CHAPTER

1 INTRODUCTION

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1.1 INTRODUCTION

With the rapid development of engineering and science, the technological debate often begins with an understanding of the material system. It is usually argued that material attributes are determined by the material composition. However, it is not just the material composition that determines their behavior, but also their size and dimensionality. This is particularly true for some materials when their size may be expressed in terms of nanometers, or when their dimensions are reduced to the nanoscale. In the current nanotechnology era, materials can be easily modified at the nanoscale (Prasad *et al.*, 2021). Materials are analyzed by having at least one dimension at the nanoscale. The dimensionality of the nanoscale materials allows them to be categorized as shown in Fig. 1.1 (Khan *et al.*, 2019). A dimension constraint results in a two-dimensional material with a sheet structure. When the size of two dimensions is restricted, 1D material is generated, and when all dimensions are restricted to nanoscales, 0D materials or quantum dots are formed (Prasad *et al.*, 2021). During his speech "there is plenty of room at the bottom," Richard P. Feynman (Nobel Prize laurate) explored the possibility of layered materials along with the concept of nanotechnology in front of researchers with two questions: (1) What could we accomplish with just the necessary layers of layered structures? (2) What would be the material property if we could truly arrange the atoms wherever we wanted? (Zhao and Ryu, 2022). This talk inspired scientists like Frindt to isolate thin sheets of MoS_2 using the micromechanical peeling process and later chemical routes such as lithium intercalation to synthesize a single layer of MoS_2 (Joensen *et al.*, 1986). Finally, Novoselov and co-workers demonstrated the ability to isolate and measure electrical properties of single atom-layered graphene (Novoselov *et al.*, 2004). Additionally, they showed that repeated mechanical peeling might isolate other single-layered 2D materials. Over fifty years after Feynman's lecture, it is interesting to note that we now understand that changing the number of layers in these materials enables us to design incredible physicochemical features (Prasad *et al.*, 2021). The library of these 2D-layered materials develops year after year, and it now has over 150 interesting families. 2D material research is in its early stages, with several new materials being presented and added to the list each year. Generally, 2D materials are classified according to their structure: such as graphene, transition metal oxides (TMOs-MoO_3, MnO_2) (Guo *et al.*, 2015; and

