

Advanced Analytical Techniques for Characterization of 2D Materials

Advanced Analytical Techniques for Characterization of 2D Materials

Edited by

Chandra Sekhar Rout

Centre for Nano and Material Sciences

Jain University

Jain Global Campus

Ramanagaram, Bangalore 562112, India

Dattatray J. Late

Centre for Nanoscience and Nanotechnology

Amity University, Maharashtra

Panvel 410206, India



AIP Publishing Books

A publication of AIP Publishing

Melville, New York

Copyright © 2022 AIP Publishing LLC

All rights reserved. No part of this publication may be reproduced, stored in a retrieval system, or transmitted, in any form or by any means, electronic, mechanical, photocopying, recording or otherwise, except as permitted by law.

To obtain permission to reuse material from this title, contact AIP Publishing at rights@aip.org. Additional rights and permissions information is available at: <https://publishing.aip.org/resources/researchers/rights-and-permissions/permissions/>.

First edition, published 2022.

Library of Congress Control Number: 2022943018

ISBN: 978-0-7354-2540-8 (Softcover)

ISBN: 978-0-7354-2542-2 (Online)

ISBN: 978-0-7354-2543-9 (ePub)

ISBN: 978-0-7354-2541-5 (ePDF)

Cover design by Nanci Kappel.

Set in 10/13pt MinionPro by Nova Techset Private Limited, Bengaluru & Chennai, India, and bound by The Sheridan Group, USA

The publisher and authors cannot guarantee the accuracy of this work. Readers of scientific texts should not rely on any third-party text, or the statements and conclusions embodied in such work, without independently confirming hypotheses, factual statements, and conclusions. The publisher does not endorse organizations, institutions, or companies that may be referenced herein.

Published by AIP Publishing

1305 Walt Whitman Road, Suite 110, Melville, NY 11747-4300, USA

To everyone who pursues research in materials science.

ACKNOWLEDGMENTS

This treatise on *Advanced Analytical Techniques for Characterization of 2D Materials* is nothing but a tough grind by the many experts who compiled the chapters and have gone through a lot of backbreaking assignments. We are obligated and thankful to the anonymous reviewers who not only appraised the work thoroughly but also enhanced its quality. The immense support from the AIP Publishing team, especially Martine Felton, Claire Gordon, and Dr. Benjamin Johnson, was instrumental and significant. The editors are most grateful to the love of their families, friends, and, above all, the grace from above.

We would like to acknowledge freepik.com for the cover page illustration.

PREFACE

Marie Curie precisely articulated “Have no fear of perfection, you will never reach it.” This quote always acts as a stimulus to scientists and researchers to bend over backwards for uplifting the standard of research and development over the years. The consideration of nanomaterials over their bulk counterparts has not only opened the gates of endless opportunity but also diminished the struggle for real time application. In this context, two-dimensional layered materials have gained tremendous attention in recent years owing to their great promise in the 21st century solid state device technology. After the discovery of graphene, other graphene analogous 2D nanomaterials with novel tunable properties associated with their metallic, semimetallic, semiconducting, insulating, superconducting, Mott-insulating nature, etc., are being explored. The tunable properties, flexibility, good mechanical properties, high packing densities, etc., make them suitable candidate for the development of different thin, flexible, and wearable devices. Transition metal dichalcogenides (TMDs) are one class of 2D materials and are being investigated extensively due to their versatile and interesting properties. They own lamellar crystal structure, where each MX_2 layer is composed of one layer on transition metal atoms M (Mo, W, V, Sn, Nb, Ta, Ti, etc.) sandwiched between two layers of chalcogen atoms X (S, Se, Te) with covalent bonds. The neighboring MX_2 layers are weakly coupled together by van der Waals interactions. Nanomaterials with one atom thickness and infinite lateral size are the ideal 2D material that holds the novel properties associated with it due to their quantum confinement effect. Hence, the characterization of these 2D materials is the first step to investigate their layer-dependent changes in their properties and to use them in different multitude applications. Since the number of layers, the size of the flakes, crystallinity, the presence of defects and vacancies, or adsorbed molecules significantly affect these material properties, advanced analytical techniques are essential to address its morphological and functional properties.

Depending on the requirement, various characterization techniques, such as optical microscopy, atomic force microscopy (AFM), scanning electron microscopy (SEM), transmission electron microscopy (TEM), Raman spectroscopy, x-ray diffraction (XRD), and x-ray photoelectron spectroscopy (XPS) have been widely used to characterize the 2D materials. Each technique has its own advantages and limitations. Therefore, combining different characterization techniques is highly desirable to understand different structural features and properties of the as-synthesized 2D materials. Furthermore, different operando spectroscopic techniques such as Raman spectroscopy, UV-Vis, XRD, and XPS are being used to understand the mechanism involved in the growth process and device performance of the 2D materials by *in situ* experimental investigations. The probe based analytical techniques such as AFM and STM can provide the information on the orientation, smoothness, the number of layers, grain size, depth profile, and dielectric and mechanical properties of the 2D materials. Electron diffraction-based techniques such as TEM, SEM, and EDS are useful for determining thickness, number of

