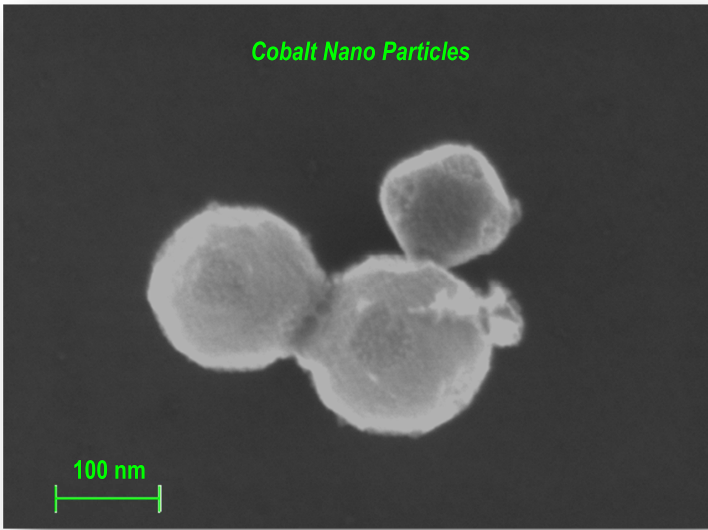
Plasma Processing Update

A newsletter from

Facilitation Centre for Industrial Plasma Technologies
Institute for Plasma Research

Issue 71: Jan – June 2015



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Editor's Note

In the Jan 2015 issue of Plasma Processing Update, we discuss about Plasma Carburizing Process Development at FCIPT, Synthesis of Cobalt Nano Particles' and 'Surface Modification of Merino Wool' by plasma methods. Preliminary experimental results are encouraging and may lead to potential application development in the field of Steel industry, Nano Technology and Textile industry.

Mrs. Alphonsa Joseph discusses in detail about Plasma Carburizing process development at FCIPT. A prototype setup for this process has been developed for the first time in India.

Mrs. Nisha Chandwani talks about plasma surface modification of Merino Wool fibers at FCIPT by atmospheric pressure plasma treatment. She discusses about the properties and limitations of Merino wool fibers and how plasma treatment helps to overcome those limitations. The intial results are quite promising and we expect the textile industry to take benefit of this emerging eco-friendly technology for wool processing.

Ms. Prachi Orpe explains about potential industrial applications of Plasma synthesized Cobalt Nano Particles. She also describes about various physical properties which are associated with nano particles and how they are important for certain applications.

In addition to these articles, this issue speaks about recent news and events such as a Book Launch by our scientists under title 'Plasma Technologies for Textile and Appeal', Installation of 'Atmospheric Pressure Plasma Process System for Angora Wool' at SHHDC, Sikkim and Technology Transfer of 'Plasma Pyrolysis'.

Do stay in touch with us on www.plasmaindia.com.

Yours sincerely,

Dr. S. Mukherjee

Editor

Co-Editors: *Mrs. Purvi Dave & Mrs. Nisha Chandwani*

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ABOUT FCIPT

Facilitation Centre for Industrial Plasma Technologies

Institute for Plasma Research is an autonomous R & D organization under the authority of Department of Atomic Energy (DAE), Government of India. This institute is largely involved in theoretical and experimental studies in plasma science including basic plasma physics, magnetically confined hot plasmas and plasma technologies for industrial application. The institute owns two operational tokamaks (a machine for controlling thermonuclear fusion) - ADITYA and Steady State Tokamak (SST) - 1. FCIPT, ITER-India and CPP-IPR, located in Gandhinagar and Guwahati are three divisions under IPR.

The Facilitation Centre for Industrial Plasma Technologies (FCIPT) links the Institute with the Indian industries and commercially exploits the IPR 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 important areas in which FCIPT has been working on include Plasma Surface Engineering, Plasma Pyrolysis/ Gasification/ Energy Recovery, Plasma Diagnostics, Plasma Based Nano Patterning and Nano Synthesis, Textile Engineering, Solar Cell Development, etc. This newsletter is designed to update the readers with the latest developments in the important field of plasma processing and plasma based technology development and to look for new industrial opportunities. FCIPT's plasma processing expert team will be happy to interact with the industries and come up with the eco-friendly plasma based industrial solutions.