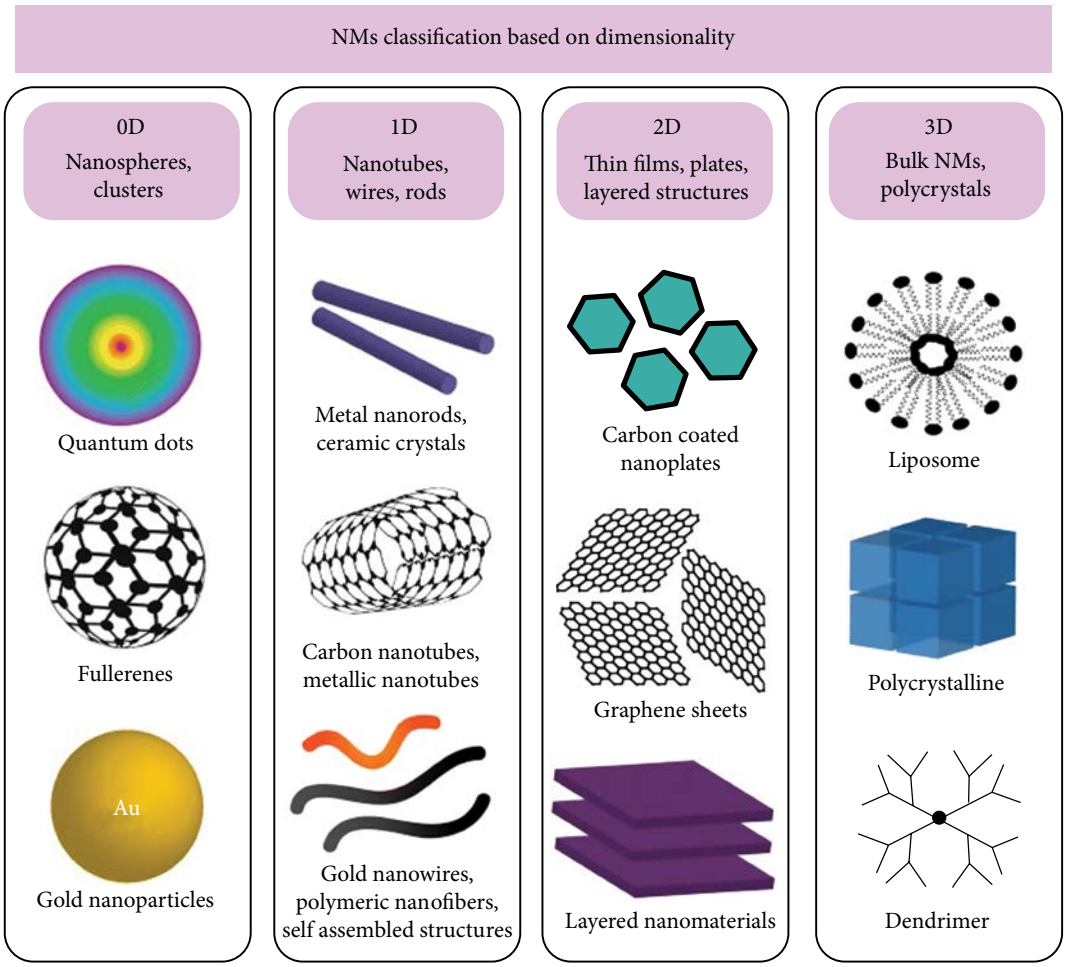

FIG. 1.1
Classification of nanomaterials. Reprinted with permission from Poh *et al.*, Part. Fibre Toxicol. **15**(1), 46 (2018). Copyright 2018 Author(s), licensed under a Creative Commons 4.0 License.

Yadav *et al.*, 2022), halides (TMHs) (Huang *et al.*, 2021) and dichalcogens (TMDCs-MoS_2, $MoSe_2$, WS_2 and WSe_2) (Yang *et al.*, 2020), hexagonal boron nitride (h-BN) (Song *et al.*, 2010; and Zhang *et al.*, 2017), a class of monoelemental compounds (Xenes), metal carbides/nitrides (MXene) (Gogotsi and Anasori, 2019), perovskite-type oxides, and 2D polymers (Evans *et al.*, 2022). These 2D-layered materials, which have weak van der Waal forces and strong in-plane bonding, look like a huge sheet with single or few atom thickness layers (similar to a sheet of paper) (Vargas-Bernal, 2016).

The linearity of a monolayer graphene at the Dirac point causes a quantum Hall effect, which aids in the exploration of a new subject of "Fermi–Dirac" physics (Liu *et al.*, 2020). Even at a thickness of one atom, this material is an amazing thermal and electrical conductor and is proposed for various applications ranging from thermal-interface materials to barrister transistor-like devices to transistor transparent conductors (Agrawal, 2018). These layered materials also have more active sites due to their high aspect ratio, making them suitable for surface active applications such as catalysis (Zhang, 2015).

