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

Solar power is the cleanest power available on the earth and the efforts are on from all over the world to more efficiently convert the solar energy into usable electrical energy. A solar cell is a device that converts sunlight directly into electricity by the photovoltaic effect. The efficiency of a solar cell is described in terms of the electrical output per unit incident light energy (watt/watt). Deposition of various thin films on the semiconductor elements like silicon has proven to improve the efficiency. Hydrogenated silicon is one of such materials. These thin films are actively studied, now a days, for their use in thin film solar cells, TFT displays, and photodiodes etc. Several techniques have been tried to obtain high quality Si:H at higher deposition rates. At FCIPT, studies have been conducted to successfully deposit high quality Si:H thin films using very high frequency (55 MHz) PECVD at low substrate temperatures (60°C) using a novel multi-hole cathode configuration. Mr. Chetan Jariwala gave the details of the experiments and the results.

Tokamak is a type of magnetic confinement device and is also one of the most researched devices to produce controlled thermonuclear fusion power. Very hot plasma needs to be confined away from the inner/plasma facing walls of the Toakamak and very high magnetic fields are used for that purpose. Carbon is one of the materials chosen as a plasma facing component (PFC) and in order to efficiently remove the heat loads, these PFCs are joined with proper heat sinks like copper or its alloys. However proper joining of the PFCs with the heat sink materials is a very challenging task. Dr. Sameer Khirwadkar has been working on this problem and has presented certain details.

Editor : Alphonsa Joseph
Co-editor : A. Satyaprasad

Conference Presentations from FCIPT

Name of the Author	*Topic*	*Date*	*Place*	*Conference*
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	Plasma Nitriding and Plasma Implantation	16th Sep.08	DMRL, Hyderabad	Invited lecture (in 'Continual Education Program')
Dr. S.K.Nema	1. Plasma Polymerization & its Applications 2. Safe Disposal of Plastic/Polymer Waste and Energy Recovery using Plasma Pyrolysis Technology"	21st & 22nd October 2008	Inst. Advance Study in Science & Technology, Guwahati, Assam	National Workshop on Recent Trends in Polymer Science
Dr. S. Mukherjee	Design aspects in low pressure plasma surface engineering systems	17-20 Dec.08	BIT, Ranchi	Invited lecture (in DST-SERC school)
Dr. P.M.Raole	Fundamentals of Plasma processing and applications	17-20 Dec.08	BIT, Ranchi	Invited lectures (in DST-SERC school)
Dr. S.K.Nema	Understanding Low & Atmospheric Pressure Plasmas and Their Applications	17-20 Dec.08	BIT, Ranchi	Invited lectures (in DST-SERC school)
Dr. P.M.Raole	Surface characterization of carbon related materials	23-24 Dec.08	S.P.University, Vallabh Vidyanagar	Nation Workshop on Characterization techniques for Carbon materials

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*

Research at FCIPT

Hydrogenated Silicon Thin Film Processing by Multi-hole-cathode Very High Frequency-Plasma Enhanced Chemical Vapor Deposition

Mr. Chetan Jariwala is an expert in Material characterization (XPS & AES) and Si:H thin film deposition by VHF(55 MHz) PECVD

Introduction

Si:H thin films are actively studied for their use in a range of devices such as thin film solar cells, thin film transistor (TFT) displays and other devices such as photodiodes [1,2]. Several fabrication techniques have been used for the deposition of Si:H and new developments are aimed at improving material quality and achieving higher deposition rates. Of these techniques, PECVD is considered the most important for large area depositions, which also allows the use of cheap substrates such as glass, stainless steel, and polymers for Photovoltaic modules. While early studies utilized RF (13.56 MHz) PECVD, the advantage of Very High Frequency (VHF: 25-150 MHz) PECVD for higher deposition rates has been recognized, based on the pioneering work at the University of Neuchâtel [3]. Curtins *et al* showed that the deposition rate can be increased to 20 A°s^{-1}, without increasing power density, when the frequency is raised from 25 to 70 MHz [3]. Also, this could be achieved without sacrificing the quality of the films compared to RF PECVD [3].

In a recent significant study, a novel multi-hole cathode (MHC) with VHF (60 MHz) PECVD has demonstrated the highest deposition rate of 77 A°s^{-1} at substrate temperatures of 200-400 C for device grade micro-crystalline (μc)-Si:H thin films [4]. It is well known that Si:H films deposited by PECVD at a substrate temperature of around 250 C show the best optoelectronic properties such as photoconductivity and photo-response for solar cell device applications. However, deposition of Si:H at low temperature (40-150°C), well below typical processing temperatures of 200-350°C have also attracted interest [5,6] with the aim of growing devices on polyethylene (PE) or polyethylene terepthalate (PET) substrates.