Plasma Carburizing Process- Development at FCIPT

Mrs. Alphonsa Joseph

Carburizing is the addition of carbon to the surface of low carbon steels at temperatures generally between 850-950 °C. Surface hardening is accomplished when the high-carbon surface layer is quenched to form martensite so that a high carbon martensite case with good wear and fatigue resistance is superimposed on a tough, low carbon steel core.

Carburizing steels for case hardening usually have base carbon contents of about 0.2%. After carburizing the carbon content of the carburized layer generally lies between 0.8 - 1 %C. Carburizing can be done by introducing carbon by use of atmosphere (Gas, Plasma and Vacuum), Liquid (Salt bath) and solid compound (Pack). Amongst these processes, vacuum and plasma carburizing utilize a vacuum chamber with a partial pressure of hydrocarbon gas as a source of carbon thereby eliminating the presence of oxygen in the furnace atmosphere. Plasma carburizing process is superior compared to vacuum carburizing and gas carburizing process in terms of low gas consumption and low processing times. For eg., a case depth of 100 microns is achieved in 9-10 hrs after gas carburizing and the same in 8 hours after vacuum carburizing process.

Though plasma carburizing has been discovered in 1934, in the last decade much effort has been devoted to the development of a plasma carburizing process. In India, plasma carburizing process has not yet been developed and efforts were made to develop a prototype plasma carburizing system in FCIPT with the assistance of Department of Science and Technology, New Delhi.

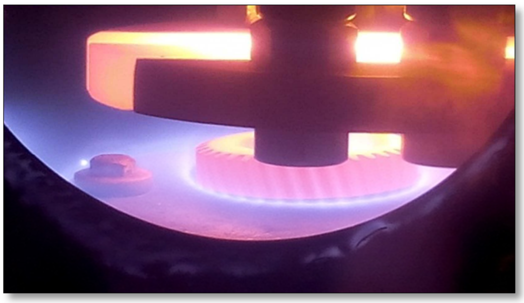
At FCIPT, a plasma carburizing process was developed using acetylene gas which is cheap compared to methane gas usually employed in plasma carburizing process. The plasma carburizing system is shown in fig. 1.

Fig. 1: Plasma carburizing system installed at FCIPT

This system consists of a vacuum chamber made of stainless steel. It consisted of heaters that that can go to maximum temperature of 1050 ^{o}C. Below the vacuum chamber is the quenching chamber which is filled with quenching oil. These chambers are separated by a gate valve. Plasma carburizing is usually carried out at 950-1050 ^{o}C. The substrate holder is given a negative bias and the vacuum chamber is given a positive bias. Samples of SAE 8620 are placed on the substrate holder and heated to 950 ^{o}C. During the process of heating the entire samples became red hot as shown in fig. 2a.

After attaining the required temperature, a gas mixture of acetylene and hydrogen in a fixed ratio are introduced in to the chamber via mass flow controllers. The pressure is maintained between 5-10mbar. Plasma is generated using a pulsed DC power supply having a rating of 20kW. Plasma was formed uniformly on the sample indicating diffusion of carbon ions into the sample as shown in Fig. 2b. After the required duration the voltage is decreased and in the absence of plasma the samples are heated to 850 ^{o}C for a required time to allow carbon to diffuse to higher depths. This process is known as diffusion process. Thereafter the gate valve is opened and the sample is dropped in the quenching chamber containing oil.

Fig. 2: (a) The sample became red hot during the heating process.



(b) Plasma surrounding the samples during plasma carburizing process

The micro hardness profile as shown in fig. 3 measured with Vickers hardness tester indicates that the surface hardness has increased to 780HV from the core hardness of 500 HV. The depth of hardening is defined as the depth where the hardness is 550HV. The structure is then 50% martensite and 50% pearlite. The case depth measured is 1000 microns. There is a gradual decrease in hardness from the surface towards the core. The increase in hardness is attributed to the increase in carbon concentration at the surface and the formation of Fe_3C phase during the plasma carburizing process.

