Recent Progress in Contact Probing Methods of Two-Dimensional Materials and Van Der Waals Heterostructures

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Due to the unique properties, two-dimensional materials and van der Waals heterostructures play an important part in microelectronics, condensed matter physics, stretchable electronics and quantum sciences. But probing properties of two-dimensional materials and van der Waals heterostructures is hard as a result of their nanoscale structures, which hinders their development and applications. Therefore, the progress of contact probing measurement in recent years including mechanical properties, interfacial properties, tribological properties, as well as electrical properties are summarized in this paper. It is found that useful properties such as Young's modulus, adhesive energy, friction coefficient and so on can be well estimated from contact probing methods. We believe that the contact probing methods will be more advanced to promote the blooming applications of two-dimensional materials and van der Waals heterostructures.

Keywords: 2D materials, van der Waals heterostructure, contact probing, adhesive energy, friction

1 INTRODUCTION

Compared with bulk materials, two-dimensional (2D) materials (Bhimanapati et al., 2015; Cheng et al., 2019; Su et al., 2021) are of typical membrane structure with thickness of only a few angstrom. Since graphene was first discovered by Novoselov et al. (2004), new members of 2D material family, e.g., h-BN (Golberg et al., 2010), transition metal dichalcogenides (TMDs) (Chhowalla et al., 2013), black phosphorous and MXenes (Tran et al., 2014; Lu et al., 2015; Bu et al., 2020; Ihsanullah, 2020) have been fabricated and developed. Due to the versatile electrical, chemical, thermal, optical and mechanical properties (Kang et al., 2016; Lee and Park, 2017; Wu et al., 2018a; Jiang et al., 2019; Du et al., 2020; Zhao and Qin, 2021), the 2D material family has drawn great attentions of scientists and engineers and become the research hotspot in scientific studies of various fields. More recently, by assembling 2D materials *via* van der Waals (vdW) interactions, vdW heterostructures (Jariwala et al., 2017; Liao et al., 2019b; Geng et al., 2019; Liu et al., 2020; Onodera et al., 2020) are proposed and fabricated, which provide a platform of combining distinct two-dimensional materials and designing novel devices of special mechanical, electrical and magnetic properties. For example, the graphene/TMDs heterostructure exhibits totally different mechanical and electronic properties compared with single graphene and TMDs. In order to enhance our knowledge and accelerate applications of 2D materials and vdW heterostructures, it is crucial to measure and characterize their mechanical,

FIGURE 1 | Some probing methods of 2D materials and vdW heterostructures and their application scenarios. AFM can be used to test the elastic properties, interface properties and fracture properties of 2D materials. FFM can be used to measure the friction of 2D materials and draw the surface topography. SEM can provide nanoscale high-resolution images. CAFM can be used to measure the electrical properties of 2D materials. Inserted figures are reproduced with permission (Huang et al., 2011; Ye et al., 2012; Tao et al., 2015).

interfacial, tribological, and electrical properties properly through suitable strategies (Das Sarma et al., 2011; Britnell et al., 2013; Hong et al., 2014; Deng et al., 2016).

In past decades, many efforts have been done on contact probing methods of two-dimensional materials and vdW heterostructures. With the development of scientific instruments, e.g., atomic force microscopy (AFM), friction force microscopy (FFM), scanning electron microscopy (SEM), as shown in **Figure 1**, contact probing measurement has become key methods in characterizing properties of 2D materials and vdW heterostructures. Specifically, by using AFM, the indentation load-displacement relationships of 2D materials and the corresponding substrates can be measured, and thus the mechanical properties can be measured with the help of analytical models. What is more, combined with AFM, the blister test can be used to measure interfacial adhesive energy. Derived from AFM, FFM exploits the AFM tip to make in-plane scratch on 2D materials and vdW heterostructures, and makes use of the topographic map and friction curve to explore the interfacial and tribological properties. Different from AFM and FFM, SEM is not used independently but as an auxiliary means to measure 2D materials. For instance, obtained results will be more accurate by combining the probe of AFM and the assisted image of SEM.