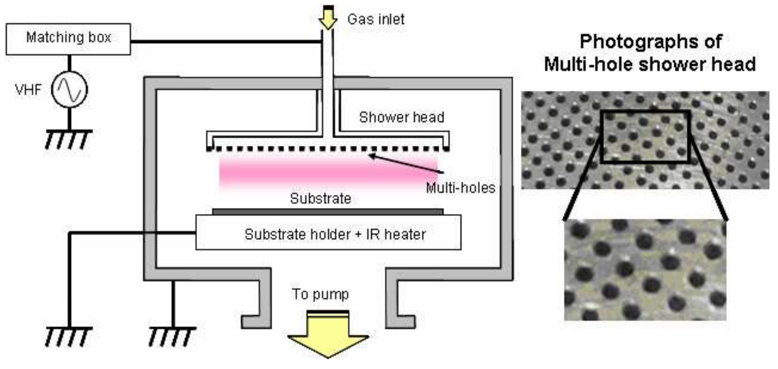

Fig.1 A schematic diagram for VHF PECVD system for amorphous silicon thin film deposition with photographs of multi-hole cathode.

In the present work, we study Si:H thin films grown by 55 MHz VHF PECVD, using a multi-hole-cathode geometry at a low substrate temperature of 60°C. We report LP measurements of the ion density and electron temperature profiles, which identify the origin of high deposition rates achieved for Si:H films grown by VHF-PECVD. We have characterized the films using XPS and AFM for chemical composition and surface morphology analysis, respectively. Optical reflectance measurements were done to determine the thickness and deposition rates.

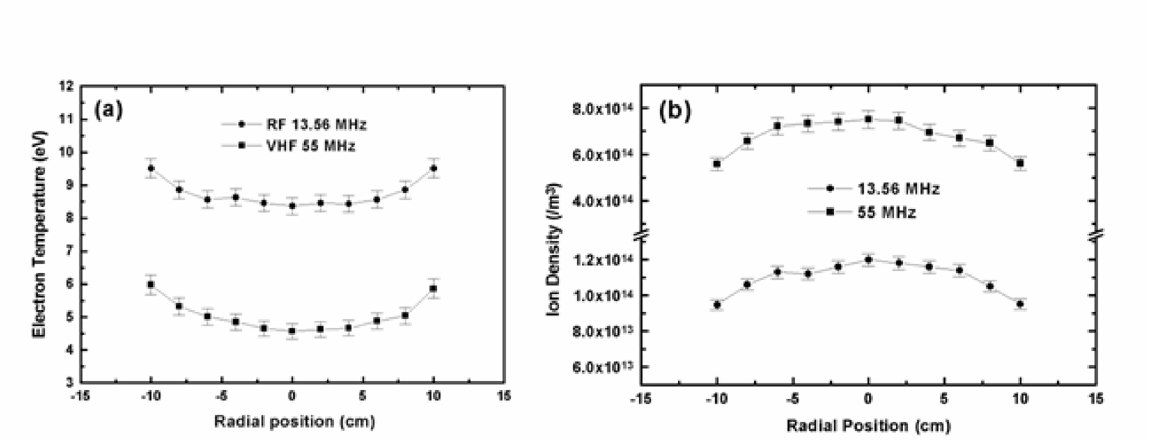

Fig.2 (a) Radial electron temperature (T_e), and (b) ion density (N_i) profiles comparison for VHF (55 MHz) and RF (13.56 MHz) H_2 plasmas at pressure 0.6 mbar with 20 mW/cm^2 power density in same reactor.

Experimental Setup

Figure 1 shows a schematic of our diode-type PECVD system along with pictures of MHC, which employs an excitation frequency of 55 MHz and a MHC shower-head [4] of 22 cm diameter. The separation between electrodes was fixed at 3 cm and [H_2]/[SiH_4] ratio, R_H = 11.5 for all the depositions. The depositions were performed at a pressure of 0.6 mbar and a substrate temperature of 60°C. The power density was varied from 20-42 mW/cm^2 and the total gas flow rates were varied from 20 to 50 sccm. The substrates were then cleaned in-situ for 20 min using nitrogen plasma, just before deposition of the thin films. The substrates were placed on the ground electrode for the depositions.