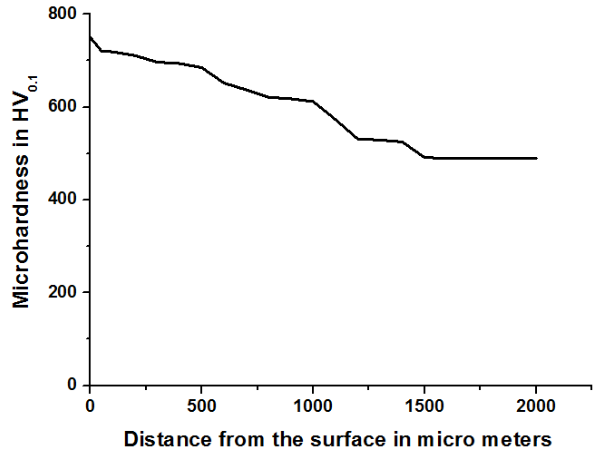


Fig. 3: Micro hardness depth profile of plasma carburised AISI 8620 sample for 0.5 h followed by 2 h diffusion process.

Plasma carburizing is mainly used for applications where hardness, wear resistance and toughness of the surface as well as toughness of the core material are required. Examples are automobile machine parts, such as axles, bolts and gears. From the results we are able to conclude that the plasma carburizing process is able to provide case depths at faster rate as compared to that obtained by the conventional gas and vacuum carburizing processes.

Atmospheric Pressure Plasma Processing of Merino Fiber for Improving Anti-Felting Properties

Mrs. Nisha Chandwani

Wool, which is one of the natural fibers, has a number of advantageous characteristics, such as durability, high tensile strength, good insulation and flame resistance. However, one of the drawbacks of wool fiber is felting. Felting is the process of the entanglement of fibers, when exposed to aqueous medium. Felting is the process of the entanglement of fibers, when exposed to aqueous medium. The felting results in shrinkage and pilling of woolen products, while laundering. Wool fiber consists of multiple layers with unique properties. A schematic diagram of wool fiber structure is shown in Figure1.The outer-most layer of wool fiber called cuticle is hydrophobic and scaly in nature. The scales are overlapping each other like tiles on a roof; they have raised edges and ridges. When fiber is immersed in water, each layer absorbs water more or less and swells. The outer layer being hydrophobic swells less as compared to inner layer cortex. This difference in expansion leads to uprising of scales and thus entanglement of fibres.

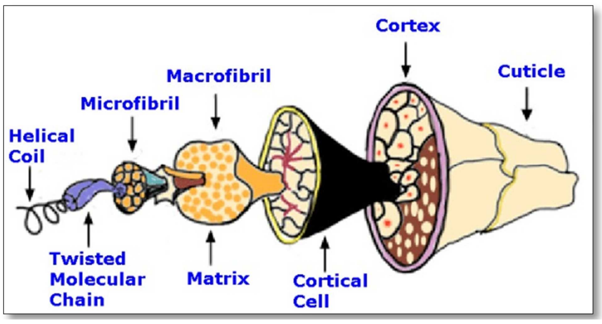


Fig.1: Schematic Diagram of Wool Fiber Structure

Presently, the most common method used to confer dimensional stability to woolen garments is chlorination/Hercosette treatment. This process uses large amounts of water as well as dangerous substances, which leads to significant wastewater pollution with organic halogen compounds (AOX) and exceed the maximum permitted limit stated by many countries. Hence there is a need of completely AOX free shrink-proofing treatment for wool.

FCIPT has explored an eco-friendly alternative to chlorination process, by way of Atmospheric pressure plasma processing of Merino wool fibers for shrink proofing application. A Dielectric Barrier Discharge (DBD) system has been used for wool fiber surface modification. The schematic diagram of experimental set-up is shown in figure 2. The plasma is generated in air at atmospheric pressure, between a pair of cylindrical Electrodes separated by about 2mm gap. A high voltage high frequency AC source is used to generate discharge. The Merino wool fibers are exposed to plasma for few seconds.

