A training session report on Analytical Scanning Electron Microscopy

Under STUTI program funded by DST.



Co-ordinated by

Prof. Pradipta Ghosh Prof. Emila Panda

Department of Materials Engineering
Indian Institute of Technology
Gandhinagar, Gujarat

25th April to 1st May 2022

Acknowledgement

We convey gratitude for the encouragement and support received from multiple sources

during the execution of this training since its beginning. First and foremost, I want to express

my sincere appreciation to the Department of Science and Technology (DST) for entrusting

me with this project.

The workshop was coordinated by Prof. Pradipta Ghosh and Prof Emila Panda. The

workshop was conducted on the analytical scanning electron microscope funded by the FIST

program (Project No.: RES/DST/MSE/P0068/1718/0046 dated 18-03-2018). Organizing

team acknowledge the contributions of the training committee, in the implementation and the

execution of the program to achieve the objectives of the project, particularly, Prof. Satyam

Suwas (Materials Engineering, IISc Bangalore), Prof. Dhiraj Bhatia (Biological Engineering),

Prof. Superb Misra (Materials Engineering), Prof. Abhay Gautam (Materials Engineering),

Mr. Mangesh Kulkarni (Oxford Instruments, India), Dr. Renjith (Oxford Instruments, India),

and Mr. Pattrick (JOEL, Singapore).

I also acknowledge all the project staff and IITGN staff contributions without which these

sessions could not have been possible.

Principal Investigators

Dr. Emila Panda

Dr. Pradipta Ghosh

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Summary

The goal of this training session is to popularize analytical scanning electron microscopy (ASEM) facility among students, faculty, scientists and industry professionals through a week-long training workshop. The workshop was conducted at IIT Gandhinagar from 25th April to 1st May, 2022 and comprised of lectures and hands on training sessions. This initiative is funded by Department of Science & Technology under the program *STUTI* (Synergistic Training Program Utilizing the Scientific and Technological Infrastructure). This workshop is aimed to provide an insight into the functioning of analytical scanning electron microscope (SEM) for advanced imaging (secondary electron, back scattered electron, electron backscattered diffraction) and spectroscopy (energy dispersive spectroscopy, wavelength dispersive spectroscopy). The participants were introduced to the basic concepts of image generation, instrumentation, image analysis and the advanced modes of operation. The focus of this workshop was to have "a balance between theory and practical training on the equipment. "Emphasis is on hands-on use of equipment for demonstration/characterization by each participant and analysis of participant's samples".

Introduction

Indian Institute of Technology Gandhinagar (IITGN) conducted 7-day long workshop on DST-FIST funded instrument 'Analytical Scanning Electron' workshop in its campus for participants from various backgrounds such as Post Graduate, B.Tech., Professors, Scientists, Post-Doc Fellows, Ph.D. Fellows and Industry persons were invited (Annex-1). The following workshop's activities took place from April 25th to May 1st, 2022 (Annex-2 and 3). This report provides a quick overview of both the lecture and technical sessions.

• <u>Lecture Sessions</u>

Prof. Dhiraj Bhatia (Biological Engineering, IITGN) provided detailed information on the DST's functions and funding schemes, which are aimed on educating and encouraging scientific/industrial R&D projects in India. The goals and objectives of the STUTI workshop was presented by Prof. Pradipta Ghosh (Materials Engineering, IITGN). Dr. Ghosh also discussed the operational concepts of SEM, EDS, WDS, EBSD, and TEM, as well as gave an overview of sample preparation. Features of SEM setup and its micrographs, sample charging, matter interaction with X-rays, image formation in SE and BSE modes, and beam interactions was explained by Prof. Emila Panda (Materials Engineering, IITGN). Prof. Abhay Raj Gautam (Materials Engineering, IITGN) gave a discussion about the various types of probes and detectors used in SEM setups, which play a critical role in producing high-quality images by preventing errors during sample scanning and data collecting. Mr. Mangesh Kulkarni (Oxford Instruments, India) discussed the significance of using EDS/WDS on a SEM as well as other aspects of SE imaging methods. Dr. Ranjith (Oxford Instruments, India) presented a comprehensive overview of SEM-EBSD modes and their mapping. Mr. Pattrick (JOEL, Singapore) spoke about advancements in SEM methods of evaluating specimen features, as well as the development of advanced SEM equipment. Prof. Superb Misra (Materials Engineering, IITGN) spoke on how SEM helps in answering key research problems in the field of carbon nanotubes, materials development, tissue regeneration, and materials safety. Prof. Satyam Suwas (Materials Engineering, IISc Bangalore) led an online session on critical analysis of metallurgical problems. Dr. Satyam

also highlighted a range of research challenges that are effectively solved employing EBSD methods during SEM investigation.