The plasma parameters were measured by a self compensating disc LP designed to work in 55 and 13.56 MHz plasmas. Suitable VHF and RF chokes were used to filter the fundamental of the VHF and RF frequencies, respectively. In addition, a large auxiliary compensated electrode was used to sense the voltage fluctuations near the probe [7]. Before each set of I-V measurements, the probe tip was sputter cleaned using low energy (280 eV) Ar ions.

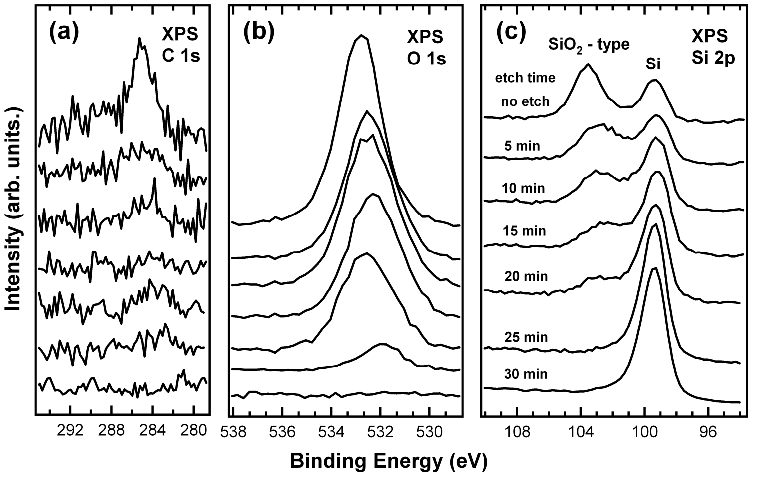

Fig.3 XPS depth profile study of Si:H film deposited on SS sample. The fig (a), (b), and (c) shows C 1s, O 1s, and Si 2p core level spectra as a function of etching time.

XPS measurements of Si:H thin films were carried out in a Multitechnique Surface Analytical System (5702) supplied by Physical Electronics USA. XPS analysis was done at a base vacuum of 5.5 x 10^{-9} Torr with the pressure rising to 1.5 x 10^{-8} Torr during etching with Xe ion gun. The measurements were performed using monochromatic Al Kα source with resolution of 0.5 eV FWHM of the Ag $3d_{5/2}$ peak, which was also used to calibrate the binding energy scale. The AFM study was carried out using a scanning probe microscope SPA 400 made by SII Nanotechnology Inc., Japan. The optical reflectance of the deposited films for determining thickness has been measured using a spectroradiometer of M/s. Optronics Laboratories Inc, USA. Its wavelength resolution is ± 1nm and reflectance accuracy is 2-3%. The average angle of incidence was within 5° of the normal, within the accuracy of the measurements, and the measured reflectance is thus considered to be normal incidence reflectance.

Results and discussion

1. Langmuir probe measurements

LPs are widely used to investigate the key plasma discharge parameters such as electron temperature (T_e), ion density (N_i), plasma potential (V_s) and EEDF [8]. In the present study, a self compensating LP was used to measure radial profiles of T_e and N_i for comparison of RF and VHF H_2 plasmas. The LP was moved along a diametric axis across the centre, between the capacitively coupled multi-hole shower head and electrode. Radial electron temperature profile (see fig 2(a)) indicates an average T_e ~ 5.4±0.27 eV, whereas the ion density profile (see fig 2(b)) indicates an average N_i ~ 5.81±0.3 x 10^{14} m^{-3}, for the 55 MHz VHF H_2 plasma, at a fixed pressure of 0.6 mbar and 40 mW/cm^2 power density. In comparison, a 13.56 MHz RF H_2 plasma gives an average T_e ~ 9.7±0.5 eV and average N_i ~ 9.43± 0.47 x 10^{13} m^{-3}. Similar results were reported in a recent study frequency dependent (13.56 to 100 MHz) study of T_e and N_i for a H_2 plasma [9]. The decrease in T_e for VHF plasmas is known to give better quality Si:H films due to increase in SiH_3 radicals in the plasma [10]. As the frequency is increased from 13.56 to 55 MHz at constant power density, a significant increase in the ion saturation current has been observed, which is proportional to N_i. Therefore, the higher Ni in case of VHF would result in a high deposition rate for thin films with respect to RF plasma.