So far, many works have reported and summarized the contact probing methods for bulk materials (VanLandingham, 2003; Cheng and Cheng, 2004; Chen, 2012). Meanwhile, increasing scientific literatures and works are developing on probing methods for 2D materials and vdW heterostructures. However, due to high precision requirement and the difficulty of sample preparation, it is impossible for scientific researchers and engineers to have a comprehensive understanding of the new progresses in contact probing methods for 2D materials and vdW heterostructures, which hinders their development and

applications. What is more, contact probing measurement of properties of 2D materials and vdW heterostructures is hard as a result of their nanoscale structures, which is different from the characterization of bulk materials. Therefore, it is key to introduce and summarize representative works of contact probing properties of 2D materials and vdW heterostructures, which are of importance in microelectronics, condensed matter physics, stretchable electronics and quantum sciences.

In this mini review, we focus on recent progress of contact probing measurement of 2D materials and vdW heterostructures. The working mechanism of contact probing method and the measurement of mechanical properties of 2D materials and vdW heterostructures are introduced first. Then, the measurement of interfacial properties is discussed in **Section 3**. **Section 4** introduces the measurement of tribological behavior of 2D materials. In **Section 5**, the contact probing measurement of electrical properties of 2D materials and vdW heterostructures are given. Finally, conclusion and perspectives of contact probing methods are presented. The results of the paper should be helpful for the blooming scientific researches and applications of 2D materials and vdW heterostructures.

2 THE MEASUREMENT OF MECHANICAL PROPERTIES

To obtain mechanical properties of bulk materials such as Young's modulus, shear modulus, Poisson's ratio, strength, fracture toughness, hardness, etc., traditional measurement methods include the tensile test, the compressive test, impact test as well fatigue test and so on. However, due to the difficult manipulation and other restricted conditions of 2D materials and vdW heterostructures, these traditional methods cannot function very well. Thus, contact probing methods are thus be designed and employed to measure mechanical properties of 2D materials and vdW heterostructures with the help of the force feedback from direct contact and optical measurement.
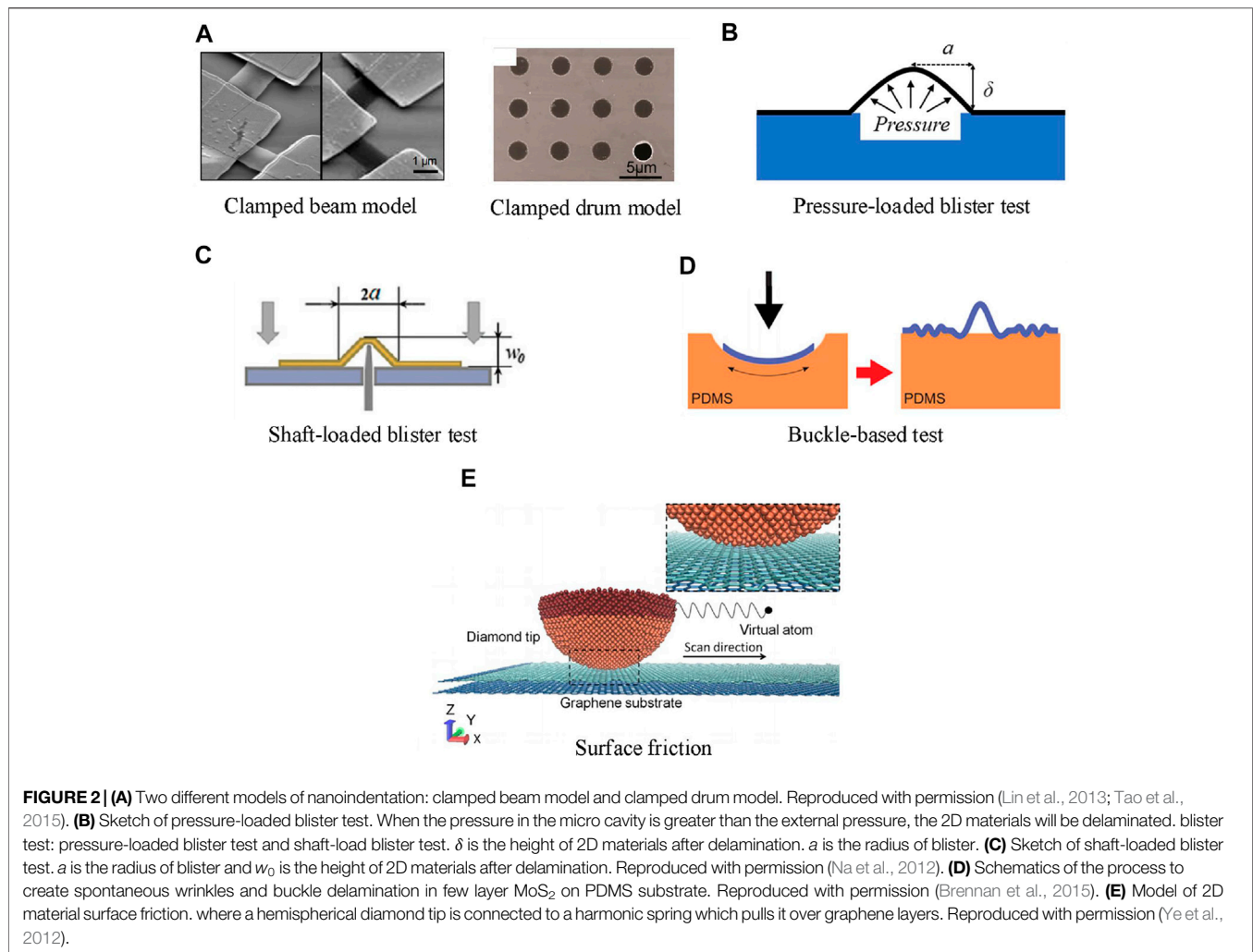
2.1 Nanoindentation Model
The mechanical properties of 2D materials can obtained by indentation. During the loading process, the indentation load-displacement relationship is recorded. According to existing research, the applied force F and the probing depth d have the following relationship (Cao and Gao, 2019),