layers, high-resolution imaging, surface properties, composition, and morphology. X-ray diffraction (XRD), x-ray fluorescence (XRF), x-ray absorption fine-structure (XAFS), and x-ray photoelectron spectroscopy (XPS) deliver rapid qualitative as well as quantitative determination of a wide variety of 2D materials. Similarly, spectroscopy-based analytical tools such as Raman spectroscopy, UV-Vis, and zeta potential analyzer are useful to determine the composition, quality, bandgap, doping, surface charge, thickness, the number of layers, orientation, chemical bonding, etc. By considering these aspects, in this book, we aim to provide detailed information on these analytical techniques, their importance, and recent progress, which have been widely used to characterize different 2D nanomaterials. This book will be useful for the researchers and scientists in the areas of materials science and engineering. Furthermore, this book will serve as a textbook for both beginners and experienced researchers who are pursuing their research in 2D layered materials and their advanced applications.

Professor Chandra Sekhar Rout

Email: r.chandrasekhar@jainuniversity.ac.in; csrout@gmail.com

Professor Dattatray J. Late

Email: datta099@gmail.com


CONTRIBUTORS

Levna Chacko 


Department of Physics and Electronics, CHRIST (Deemed to be University), Bangalore, Karnataka 560029, India

Shobhnath P. Gupta 

National Centre for Nanosciences and Nanotechnology, University of Mumbai, Mumbai 400098, India

Sayed M. Hasnain 

National Centre for Nanosciences and Nanotechnology, University of Mumbai, Mumbai 400098, India

Rajesh Jaiswar 

National Centre for Nanosciences and Nanotechnology, University of Mumbai, Mumbai 400098, India

Namsheer K 

Centre for Nano and Material Sciences, Jain University, Jain Global Campus, Ramanagaram, Bangalore 562112, India

Riddhi Kadrekar 

Amity School of Applied Science, Amity University Maharashtra, Mumbai and Centre for Nanoscience and Nanotechnology, Amity University Maharashtra, Mumbai-Pune Expressway, Bhatan, Post—Somathne, Panvel, Mumbai, Maharashtra 410206, India

Dattatray J. Late 

Centre for Nanoscience and Nanotechnology, Amity University Maharashtra, Mumbai 410206, India

Abhinandan Patra 

Centre for Nano and Material Sciences, Jain University, Jain Global Campus, Ramanagaram, Bangalore 562112, India

Sithara Radhakrishnan 


Centre for Nano and Material Sciences, Jain University, Jain Global Campus, Ramanagaram, Bangalore 562112, India

S. Reshmi 

Institute of Physics, Bhubaneswar, Sachivalaya Marg, Gajapati Nagar, Bhubaneswar, Odisha 751005, India

Chandra Sekhar Rout 


Centre for Nano and Material Sciences, Jain University, Jain Global Campus, Ramanagaram, Bangalore 562112, India

Rutuparna Samal 


Centre for Nano and Material Sciences, Jain University, Jain Global Campus, Ramanagaram, Bangalore 562112, India

Aditya Sharma 


Centre for Nano and Material Sciences, Jain University, Jain Global Campus, Ramanagaram, Bangalore 562112, India

Pratik V. Shinde 

Centre for Nano and Material Sciences, Jain University, Jain Global Campus, Ramanagaram, Bangalore 562112, India

Vitthal M. Shinde 

Department of Chemistry, Annasaheb Waghire College, Otur Post, Junnar Taluka, Pune District, Pune 412409, India

R. Sundheep 

Department of Chemistry, Chungnam National University, 99 Daehak-ro, Yuseong-gu, Daejeon, South Korea

Pravin S. Walke 

National Centre for Nanosciences and Nanotechnology, University of Mumbai, Mumbai 400098, India



AIP PUBLISHING BOOKS

Advanced Analytical Techniques for Characterization of 2D Materials

16 October 2024 08:21:47

TABLE OF CONTENTS

Chapter 1	Introduction	1-1
	<i>Chandra Sekhar Rout, Sithara Radhakrishnan, and Dattatray J. Late</i>	
	1.1 Introduction	1-1
	1.2 History of 2D Materials	1-3
	1.3 Significance of 2D Materials	1-5
	1.4 Characterization Techniques of 2D Materials	1-7
	References	1-10
Chapter 2	Optical Based Techniques for 2D Layered Materials	2-1
	<i>S. Reshmi, R. Sundheep, and Dattatray J. Late</i>	
	2.1 Optical Microscopy and Imaging Techniques	2-1
	2.2 Nonlinear Optical Property-Based Approaches	2-7
	2.3 UV-Visible and Photoluminescence	2-10
	2.4 Dynamic Light Scattering and Zeta Potential	2-15
	2.5 Conclusion	2-17
	References	2-18
Chapter 3	Raman Spectroscopy-Based Techniques for 2D Layered Materials	3-1
	<i>Levna Chacko and Dattatray J. Late</i>	
	3.1 Introduction	3-1
	3.2 Raman Characterization of Different Types of 2D Materials	3-2
	3.3 Phase Transition Triggered by Defects	3-5
	3.4 Effects of Doping and Temperature	3-6
	3.5 Thickness-Dependent Interlayer and Interlayer Modes	3-8
	3.6 Two-Dimensional Alloys with Tunable Compositions	3-11