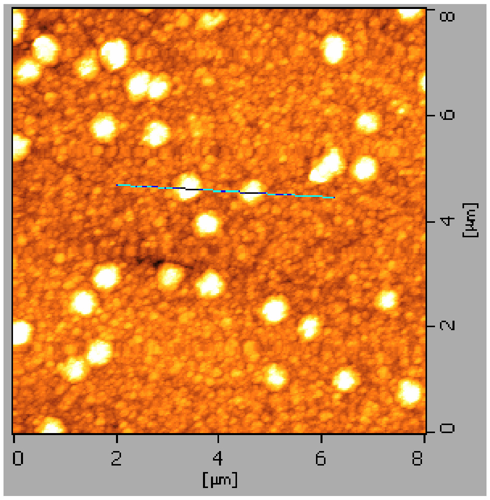

Fig. 4 AFM image shows a μ-Si:H phase embedded in a-Si:H matrix

2. Thin Film Characterizations

Fig. 3(a-c) shows XPS depth profiles obtained as a function of etching time for the C 1s, O1s and Si 2p core levels. While a small amount of Carbon is present on the as-obtained thin film surface, the etching results in a reduction and eventual absence of the carbon in the films. Similarly the oxygen present on the surface as an oxide (Fig. 3b and 3c), is strongly reduced after etching the surface for 30 min (~ 80 A°). Simultaneously, a clean Si 2p spectrum is obtained in the bulk of the film.. AFM image shown in Fig. 4 confirms the mixed phase in the deposited films, showing Si micro-crystallites of 500-600 nm embedded in the amorphous-Si:H matrix, consists of 40-70 nm grains. Further, our recent study of high resolution photoemission (HRPES) confirmed the mixed phase present in the grown films [11].

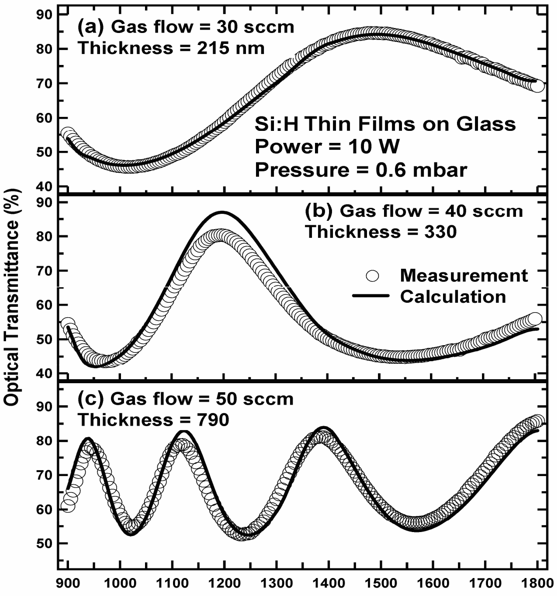

Fig. 5 Optical reflectance spectra measured for various Si:H thin films deposited on glass fitted with calculated spectra.

In order to determine thicknesses of the deposited films, we measured the optical reflectance of Si:H thin films of different thicknesses grown on glass substrates in the IR region: 900-1800 nm. The experimental data are shown in Fig. 5 for three different flow rates, from 30 to 50 sccm during deposition, which leads to different thicknesses of the Si:H films. The reflectance of Si:H films on glass can be simulated in terms of Fresnel equations for a three-layer system [12], consisting in the present case of air/a-Si:H/glass.

Using a least squares error analysis, we have compared the calculated reflectance spectra (superimposed) with the measured spectra and determined the thickness values. They range from 2100 to 8000 A, with a fixed refractive index η ~ 3. The deposition rate has been calculated, and is found to vary from 70 to 110 A/min as gas flow increases from 30 to 50 sccm, with all other process parameters kept constant (fig. 6).

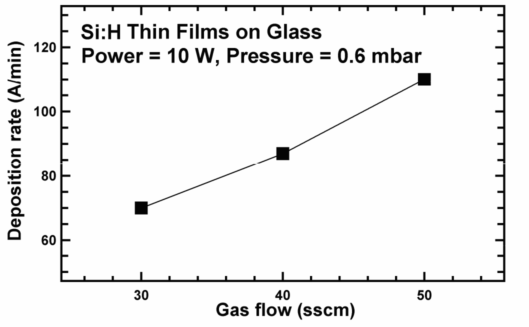

Fig. 6 The deposition rates of Si:H thin films on glass for varying gas flow from 30 to 50 sccm.