• Technical Sessions

All the participants were invited to visit the several characterization laboratories in the institute on the first day. Separate batches were set up on the second day to demonstrate the FIST SEM and CIF SEM setups in the institute. Various components of the SEM were also demonstrated to the participants during the laboratory presentation in order to provide an understanding of the working principle of the SEM technique. On the third day, participants were shown how to prepare samples for SEM characterization. Four distinct types of samples used to explain the procedure of sample preparation before charging into the SEM equipment such as, metallic samples, cross-sectional specimens, powder samples, biological material samples. On the **fourth day**, the participants were shown image processing labs. The purpose of this lab is to illustrate that clear and high-quality SEM images are required for a more accurate interpretation of the results. On the fifth day, participant was allowed to prepare their samples in the lab with the assistance of assigned TAs. Participants from diverse institutes and disciplines bought four types of samples for evaluation during the sample preparation session: powder samples (biological application), nano-fibers, cross-sectional samples, and thin film samples. On days six and seven, the participants' samples were characterized using the CIF SEM and the FIST SEM. Both SEMs were used to characterize a vast number of samples provided by participants in order to aid them in their research.

• Types of samples tested

During the technical session, all of the participants expressed an interest in learning from the workshop and characterized more than 25 no. of samples. In addition, it was observed that, three categories of samples were characterized utilizing FIST-SEM and CIF-SEM setups during the technical sessions: 1). Metallic samples (for microstructure analysis and mapping), 2). Thin film samples (for investigating cross-sectional as well as surface morphology) and

3). Powder sample analysis (comprises of biological and nanomaterials).

Outcome of the workshop

The workshop attracted 65 % male and 35 % female participants from 15 different institutes (Figure 1). About 46 participants enrolled for the ASEM workshop, while 35 people attended the STUTI-funded training sessions. The goal of this training event was to bring together participants from many disciplines and raise awareness of the institute's advanced facilities. Throughout the sessions, participants asked major questions regarding theoretical and practical aspects of SEM setup and its procedures. The experts gave suggestions for possible solutions and invited participants to future collaborations. Finally, the feedback from the participants was considered in the evaluation of the workshop (Annex4). The majorities of the participants were pleased with the training session and suggested that more workshops be held in the future. Few participants suggested organizing such a workshop/training session on more advanced characterization techniques.





Figure 1: Participants registered for the 'Analytical Scanning Electron Microscopy' workshop from 15 different institutes.

Annex 1: Brochure for the program.

Speakers



Prof. Emila Panda is Associate Professor in Materials Engg. at IIT Gandhinagar. Her work focusses on understanding. optimizing and fabricating coatings

for optoelectronic applications.



Prof. Pradipta Ghosh is Assistant Professor in Materials Engg. at IIT Gandhinagar. His expertise is in synthesis of nanocrystalline alloys and their microstructure

evaluation using SEM, TEM, EBSD, XRD techniques.



Prof. Abhay Gautam is an Assistant Professor in Materials Engg. at IIT Gandhinagar. He works on interface structure and dynamics of solidsolid systems. His

expertise is in TEM, SEM, material processing.

Registration & Contact Details

Interested participants must register and only selected candidates would be invited for the workshop.

For selected candidates Registration fees, local travel, Boarding and lodging will be covered by IIT Gandhinagar.

Interested participants should register using the following link: https://forms.gle/rchDKRtz9wRyhN4D9

Registration Deadline: 15th April 2022

Shortlisted candidates will be intimated by email, latest by 17th April 2022.

