PLASMA PROCESSING UPDATE

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Facilitation Centre for Industrial Plasma Technologies, Institute for Plasma Research

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Contents

Editor's note

Safe disposal of various types of waste including medical, industrial, chemical, municipal, and nuclear waste is becoming critical and a technology which can do this is in great demand. Plasma Pyrolysis is an upcoming technology in INDIA and has very bright future due to its excellent advantages. FCIPT is the first institute in the country to start the work in this area and has already developed and installed plasma pyrolysis systems of low capacity (12 -15 kg/hr) at various places. Armed with the knowledge gained through the operation of these low capacity systems, efforts were on to develop more efficient and higher capacity systems. As a part of that, FCIPT has developed a 50 kg/hr system which is more efficient than the low capacity system. Various hospitals, chemical & petroleum industries, municipal corporations can make use of this system. Mr. Vishal Jain has explained about the development of this system.

Most of the instruments and machines have such parts which have relative motion while contacting with each other. The wear & tear, and friction caused by this relative motion leads to the loss of energy and the breakdown of machines. Deposition of tribological coatings on these parts can reduce the damage to a large extent. TiN is one of such coating material, with very low coefficient of friction and very high hardness. FCIPT has developed a TiN deposition system, using magnetron based ion sputtering process. Mr. Rane, who has led this project team, describes about the development of this process at FCIPT.

Plasma Ion Nitriding (PIN) technology has been one of the flag bearer technologies of FCIPT for so long. This clean and environment friendly technology is really an asset to universities and small institutes for pursuing research activities. A PIN system that was supplied & installed – by FCIPT – at BIT, Jaipur was unveiled and inaugurated on the National Technology Day (11th May). The details of the program are presented by Dr. Ramprakash.

Editor : *Alphonsa Joseph*
Co-editor : *A. Satyaprasad*

Conference Presentations from FCIPT

Name of the Author	*Topic*	*Date*	*Place*	*Conference*
Dr. S. Mukherjee	Low Pressure Plasma Applications : Eco friendly plasma nitriding process for surface hardening of industrial component	11th March 2008	BIT, Jaipur Campus, Jaipur	Workshop on ‘Environment Friendly Plasma Nitriding Process for Surface Hardening of Industrial Components’
Nirav Jamnapara	Hot Dip Aluminizing Process for TBM Applications - An Overview	21-22 July 2008	Institute for Plasma Research, Gujarat	Work-shop on steels and fabrication technologies 2008
Dr. S. Mukherjee	Surface modification with non-equilibrium plasmas	23 -25 July 2008	AMC, VIT, Vellore	Invited Talk
Dr. S. Mukherjee	plasma science and its industrial applications	25 July 2008	LPSC, Bangalore	Invited Lecture

About FCIPT

Facilitation Centre for Industrial Plasma Technologies

The Institute for Plasma Research (IPR) is exclusively devoted to research in plasma science, technology and applications. It has a broad charter to carry out experimental and theoretical research in plasma sciences with emphasis on the physics of magnetically confined plasmas and certain aspects of nonlinear phenomena. The institute also has a mandate to stimulate plasma research activities in the universities and to develop plasma-based technologies for the industries. It also contributes to the training of plasma physicists and technologists in the country. IPR has been declared as the domestic agency responsible in INDIA to design, build and deliver advanced systems to ITER (International Thermonuclear Experimental Reactor), to develop nuclear fusion as a viable long-term energy option.

The Facilitation Centre for Industrial Plasma Technologies (FCIPT) links the Institute with the Indian industries and commercially exploits the IPR's knowledgebase. FCIPT interacts closely with entrepreneurs through the phases of development, incubation, demonstration and delivery of technologies. Complete package of a broad spectrum of plasma-based industrial technologies and facilitation services is offered. Some of the notable achievements of FCIPT are: plasma nitriding of industrial components to increase wear resistance and hardness, coating of quartz-like films on brassware to inhibit oxidation and tarnishing, thermal plasma technologies for waste treatment, plasma processing for textile industries, deposition of TiN coatings to increase abrasion resistance, deposition of amorphous silicon coatings for anti-reflection properties, etc. The Centre has process development laboratories, jobshops and material characterisation facilities like Scanning Electron Microscope, X-ray Diffractometer, Microhardness testing facilities, which are open to users from industry, research establishments and universities.

This newsletter is designed to help you keep abreast with the developments in the important field of plasma assisted manufacturing and to look for new industrial opportunities. We would be very happy to have you write to us on ways of improving this service or visit us for further discussions.