$$F(d) = \alpha d + \beta d^3 \qquad (1)$$

Where α and β are functions of elastic parameters determined based on different theoretical models.

Though the working mechanism of contact probing measurement is simple, while accurate measurement of mechanical properties of 2D materials and vdW heterostructures highly depends on the setup of contact probing experiment and the choice of theoretical models. In existing theoretical models, the effect of the tip is treated as a point load except cases that the ratio of sample size to tip radius is not large enough.

FIGURE 2 | (A) Two different models of nanoindentation: clamped beam model and clamped drum model. Reproduced with permission (Lin et al., 2013; Tao et al., 2015). **(B)** Sketch of pressure-loaded blister test. When the pressure in the micro cavity is greater than the external pressure, the 2D materials will be delaminated. blister test: pressure-loaded blister test and shaft-load blister test. δ is the height of 2D materials after delamination. a is the radius of blister. **(C)** Sketch of shaft-loaded blister test. a is the radius of blister and w_0 is the height of 2D materials after delamination. Reproduced with permission (Na et al., 2012). **(D)** Schematics of the process to create spontaneous wrinkles and buckle delamination in few layer MoS_2 on PDMS substrate. Reproduced with permission (Brennan et al., 2015). **(E)** Model of 2D material surface friction. where a hemispherical diamond tip is connected to a harmonic spring which pulls it over graphene layers. Reproduced with permission (Ye et al., 2012).

2.2 Clamped Beam Model

As shown in **Figure 2A**, the clamped beam is a typical experimental setup in contact probing experiments. For the clamped beam model, the parameters in **Eq. 1** should be $\alpha = \left(\frac{\pi^4 Ewt^3}{6L^3} + \frac{T}{L}\right)$ and $\beta = \frac{\pi^4 Ewt}{8L^3}$, where E is the Young's modulus, w, L, and t are the width, length and thickness of the sample, respectively, and T is the pretension in the suspended film (Pruessner et al., 2003; Nadler and Steigmann, 2006).