Eligibility criteria:

(a) Minimum qualification: Post Graduate (Science) or B.Tech. (Technology)

(b) Professors/Scientists/ Post-Doc Fellows/ Ph.D. Fellows/ Industry persons who are actively involved in

(c) Not more than 3 people from one institute

For more information:

Access: https://events.iitgn.ac.in/stuti/

Mail: stuti@iitgn.ac.in

Address: Block 5-208, IIT Gandhinagar, Palai,

Gandhinagar 382055

Acknowledgements

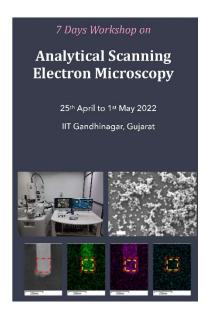






Department of Science & Technology (DST) funded

Training workshop under STUTI (Synergistic Training Program Utilizing the Scientific and Technological Infrastructure)



Contents of the workshop

- · Inauguration and Welcome note
- · Introduction of the Participants and the Host.
- · Overview of various DST sponsored Programs.

Session 2 A-B

- Physics of Electron Microscopy
- · Overview of electron-material interaction

Session 3 A-B

- Fundamentals of image generation in SEM
- · Working of different Imaging modes (SE, BSE)
- · Case studies to illustrate the imaging modes.
- · Sample Preparation (tools and techniques)

Session 4 A-B

- · Fundamentals of various analytical modes in SEM
- · Working of spectroscopy modes (EDS, WDS)
- · Instrumentation and Software

Session 5 A-B

- · Fundamentals of EBSD
- · Instrumentation and indexing

Session 6 A-B

- · Application and Discussion: Case studies on Structural materials, Biomaterials, Nano Materials, High Entropy materials
- · Advanced modes of Microscopy

Session 7 A

· Analysis of participant's samples

Overview of STUTI and Objectives of Workshop

DST welcomes all their participants for the workshop on Analytical Scanning Electron Microscopy organised under STUTI. The STUTI program envisions hands-on-training and sensitization of the state-of-the-art equipment as well as towards sharing while ensuring transparent access to S&T facilities. Department of Science and Technology has identified IIT-Gandhinagar to function as a Project Management Unit (PMU) and as co-ordinator for this workshop.

This workshop is aimed to provide an insight into the functioning of Scanning Electron Microscope (SEM) for advanced imaging (Secondary electron-SE, Back scattered electron-BSE, Electron backscattered diffraction-EBSD) and spectrocopy (Energy dispersive spectrocopy-EDS, Wavelength dispersive spectroscopy-WDS). The participants will be introducted to the basic concepts of image generation, instrumentation, image analysis and the advanced modes of operation. The participants will be provided with hands on experience on the operation of the instrument and will have a chance to interact with subject experts and also analyse their own samples (with prior approval).

Schedule

Day 1			Day 2			Day 3	
08:30	Registration		09:00	Sessio	n 2A	09:00	Session 3A
09:00	Innaugral Session	า	11:00	Break		11:00	Break
09:30	Session 1A		11:30	Sessio	n 2B	11:30	Session 3B
10:00	Session 1B		13:00	Lunch		13:00	Lunch
12:00	Lunch		14:00	Lab se	ssion 1	14:00	Lab session 2
13:00	IIT Gandhinagar I	_ab Visit	17:30	Coffee	Break	17:30	Coffee Break
15:30	User requirement of participants						
16:00	Coffee Break						
Day 4		Day 5		Day 6		Day 7	
09:00	Session 4A	09:00	Session 5A	09:00	Session 6A	10:00	Session 7A
11:00	Break	11:00	Break	11:00	Break	13:00	Lunch
11:30	Session 4B	11:30	Session 5A	11:30	Session 6B	14:00	Site visit
13:00	Lunch	13:00	Lunch	13:00	Lunch		
14:00	Lab session 3	14:00	Lab session 4	14:00	Lab session 5	Netv	vorking Dinner

Annex 2: List of participants registered and their attendance for the Analytical SEM workshop.