Please visit our website: *http://www.plasmaindia.com or http://www.ipr.res.in/fcipt*

Technology developed at FCIPT

Development of 50 kg/hr Plasma Pyrolysis System

Mr. Vishal Jain is an electrical engineer and is an expert in the design of plasma power supplies and control & automation systems.

Plasma Pyrolysis is the process of thermal disintegration of organic mass into combustible gases like methane, ethane, hydrogen, carbon monoxide etc. precisely in oxygen starved environment. This process is very useful to dispose the waste which has good calorific value e.g. plastics.

The Plasma Pyrolysis System comprises of primary chamber, secondary chamber, scrubber, ID fan, feeder chamber, purging system and plasma torch system. Earlier FCIPT has developed and demonstrated plasma pyrolysis systems that are of 12-15 kg/hr capacity. The present article, however, describes about the development of more efficient and of higher capacity (50 kg/hr) plasma pyrolysis system.

In this plasma pyrolysis system the pyrolysis reactions take place in the primary chamber. The temperature of the primary chamber is maintained at > 600 degree C (however the temperature near the plasma zone could be > 1000°C) by plasma torch system. The Pyrolyzed gases which are generated in primary chamber are passed through secondary chamber. In secondary chamber, these gases are mixed with air for producing flame. The flame temperature is maintained at 1050° C. The residence time of gases in the secondary chamber are more than one second and hence, any dioxins and furans, if any are formed in the primary chamber because of chlorinated plastic waste, are decomposed into the combustible gases. The product gases after combustion are passed through scrubber where the temperature of the gases is reduced to 70° C. In scrubber, the alkaline water is used which eliminates the chlorine from the product gas and converts it in to salt which remains in the scrubber water and finally the ID fan releases the clean gases into the atmosphere.

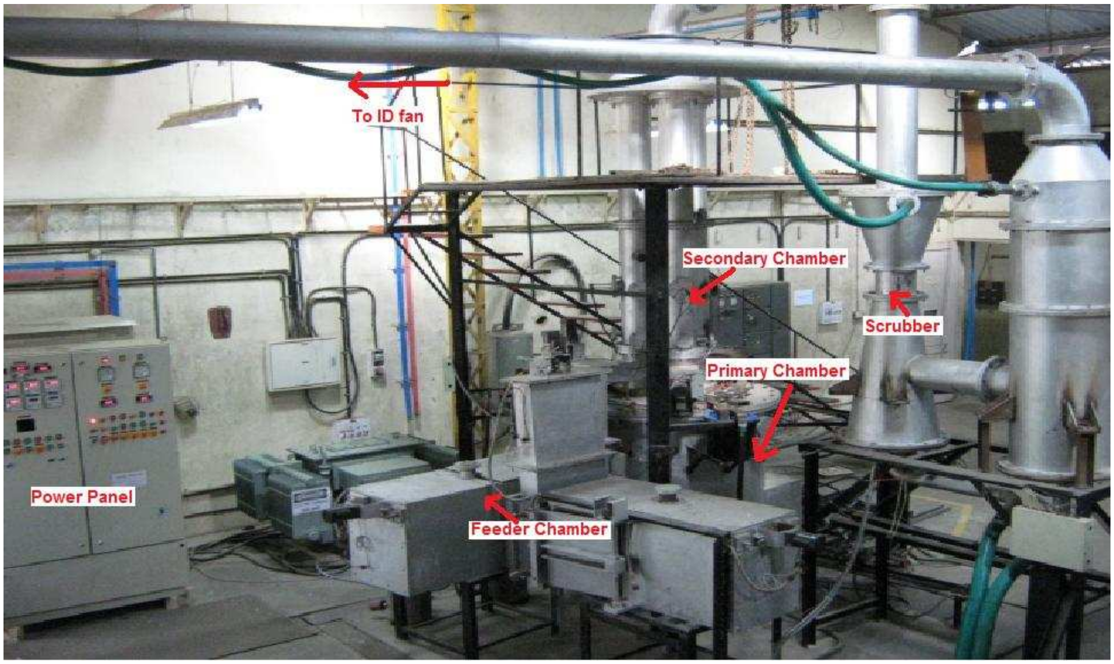

Figure 1: Photograph of the plasma Pyrolysis System of 50kg/hr capacity

This higher capacity (50 kg/hr) plasma pyrolysis system is a better version of the earlier system which FCIPT has developed for the disposal of medical and plastic waste. The photograph of the system is shown in figure 1. The previously developed system was of 12-15 kg/hr capacity. There were following objectives before starting work on the new system:

1. Improvisation in the electrical efficiency of the system. This was the important task to avoid losses in the system for reducing the power requirement of the system from the mains, allow the system to run on continuous basis and also reduce the per kg treatment cost of the waste.
2. To feed bigger waste packets per feed. The waste packet size was calculated in such a way that it should not require any further segregation of medical waste as per the BMW rule.
3. The electrodes feeding is done from outside. This was important to avoid the opening of the primary chamber to change the electrode and subsequently, the consumption rate of the electrodes also been improved.

The operating procedure of the new system is almost similar to that of the earlier system. The comparison of both the old and new systems is presented in table1.

Table1 : Comparison of 12-15 kg/hr and 50 kg/hr plasma pyrolysis systems.

S. No.	Particulars	Earlier DST systems (12-15 kg/hr)	DST-50 System
1	Electrical Efficiency	~ 60%	~ 90%
2	Feeder capacity	1.5 kg per feed	5 kg per feed
3	Automation	Relay, Contactor based	PLC based
4	Pre- heating time (primary chamber temp > 550 Deg C)	30-35 minutes	20-25 minutes
5	Primary chamber volume	0.18 m^3	0.6 m^3
6	Scrubber	shower mechanism	Venturi Scrubber mechanism + shower mechanism
7	Capacity of waste disposal	**12 – 15 kg/hr**	**~ 50 kg/hr**
8	Input power	**65 kW**	**85 kW**

This project is funded by DST, New Delhi for generating a medium scale system for disposal of plastic and medical waste in their TSG program.

The analysis of the effluent gases from the earlier low capacity system had shown that the emissions are very much under control and are in line with the pollution control board's norms. Similar analysis is underway for the new system also, and we are confident that the emissions will be under control in this system too.

Typical industries or organizations that can benefit from this system are : Biomass energy industries, municipal corporations (to treat municipal solid waste), chemical industries, petroleum industries (to treat the residual waste), hospitals etc.

Plasma Based Processes

Development of Titanium Nitride (TiN) coating for tribological applications

Mr. R.S. Rane is an expert in thin film deposition and plasma technology development

Tribology is the branch of science and technology concerned with contacting surfaces having relative motions against each other. It deals with the phenomenon of friction and wear of surfaces with and also without lubrication. One way to reduce friction and wear is to use lubricants; another way is to modify surfaces by deposition of coatings or by special surface treatments. A large number of tribological coatings are known. Titaniun Nitride(TiN) coating is one of them. The application of hard TiN coatings to metal cutting tools has been hailed as one of the most significant technological advances in the development of modern tools. Although cutting tools have been the primary target for the development of such coatings, TiN has also found other tribological applications such as in bearings, seals and as an erosion protection layer. Various techniques like CVD, PVD, PLD etc. have been used to deposit TiN coating.

PVD techniques are atomistic deposition processes which operate at relatively low temperatures. These techniques are based on the principle of mass transfer from a source to the substrate. The PVD coatings have certain micro structural advantages over their CVD counterparts. The PVD films are extremely fine grained and individual crystallites contain a large concentration of point defects due to the non equilibrium deposition process. Consequently PVD coatings are harder than CVD coatings. PVD methods for producing coatings in vacuum environment can be separated in two main groups (i) those involving thermal evaporation techniques and (ii) those involving ion sputtering technique. In Evaporation PVD technology, the coating material is placed in a crucible and heated under low pressure conditions until its vapour pressure is greater than ambient pressure. Currently there are four main methods for evaporation i.e. resistive heating, Induction heating, Arc and electron beam source. Where as in the case of pvd coatings based on ion sputtering techniques, an ion source would be used to produce massive ions which are made to bombard the target material and due to mass transfer the target material gets sputtered and deposited on the substrate. Though there are several ways of producing ion fluxes, most of the recent techniques use plasma as the ion source and these processes are known as plasma assisted physical vapour deposition (PAPVD) processes.