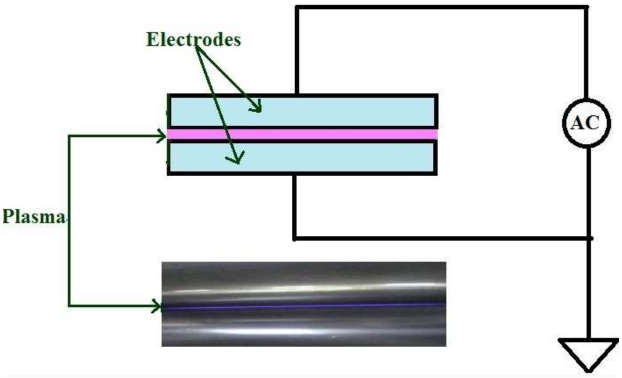


Fig.2 Schematic Diagram of DBD experimental set-up & Discharge

The surface morphology of the fibers is studied by SEM (Scanning Electron Microscope). . The SEM image of untreated and plasma treated fibers is shown in figure3. The untreated fiber has scales with sharp edges, while the edges of plasma treated fiber have been smoothened. This helps in reducing the directional friction and hence the felting. Also, some nano-scale roughness pattern is induced on the surface of plasma treated Merino wool fiber. The nano-scale roughness pattern results in higher surface area and therefore gives better wettability and dye-uptake properties.

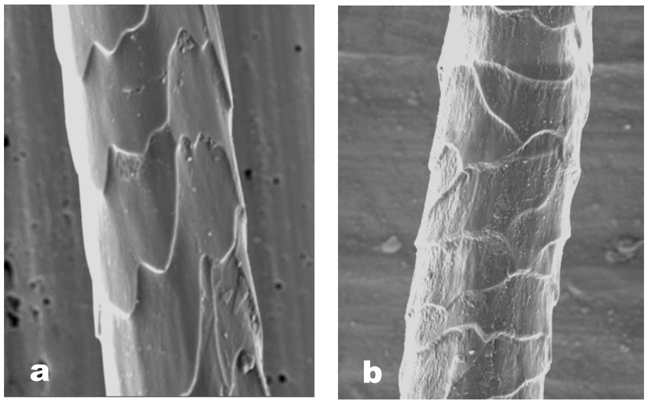


Fig. 3 SEM Image at 5000x Magnification (a) Untreated (b) Plasma Treated Fiber

We have used FTIR (*Fourier Transform Infrared*) Spectroscopy , to study the surface chemistry of the wool fiber. The absorbance ratio of various species as compared to Amide band is shown in Table1. It can be seen, there is an increase in absorbance ratio of Bunte's salt, Cysteic acid and Cysteine Monoxide, after plasma treatement. When wool fibers are exposed to plasma, the disulphide bonds (-S-S-) in the cuticle are broken. Also fiber surface is oxidised. This results in formation of bunte's salt. Also protein-cysteine is converted to cysteic acid. Cystine monooxide and Cystine dioxide are inter-mediate products when cysteine is converted to cysteic acid. The Bunte's salt helps in improvement of shrink resistance properties of wool. The cysteic acid along with the Bunte salt provides polar surface, which improves wettability of the wool fabric. Cystine monooxide and Cystine dioxide being more reactive, provide suitable sites for dyes and softeners.

Sr. No	IR Peak cm^{-1}	Species	Group	Absorbance Ratio	
				Untreated	Plasma Treated
1	1020	Bunte's salt	(-S-SO3)	0.38	0.98
2	1043	Cysteic acid	(-SO3-)	0.53	2.08
3	1076	Cystine Monoxide	(-SO-S-)	0.51	1.00
4	1121	Cystine Dioxide	(-SO2-S-)	1.1	0.71
5	1232	Amide III	(-N-H)	1	1

The change in wettability of fibers surface is observed using Potassium Permagnate solution (KMnO4).The untreated and plasma treated fibers are dipped in KMnO4 solution for 30 minutes and afterward fibers are allowed to dry in ambient air. The photograph of untreated and plasma treated dried fibers is shown below. The color of plasma treated fibers is much darker as compared to untreated fibers,it means more number of KMnO4 molecules have adsorbed at the surface of plasma treated Merino wool . This indicates the wettability of fibers has improved significantly after plasma treatement.