Sr. No.	Candidate Name	Gender	Educational Qualification	Email address	University/Institute	
1	Vijay Dhanabal M H	Male	MSc. (Physics)	vijaydhanabal1998@gmail.com	Dhonothion II.::	
2	Kavitha E R	Female	M.Phil. (Physics)	kavier.er@gmail.com	Bharathiar University, Coimbatore	
3	Kumaresan L	Male	M.Phil. (Physics)	kumaresh.msc55@gmail.com	Combatoic	
4	Ajay Kumar Mishra	Male	M.Tech (Metallurgy)	akm.18mm1103@phd.nitdgp.ac.in	National Institute of	
5	SK Md Arif	Male	M.Tech (Metallurgy)	sma.18mm1101@phd.nitdgp.ac.in	Technology, Durgapur	
6	Rahul Bhandari	Male	M.Tech (Manufacturing)	rb.18mm1102@phd.nitdgp.ac.in		
7	Ms. Kinjal K. Joshi	Female	MSc. (Physics)	kinjaljoshi38@gmail.com	P.D.Patel Institute of Applied Science, Charusat University,	
8	Patel Meswa Harshadkumar	Female	MSc. (Physics)	meshpatel96@gmail.com		
9	Gayatri Dave	Female	Ph.D. (Microbiology)	gayatridave.bt@charusat.ac.in	Change	
10	Himaben Patel	Female	MSc. (Physics)	20drphy004@charusat.edu.in	Dr. K. C. Patel Research and Development Centre, Anand	
11	Anjali Mishra	Female	Ph.D. (Chemistry)	anjali.mishra@iar.ac.in		
12	Meghna Goswami	Female	M.Tech (Bio- Technology)	meghnagoswami.phd2021@iar.ac.in	Institute of Advanced Research, Gandhinagar	
13	Dr. Tvarit Patel	Male	Ph.D. (Materials Sci & Engg.)	tvarit.patel@iar.ac.in		
14	Vivek Pachchigar	Male	M.Tech (Nuclear Eng.)	vivek.pachchigar@ipr.res.in	Institute for Plasma Research (IPR-FCIPT), Gandhinagar	
15	Sukriti Hans	Female	MSc. (Physics)	sukritihans21@gmail.com		
16	Sudheer	Male	Ph.D. (Physics)	sudheer@ipr.res.in		
17	Ariful Hoque	Male	MSc. (Nano Technology)	arifhoque0112@gmail.com	Central University of Gujarat	
18	Mrinal talukdar	Male	MSc. (Chemistry)	mrinaltalukdar220@gmail.com		
19	Biplob Borah	Male	MSc. (Chemistry)	biplobbora18@gmail.com		
20	Debi Prasad Panda	Male	Ph.D. (Electrical)	dppanda.ece@gmail.com	Physical Research Laboratory, Ahmedabad	
21	Surendra Vikram Singh	Male	Post Doc Fellow (Physics)	surendra@prl.res.in		
22	Arijit Roy	Male	MSc. (Physics)	arijit.roybwn11@gmail.com		
23	Devanshi Zala	Female	M.Phil. (Physics)	devanshiba.zphd21@sot.pdpu.ac.in		
24	Monika Mukeshkumar Patel	Female	MSc. (Chemistry)	monikapatel1047@gmail.com	Pandit Deendayal Energy University, Gandhinagar	
25	Chandra Babu	Male	MSc. (Physics)	chandrababu88badampudi@gmail.com		
26	Dr. Hardik Bhatt	Male	Ph.D. (Pharmacy)	hardikbhatt23@nirmauni.ac.in	Nirma University,	
27	Dr. Ankur Pandya	Male	Ph.D. (Physics)	ankur.pandya@nirmauni.ac.in	Ahmedabad	
28	Rohan Kinger	Male	MSc. (Physics)	rohan.kinger98@gmail.com	Vishwakarma Gov. Eng.	
29	Maitri Patel	Female	MSc. (Physics)	maitripatel1701@gmail.com	College, Ahmedabad	
30	Ankitkumar shah	Male	M.E. (Metallurgy)	ankit.destiny89@gmail.com	Gujarat Technical Univ, Ahmedabad	
31	Bhargav Pathak	Male	M.Phil. (Physics)	bhargavpathak.phy@gmail.com	Gujarat University	
32	Manjit	Male	M. Pharmaceutics	manjit.rs.phe20@itbhu.ac.in	IIT-BHU, Varanasi	
33	Dr. Achintya Jana	Male	Ph.D. (Chemistry)	achintya.jana@iitgn.ac.in	IIT Gandhinagar	
34	Priyanka Jayeshbhai Hemani	Female	MSc. (Physics)	priyankaj.hemani710@gmail.com	Marwadi Univ, Rajkot	
35	Jaimin Umesh Trivedi	Male	MSc. (Physics)	jaimint@nuv.ac.in	Navrachana University, Vadodara	

Annex 3: Schedule date and activities during the workshop.