The coatings deposited by PAPVD method are found to be very dense. These coatings contain only few macroscopic defects and their adhesive strength is relatively very good. In this process, glow discharge plasma is used to generate a flux of ions (e.g. Ar^+) and by applying a negative bias to the target, they are allowed to incident on the target surface. These ions cause atoms and occasionally cluster of atoms to be ejected from the target surface through the transfer of their momentum. However by using magnetrons, an external magnetic field could be coupled with the existing electric field and by this way, electrons are kept in trajectories close to the target surface. This increases the number of ionizing collisions of electrons with argon atoms and therefore enhancing the sputter and deposition rates. To enable the sputtering process as a useful coating process a number of criteria must be satisfied. Firstly ions of sufficient energy must be created and directed towards the surface of target to eject atoms form the target material. Secondly, ejected atoms must be able to move freely towards the objects to be coated with little impedance to their movement.

At FCIPT we had developed a magnetron based ion sputtering system to deposit Titanium nitride (TiN) coating on various objects. Pure Titanium disc of 3” diameter and 5 mm thick was used as the target material. This target was clamped with a standard 3” planar magnetron. This magnetron setup was mounted on to a vacuum compatible reaction chamber. A combination of rotary and diffusion pump was used to obtain a base pressure of approximately 2 X 10^{-5} mbar. An operating pressure of 5 X 10^{-3} mbar was obtained by admitting both Argon and Nitrogen in a proper ratio. The target Ti disc was biased with a D.C. power Supply (1000V, 3A) to form the required glow discharge plasma. When the plasma was generated, the energetic argon ions sputter the Titanium target, and the ejected Ti atoms react with the nitrogen present in the chamber and forms TiN which gets deposited on the object that was appropriately placed in the reaction chamber. A photograph of the actual system with all the accessories is shown in figure 1. And the typical operating parameters are shown in table 1.

Fig.1 Photograph of the PVD based Titanium Nitride Coating system developed at FCIPT

Table 1 : Typical operating parameters used to deposit TiN coating

1. Operating pressure : 5 x10^{-3} mbar (Ar + N_2)
2. Power density : 5-6 W/cm^2
3. Substrate bias : 0 to -100 V
4. Substrate Temp. : 300^0C to 500^0C
5. Deposition rate : 4-5 A^0/s

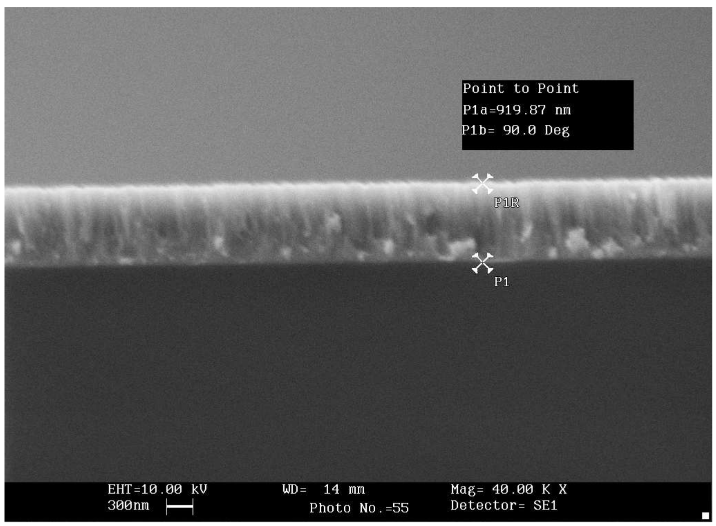

Fig.3 SEM image of TiN coating on silicon substrate (cross section)

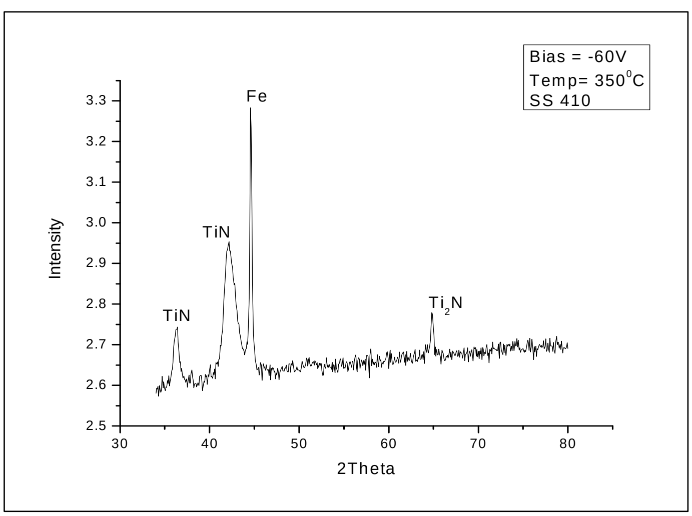

Fig.2 XRD spectrum of the deposited TiN coating

Results

By using magnetron based ion sputtering system, TiN was successfully deposited on stainless steel (SS) objects. X-Ray Diffraction (XRD) was used to confirm that the deposited coating was TiN. The XRD spectrum of the deposited coating is shown in figure 2. Scanning Electron Microscope was used to measure the thickness of the deposited coating. The cross sectional SEM image of the TiN deposited for one hour is shown in figure 3. The deposited coating was also studied for its adhesion strength by using an elcometer and the strength was found to be in the range of 8-10 Mpa .