Conclusion

In conclusion, Si:H thin films were grown on single crystal silicon, stainless steel and glass substrates, using a 55 MHz PECVD process with a multi-hole cathode geometry at a low substrate temperature of 60°C. LP profiles of ion density and electron temperature suggest that the origin of higher deposition rates compared to RF PECVD is due to higher ion density and a lower electron temperature in VHF PECVD. XPS was used to confirm the high quality of the deposited films where as AFM reveals typical mixed phase of the Si:H films. The thickness and deposition rates have been obtained from optical reflectance measurements of the films, which are in the range of 2100 A to 8000 A and 70-110 A/min, respectively.

Acknowledgement

I am grateful to my colleagues Mr. S.Bhatt, Dr. A.Chainani for their involvement in various aspects of this work; and Dr. R.Eguchi, Dr. M.Matsunami & Prof. S.Shin for AFM and XPS measurements. I thank Professor P. K. Kaw, Professor V.L.Dalal and Professor K. L. Narasimhan for supporting the VHF PECVD project at IPR. I also thank Mr. Sunil Kumar for the design and development of the VHF power supply.

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Studies on development of plasma facing components for Tokamak at IPR

Dr. S.S.Khirwadkar is a senior scientist at IPR

Tokamak is a device used for confinement of hot and dense plasma using toroidal vacuum chamber and toroidal magnetic field. It is of specific interest for production of nuclear energy using controlled thermonuclear fusion of Deuterium and Tritium nuclei. Plasma facing materials in a tokamak device have to withstand extreme heat loads and particle loads from plasma. Beryllium, Carbon and Tungsten are popular choices for plasma facing materials. Typical heat energy deposition on plasma facing materials of a tokamak is extremely high e.g. steady-state heat-flux of the order of 10 MW/m^2 and transient heat flux ($\Delta t \leq 1$ sec) can exceed 20 MW/m^2. Efficient removal of heat energy from plasma facing materials by joining them with water-cooled copper-alloy heat-sink material is a challenging task. Stress concentration at the interface due to large difference in coefficient of thermal expansion between plasma facing material (Carbon or Tungsten) and copper-alloy is reduced by introducing soft Oxygen Free High Conductivity (OFHC) copper layer at the interface. Techniques used for joining of plasma facing materials are: Brazing, Hot Isostatic Pressing (HIP), Hot Pressing, E-Beam Welding, Plasma Spray Coating.

Experimental studies on joining Carbon (Graphite or Carbon-Fiber-Composite) and Tungsten materials with copper-alloy heat-sink materials for plasma facing components of tokamaks are in progress at IPR. Joining of copper and carbon is a challenging task due to poor wettability. Experimental study[1] for joining Graphite with OFHC Copper using Electron Beam assisted Physical Vapor Deposition & Casting process is shown in Figure A. Further joining of OFHC copper to copper-alloy heat-sink can be done by standard joining techniques such as Brazing or EBW.

Experimental studies on brazing of Carbon/Tungsten materials with copper-alloy heat-sink are also attempted using active-brazing-filler materials. Figure B shows vacuum brazing furnace at IPR and SEM image of interface of good quality joint obtained using Ticusil® filler material. Further studies on brazing with silver-free filler materials (suitable for neutron environment) are underway.

Collaborative researches with other organizations are also actively pursued by IPR. In this regard, National Aerospace Laboratories (NAL, Bangalore) and Non-Ferrous Materials Technology Development Centre (NFTDC, Hyderabad) are already contributing to the development of technologies for fabrication of plasma facing components. Indigenous development of tungsten based plasma facing materials is also presently being discussed with some organizations.

Helium gas cooled plasma facing components is a new technological area that will be taken up at IPR in near future.

Figure A: Graphite to Copper joining studies using EB-PVD

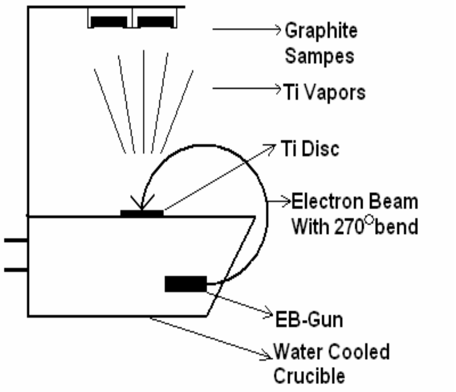

Figure 1: Schematic of EB assisted Physical Vapor Deposition of Titanium on Graphite

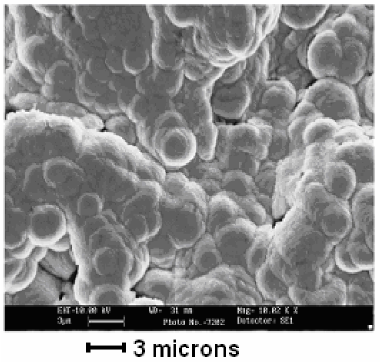

Figure 2: SEM image of pure titanium deposition (of thickness 3.6 microns) on Graphite surface.

