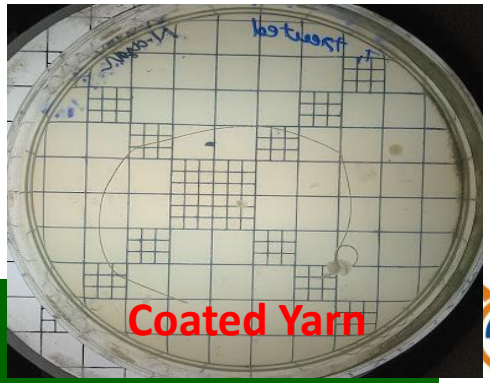




Schematic of Cylindrical magnetron sputtering set up

- Deposit CuO coating in nanometer range (50-100nm) on moving yarn and fabric (woven/non-woven).
- Indian Patent application number: 202321003767 (2022)

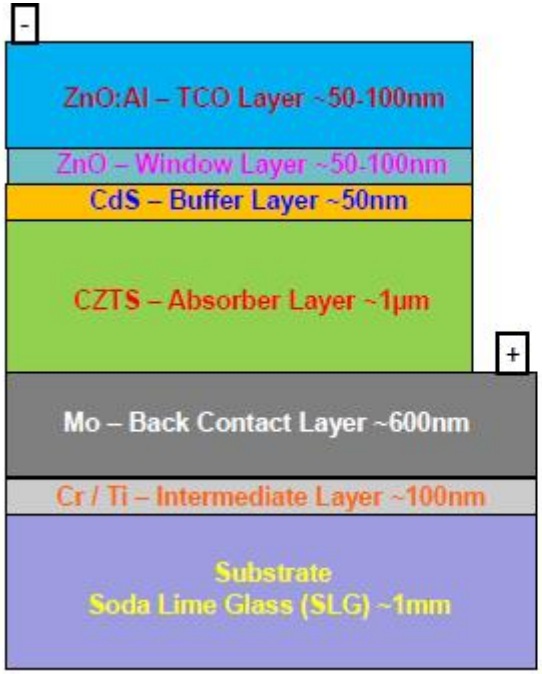


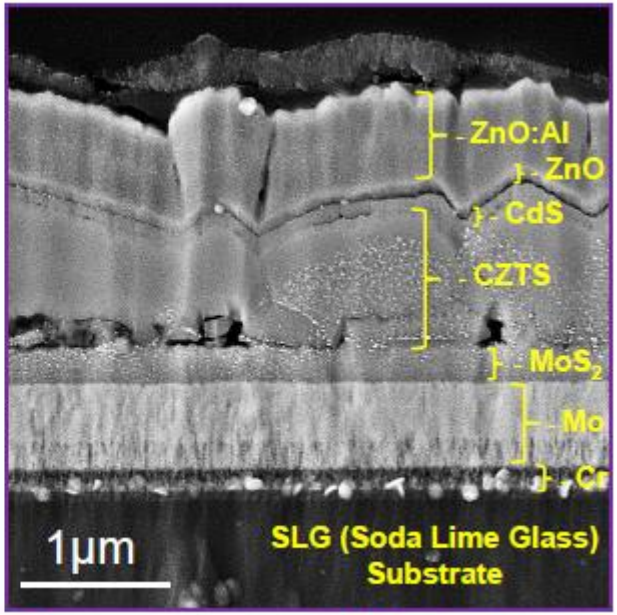


Plasma Assisted PVD

Multi-magnetron sputtering system in IPR

Development of CZTS (Cu_2ZnSnS_4) absorber based thin film solar cell on glass & flexible substrate