Development of Teflon like coatings for LPSC, Trivandrum

Ms. Purvi is an expert in thin film deposition using PECVD

Liquid Propulsion System Center (LPSC), – a center of Indian Space Research Organization (ISRO) –, Trivandrum, is engaged in development of liquid and cryogenic propulsion stages for launch vehicles, and auxiliary propulsion systems for both launch vehicles and satellites. In a spaceflight, a launch vehicle or carrier rocket is a rocket used to carry a payload from the Earth's surface into outer space.

Ball bearings used in cryogenic turbo pumps in launch vehicle have to function at very low temperatures of about −130° C to −270° C and also they are in direct contact with cryogenic fluids. In order to ensure high reliability of bearing performance under gross slip conditions during high transient periods at start-up and shut down conditions, a pre-lubrication of the bearings with suitable solid lubricant coating is adopted. Different kinds of solid lubricants in the form of coatings have been used in various international space programs. For example Au coating and RF sputtered PTFE coating are used for the cryogenic turbo pumps of Japanese space programs, where as MoS_2 coating, Ag coating and Si_3N_4 balls in SUS 440C steel bearings are used in turbo pumps of a different programs by NASA.

Poly Tetra Fluoro Ethylene (PTFE), which is also known as Teflon, is considered one of such solid lubricating material. Teflon is a chemically inert material with low surface energy, and good dielectric and lubrication properties. The coefficient of friction of the Teflon is generally in the range of 0.05 to 0.20 depending on the load, sliding speed and particular coating used. Further its performance at extremely low temperatures is observed to be outstanding.

FCIPT has used Plasma Enhance Chemical Vapor Deposition (PECVD) technique to deposit Teflon like coating on stainless steel (SS) samples. Samples of SUS440C stainless steel, have been used for the feasibility study, as it is the most widely used steel for manufacturing ball bearings that are used for cryogenic applications. The schematic of the experimental setup is shown in figure 1.