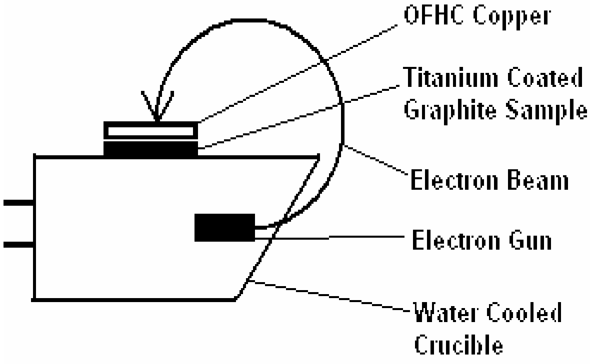

Figure 3: Schematic of EB assisted casting of OFHC copper on Titanium coated Graphite

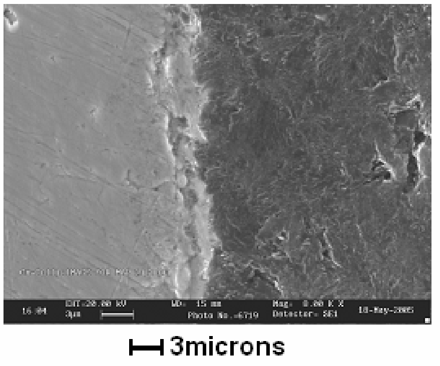

Figure 4: SEM Photograph showing Carbon-OFHC Cu Joint interface.

Figure B: Graphite to Copper alloy joining studies using vacuum brazing

Figure 5: Vacuum brazing furnace at IPR

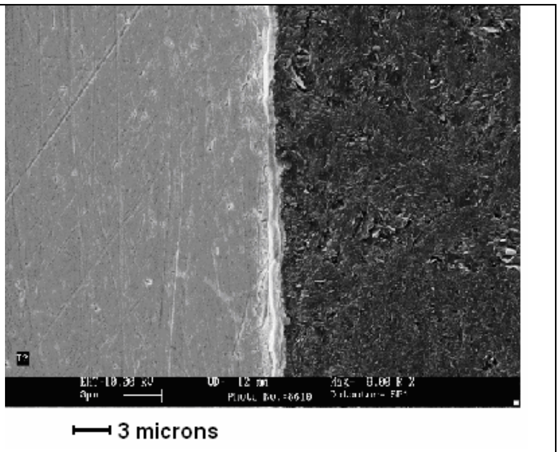

Figure 6: Graphite to CuCrZr copper alloy joint obtained using Ticusil® brazing filler material

Acknowledgement: The author would like to thank Dr. Raole, his team and the concerned members at FCIPT, IPR, for their contribution in the characterization of the joints.

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1 work done in collaboration with Laser and Technology division, BARC, Mumbai

Plasma displays and research at Samtel Color Limited

Mr. Suraj K Sinha is an experienced scientist from m/s samtel color ltd.

In the last 25 years, the technology has managed to reach Indian villages and has considerably changed the life of common man. In mid 80es to mid 90es, two famous serials broadcasted on the Indian television (TV) viz. Ramayan and Mahabharata made people frenzy to have a Cathode Ray Tube (CRT) based TVs. This CRT boom was followed by invasion of desktop computers in mid 90es to 2005. And in the last 5 years laptops and mobile phones have been in vogue. Following the suit, Flat panel HDTV is going to be rage for the next 10 years in India and Plasma Displays are one of the competing technologies for the large screen Flat Panel displays.