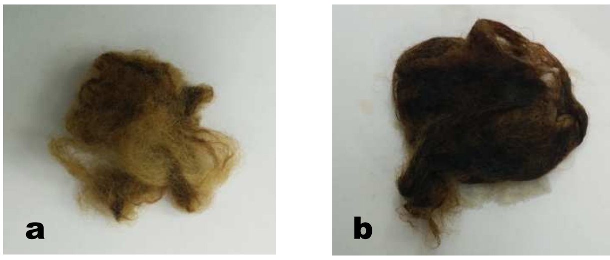


Fig.3a: Untreated Merino wool fiber, 3b: Plasma treated Merino wool fiber

The shrink resistance of wool fibers have been tested using Aachen felt ball method. The test have been carried out at Wool Research Association (WRA). The felting density of untreated fiber is found to be approximately 0.12 gm/cc while plasma treated fiber has a value of about 0.10 gm/cc. This indicates there is significant improvement in shrink resistance properties after plasma treatment. This may further be improved by application of enzyme, resin or bio-polymer

From the preliminary experiments , we can conclude that atmospheric pressure plasma can modify the surface properties of Merino wool within few seconds for improved shrink resistance and dye-uptake.

Synthesis of Cobalt Nanoparticles by plasma process

Ms. Prachi Orpe & Ms. Akshaya. B

Nanometer-scale materials with the size of 1 to 100 nm have attracted considerable interest in recent years [1]. Cobalt is a Block D, Period 4 element. Cobalt nanoparticles possess magnetic properties, which lead to its applications in various emerging areas such as;

- Medical sensors
- Biomedicine as a contrast enhancement agent for magnetic resonance imaging (MRI)
- Site-specific drug delivery agents for cancer therapies

- Coatings, plastics, nanofibers, nanowires, textiles, and high-performance magnetic recording materials
- As a magnetic fluid - made of iron, cobalt, nickel and its alloy nanoparticles
- Microwave-absorption materials

Conventional methods for synthesis of Cobalt nano structures are mostly low temperature based. This route is mainly adopted to synthesize highly stable magnetic nanoparticles. However, synthesizing nanoparticles using high temperature route has many benefits over the low temperature chemical routes.
At FCIPT, IPR we have synthesized, cobalt based magnetic nanostructures using commercially available cobalt powder (particle dimension< 150 micro meter) from Sigma Aldrich by thermal arc plasma method under ambient atmospheric conditions. Schematic of the system is shown in fig.1.

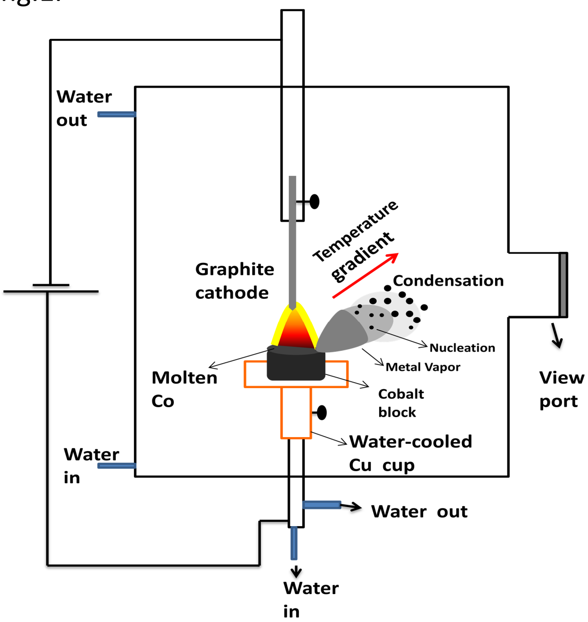


Fig.-1 Schematic of Plasma Processing system

This high temperature method, unlike conventional low temperature synthesis methods, will give rise to thermal stresses and consequently affect the local atomic ordering/disordering. The magnetic character is predominantly dependent on the size and shape of the material. We have obtained cobalt nano particles in the range of 40- 100 nm. Fig. 2 shows HR-SEM image of plasma synthesized cobalt nano particles at FCIPT, IPR. Detail study in this topic is still going on.

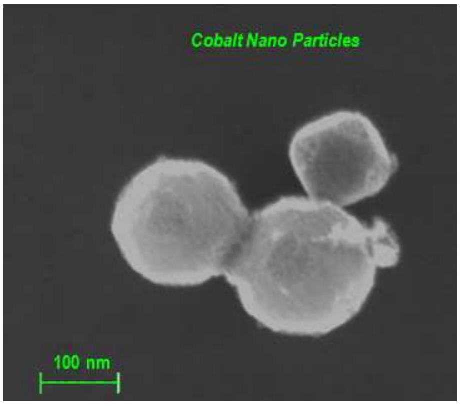


Fig.2 HR-SEM Image of Cobalt Nanoparticles produced at FCIPT, IPR by thermal plasma process.

