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**WORKSHOP ON PLASMA DIAGNOSTICS AND
INDUSTRIAL APPLICATIONS OF PLASMAS**

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***INDUSTRIAL APPLICATIONS OF PLASMAS: CASE
STUDIES IN COMMERCIALISATION***

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INDUSTRIAL APPLICATIONS OF PLASMAS: CASE STUDIES IN COMMERCIALISATION

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Abstract

The paper describes an initiative by the Institute for Plasma Research, India in establishing links with the Indian industry for developing and commercialising advanced plasma-based industrial technologies. This has culminated in the creation of a self-financing technology development, incubation, demonstration and delivery facility. A business plan for converting the knowledge base to commercially viable technologies conceived technology as a product and the industry as the market and addressed issues like resistance to new technologies, the key role of entrepreneur, thrust areas and the necessity of technology incubation and delivery. Success of this strategy is discussed in a few case studies. We conclude by identifying the cost, environmental, strategic and techno-economic aspects, which would be the prime drivers for plasma-assisted manufacturing technology in India.

1. IPR and Plasma Physics

IPR is an autonomous research institute under the Department of Atomic Energy, Govt, of India, primarily engaged in research on the science and technology of plasmas. The thrust of the institute is on fusion research, focussed on Tokamak devices. The first Indian Tokamak Aditya has been operating since 1989 and the second machine with superconducting magnets is in the engineering stage, scheduled to become operational in 2002. A wide range of fundamental and applied plasma physics problems are addressed at the institute through experimental, theoretical and computational programmes. The questions dealt with in basic plasma and fusion research are related to production and confinement of plasmas, the equilibrium and stability properties, interaction with electromagnetic fields, manipulation and control of the parameter space etc. To answer these questions, an extensive technological base of plasma sources, pulsed power, ultrahigh vacuum, magnetics, plasma diagnostics, computer-modelling etc. has been created.

The initiative on developing and commercialising plasma-based industrial technologies was taken in 1990 and has culminated in the creation of a financially self-reliant technology development, incubation, demonstration and delivery facility. This activity has brought to the surface some of the fundamental problems in indigenous development and transfer of advanced technologies to industry. In this talk, I wish to share with you some experiences and the lessons learned from this interaction.

2. Plasma Assisted Manufacturing

We realised that our knowledge base in plasma sciences and associated technologies has immense potential for near-term application. This also reflects the contemporary international perspective that the plasma science offers unique and novel opportunities in high energy density and high value added material processing.

Plasma assisted manufacturing exploits plasma as an industrial tool [1]. Plasmas are ionized gases with free electrons, ions and excited neutrals. The charged particles can respond to external electromagnetic energy fields and transport energy. The fluid properties are enhanced by the particles setting up internal self-consistent electric and magnetic fields, resulting in collective effects like flows, waves, instabilities and self-organization. Each specie may have independent

energy distribution, not necessarily in equilibrium with other species. The internal energy is composed of thermal, electric, magnetic and radiation fields, whose relative magnitudes allow the plasma state to exist in an extended, multi-dimensional parameter space.

Electrical energy can be converted to high temperature plasma in a plasma torch. Gas flowing between two electrodes is ionised and ohmically heated to temperatures more than 10,000 degrees and ejected. The conversion efficiency from electrical to heat energy is close to 80 per cent. Plasma enthalpy is independent of the oxygen potential giving oxidising, reducing or inert environment options. This plasma stream can melt or vaporise any material. High temperature gradients aid in rapid quenching and selectivity of reactions. Plasma metallurgy, synthesis of ceramics, waste pyrolysis etc. results from these properties.

Non-equilibrium plasmas can be formed in low-pressure gases by glow discharges. In such plasmas, the electrons are hot, with temperatures of the order of 10-50,000 degrees while the ions and neutrals are cold. Non-equilibrium plasmas of molecular gases are chemically reactive due to the presence of radicals, energetic electrons, microscopic electric fields and ultraviolet flux. The products of this are surface modification, synthesised films and surface coatings.

Plasma can localize electric fields in sheaths or virtual electrodes to generate high flux beams of energetic ions and electrons. These beams are used in applications like ion implantation, etching and micromachining.

A variety of physical, chemical and metallurgical transformation of materials can take place when they are exposed to plasmas. PAM [2] is an enabling technology, which integrates processes associated with plasma-material interaction with manufacturing. PAM technology adds value to conventional materials and makes new types of materials and material processing techniques possible. The striking example of a plasma product is the Pentium chip where 500 million micron sized transistors, capacitors, resistors and diodes are formed on a silicon substrate and wired together to form circuits in approximately one square cm.