		Day 1		
	08:30	Registration		
25th April 2022	09:00-09:15	Welcome speech (Prof. Ghosh)		
	09:15-10:15	Overview of various DST sponsored programs (Prof. Bhatia)		
	10:15-10:45	TEA/COFFEE SESSION		
	10:45-13:00	Participants interaction session		
	13:00-14:00	LUNCH		
	14:00-17:00	LAB Session-1: IITGN Lab Visit and FIST-SEM Demonstration		
	11.00 17.00	END Session 1. HTGIV East Visit and FIST SERVI Demonstration		
		Day 2		
26th	09:00-11:00	Module 2A: Physics of Electron Microscopy (Prof. Panda)		
April	11:00-11:30	TEA/COFFEE SESSION		
2022	11:30-13:00	Module 2B: Electron-material interaction (Prof. Panda)		
	13:00-14:00	LUNCH		
	14:00-17:00	LAB Session-2: Interaction and FIST-SEM Demonstration		
		Day 3		
27th	09:00-11:00	Module 3A: Fundamentals of image generation (Prof. Gautam)		
April	11:00-11:30	TEA/COFFEE SESSION		
2022	11:30-13:00	Module 3B: Working of SE and BSE modes (Prof. Panda)		
	13:00-14:00	LUNCH		
	14:00-17:00	LAB Session-3: SEM sample preparation and SEM Demonstration		
		Day 4		
28th	09:00-11:00	Module 4A: Fundamentals of analytical modes in SEM (Prof.Ghosh)		
April	11:00-11:30	TEA/COFFEE SESSION		
2022	11:30-13:00	Module 4B: Working of EDS and WDS modes (Mr Mangesh)		
	13:00-14:00 14:00-17:00	LUNCH LAB Session-4: Image processing and FIST-SEM Demonstration		
	14.00-17.00	LAB Session-4. Image processing and F151-3EM Demonstration		
-		Day 5		
29th	09:00-11:00	Module 5A: Fundamentals of EBSD (Prof.Ghosh)		
April	11:00-11:30	TEA/COFFEE SESSION		
2022	11:30-13:00	Module 5B: Fundamentals of EBSD and Indexing (Mr. Renjith)		
	13:00-14:00	LUNCH		
	14:00-17:00	LAB Session-5: Sample preparation for participants and FIST-SEM Demonstration		
-		Day 6		
	09:00-11:00	Module 6A: Applications (Prof. Misra)		
30th	11:00-11:30	TEA/COFFEE SESSION		
April	11:30-13:00	LAB SESSION		
2022	13:00-14:00	LUNCH		
	14:00-15:30	Module 6B: Applications (Prof. Satyam Suwas)		
	15:30-17:00	LAB Session-6: Participant sample analysis and FIST-SEM Advanced mode		
		Day 7		
	10:00-11:30	LAB SESSIONS-7a: Participant sample analysis and interaction		
1st	11:00-11:30	TEA/COFFEE SESSION		
May	11:30-13:00	LAB SESSIONS-7b: Participant sample analysis and interaction		
2022	13:00-14:00	LUNCH		
	14:00-17:00	LAB Session-7c: Participant sample analysis and interaction		
		1 1 2		

Annex 4: Feedback summary

Sr. No.	Content	% Rating
1	Overall grading of the Programme with reference to relevance of course, module/content etc.	100 % rated above 8 points
2	Overall grading of the facilities provided by the institute, i.e., Hostel, Mess, Class Rooms, Transport/infrastructure etc.	95% rated above 8 points
3	Overall grading of the faculty members conducting the training	95% rated above 8 points
4	How do you rate the overall training methodology	95% rated above 8 points
5	How far the field visit is relevant and related to your research study	80% rated above 8 points
6	Usefulness of this training in your current role	85% rated above 8 points
7	Usefulness of this training in future work/job you may handle	90% rated above 8 points
8	How far have you benefitted from interaction with the fellow participants of the training	85% rated above 8 points
9	How far the course material supplied relevant and related to the training curriculum	92% rated above 8 points
10	Overall grading of the process of training	92% rated above 8 points
11	Your recommendation to your peers/ colleagues for the training Programme	92% rated above 8 points