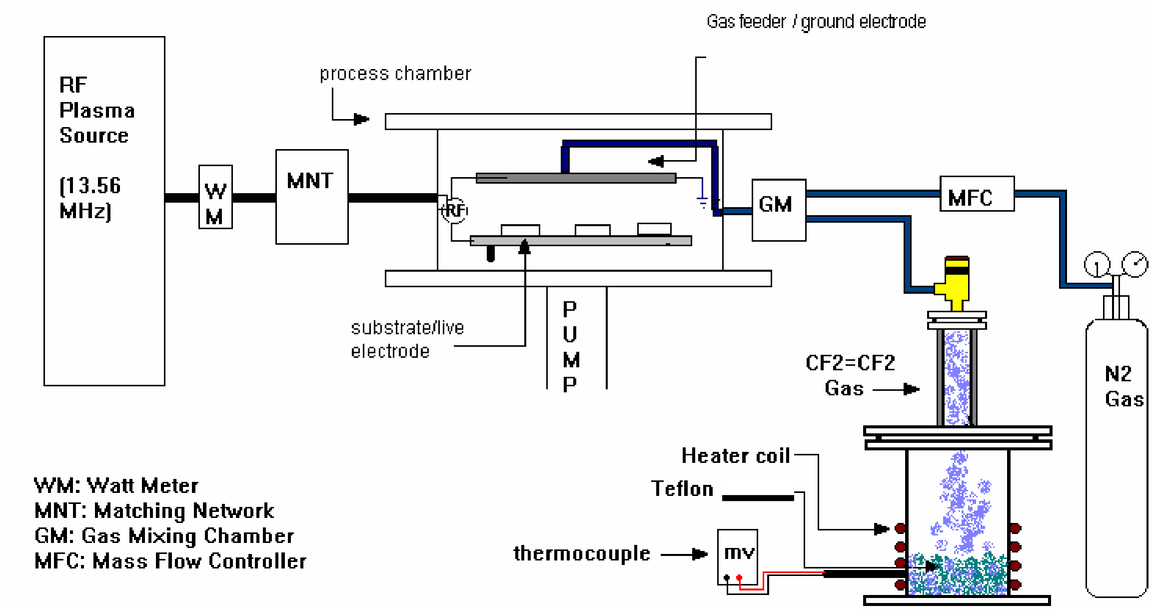

Figure 1: Schematic of the experimental setup for depositing Teflon like coating

The precursor gases necessary for depositing the coating were generated by pyrolyzing Teflon powder at 450° C, in the absence of oxygen. A parallel plate electrode configuration was used the electrodes were powered using a RF (13.56 MHz) power source. The precursors mixed with nitrogen are introduced in to the RF plasma where they get fragmented into F, CF, -C-CF, CF_2, and CF_3 radicals, and reach to the substrate surface. At the substrate surface these species combine and polymerize to form long chain molecules.

The feasibility study was carried out on SUS440C samples of 30mm diameter and 5 m thick. The required thickness of the coating was around 0.7 μm, and the operating parameters have been optimized accordingly. The deposited samples were subjected to X-ray Photo electron Spectroscopy (XPS) and Scanning Electron Microscope (SEM) studies. The XPS spectrum is shown in figure 2 and from the figure it is clear that the deposited coating is in fact Teflon like. SEM was used to study the morphology of the coating and to find its thickness. The morphology of the deposited coating is shown in figure 3, and from the figure it is clear that the deposited film is pinhole free. To find the thickness, the deposition was done over a silicon wafer and its cross section was studied with SEM. The result is shown in figure 4.

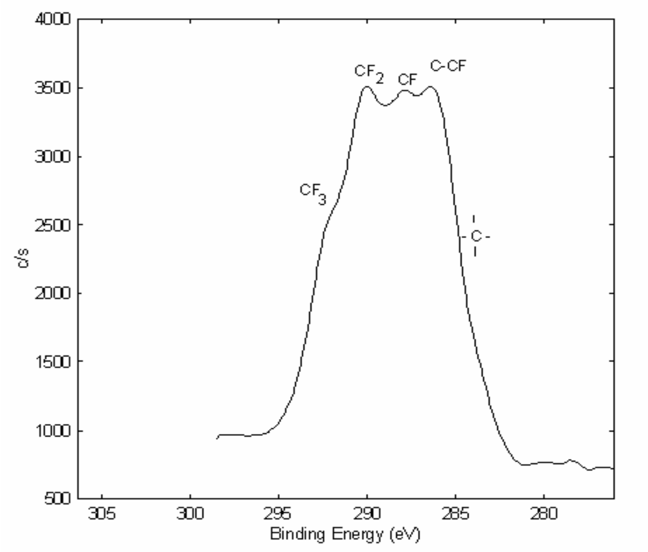

Figure 2: XPS Spectra of Teflon Like coating deposited at FCIPT.

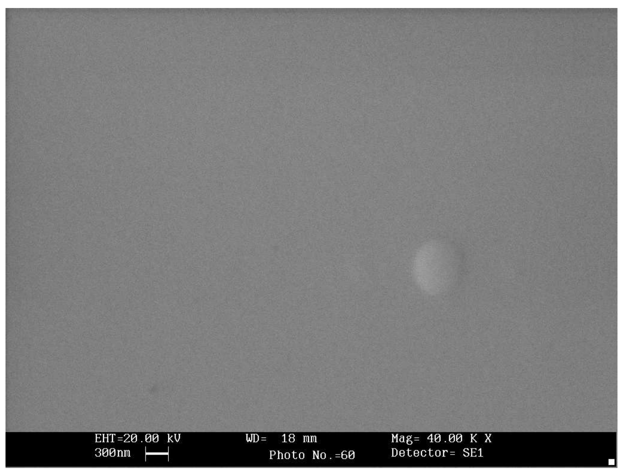

Figure 3: SEM micrograph of Teflon Like coating deposited at FCIPT.