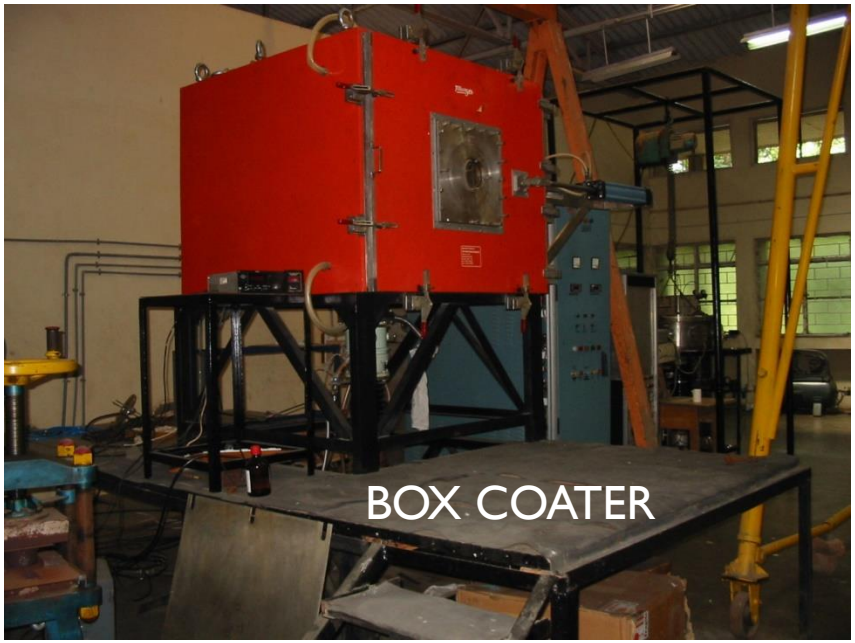
Cross Section SEM of Device

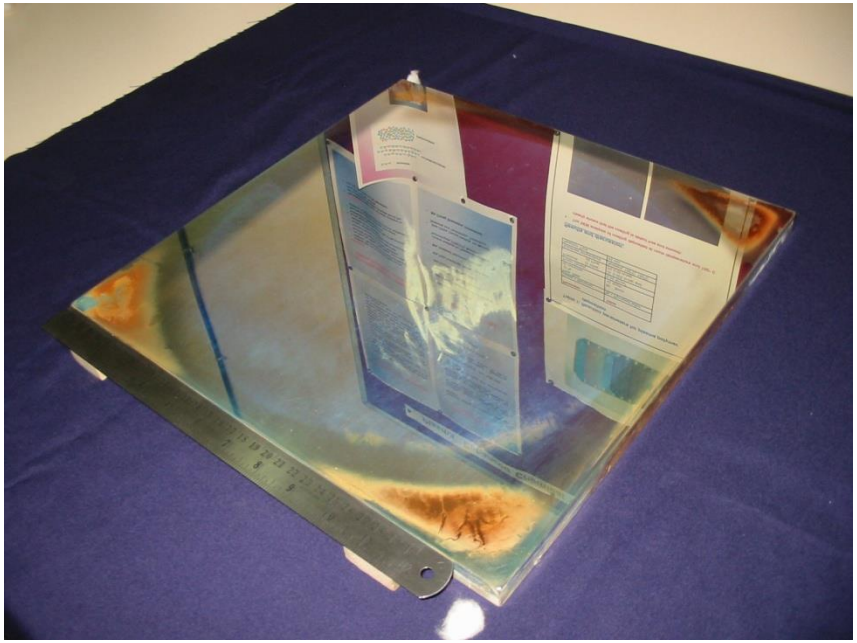
Challenge on adhesion was overcome by chemical and plasma cleaning for depositing CZTS based solar cell on Flexible steel substrate

This can be used portable charging devices and to convert roof tops into solar cell for remote areas after achieving higher efficiency

Plasma Assisted Chemical Vapour Deposition at IPR

SiOx Coating by Plasma Polymerization





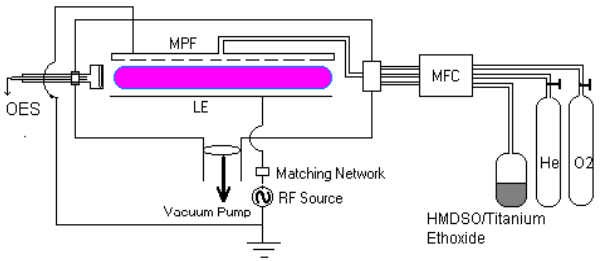


Fig. 1:Schematic of the experimental set-up. (i) Lp= langmuir probe, (ii) LE= live electrode, (iii) MPF= multi-point gas freeder, (iv) MFC= mass flow controllers

Deposition experiments were carried out using an organic precursor with nitrogen gas on Aluminium mirrors.

Thickness of the deposited film were few hundred A°, with good uniformity over the substrate area.