3. Relevance of PAM to India

According to a recent World Bank survey, amongst all major countries, India has the lowest share in manufacturing: 25 % in agriculture, 19 % in manufacturing and 45 % in services. Even this manufacturing is concentrated in low value addition sectors in the absence of more productive, advanced technologies. India has to upgrade traditional manufacturing and value addition substantially for it to be a generator of wealth.

The world over, manufacturing is undergoing fundamental structural changes[3] due to a variety of factors. The new technologies are characterised by the following features.

- Amalgamation of research and production: perpetual innovation and incremental technological advancement due to integration of intellectual and manual labour and disappearance of the laboratory-shop floor boundary.
- Flexibility: Innovator, entrepreneur and consumer form a triad, responding to rapid change in markets, raw materials, processes and products. Cluster tooling and software driven flexible process cycles are now integrated with many manufacturing systems.
- Eco-friendliness: Ecological concerns and new concepts of industrial ecology stressing recycling and productive use of waste almost imitating nature are driving technologies which minimise raw materials and energy requirement.

Emergent technologies like plasma-based manufacturing are enabling this transformation from the smokestack to knowledge-based manufacturing.

4. Business Plan

Our approach to the task of converting the knowledge base to commercially viable technologies was to conceive technology as the product and the industry as the market. What is needed to realise this is a business plan focussed not only on the creation of the product by research and development, but also on how to market them to industry. In the absence of pre-existent models of such an activity within the constraints of a research organisations in India, the details of the business plan evolved alongwith our learning curve, although some basic elements were realised right in the beginning.

- **Market Resistance to New Technologies**

Despite the common belief that markets have insatiable hunger for new technologies, there is considerable resistance to technological change[4]. The reason is that new technologies mean additional capital investment, new work practice, new personnel etc. Even in the west, this resistance is substantial. Xerography was invented in 1937 but was accepted only in 1954; transistor was born in 1947 but became a valued product in 1956. A very recent example is the transverse flux induction heating whose principle was expounded in 1967 but was accepted as an industrial practice in 1983.

- **Entrepreneur as the Key**

We decided that the programme should be market driven to make it agile and responsive to rapid changes. This means careful selection and focussing on areas where immediate impact would be possible. We used many platforms like meetings and workshops for industry and consultants, participation in trade fairs etc. for this. A powerful tool in stimulating the market was the quarterly newsletter Plasma processing Update, which now goes to more than 2000 industries

- **Focus on Thrust Areas**

The assessment culled from our interaction with industry through various fora such as market surveys, consultants meetings and industrial exhibitions, identified the following trust areas:

Surface Engineering: Engineering goods like plastic dies/moulds, automobile parts, tools and brassware form a major part of export from India. Performance enhancement in wear/corrosion resistance, hardness and finish would be vital in competing in international markets. Technologies selected for development were Ion Implantation, Plasma Nitriding, Silicon Dioxide coating etc.

Processing of Minerals: India has large deposits of ilmenite, zircon etc. with no modern technology to process these. Thermal plasma processing like in-flight benefaction and reduction are considered important to this sector.

Advanced Ceramics: Emerging need for advanced ceramics and nanocomposites is now met entirely through imports. Indigenously manufactured low value ceramics can also benefit from property modification.

Photovoltaics: India is the fifth largest producer of photovoltaic cells. Considerable potential for technology upgradation through surface passivation, efficiency improvement exists. Emerging technologies like high frequency plasma CVD and advanced heterojunction devices would be important.

Waste Remediation: Rapid industrialisation, increasing affluence etc. are strong factors in the increase of per capita waste generation. Increasing public and judicial activism is growing against waste disposal by conventional remediation technologies.

- **Technology Incubation and Delivery**

A fourth aspect of the business plan is an effective delivery system. Industries and research institutes have divergent perception of what is expected in technology transfer. Scientists believe that proof of principle of a process or manufacturing idea is sufficient to validate technology. Industry, on the other hand, wants a finished product, ready to be put on the factory floor and integrated with the production line. Adding value to an innovation by dressing it in all the embodiments of technology, needed by the user, is essential. The embodiments are in the form of process optimisation, process equipment and instrumentation, reliability assessment, and techno-economic data. This step can be called, 'technology incubation, 'technology manufacturing' or 'technology facilitation'.