1.2 HISTORY OF 2D MATERIALS

Two-dimensional materials were originally thought to be impossible to find in nature due to their environmental instability. However, there are already about 700 stable 2D materials that are theoretically recognized. This is despite the fact that some of them have yet to be synthesized. The first investigations of the atomic and electrical structures of silicene and germanene were performed in 1994, ten years before the discovery of graphene. Carbon foils with a single layer received the suffix "ene" in 1962, according to Boehm. It is astounding to realize that graphite has been pretty well known among researchers for a very long time when the numerous carbon discoveries have been analyzed consecutively (particularly dating back to the 16th century) (Kroto *et al.*, 1985). Graphite has also found widespread industrial applications, particularly in the manufacture of steel, as brake linings and dry lubricants. However, it wasn't until the discovery of fullerenes in 1985 that the presence of many other carbon allotropes came to light. This also demonstrated the presence of 1D carbon nanotubes. The studies regarding carbon nanotubes were first presented in 1991 (Iijima, 1991). These discoveries have fueled massive amounts of research, enabling scientists to create 1D nanoribbons or extract 2D graphene materials from 2D graphite crystals. Since then, numerous discoveries on nanotubes, graphite, and fullerenes have been made, with graphite (single-layered) being the oldest substance. Several efforts have been made in the last 50 years to achieve graphene sheets. However, its success rate appears to be modest because, prior to the 1990s, people utilized a process known as intercalation, in which atoms are switched between graphitic layers to weaken interplanar forces and accelerate layer separation (Gupta *et al.*, 2015). Finally, Novoselov *et al.* mechanically exfoliated a single sheet of graphene in 2004. This is a significant step in developing a new class of materials called 2D materials. Graphene was given its name for the solitary, isolated graphite layers that it has created (in bulk form). Consequently, graphene was the first contemporary carbon-based two-dimensional substance, and its synthesis proved that it is possible to create materials with a single, stable, and few atoms thick layer with a variety of amazing technologically useful properties (Vargas-Bernal, 2016). Researchers are now concentrating on the analysis and synthesis of non-carbon-based 2D materials as a result of the discovery of graphene. Additional 2D materials have been identified since then. TMDs are the primary and extended lineage of two-dimensional materials that have sparked research interest. Due to its distinctive electrical, optical and mechanical qualities, it has drawn considerable attention (Butler *et al.*, 2013). TMD bulk crystal, like graphite, is made up of monolayer-sheets bound by van der Waal forces. However, these characteristics differ from those of graphene semimetals. Because of

their direct-bandgap nature, they can be used in electronics as transistors and in optics as detectors and emitters. TMDs find applications in spintronics due to their high spin–orbit coupling and 2D nature. TMDs differ from graphene in that they have a sandwich structure with two chalcogens sandwiching the transition metal atomic layer (Choi *et al.*, 2017). Graphene, on the other hand, has a carbon layer that is only one-atom thick. The rediscovery of graphene and TMD paves the way to understand the differences in the properties of 2D and 3D materials, which opens the door of understanding the properties of 2D carbides, oxycarbides, nitrides and other related structures. The MAX phase is a large family of materials and are well known for the last 25 years. These layered ceramic materials have always been produced as a 3D material, until researchers from Draxel University in 2011, selectively etched Al from Ti_3AlC_2 to produce MXene nanosheets (Gogotsi and Anasori, 2019). The first reported MXene was $Ti_3C_2T_X$. Many other 2D materials, such as Ti_3AlCN, Ti_2AlC, $(V_{0.5}Cr_{0.5})_3AlC_2$, $(Ti_{0.5}Nb_{0.5})_2AlC$, and Ta_4AlC_3, were exfoliated within a few months. MXenes have been manufactured in over 30 different variations. It is one of the 2D material families whose theoretical predictions of the most have been confirmed (Anasori *et al.*, 2017).

In 2012, the process known as molecular beam epitaxy (MBE) was used to synthesize a 2D allotrope of silicone termed silicene over a silver crystal. A targeted search for related 2D molecules created using similar methods, such as MBE, was sparked by the synthesis of silicene. Silicene's characteristics differ significantly from graphene's. Their two-dimensional structure is the sole thing that unites them. These substances neither arise naturally nor have a parent crystal with a three-dimensional layer. Thus, exfoliation is no longer a viable option, leaving chemical manufacture as the only alternative. The fabrication can also be performed on a substrate using the epitaxial growth approach. As a result, the electrical and structural properties may be substrate dependent (Vogt *et al.*, 2012). Exfoliated black phosphorus (BP) appears to be particularly intriguing because of its bandgap, the value of which relies on the number of stacked layers discovered as a result of graphene research. In this regard, the direct bandgap can be adjusted from 0.3 eV for bulk BP to around 2 eV for monolayer phosphorene. Despite the fact that the BP was discovered many years ago, its significance and interest arose around the beginning of 2014. Two groups concurrently reported the isolation of BP with thicknesses ranging from 2 to 10 nm using the Scotch tape approach, which was also employed for graphene exfoliation by Novoslov and Geim in 2004 (Li *et al.*, 2014; Liu *et al.*, 2014; and Peruzzini *et al.*, 2019). In the same year 2D germanium was reported by Guy Le Lay at France's Aix and his team (Dávila *et al.*, 2014). Other layered materials, such as antimonene, are also anticipated theoretically. According to the theoretical calculation, the semimetallic nature of the antimonene in bulk form will convert into a semiconductor in nature while being thinned into one-atom layer. In 2016, Lei *et al.* synthesized a monolayer of antimonene using MBE. However, because the products have small domains, further research has been limited. Mechanical exfoliation was used to obtain antimonene flakes, although the amount obtained was quite small. In a similar vein, 2D Tin, also known as Stanene, was also found in 2016 (Zhu *et al.*, 2015). There may be a large number of undiscovered 2D materials, according to an analysis of exfoliatable 3D materials. Among the new systems developed by researchers are hexagonal boron nitride and various transition metal chalcogenides (VS_2, VSe_2, VTe_2, $CrSe_2$, and so on). The history of 2D materials can be summarized and shown in Fig. 1.2.