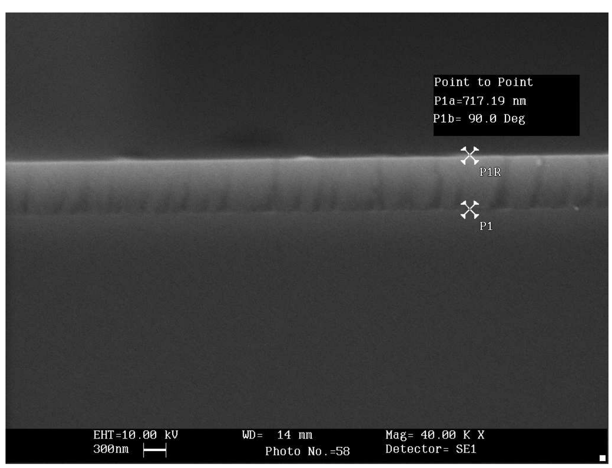

Figure 4: SEM Image of silicon wafer cross section having Teflon like coating

The deposited samples were subjected to adhesion test also using a pull pin tester, as per ASTM 4541. The adhesion strength of the coatings was observed to be 3-5 MPa. The tribological tests are underway.

The deposition of the Teflon like coating on the actual bearings is in progress.

Inauguration of Plasma Ion Nitriding (PIN) system at Birla Institute of Technology (BIT), Jaipur Campus on National Technology Day (11^{th} may 2008)

Dr. Ramprakash is a scientist at BIT, Jaipur campus, Jaipur

To commemorate the National Technology day in Rajasthan, a one-day workshop was organized on “Environment Friendly Plasma Nitriding Process for Surface Hardening of Industrial Components” at BIT, Jaipur campus on the 11^{th} May 2008, in association with the Department of Science and Technology, Govt. of Rajasthan. Around 100 participants from various scientific and academic institutions and industries of Rajasthan and neighboring sates attended the workshop. The objective of this workshop was to inaugurate, demonstrate, and help promote the environment friendly plasma nitriding technology to the industries of Rajasthan, and also to facilitate training on this clean technology. This technology would enable the local industry to deliver the plasma nitrided products/components of such quality that they can be competitive in the international market. It would also help the state of Rajasthan in replacing existing hazardous technologies with an environment friendly technology. The commemoration was followed by dedicating this new technology to the state of Rajasthan. Further, arrangements were made to provide one-day training to the industrialists and academicians of the state on Plasma Ion Nitriding (PIN) system.

The PIN system was installed on 22^{nd} Feb, 2008 at BIT Jaipur Campus under the guidance and collaboration with Facilitation Centre for Industrial Plasma Technologies (FCIPT), Gandhinagar and was financially assisted by the Department of Science and Technology (DST), Government of India, National Engineering Industries (NEI) Ltd., Jaipur and BIT, Jaipur campus. PIN is basically an environment friendly surface hardening technique. The conventional nitriding systems use poisonous chemicals like cyanides and ammonia where as the PIN system uses non-toxic and precisely controlled gas mixtures. The installed PIN system is a fully automatic PLC/PC based system and is capable to plasma nitride the components (of around 500 kg) from automobile, textile, plastics, power and moulding & die industries.

The workshop began by lighting the plasma lamp by the honourable chief guest Shri. Damodar Sharma, principle secretary, Department of Science and Technology (DST), Government of Rajasthan. Shri Sharma also inaugurated the PIN system on this occasion. Dr. R. Venkatramani, a distinguished scientist and ex-director of Beam Technology Division, Bhabha Atomic Research Centre (BARC), mumbai was the guest-of-honour for the event. Prof. P. K. Barhai, the honourable vice-chancellor of BIT, chaired the inaugural session and also delivered the welcome address. Dr. S. Mukherjee, Head, FCIPT division, IPR, Gandhinagar delivered the keynote address and introduced the theme of the workshop. Dr. Amita Gill summarized the activities of the DST, Government of Rajasthan and their role in promotion of new technologies in the state of Rajasthan. Mr. K. S. Boob, General Manager, NEI Ltd., has spoken on the need of plasma nitriding process for the industries in Rajasthan. Dr. Abhinav Dinesh, Director BIT Jaipur, and the Chairman Local organizing committee delivered the vote of thanks. Dr. Ram Prakash, Convenor, conducted the inaugural session.

The workshop had two technical sessions followed by the inaugural session. The first session was focussed on "Plasma Nitriding Process for Surface Hardening of Industrial Components" which consisted of three presentations. The second session was a practical session and was conducted in the later half of the day.