5. FCIPT

In 1997 IPR set up the Facilitation Centre for Industrial Plasma Technologies (FCIPT) to realise the above strategy and consolidate all activities related to technology development, incubation and delivery.

FCIPT has process development laboratory to exploit the areas of expertise in plasma and other allied fields of the institute in developing new plasma based technologies for the industry. It has prototypes and pilot plants covering a range of plasma technologies and utilises them for extensive job working of industrial components to generate the database on instrument and process reliability and economics. The material characterisation facility, consisting of advanced instruments, is open to users from industry, research establishments and universities. Manufacturing complete reactors is an integral part of the technology transfer activity.

In two years of operation, FCIPT has become close to financial self-sufficiency. More than 100 industries use the technology demonstration facilities for jobworking and process and prototype development. Equipment has been supplied to industries and research institutions and technology has been transferred for commercialization. The clientele for contract research and technology transfer includes multi-nationals.

6. Case Studies

I shall now describe some specific achievements, which have vindicated this strategy.

• Plasma Processing of Minerals

India has large deposits of ilmenite, zircon etc. with no modern technology to process these. We will examine how a single step plasma dissociation process of zircon sand into zirconia is viable and efficient.

Zirconia has high melting point, good chemical resistance and high strength. The typical sintered products are scissors and shears, wire drawing dies and hot extrusion dies, seals in chemical valves, automobile engine parts, bio-medical components, piezo-electric crystals and high-temperature refractories. It is used for thermal barrier coatings in turbine blades and for anti-corrosion coating in harsh environments. Stabilized zirconia and zircon is used as refractory lining in the melting vats, polishing of glass and as an opacifier in enamels.

Zircon is basically a very stable compound and to extract zirconia requires chemical or thermal dissociation. In the chemical route, zircon is fused with sodium hydroxide above 600 degree C to produce sodium silicate and sodium zirconate from which sodium silicate is removed by leaching. Sodium zirconate is hydrolysed to produce hydrated zirconia, which after calcination produces an impure oxide, which is further, purified by acid leaching. Chlorination of zircon produces zircon tetrachloride, which after hydrolysis and calcination also yields zirconia powders. Indian Rare Earths, Kerala a company that produces about 400 tons of zirconia is following the chemical route.

Thermal dissociation of the Indian Zircon sand in an in-flight reactor yielded 90-95% dissociated product regularly under optimum conditions with a specific energy consumption of 4-6 kWh/kg of zircon. The SiO₂ phase is in amorphous state and highly reactive and can be leached out in one step to yield ~95% monoclinic ZrO₂ phase.

- **Surface Engineering to Enhance Wear Resistance**

Nitrogen reacts with metals and form thermodynamically stable metal nitrides when activated chemically or electrically. Metal nitrides exhibit high hardness and wear resistance. Plasma can assist the nitriding process through both surface and volume chemical reactions. Nitrogen plasma is rich in NH radicals and excited states and reacts with metals and form thermodynamically stable metal nitrides, which exhibit high hardness and wear resistance. Nitrides can be produced as surface or thermochemical diffusion coatings. Plasma nitriding has no toxic by-products, reduces processing times with a concurrent saving in energy, reduces mechanical distortion of the components, and produces smoother finish.

The relevance of nitriding technology to India can be seen by the following example. Hydropower components like turbine runner, guide vanes, draft tube cones, check plates and labyrinth rings undergo considerable erosion because of the action of sand particles and silt. This is particularly true of the power stations tapping the hydropower potential of the Himalayan rivers, inspite of the complex de-silting procedure adapted to remove particles above 0.2 mm size. The rate of erosion is so large that the life of these components do not last more that a year. The National Hydro Power Corporation of India now spends more than \$ 2 Million annually for the replacement of these parts. NHPC has been exploring a variety of remediation techniques to protect the components from erosion. A campaign of plasma nitriding of critical hydropower components was taken up last year and the nitrided components are undergoing field trials at present.

The plasma nitriding reactor that we developed has many features like the computer controlled process automation and menu driven process cycle, which make it comparable to the best reactors available in the international market. The reactor technology had to be supplemented with extensive application database to make the technology meaningful to the user community. To demonstrate indigenously developed pulse plasma nitriding technology to the user industry in a technically convincing manner and convince them on the commercial viability of nitriding, a commercial scale reactor capable of nitriding industrial scale components in large quantities, was set up. This also yielded techno-commercial data necessary for commercialization.