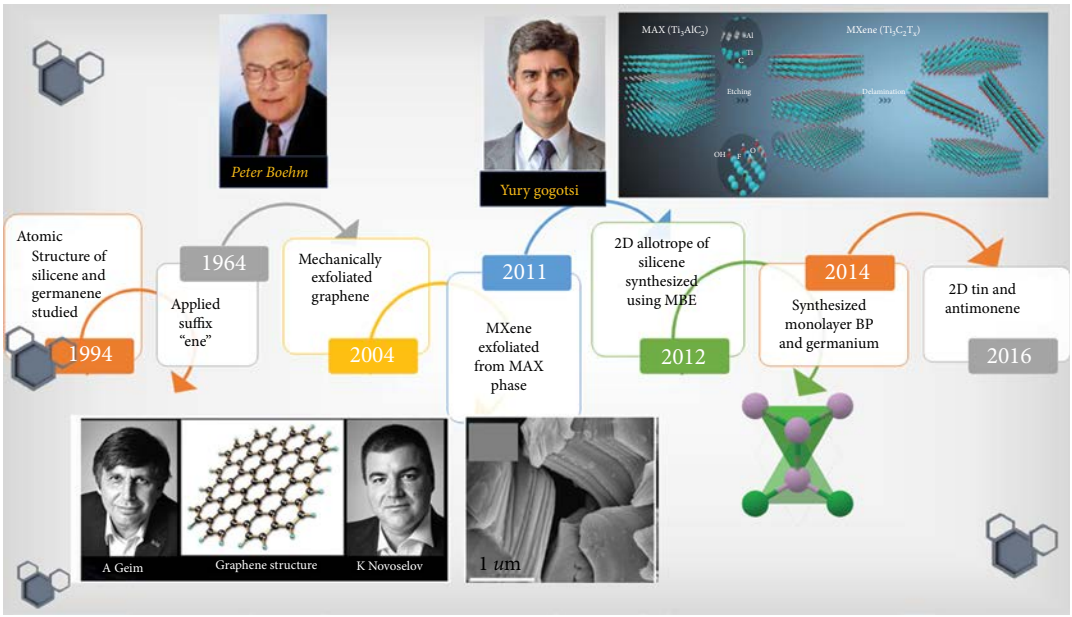

FIG. 1.2
History of 2D materials. Top right panel reprinted with permission from Zhang *et al.*, Chem. Soc. Rev. **49**, 7229 (2020). Copyright 2020 Author(s), licensed under a Creative Commons Attribution 3.0 Unported License.

1.3 SIGNIFICANCE OF 2D MATERIALS

Regarding 2D materials, there are many different questions that might be raised. What makes 2D materials unique? Why are they more attractive? Why do 2D materials' properties differ from their bulk structure? Is it possible that the next major technological advance will involve 2D nanomaterials? As we all know, the unusual physical properties of 2D inorganic materials brought about by the quantum effect and coupled to their nanosized thickness have recently attracted a lot of interest. Layered materials experience certain unexpected changes in their optical and electrical properties, presumably as a result of electron confinement, when the layers are in close proximity to one another to create interfaces. Their band structure is largely determined by this interlayer interaction. Mechanical and chemical responses are crucial in altering their properties because of quantum effects and a high surface-to-volume ratio (Gupta *et al.*, 2015).

