A training session report on

Application of XRD Technique in Materials Science and Engineering

Under STUTI program funded by DST

In association with
Indian Institute of Technology, Gandhinagar
(Project Management Unit)

Coordinated by

Prof. Santosh Kumar Sahoo

Prof. Syed Nasimul Alam

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National Institute of Technology Rourkela, Odisha

22nd to 28th August 2022

Acknowledgement

Summary

The goal of this training session is to popularize application of XRD technique in materials science engineering among students, faculty, scientists and industry professionals through a week-long training workshop. The workshop was conducted at NIT Rourkela from 22nd to 28th August, 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 different application of XRD technique in materials science and engineering. The participants were introduced to the basic crystallography of materials, XRD instrumentation, and characterization of crystal structure, phases, crystallite size, lattice strain, residual stress, texture & rietvelt refinement through XRD technique. Some case studies have also been provided to the participants to include the XRD technique in their research works. 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

National Institute of Technology Rourkela (NIT Rourkela) conducted 7-day long workshop on DST-FIST funded instrument, 'XRD (with texture cradle)' in its campus for participants from various backgrounds such as Post Graduate, B.Tech., Professors, Scientists, Ph.D. and Post Doc Fellows and Industry persons were invited (**Annex-1**). The following workshop's activities took place from August 22nd to 28th, 2022 (**Annex-2 and 3**). This report provides a quick overview of both the lecture and technical sessions.

- *Lecture Sessions*

Prof. Santosh Kumar Sahoo (Dept. of Metallurgical and Materials Engg.) provided the goals and objectives of the STUTI workshop in the inaugural session. This session was followed by a talk of **Prof. Dilip Kumar Pradhan** (Dept. of Physics and Astronomy). **Prof. Dilip** provided knowledge on symmetry in crystalline materials. He has extensively covered: Introduction to point symmetry operations and their notations; Presentation of seven crystal systems based on the hierarchy of point symmetry operations; Classification of 14 Bravias Lattices for seven crystal systems; Development of 32 crystallographic point groups based on the group theoretical calculations; Basic introduction to Space groups, i.e., basic notions and the Hermann-Mauguin symbolism. He has also discussed on description of space groups in International Tables for Crystallography, Vol. A. He further given a lecture was on the Rietveld Refinement analysis of the XRD patterns. **Prof. Debasis Chaira** (Dept. of Metallurgical and Materials Engg.) provided an insight on generation of X-ray, characteristic X-ray used for X-ray diffraction. **Prof. Syed Nasimul Alam** (Dept. of Metallurgical and Materials Engg.) presented the basics of crystallite size and lattice strain analysis using XRD diffraction peaks. Different measurement parameters with their importance in the measurement/analysis were also discussed. **Prof. Anindya Basu** (Dept. of Metallurgical and Materials Engg.) gave a talk on application of XRD technique on characterization of coating materials which includes discussion on glancing angle (or grazing angle) XRD (GXRD or GIXRD), with a case studies on nitriding of Cr coating and composite deposition of Cu-ZrO$_2$. Characterization of refractory materials by XRD has been discussed by

Prof. Anshuman Patra (Dept. of Metallurgical and Materials Engg.), which includes information on various properties, applications, drawbacks of refractory alloys in high temperature applications followed by different fabrication techniques. **Prof. Rajesh Kumar Prusty** (Dept. of Metallurgical and Materials Engg.) showed the importance of XRD for characterization of polymers, where the crystalline behavior of polymers compared with amorphous, semi-crystalline and high crystalline structures. **Prof. Santosh Kumar Sahoo** (Dept. of Metallurgical and Materials Engg.) provided the basics of crystallographic textures in materials, their representation through pole figure and ODF (Orientation distribution function), development of textures in materials, and the characterization of texture through XRD and EBSD (Electron backscattered diffraction) techniques. **Prof. Kumud Kant Mehta** (Dept. of Metallurgical and Materials Engg.) gave a talk on the role of texture on design and development of aerospace alloy for formability application. A case study of regular failure of aerospace turbine casing made of nickel based commercial alloy (Hastelloy C276) was also discussed. **Prof. Santosh Kumar Sahoo** (Dept. of Metallurgical and Materials Engg.) also presented the basics of residual stress measurement through XRD technique using $Sin2\psi$ method. He extensively discussed the different measurement parameters to be considered while calculating the residual stress in a material through XRD technique.