An Ahmedabad based heat treatment Company was supplied a large plasma nitriding system specifically designed to treat screws and barrels. Specialized research systems are also being delivered to National Aerospace Laboratories and the Indira Gandhi Centre for Atomic Research. The innovation cycle was completed with the licensing of know-how for the manufacturing of Plasma Nitriding Systems to Fluidtherm Technologies, a Chennai based company.

- **Plasma Chemical Vapour Deposition Coatings**

The general area of metal finishing using galvanic processes is being phased out because of the use of such materials as zinc and chromium, which are considered environmentally hazardous. Plasma based processes like physical and chemical vapour deposition are beginning to be competitive with galvanic processes.

Brassware and aluminised automobile reflectors lose their shine due to oxidation and corrosion when exposed to atmosphere. Conventionally, lacquer is coated to protect the surface from corrosion, which offers only a temporary reprieve. An ideal solution would be to deposit transparent, non-reactive, corrosion resistant and scratch-proof glass coatings on surfaces.



Glass-like coatings are thin films deposited using plasma enhanced chemical vapour deposition (PECVD) technique. The technique involves introduction of the required monomer into a plasma. The monomer dissociates and releases silicon leading to formation of Si-O chains, which deposit as glass-like coating on substrates. The coating consists of highly coherent, crosslinked, pinhole free Si-O-Si bonds and has good adherence to the substrates. The coating is uniform and provides excellent corrosion resistance to the components coated. Solvents like acetone, petroleum ether, water, dilute acid and base have no effect on the coating.

This technology is also supported by a jobshop and extensive database for specific applications.

- **Efficiency Improvement of Solar Photovoltaic Cells**

We had taken up a joint project with BHEL to develop a process to synthesise plasma polymerised silicon oxynitride films to use as anti-reflection/passivation layers for silicon solar cells using a safe organo-silicon precursor. We have been able to optimise process parameters so as to reduce carbon content significantly (< 1%) in the films. An improvement in the efficiency of the solar cells is obtained.

Silicon oxynitride films were grown on textured silicon solar cells made by BHEL (Electronics Division), India, with a thermal oxide layer of approximately 400 angstroms. Thickness of the film is found to be about 860 angstroms. This implies a deposition rate of nearly 15 Å per minute. The chemical composition of the film is $\text{SiO}_{1.85}\text{N}_{0.1}$.

- **Plasma Pyrolysis of Hospital Waste**

Public concern over disposal and treatment of medical waste has resulted in an increase in regulations and court actions. The basic reason is the phenomenal growth in the quantity of medical waste generated in the hospitals, attributed to the growing use of disposable, as precautions against exposure to infectious diseases and in general to the growth of medical and public health facilities. A rule of thumb for medical waste production in India seems to be 1 kg per bed per 8-hour shift.

Safe disposal of medical waste is a critical problem. Landfilling is easy and cheap; positive correlation with groundwater contamination has made this option unacceptable. Burning the waste in open air can never be complete, with small quantities of many organic and chlorinated organic compounds as well as pathogens surviving and leading to dispersal of dangerous diseases through air. Incineration is currently used to destroy biomedical waste. The essential problem of conventional incineration is that the generation of heat is closely coupled to the reaction chemistry. Oil fired incineration produces harmful products due to insufficient temperature in the process chamber. This can cause air pollution or the toxic pollutants can remain at the bottom ash, eventually finding way into landfills.

In plasma pyrolysis, electrical energy is converted by a plasma torch to a plasma stream with temperatures more than 10,000 degrees. When applied to medical waste the intense plasma heat causes the molecules to disintegrate forming fragments of compounds. The highly reactive plasma environment can catalyse homogeneous and heterogeneous chemical reactions. When optimized, the most likely compounds to form from carbonaceous matter are methane, carbon monoxide, hydrogen, carbon dioxide and water. These gases are burnt in a secondary chamber at a temperature of the order of 1200 degrees. Hot byproduct gases are scrubbed and quenched in the quenching system. The recombination of the plasma produces intense ultraviolet radiation, which can destroy pathogens completely. This is an advantage unique to plasma pyrolysis.

Plasma pyrolysis has many advantages over conventional incineration:

- The quantity of toxic by-products formed is much below the emission standards
- Waste can be dry or wet
- Segregation of hazardous waste is not required

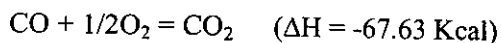
- Recovery of energy in the form of CO and H₂ is possible.