OTHER NEWS

Technology Commercialization

Dr. Nirav Jamnapara
In charge- Technology Commercialization Cell, FCIPT

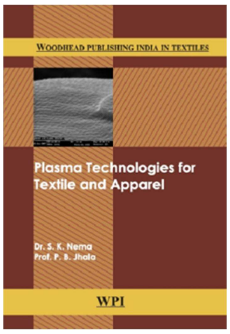
Dr. Nirav Jamnapara tells about recent technology commercialization at FCIPT, IPR. Plasma pyrolysis is an environment friendly technology for safe disposal of organic and biomedical waste. It uses extremely high temperatures of plasma arc in oxygen starved environment to completely decompose waste material into simple molecules.

FCIPT, Institute for Plasma Research (IPR) has given Plasma Pyrolysis technology rights to B.L. Engineering, Ahmedabad for manufacturing of Plasma Pyrolysis Plants for disposal of organic waste. For commercial exploitation of the technology, an agreement has been signed between B.L. Engineering and Institute for Plasma Research on 18th March 2015, at board room- IPR.

Prof. Dhiraj Bora, Director IPR and Mr. B.L..Suthar, B.L.Engineering on the occasion of signing of technology transfer agreement.

Book Release

A book titled “Plasma Technologies for Textile and Apparel” by Woodhead publishing India, has been **released on 10th Dec 2014 by Mr. Saurabh Patel, Hon'ble Minister of Finance, Environment & Petrochemicals, Govt. of Gujarat** , during the Inaugural session of INTEXCON 2014 .The book has been edited by **Dr.S.K.Nema, Sr. Scientist, FCIPT**, **IPR** and Prof. P.B.Jhala, Research Advisor, NID.



Book Cover Page

Book released by Shree. Saurabh Patel

Dr. S. K. Nema and Prof. Jhala in the audience

This Book’s main objective is to popularize plasma based technologies in textile industries and dissemination of knowledge gained over the years by Indian Institutes and organizations in the arena of plasma based applications for textiles. The book describes basics of low temperature plasma production in vacuum as well as at atmospheric pressure and various applications of plasma in textile particularly in Indian context.

Book is available at:
Woodhead Publishing India Pvt. Ltd.
303, Vardan House, 7/28 – Ansari Road,
Garyaganj, New Delhi, India.

Plasma System Installation

FCIPT has developed and disseminated an atmospheric pressure plasma processing technology for surface modification of Angora wool, which has made possible to weave 100% Angora Garments.

Recently, an atmospheric pressure plasma processing plant has been installed and commissioned at Sikkim Handicraft & Handloom Development Corporation (SHHDC), Sikkim. The project has been funded by Department of Science & Technology. The plasma system has been synchronized with the carding machine and the processing speed of the system is about 4m/min. FCIPT Engineers have given training to SHHDC personnel's for operation and maintenance of the plasma system. The plasma processing plant would be beneficial to Angora wool farmers and weavers in Sikkim region. SHHDC shall provide the service to weavers on chargeable basis.

Plasma System installed at SHHDC

Seminar / Workshop

A one day seminar on "Industrial applications of Plasma based Technologies", was held in association with Gujarat Council on Science and Technology (GUJCOST) and ASM International, Gujarat Chapter on 27th March 2015 at AMA, Ahmedabad. Dr. Neeraj Sharma (Advisor and Head TDT) and Mr. Ravinder Gaur from DST and Professor Dhiraj Bora (Director-IPR) attended the seminar.

Inaugral talk by Professor Dhiraj Bora (Director-IPR)

The seminar was well attended by enthusiastic 40 participants from different industries mainly automobile, heat treatment, textile, tool and die, waste management industries and environmental sectors. The talks were delivered by scientists and engineers from FCIPT. A wide spectrum of talks ranging from surface modification by plasma based coatings, waste destruction by plasma pyrolysis, nanopatterning and plasma processing on textiles were covered during the sessions. In the concluding session, there was an appreciable response from many industrialists showing interest in many of the technologies.