- *Technical Sessions*

Each day's second half session was held for the technical exposure of the institute. During this session participants visited the laboratory of important instruments procured from DST-FIST grant by the Institute. This includes X-ray diffractometer (Dept. of Physics and Astronomy), Single crystal XRD (Dept. of Chemistry), X-ray Fluorescence Spectroscopy (XRF) (Dept. of Earth and Atmospheric Science), and Field Emission Scanning Electron Microscope (FESEM) (Dept. of Ceramic Engg). The participants were given detailed basics and functioning of these instruments, and also information was provided on how they can avail the facility for the characterization of samples from outside NIT Rourkela.

Various components of the XRD were also demonstrated to the participants during the laboratory presentation in order to provide an understanding of the working principle of the XRD technique. On the **third** day, participants were shown how to characterize the crystal structure of materials using XRD technique. They were taught to estimate the crystal structure of BCC, FCC and HCP materials manually, and also through *Xpert HighScore* software. They were also trained to estimate the crystal structure of phase mixtures. On the **fourth** day, the participants were trained how to estimate the crystallite size and lattice strain using XRD technique. A high energy ball milled powders were utilized for estimation of the crystallite size and lattice strain. On the **fifth** day, participant was allowed to characterize the crystallographic texture in a material through XRD technique. They were provided how to set-up programs in XRD system for texture measurements, and subsequently allowed to analyze the texture of the samples using *Labotex* software. A rolled Mg sheet was used for the texture analysis. On day **six**, the participants were trained on residual stress characterization through XRD $Sin^2\psi$ method. They were provided how to set-up stress programs in the XRD system, and subsequently allowed to analyze the texture of the samples using $Sin^2\psi$ method. On the same day, they were trained how to perform Rietveld refinement analysis through Fullprof software. On day s**even**, the participant's samples were characterized which also includes hands-on session for each of the participants.

Outcome of the workshop

The workshop attracted 59 % male and 41 % female participants from 24 different institutes (**Figure 1**). About 32 participants attended the DST-STUTI training workshop on XRD. 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 XRD technique used for different characterization. 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 (**Annex 4**). The majority of the participants was pleased with the training session and suggested that more workshops be held in the future.
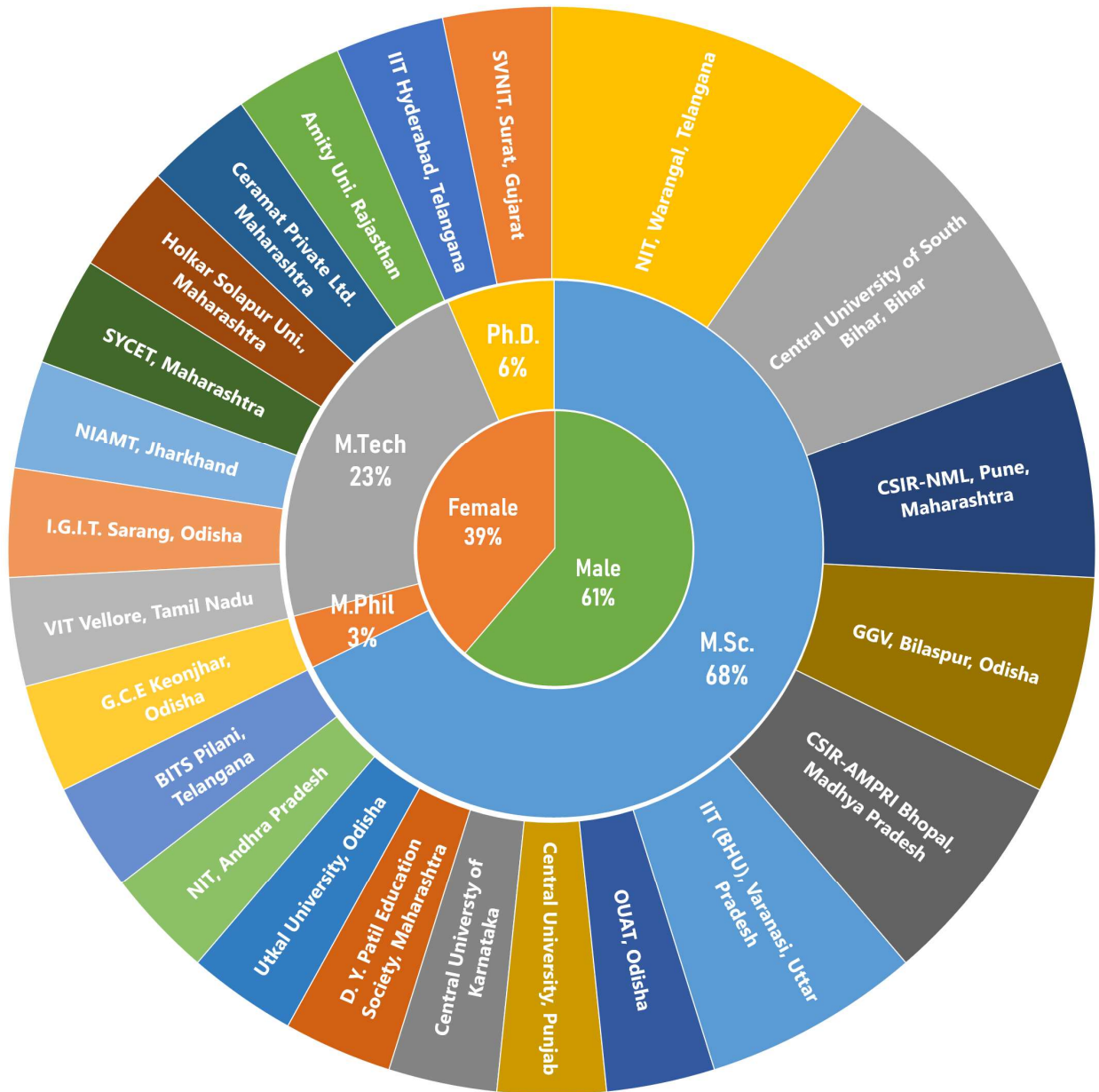
Figure 1: Participants registered for the training program from 16 different institutes.