A **plasma display panel (PDP)** is a type of flat panel display and is common to large TV displays (32 inch or larger). In PDPs, many tiny cells located between two panels of glass hold an inert mixture of noble gases as shown FIG. (b). The gas in the cells is electrically turned into a plasma which then excites phosphors to emit light, the typical dimensions and discharge are shown in FIG (a). Plasma Displays work by applying a voltage between 2 transparent display electrodes on the front glass plate of the display. The electrodes are separated by an MgO dielectric layer and surrounded by a mixture of neon and xenon gases. When the voltage reaches the ‘firing level’, a plasma discharge occurs on the surface of the dielectric resulting in the emission of ultra violet light. This UV light then excites the phosphor at the back of the cell and emits visible light. Each cell or sub-pixel has red, blue or green phosphor material and 3 sub-pixels combine to make up a pixel. The intensity of each color is controlled by varying the number and width of voltage pulses applied to the sub-pixel during a picture frame. This is implemented by dividing each picture frame into sub-frames. During a sub-frame, all cells are first addressed – those to be lit are pre-charged to a specific address voltage – then during the display time the display voltage is applied to the entire screen lighting those which were addressed. Each sub-frame has a weighting ranging from 1 time unit to 128 time units for a typical eight sub-frame arrangement (Time Unit depends on size and number of pixels on the screen). This is a purely digital Pulsed Width Modulated control mechanism, which is a key advantage as it eliminates any unnecessary digital to analogue conversions, making the PDP technology ideal for the all-digital age. PDP have following features:

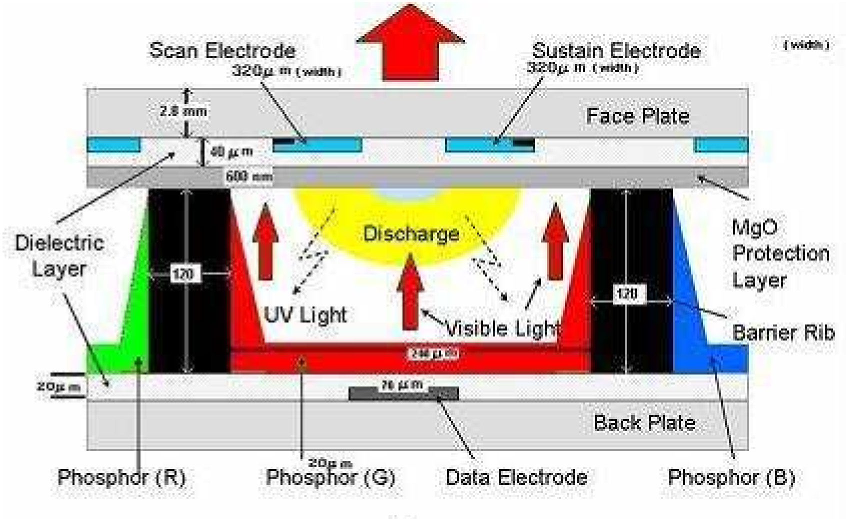
- Brilliant picture quality
- Fully flat, large screen formats
- Thin (40mm) – suitable for wall hanging
- 1.07 billion (10bit / [RGB]) colors for natural color reproduction

- High brightness, high contrast
- Wide viewing angle 160° – in all directions
- Fully digital internal operation
- Light weight – 1/6 th of CRT
- Unaffected by magnetic fields
- Fully flicker-free operation
- High resolution

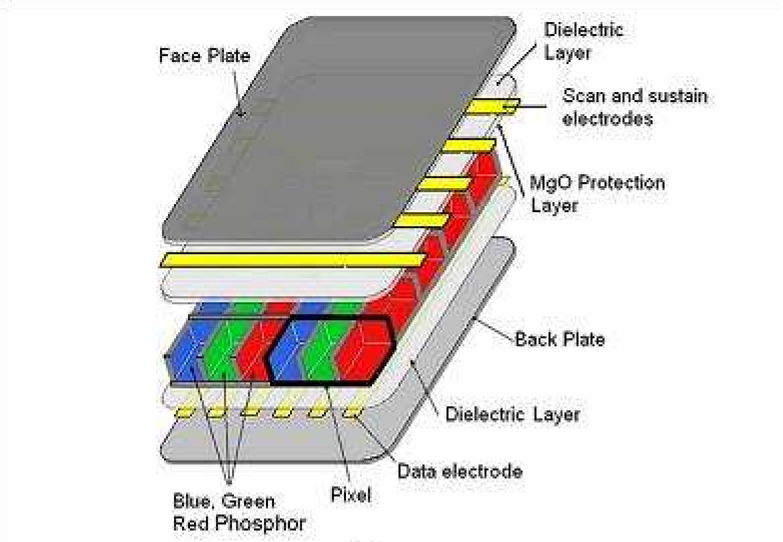
Apart from these outstanding features, however, PDP is a low efficiency device. In PDP, only ~ 1 % of the total applied power is converted into visible light. Achieving high luminous efficacy is the main challenge for the PDP technology. Intense investigation on basic discharge phenomena and related issues is required for the development of next generation PDPs with high luminous efficacy, low cost and larger size. The basic challenges are ion heating reduction, maximum utilization of electron energy, improvement of VUV to visible light conversion efficiency, response time reduction, new structure development etc.