3.7	Layer-Dependent, Temperature, and Pressure Dependent Raman Spectroscopy	3-14
3.8	Raman Spectroscopy to Probe the Moiré Phonons	3-16
3.9	SERS in 2D Materials	3-17
	References	3-18
Chapter 4	X-Ray-Based Techniques for 2D Materials	4-1
	<i>Pratik V. Shinde, Vitthal M. Shinde, and Chandra Sekhar Rout</i>	
4.1	Introduction	4-1
4.2	X-Ray Diffraction (XRD)	4-2
4.3	X-Ray Fluorescence (XRF)	4-9
4.4	X-Ray Absorption Fine-Structure (XAFS)	4-11
4.5	X-Ray Photoelectron Spectroscopy (XPS)	4-12
4.6	Summary and Future Scope	4-18
	Acknowledgments	4-19
	References	4-19
Chapter 5	Probe-Based Techniques for 2D Layered Materials	5-1
	<i>Aditya Sharma and Chandra Sekhar Rout</i>	
5.1	Introduction	5-1
5.2	Scanning Tunneling Microscopy	5-3
5.3	Atomic Force Microscopy	5-5
5.4	Kelvin Probe Force Microscopy (KPFM)	5-8
5.5	Conclusion	5-11
	Acknowledgments	5-11
	References	5-12
Chapter 6	Electron Diffraction-Based Techniques for 2D Layered Materials	6-1
	<i>Abhinandan Patra and Chandra Sekhar Rout</i>	
6.1	Introduction	6-1
6.2	Scanning Electron Microscopy (SEM)	6-2

6.3	Transmission Electron Microscopy (TEM)	6-5
6.4	Electron Diffraction Technique	6-7
6.5	State-of-the-Art of <i>In Situ</i> , Environmental, and Cryo-TEM-Based Characterizations	6-8
6.6	Conclusion and Future Outlooks	6-12
	Acknowledgments	6-13
	References	6-13
Chapter 7	Analytical Techniques for Hardness, Nanoindentation, and Elastic Modulus	7-1
	<i>Namsheer K and Chandra Sekhar Rout</i>	
7.1	Introduction	7-1
7.2	Analytical Techniques for Nanohardness	7-2
7.3	Analytical Techniques for Nanoindentation	7-3
7.4	Elastic Modulus of 2D Materials	7-9
7.5	Theoretical Models	7-11
7.6	Conclusion	7-11
	Acknowledgments	7-11
	References	7-12
Chapter 8	Analytical Techniques for Pore Size and Specific Surface Area Analysis	8-1
	<i>Rutuparna Samal, Sithara Radhakrishnan, and Chandra Sekhar Rout</i>	
8.1	Introduction	8-1
8.2	General Terminology	8-3
8.3	Analysis of Pore Size and Surface Area	8-3
8.4	Surface Areal Analysis of State of the Art 2D Materials	8-10
8.5	Limitations of BET	8-17
8.6	Conclusions	8-20
	Acknowledgments	8-21
	References	8-21

Chapter 9	Analytical Techniques for the Wettability and Contact Angle	9-1
	<i>Shobhnath P. Gupta, Sayed M. Hasnain, Rajesh Jaiswar, Dattatray J. Late, and Pravin S. Walke</i>	
	9.1 Introduction	9-1
	9.2 Concept of Wettability	9-2
	9.3 Factors Affecting Wetting	9-3
	9.4 Contact Angle	9-4
	9.5 Types of Contact Angles	9-5
	9.6 Analytical Methods for Surface Wetting	9-7
	9.7 Wettability of 2D Materials	9-11
	9.8 Electrical Double Layer (EDL) of 2D Materials	9-15
	9.9 Summary	9-16
	References	9-17
Chapter 10	Twistronics: Investigating Twist Angles in 2D Materials	10-1
	<i>Riddhi Kadrekar and Dattatray J. Late</i>	
	10.1 Introduction	10-1
	10.2 Synthesis Techniques	10-3
	10.3 Techniques to Control and Manipulate the Twist	10-4
	10.4 Characterization Techniques to Understand Twist Materials	10-5
	10.5 Conclusion	10-10
	References	10-10
Chapter 11	Density Functional Theory: An Investigative and Predictive Tool for the Study of 2D Materials	11-1
	<i>Riddhi Kadrekar and Dattatray J. Late</i>	
	11.1 Introduction	11-1
	11.2 Structural Optimization and Material Stability	11-2

	11.3 Study of Material Properties	11-3
	11.4 Material Engineering	11-11
	11.5 Conclusions	11-19
	References	11-19
Chapter 12	Summary	12-1
	<i>Dattatray J. Late, Abhinandan Patra, and Chandra Sekhar Rout</i>	
Index		I-1