Annex 1: Brochure for the program.

Department of Science & Technology (DST) funded

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

7 days workshop on
Application of XRD Technique in Materials Science and Engineering

📅 **22 - 28 August 2022**

📍 **Department of Metallurgical and Materials Engineering, NIT Rourkela**
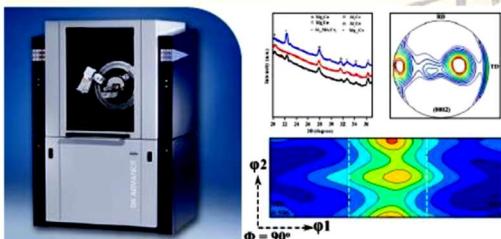
Overview of STUTI and objectives of Workshop

DST welcomes all their participants for the workshop on X-RAY DIFFRACTION organized 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 NIT Rourkela has been chosen as the coordinator for this workshop.

The workshop is designed for a balance between theory and practical training on the equipment. Emphasis is on hands-on use of equipment for demonstration/characterization by each participant. Participants may be allowed to bring their samples.

Eligibility criteria for participants for the Training Program:
a) Person of Indian origin;
b) Minimum qualification should be Post Graduate (Science) or BTech (Technology);
c) Professors/Scientists/ Post-Doc Fellows/ PhD Fellows/ Industry persons who are actively involved in research and development (R&D);
d) Not more than 3 people from one institute per training should be allowed from outside the host institute.

Registration Details
Interested candidates must register and only selected candidates would be invited to the workshops. The total no. of participants is limited to 30. For selected candidates registration fees, travel (by train), boarding and lodging will be covered by NIT Rourkela
interested candidates should register using the following link:
🔗 **https://forms.gle/8A2KN6RdzWwLAtVp9**
Registration Deadline: 24 July 2022
Shortlisted Candidates will be intimated by email, latest by 29 July 2022

Tentative Schedule

Session 1: 9:00 AM – 1:00 PM; Tea Break: 11:00 AM – 11:30 AM;
Lunch: 1:00 PM – 2:00 PM and
Session 2: 2:30 PM – 5:30 PM; Tea: 5:30 PM – 6:00 PM

DAY–1
Session 1: Inauguration and Welcome note followed by High Tea
Basic Crystallography: Symmetry Operations; Development of 7 Crystal Systems,
14 Bravais Lattices, 32 Point Groups; Introduction to Space Group.
Speaker: Prof. Dillip Pradhan, Dept. of Physics and Astronomy, NIT Rourkela
Session 2: NIT Rourkela Lab Visit; Facilities supported by DST

DAY – 2
Session 1: Basic Crystallography: Continued….
Session 2: Lab Session – 1 (Demonstration of X-ray Diffractometer)

DAY – 3
Session 1: X-ray Diffraction:X-ray Generation;Properties;Diffraction;
Crystal Structure Determination; Phase Analysis.
Speaker: Prof. Debasis Chaira and Prof. Santosh Kumar Sahoo, Dept. of Metallurgical and Materials Engg., NIT Rourkela
Session 2: Lab Session – 2 (Phase Analysis)