SAMTEL, a pioneer and the largest manufacturer and exporter of cathode ray TV picture tubes in the country, has already initiated efforts in the area of flat panel displays, having developed the basic technology for Standard Definition (SD) PDP at Samtel Technology Labs at Ghaziabad, Uttarpradesh, India. The R&D efforts, in the field of PDP, at Samtel are being carried out over last seven years with the main focus being on the development of basic technologies. SAMTEL has established its capability in gas discharge physics (for basic design of pixels/cells), thin film technology (for coating of 600nm thick emissive layers on large glass plates), printing technology (for deposition of electrode structures of micron size thickness and width), patterning (using photolithographic techniques on large glass plates), assembly and processing of PDP, discharge characteristics of single pixels/group of pixels under different voltage conditions and driving systems. This has been demonstrated in the form of development of a 42” SD PDP laboratory model. Encouraged with the results of 42” SD PDP and with realization of need of breakthroughs in this technology, SAMTEL considers that it is imperative for the country to make a determined bid to become a significant player among the international (South Korean & Japanese) players in the field of PDPs. Towards this end, SAMTEL envisages to take up **Development of Next Generation Plasma Display Panel (PDP) Technology** in a comprehensive way, working on the various unit processes, optimizing and integrating these towards development of a 50” HD (1366 x 768 pixels) PDP TV demonstrator of Luminous Efficacy ≥ 5 lumens/watt with life times of 60,000 hours and using eco-friendly (lead free) materials.

Performance of economy is heavily dependant on industrial growth and manufacturing is considered as the engine of industrial growth. Moreover, manufacturing of an item is defined by demand, which is solely dependant on superior, viable and low cost technologies that are available to manufacture that item. Flat panel manufacturing industry is unable to meet the strong global demand including India's, which is expected to grow further. Major international players have already invested in billions of dollars in the manufacturing of LCD TV. Moreover no Indian company could afford to buy the expensive LCD technology, which further needs to be followed by huge investments for their manufacturing. On the other hand, manufacturing cost of a plasma display is one third of that of LCD TV; and at the same time SAMTEL, IIT-Kanpur, CGCRI-Kolkata, NPL-Delhi and Allahabad University have already developed a considerable amount of expertise in the field of PDP technology. Hence we feel 'plasma displays' is a potential contender in the field of large size Flat Panel Displays, and India can boast of its own technology assisting the industries and maintaining the growth. Thus, it can be considered that Plasma Displays Technology could be an Indian technology, which would assist its Industrial growth, and to make this a viable technology industries require a much needed support from research institutions.



(a)



(b)

Equipment Delivery

Plasma Nitriding System installed at IGTR, Ahmedabad

Facilitation Center for Industrial Plasma Technologies (FCIPT) in collaboration with Gujarat Council of Science and Technology, GUJCOST has installed a 1000 mm diameter and 1000 mm height plasma nitriding system at Indo German Tool Room (IGTR), Vatva, Ahmedabad (Fig.1). The plasma nitriding system has advanced features like novel hot retort design, excellent three zone heater control with temperature uniformity together with complete PLC-PC based automation. It has state of the art features like SCADA package to provide recipe storage, recall resulting in process repeatability. The plasma nitriding system is funded by Department of Science and Technology, New Delhi to promote the awareness of clean technology of plasma nitriding process to the local industries in and around Gujarat.

The plasma nitriding system was inaugurated by Shri Rajubhai Shah, Managing Director of Harsha Engineers on 12th November, 2008 at IGTR and is now opened for job work from the industries. The function was well attended by many industrialists from Ahmedabad, Rajkot and Surat. The keynote lecture was delivered by Dr. S. Mukherjee from FCIPT. The function ended with a demonstration of the plasma nitriding process. Some of the major industries that can benefit from this process are automobile, textile and plastic die manufacturing sectors.

Fig.1 : Plasma Nitriding System installed at Indo German Tool Room, Vatva Ahmedabad

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