Film is transparent on the mirror surface

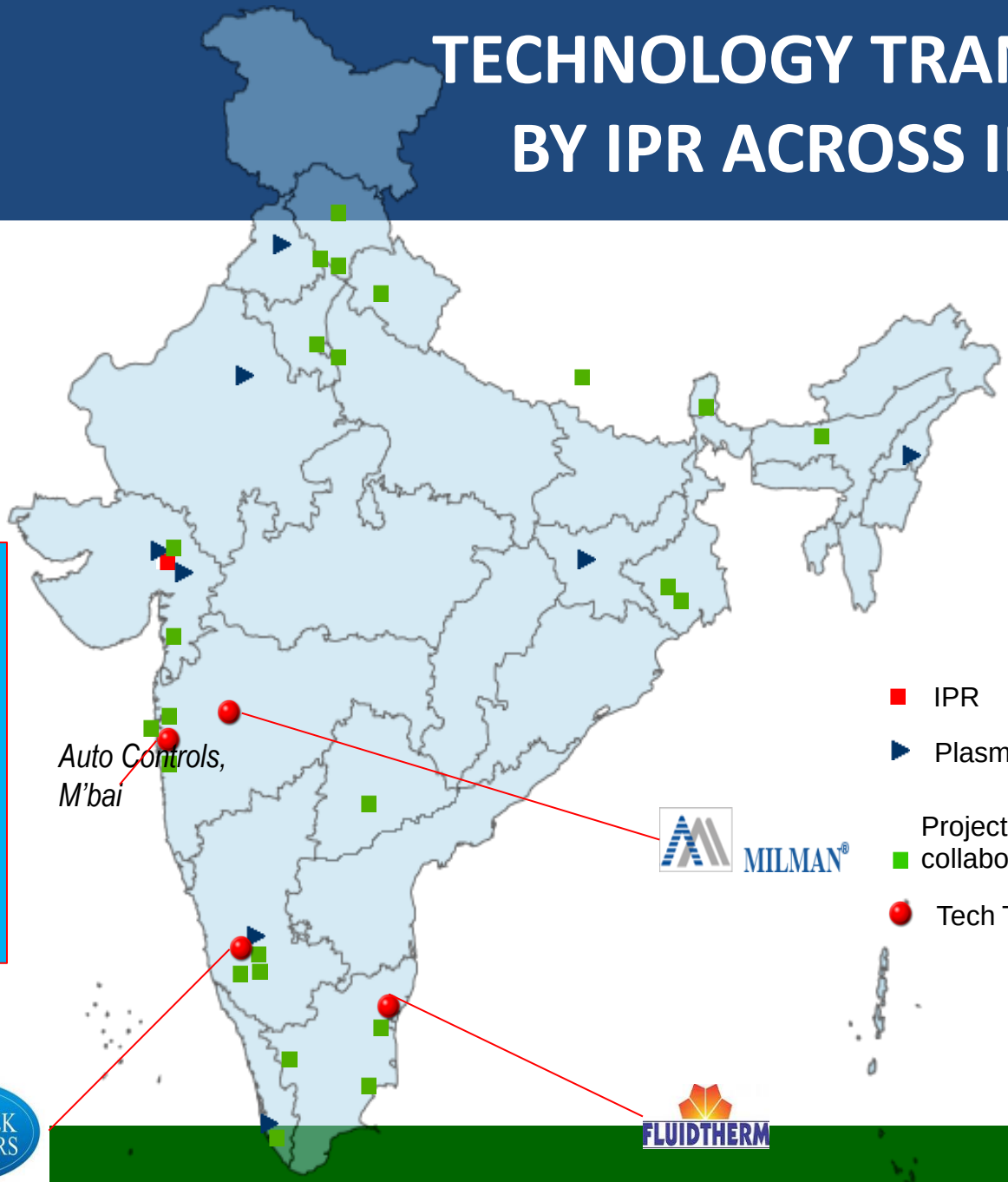
ANTI TARNISH COATINGS ON DECORATIVE ITEMS AND HEADLIGHTS

SiOx COATING ON BRASSWARE

SiOx COATING ON HEADLIGHT

System installed in Moradabad, Uttar Pradesh

TECHNOLOGY TRANSFERS BY IPR ACROSS INDIA



HR developed

BTech: 20
MTech:30
Ph.D: 4
PDF: 10

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Conclusion

- Plasma Nitriding , Plasma nitrocarburizing process and PA deposition processes have gained maturity and are being regularly used by customers.
- Plasma based surface modification - modifies material surface in an environment friendly way.
- The life of steel components improved by a factor of 2 depending on the application.
- Based on our results, the TiN coated Ti alloy Femoral head can be used for clinical trials as the wear rates are in acceptable range.
- Plasma based technologies have reached to a mature and deployable level for commercialization to fulfill our country's mission of Make-in-India.

Challenges and Opportunities ahead

Challenges

- Existing polluting technologies, as there are no strict environment regulations.
- Reduced Power consumption.
- Coating / Diffusion Techniques using atmospheric pressure plasma.

Opportunities

- Clean Technology as no residues are found after the process
- No environmental clearance required.
- Applicable to all types of Ferrous, Al, Ti, Cr materials
- Beneficial to many industrial sectors like, automobiles, dies, textile, space, machine tools etc.

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THANK YOU

HAPPY TO TAKE YOUR QUESTIONS!

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