The composition of the medical waste varies depending on the source, but the average seems to fall in the following range:

Paper and Cloth items : 50 –70 %, Plastics : 20 –60%, Metal, glass, inorganic : 7 –20%, Fluids : 1 –10%, Moisture : 8.5 – 17%

The elemental composition of the waste consists primarily of C, H, O, N, F, S and Cl. The gaseous by-products in pyrolysis include CO, SO₂, NO_x, HCl, HF, CO₂, C_xH_y, H₂ and particulates. The waste stream has an overall Heating Value of 7500-8500 Btu/lb.

The electrical energy through plasma is consumed in melting of plastics, bond dissociation and in endothermic reactions. Recovery of these gases will render this process economically attractive. The reactions producing gases in the form of [CO + H₂] are most probable at high temperatures. When the CO and H₂ gases are combusted in the presence of oxygen, the following exothermic reactions take place to liberate a specific quantity of energy in the form of heat and light:



$$\text{Total } \Delta\text{H} = -135.95 \text{ Kcal}$$

This implies that 135.95 Kcal energy is obtained by the combustion of one mole of CO gas and one mole of H₂ gas. Translating this, for example, combustion of 14 grams of polyethylene, at the rate of 860 Kcals /Kg, requires 12.04 Kcals of energy to generate 1mole each of CO and H₂ and yield 135.95 Kcals of energy which is more than 11 times the input.

For 100 kW systems, the specific energy consumption is around 0.7 units per kg. As the size of the system increases, this goes down to as low as 0.5. We expect the capital cost of a 25 kg/hr system to be around Rs. 1 Million. The operating cost including manpower, electricity, raw materials and maintenance is calculated to be approximately Rs. 8 per kg. These compare very favourably with electrical and oil fired incinerators.

7. The Next Step

FCIPT is at present a division of IPR; this presents the following problems:

- The necessity for knowledge to be treated as a commercial entity conflicts with the ambience of research laboratories where pursuit of pure knowledge is an end in itself.
- The peer pressure in the institute is to publish results, while protection of commercial interests demand secrecy and legal procedures like patenting, which delays publications.
- Confidentiality essential in interaction with industry cannot be fully maintained in an environment, which promotes openness.
- Meeting industry schedules demand highly time-sensitive work culture which conflicts with a generally relaxed atmosphere of the research institute.
- The commercial activity means compliance with statutory formalities like taxes, duties etc., which are alien to the accounting procedure of institutes.

It is important to think of an appropriate organizational structure, which will accelerate the pace of technology generation and commercialization. We believe that the solution to these problems is the creation of a corporate entity like an R&D Company in the company's act, but with an important modification that they should be allowed to manufacture process equipment. Alternatively, it can be a Section 25 company promoted by IPR.

The Unique Selling Points of such a company would be:

- **Competition Drivers:** An IPR-industry partnership can yield truly innovative, internationally competitive technologies. An example is the plasma polymerised anti-reflection coating and plasma sterilization.
- **Cost Drivers:** The lower cost of indigenously engineered system yield a price advantage. Ensuring higher up-time due to local availability of trouble shooting and maintenance is another positive feature of indigenous equipment technology.
- **Environmental Drivers:** The green initiative in industry means that solvent-based wet processing is to be replaced in five years. The need for hazardous waste minimisation will stimulate need for plasma based technologies.
- **Strategic Drivers:** many surface-engineering and thermal plasma technologies are considered dual-use and hence are on the restricted export list.
- **Techno-economic Drivers:** Plasma-based industrial technologies have the attractive features of high value addition. Many steps in conventional processes can be integrated into a single step. Alternately, a single reactor can be flexible enough to manufacture a variety of products.

8. Conclusions

We have shown that it is possible to develop and transfer advanced plasma-based technologies to industry by following a roadmap, which recognizes various factors inhibiting such an activity. This has culminated in the creation of a self-financing technology development, incubation, demonstration and delivery facility called the Facilitation Centre for Industrial Plasma Technologies. The business plan for converting the knowledge base to commercially viable technologies conceived technology as a product and the industry as the market and addressed issues like resistance to new technologies, the key role of entrepreneur, thrust areas. The critical components of this activity are technology development, incubation, demonstration and delivery. Concurrent to this are functions, such as generation of database on process, application, techno-commercial aspects and reliability. Indigenous development of relevant instrumentation pertaining to plasma, power electronics, vacuum and process automation is crucial to the credibility of technology transfer. Success of this strategy is discussed in a few case studies.

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