A new class of materials can be engineered using several examples of 2D materials that have already been realized in practice. In fact, the "all-surface" nature of 2D materials permits surface modifications

that change their properties [e.g., by chemical functionalization (Xu *et al.*, 2009)]. Furthermore, a dispersion of tiny nanoflakes can be used to make 2D materials with ease (Hernandez *et al.*, 2008). Many of its unique qualities are still present, and they can be used in tried-and-true methods to create composite materials with better performance.

The important properties for the rapid progress of 2D materials is due to the abundance of remarkable properties observed. Being a one-atomic thick material, they appear to be the best choice for manufacturing of new generation electronic devices because their 2D properties allow simple processing and immediate access to charger carriers, ultrahigh carrier mobility even at low and room temperatures, and finally the excellent thermal conductivity. Single-layer and few-layer 2D crystals are two well-known allotropes where charge carriers resemble relativistic Dirac particles and where electrons also exhibit some Dirac-like characteristics but with parabolic dispersion. In many situations, both allotropes are gapless semiconductors (Geim, 2004). Few layers are more adaptable than a single layer, where the absence of a gap is protected by the honeycomb lattice's tight symmetry. A transverse component of an electric field can create a gap, and its low-energy band structure can be altered by very small strain, studying 2D materials' relativistic effects in condensed matter, a field made all the more fascinating by the inventive behavior of electrons. Understanding the behavior of 2D materials in the presence of a strong, quantizing magnetic field is important for understanding their transport properties. The accurate quantization of the device's Hall resistance, which is a fundamental quantum Hall effect phenomenon, is illustrated by this behavior (McCann and Fal'ko, 2006). This unusual band structure becomes increasingly prevalent when more layers (more than three) are added. The band structure of 2D materials is susceptible to tiny changes, including external electric field, mechanical deformations, doping, and lastly the adsorbents, which are needed for sensing applications, because of their brittleness in one contact point (Meyer *et al.*, 2007). Nevertheless, local electric fields can also alter the charge carrier sign, thus doping is not always necessary (Novoselov *et al.*, 2004). The creation of highly mobile p-n junction transistors, the cornerstone of modern complementary metal oxide semiconductor (CMOS) technology, requires the specific properties of 2D materials. Additionally, the structural research of 2D materials reveals that they are capable of growing the finest crystals at micrometer sizes. This defect-free substance enables electrons to move freely without scattering, even on rough surfaces. High charge carrier mobilities are produced in the absence of scattering, demonstrating the essential properties of the quantum Hall effect even at room and low temperatures. FETs also employ single-layer 2D materials with direct bandgaps, yielding on/off ratios of 10^8 (Radisavljevic *et al.*, 2011).

Due to the availability of large mobile charge carriers, 2D materials are excellent candidates for high-gain photodetection. Due to photogenerated carriers close to the top layer, this material's conductivity is thus particularly sensitive to electrostatic disturbance (surface). Because of this, 2D materials are a particularly good choice for photodetection with high gain via the photogating effect. As a result, the 2D material's evidence of photodetection improvement would pave the way for a variety of applications, including integrated optoelectronic devices, optical communications, remote sensing, and biological