DAY – 4
Session 1: Characterization of Nanomaterials, refractory materials, Coating materials and Polymers/Composites using XRD Technique; Some Case Studies.
Speaker: Prof. Anindya Basu, Prof. Syed Nasimul Alam, Prof. Rajesh Prusty and Prof. Anshuman Patra, Dept. of Metallurgical and Materials Engg., NIT Rourkela

Session 2: Lab Session – 3 (Characterization of Nanomaterials and Polymers/Composites)

DAY – 5
Session 1: Texture: Basics; Pole Figure; Orientation Distribution Function; Introduction to Electron Backscattered Diffraction (EBSD); Some Case Studies.
Speaker: Prof. Santosh Kumar Sahoo and Prof. Kumud Kant Mehta, Dept. of Metallurgical and Materials Engg., NIT Rourkela
Session 2: Lab Session – 4 (Texture Analysis)

DAY – 6
Session 1: Rietveld Refinement; Residual Stress Analysis by XRD Technique; Some Case Studies.
Speaker: Prof. Dillip Pradhan and Prof. Santosh Kumar Sahoo
Session 2:Lab Session – 5 (Rietveld Refinement and Residual Stress Analysis)

DAY – 7
Session 1: Measurement and characterization of participant's samples.
Session 2: Feedback, and Valedictory Function.

Coordinators

Prof. Santosh Kumar Sahoo   **Prof. Syed Nasimul Alam**   **Prof. Rajesh Kumar Prusty**

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Annex 2: List of participants registered and their attendance for the Application of XRD Technique in Materials Science and Engineering workshop.

Sr. No.	Candidate Name	Gender	Educational Qualification	Email address	University/Institute
1	Urvashi Kesarwani	Female	M.Sc. Materials Science	urvashi.kesarwani.rs.cer18@iitbhu.ac.in	IIT (BHU), Varanasi, Uttar Pradesh
2	Manisha Sharma	Female	M.Sc. Physics	manishasharma.rs.phy19@iitbhu.ac.in	
3	Piyush A. K. Sharma	Male	M.Sc. Chemistry	i18cy029@chem.svnit.ac.in	SVNIT, Surat, Gujarat
4	Mufeeduzzaman	Male	M.Sc. Physics	mufeedzzaman@gmail.com	Central University of South Bihar, Gaya, Bihar
5	Ashutosh Kumar	Male	M.Sc. Applied Physics	ashutoshswg007@gmail.com	
6	Vijay Raj Singh	Male	Ph.D. Physics	vijayraj@cusb.ac.in	
7	Mr. Reddivari Muniramaiah	Male	M.Sc. Physics	r.muniram1995@gmail.com	NIT, Warangal, Telangana
8	T. Lavanya	Female	M.Sc. Physics	lavanyathyda1952@gmail.com	
9	M. Rakshita	Female	M.Sc. Physics	rakshitaammulu1997@gmail.com	
10	G. Neeraja Sinha	Female	M.Sc. Chemistry	cy18resch02001@iith.ac.in	IIT Hyderabad, Telangana
11	Sonam Raghav	Female	M.Sc. Physics	sonam.raghav@s.amity.edu	Amity Uni. Rajasthan
12	Sujoy Mandal	Male	M.Tech Material Science	sujoy_qc@tsl-ceramat.com	Ceramat Private Limited (A Tata Steel Enterprise)
13	Vinod Vasant Patil	Male	M.Sc. Chemistry	patil2016vv@gmail.com	Punyashlok Ahilyadevi Holkar Solapur Uni., Maharashtra
14	Gaurav Rajak	Male	M.Tech Material Science	gauravrajak007@gmail.com	CSIR-AMPRI Bhopal, Madhya Pradesh
15	Rahul Arya	Male	M.Sc. Physics	rahularyasept@gmail.com	
16	Aastha Sahu	Female	Ph.D Physics	aasthasahu672@gmail.com	GGV, Bilaspur, Odisha
17	Sourabh Gouraha	Male	M.Sc. Physics	gourahasourabh@gmail.com	
18	Shivamurthy B. P.	Male	M.Sc. Chemistry	bp.shivamurthy27@gmail.com	CSIR-NML, Pune, Maharashtra
19	Mayur P. Sangole	Male	M.Sc. Physics	mp.sangole@ncl.res.in	
20	Mr. Ankush Balajirao Khansole	Male	M.Tech Mechanical Engineering	ankush.khansole01@gmail.com	SYCET, Maharashtra
21	Tapas Pal	Male	M.Tech Nano Technology	tapaspal.ar@gmail.com	NIAMT, Jharkhand
22	July Randhari	Female	M.Tech Design	julyrandhari678@gmail.com	I.G.I.T. Sarang, Odisha
23	Manu M. Nair	Male	M.Phil Nanoscience	manunair.m2020@vitstudent.ac.in	VIT Vellore, Tamil Nadu
24	Adiraj Behera	Male	M.Tech Metallurgy	adirajbehera_fmt@gcekjr.ac.in	G.C.E Keonjhar, Odisha
25	Aarjoo Jaimin	Male	M.Tech Material Science & Engineering	p20200460@hyderabad.bits-pilani.ac.in	BITS Pilani, Telangana
26	Maddela Siva Kumar	Male	M.Sc. Physics	shiva.sk262@gmail.com	NIT, Andhra Pradesh
27	Bhagaban Kisan	Male	M.Sc Physics	bkisan@gmail.com	Utkal University
28	Ms. Sumita Suryakant Patil	Female	M.Sc. Physics	sumitapatil.physics@gmail.com	D. Y. Patil Education Society, Maharashtra
29	Deepshikha Srivastava	Female	M.Sc. Geology	deepshikhasrivastava114@gmail.com	Central Universty of Karnataka
30	Tanaya Subudhi	Female	M.Sc. Chemistry	tanayasubudhi13@gmail.com	Central University, Punjab
31	Swagatika Mallik	Female	M.Sc. Chemistry	swagatikamallik21@gmail.com	OUAT, Odisha