The first presentation of the first technical session was delivered by Dr. S. Mukherjee on "Low pressure plasma applications: Eco-friendly plasma nitriding process for surface hardening of industrial components". In his presentation, he has mentioned that plasma nitriding imparts a higher case depth in lesser time scales, uniform case depth, and it can be applicable to almost all types, shapes, and sizes of steel components. Further, he also stressed that in plasma nitriding no post grinding is required, it can be carried out at a wide range of operating temperatures, guaranteed cost-effective performance enhancement, and most importantly it is an environment friendly technology. Dr. Mukherjee illustrated the progressive advancement of plasma nitriding technology in the context of the present national need. He talked about the crucial issues related to this technology and also on other developments at FCIPT, IPR. He emphasized the role of DST, Government of India and FCIPT, Gandhinagar in promoting the plasma nitriding technology in India.

Mrs. Alphonsa Joseph of FCIPT had delivered the second presentation and it was on "Characterization of Plasma Nitriding Components". She focused on the various techniques of material characterization that can be used in the validation of plasma nitrided components. She elaborated these techniques and mentioned that the surface hardness is measured by Vickers microhardness tester, phase identification is done by X-ray diffractometer, wear resistance and anti-galling properties are measured by pin-on disc type wear tester, fatigue life is measured by fatigue fracture testing machine and corrosion resistance is measured by potentiostat method.

The third presentation was delivered by Dr. Ram Prakash and it was on "Promotion of Environment Friendly Plasma Nitriding Process in the State of Rajasthan". He highlighted the importance of this technology in the context of Rajasthan and also explained the comparative superiority of this technology over the other conventional processes. He mentioned that Rajasthan has a large number of tool and die manufacturers and the demand for heat treatment of such components is very high. Major segment of these industries currently use highly polluting technologies, posing a threat to the ecology in and around the state. Further, most of the hydro and thermal power plant components like guide vanes, turbines, ball-bearings etc can also be plasma nitrided to improve their wear and erosion resistance. Both automobile and plastics industries in Rajasthan, who manufacture or treat blades, dies, moulds etc. in large numbers can avail this technology to a large extent. Dr. Venkatramani chaired this session and at the end of the session he summarized all the presentations.

In the later half of the day a practical session was conducted on the Plasma Ion Nitriding system to let the participants understand the working mechanism of the PIN system. Finally a concluding session was organized to close the workshop.

Conclusions and Recommendations:

The training covered during this one-day workshop was well appreciated by both participants as well as speakers as noted during the feedback session. The lectures by the expert scientists and rigorous discussions on the plasma technology were extremely valuable. During the concluding session, it was felt that more industrialists should be trained on such eye catching environmental friendly technologies. It was suggested that additional workshops focused on various aspects of plasma nitriding should be organized regularly.

In the concluding session, Shri Damodar Sharma, the chief guest for the event, appreciated the combined efforts made by BIT, FCIPT, NEIL and DST, in establishing this eco-friendly technology at Jaipur. Being an expert in environment science, he mentioned in his address that the plasma nitriding technology is a specialized and advanced technology for the surface hardening of steel components without disturbing the environment. He highlighted the recent achievements of the state DST for pursuing certain environment friendly technologies.

Dr. S. Mukherjee, in his concluding remarks has expressed his commitment to promote this technology in India and he was happy to collaborate on this project with BIT Jaipur Campus and with the way this project is progressing. He mentioned that plasma nitriding is a high quality technology, and is reliable and clean compared to conventional surface hardening technologies and the demand for this technology is rising day by day in India.

Dr. R. Venkatramani, the guest-of-honor for the event, explained how plasma based advanced surface engineering plays a pivotal role in our daily lives. Further, he gave an overview of the support available from DST and other sources to support similar activities for academicians, researchers and industrialists.

Mr. Boob, general manager of National Engineering Industries Limited (a pioneer industry in the field of bearing manufacturing), mentioned that they would replace their conventional surface hardening techniques of bearings with the advanced plasma nitriding techniques.

Dr. Ram Prakash, the convenor of the workshop, mentioned that the plasma nitriding system installed at BIT is open for all industries to get their steel components nitrided at a very minimal cost. Practical session headed by Mrs. Alphonsa Joseph and computerized demo presented by Mr. Ravindra Kumar, machine operator of the plasma Nitriding system was well appreciated by the participants.

Prof. P. K. Barhai, Vice-Chancellor BIT, MESRA, Ranchi, expressed his satisfaction on the successful completion of the workshop. He underlined his association with this project and highlighted the importance of this project in view of the wealth of Rajasthan. He expressed his commitment to support the plasma lab facility at BIT Jaipur Campus.

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