imaging (Heersche *et al.*, 2007). Additionally, 2D material thickness makes it possible to quantify sample conductance field variations, which is crucial for optoelectronic and sensor applications. Due to their one-atom thickness, 2D materials' electron waves can be extensively characterized using surface characterization techniques. The 2D materials' electronic band structures are extremely fascinating. A wide range of light absorption across the electromagnetic spectrum, from infrared (IR) to ultraviolet (UV), is possible with these materials due to their minimal bandgap. Due to the numerous electrical transition choices made possible by this, high-performance optoelectronic devices as well as other fundamental investigations in photonics and optics are produced (Nair *et al.*, 2008; and Mao *et al.*, 2013). A 2D material with few layers has an absorbance of less than 10%, whereas a monolayered material has an absorbance of 2.3%, which can be related to their thickness and transparency (Nair *et al.*, 2008). They are perfect for use as transparent electrodes in a variety of electrical devices, including liquid crystal displays and solar cells, due to their extraordinary transparency and high conductivity (Eda *et al.*, 2008; and Wang *et al.*, 2008). It is necessary to comprehend how 2D material functions in real devices and how long it lasts under challenging and ambient conditions for it to be employed in practice. A specific effort is needed to explore the dependability of 2D materials-based devices, such as electric or thermal stress tests, device longevity, and so forth. It is envisaged that some 2D material and metal protection will be needed to reduce external impacts in order to preserve device performance. The strength of the 2D materials surpassed that of graphene in all tests. It has a remarkable breaking strength that is 200 times stronger than steel, and these materials' flexibility varies greatly (Lee *et al.*, 2008). As a result, the 2D material enhances the mechanical properties of lightweight polymers while also adding an appealing new component. Additionally, single layer, which is an ultrathin stretchy membrane, is a suitable material for nonlinear tunable electrochemical systems. It has a very wide range of flexibility and can withstand more than 20% elastic deformation (Meyer *et al.*, 2007). In conclusion, the material is mechanically fascinating due to its 2D nature and chemical bond strength. Two-dimensional materials must also undergo a chemical change. In this context, it has been shown that functionalization of 2D materials can lead to chemical doping with different metal ions and a metal-to-insulator conversion (Gómez-Navarro *et al.*, 2008). If there are imperfections or functional groups present, mechanical properties can be modified. All 2D material devices with different regions and varied properties can be produced thanks to these chemical qualities, which are ideal for local production (Gupta *et al.*, 2015).

1.4 CHARACTERIZATION TECHNIQUES OF 2D MATERIALS

As nanotechnology has grown quickly, some effective characterization methods for 2D materials have been created to show their size, thickness, flaws, oxidation states, composition, crystallinity, and electrical properties. A thorough and comprehensive characterization of 2D nanomaterials is necessary to comprehend the connection between structural characteristics and properties. The ideal 2D material is a nanomaterial with an infinite lateral size and an atom thickness that has novel features

due to the quantum confinement effect. Therefore, characterizing these 2D materials is the first step in examining how their properties change depending on the layer and using them for a wide range of 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 (Anand *et al.*, 2021). Microscopy, atomic force microscopy (AFM), scanning electron microscopy (SEM), transmission electron microscopy (TEM), Raman spectroscopy, x-ray diffraction (XRD), x-ray photoelectron spectroscopy (XPS), etc., 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. Further, 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. Chapters 2 and 3 discuss the optical-based techniques and x-ray based techniques to characterize 2D nanosheets and their heterostructures. The electronic properties of 2D materials can be understood by UV–visible absorption and photoluminescence emission techniques. These linear optical spectroscopic techniques can be further used to study the effect of defects and the band structure at heterostructure interfaces. Nonlinear spectroscopy techniques can be specially employed to study strain in 2D materials as they can provide intricate details about the strain existing in these materials. Optical characterization methods based on scattering techniques, especially dynamic light scattering can be utilized to study the stability of liquid-exfoliated 2D materials. Similarly, spectroscopy-based analytical tools such as Raman spectroscopy and surface enhanced Raman scattering (SERS) discussed in Chap. 3 are useful to determine the composition, quality, bandgap, doping, surface charge, thickness, the number of layers, orientation, and chemical bonding. Also, Chap. 4 deals with the nondestructive x-ray technique to characterize 2D materials. The nondestructive XRD method provides details on the phases, sizes, and crystallinity of 2D materials. Furthermore, the study of *ex situ* and *in situ* XRD analysis of 2D materials also plays a significant role in understanding their electrochemical performance. *In situ* XRD analysis gives useful information on the crystalline structure of materials as well as changes occurring within the structure at the atomic scale. XPS is a very useful technique for examining and quantifying various features of 2D materials, such as phases, chemical bonds, and chemical states. *In situ* XPS analysis is useful for analyzing electron migration or charge transfer in processes, which helps in understanding the performances of materials. X-ray fluorescence (XRF) is a useful instrument for determining the composition and concentration of elements in 2D materials. Therefore, this technique plays a crucial role in determining the atomic ratio of 2D materials that are doped or undoped. X-ray absorption fine structure (XAFS) is an important tool in determining the coordination chemistry, coordination number, valence states, etc. Because of this, it is very important to understand the performance of 2D materials in electrochemical reactions. The probe-based analytical techniques discussed in Chap. 5, such as AFM, STM can provide the information on the orientation, smoothness, number of layers, grain size, depth profile, and dielectric properties of the 2D materials. Electron diffraction-based techniques such as TEM, SEM, and energy-dispersive x-ray spectroscopy (EDS)