Annex 3: Schedule date and activities during the workshop.

Day	Session – I			Session – 2	
	9:00 AM – 11:00 AM	T E A B R E A K	11:30 AM – 01:00 PM	02:30 PM – 05:30 PM	
22 August 2022	Registration (9 – 9:30) + Inauguration		Basic Crystallography Prof. Dilip Pradhan	NIT RKL Lab Visit (CY, ER, CR, MM) Single crystal XRD, XRF, FE-SEM, etc.	
23 August 2022	Basic Crystallography Prof. Dilip Pradhan		Introduction to XRD Prof. Debasis Chaira	Lab Session – 1 (XRD System)	Basic Crystallography Prof. Dilip Pradhan
24 August 2022	Phase Analysis Prof. Debasis Chaira		Size and Strain Analysis Prof. Syed Nasimul Alam	Lab Session – 2 (Crystal Structure and Phase Analysis)	
25 August 2022	Characterization of Nano-materials and Refractory Materials Prof. Syed Nasimul Alam and Prof. Anshuman patra		Characterization of Coating Materials and Polymers & Composites Prof. Anindya Basu and Prof. Rajesh Prusty	Lab Session – 3 (Analysis of Crystallite Size and Lattice Strain)	
26 August 2022	Texture Analysis Prof. Santosh Sahoo		Texture of Materials Prof. Kumud Kant Mehta	Lab Session – 4 (Texture Analysis)	
27 August 2022	Rietveld Analysis Prof. Dilip Pradhan		Residual Stress Analysis Prof. Santosh Sahoo	Lab Session – 5 (Rietveld Analysis)	Lab Session – 6 (Residual Stress Analysis)
28 August 2022	Analysis of Participant's Samples			Feedback and Valedictory Function	

(Lunch Break column: "L U N C H B R E A K (1 PM – 2 PM)" spanning all days between Session–I and Session–2)

Annex 4: Feedback summary

Sr. No.	Content	% Rating
1	Overall grading of the Programme with reference to relevance of course, module/content etc.	97% rated 8 and above 8 points
2	Overall grading of the facilities provided by the institute, i.e., Hostel, Mess, Class Rooms, Transport/infrastructure etc.	100% rated 8 and above 8 points
3	Overall grading of the faculty members conducting the training	100% rated 8 and above 8 points
4	How do you rate the overall training methodology	91% rated 8 and above 8 points
5	How far the field visit is relevant and related to your research study	97% rated 8 and above 8 points
6	Usefulness of this training in your current role	94% rated 8 and above 8 points
7	Usefulness of this training in future work/job you may handle	97% rated 8 and above 8 points
8	How far have you benefitted from interaction with the fellow participants of the training	97% rated 8 and above 8 points
9	How far the course material supplied relevant and related to the training curriculum	94% rated 8 and above 8 points
10	Overall grading of the process of training	97% rated 8 and above 8 points
11	Your recommendation to your peers/ colleagues for the training Programme	97% rated 8 and above 8 points