discussed in Chap. 6 are useful for determining thickness, number of layers, high-resolution imaging, surface properties, composition, and morphology. X-ray based techniques such as XRD and XPS are being used to gain information on crystallinity, interlayer distance, interface information, composition, chemical bonding, layer interaction, and defects. In addition to morphological examination, probe-based analytical techniques such as AFM may analyze the mechanical properties of 2D materials, such as hardness and elastic properties. These approaches discussed in Chap. 7 offer an efficient strategy for determining the mechanical behavior of a material by applying a little force; the force displacement profile provides an overall picture of the mechanical stability of 2D materials for flexible and wearable electronic applications.

A thorough examination of the pore architecture and surface area is specifically vital in 2D materials because they influence diffusion rates, transport phenomena, rule selectivity, and performance in catalyzed reactions. Chapter 8 discusses the major aspects of the underlying mechanisms of the most commonly used Brunauer–Emmett–Teller (BET) method for evaluating the surface area and pore size of porous and finely divided 2D nanomaterials. Chapter 9 describes wettability, contact angle measurements, electric double layer (EDL) structures, and frictional interactions that arise from interactions between 2D materials and liquids. The numerous analytical methods used to

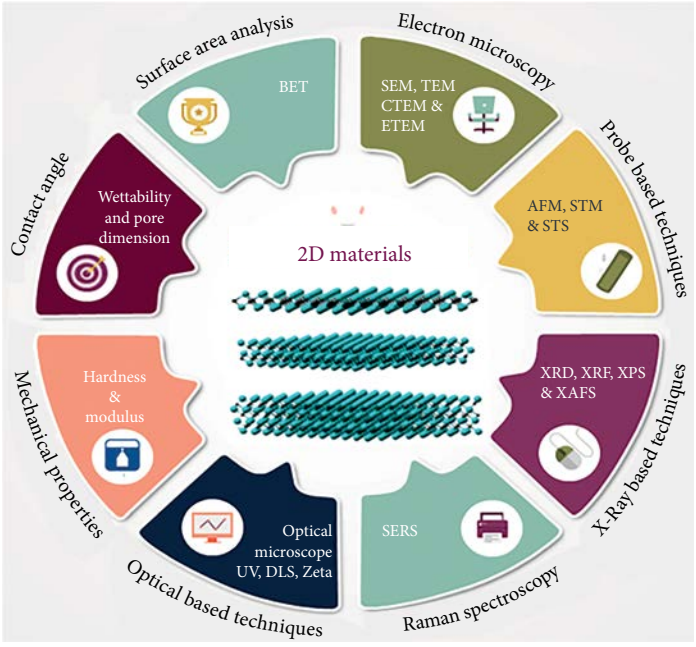

FIG. 1.3
Summary of the techniques covered in this book that are used for the characterization of 2D materials.

characterize the wetting behavior of 2D materials are described. All of the unique qualities presented by 2D materials are determined by composition, configuration, number of layers, and morphology. Identifying the optimum material entails recognizing the ideal combination of composition, configuration, number of layers, and morphology from a set of novel materials produced by slightly altering one or more of these factors. It is done experimentally using an "iterate and test" technique. A wide range of potential active materials are produced and described, and their performance is assessed separately.

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 the different 2D nanomaterials shown in Fig. 1.3. This book will be useful for researchers and scientists in the areas of materials science and engineering. This book will serve as a textbook both to beginners and experienced researchers who are pursuing their research in 2D-layered materials and their advanced applications.

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