Until now, Frank et al. (2007) used this model to measure effective spring constants of stacks of graphene sheets, which agrees well with the value of dimension analysis. With the clamped beam model, the elastic properties of thin clay tactoids were studied by Kunz et al. (2009). Recently, Tao et al. (2015) conducted the contact probing experiment of a clamped black phosphorous beam. What is more, Yang et al. (2017b) studied the brittle fracture of 2D $MoSe_2$, whose average fracture strength was 4.8 ± 2.9 Gpa.

2.3 Clamped Drum Model

Compared with the clamped beam model, it is easier to fabricate clamped drums in experiment. For the clamped drum model, the

parameters in **Eq. 1** should be $\alpha = \frac{\left[\frac{4\pi Et^3}{3(1-\nu^2)a} + T\right]}{a}$ and $\beta = \frac{E\psi^3 t}{a^2}$, where a is the radius of the drum sample, ν is the Poisson ratio, ψ is a function of ν (Pruessner et al., 2003; Wan et al., 2003; Begley and Mackin, 2004; Komaragiri and Begley, 2005; Steigmann, 2005; Nadler and Steigmann, 2006). With the help of the $F\text{-}d$ relationship, the Young's modulus E and other material parameters can be obtained.

The elastic properties of monolayer graphene and suspended MoS_2 were measured by using this model (Castellanos-Gomez et al., 2012).

Lee et al. (2008) measured the elastic properties and intrinsic strength of monolayer graphene, whose breaking strength was 42 N m^{-1}, and Young's modulus was 1.0 Tpa. Lin et al. (2013) studied the mechanical properties of graphene grown by chemical vapor deposition (CVD), and found that it is important to determine the zero-displacement point of the force-displacement curve precisely. The elastic properties of suspended MoS_2 sheet was studied by Castellanos-Gomez et al. (2012), and the Young's modulus for ultrathin MoS_2 flakes was 0.33 ± 0.07 Tpa. Bertolazzi et al. (2011) studied stretching and breaking of ultrathin MoS_2. In addition, Hatter

et al. (2020) measured elastic properties of MXene reinforced epoxy composites with this model.

It is worth noting that the clamped drum model rather than the clamped beam model are used in existing nanoindentation molecular dynamics (MD) simulation. Zhao et al. (2013) obtained the temperature-dependent mechanical properties of monolayer MoS_2. Tan et al. (2013) studied the size effect of indenter on probing results. Mechanical properties and thickness-determined fracture of h-BN were studied by Liu et al. (2021). In addition, Mallick and Elder (2018) measured the mechanical properties of graphene/h-BN heterostructures.

A final note about nanoindentation test is that, whether it is clamped beam model or clamped drum model, there will be slippage in the process of indentation. The slippage will cause errors in the experiment (Dai and Lu, 2021). However, different clamping methods may lead to different results. For example, in the work of Tao et al. (2015), the end of black phosphorus beam was sandwiched between Au and SiO_2. In the work of Lee et al. (2008), 2D materials are laid on a pre-fabricated cavity substrate *via* vdW interaction. These two different clamping methods may lead to different results.

3 THE CHARACTERIZATION OF INTERFACIAL PROPERTIES

Interfacial properties, such as adhesive energy, are key parameters in various applications of 2D materials, especially, 2D materials are usually fixed on or transferred to certain substrates. In addition, the stability of vdW heterostructures also depends on its interfacial properties. In order to assemble 2D materials and obtain stable vdW heterostructures, various contact probing methods (Jin, 2008; Zhao et al., 2010; Koenig et al., 2011; Bunch and Dunn, 2012; Na et al., 2012; Lou et al., 2020) have been developed and analyzed.

3.1 The Blister Test
As shown in **Figure 2B**, the pressure-loaded blister test is generally performed by generating a pressure difference between inside and outside of the sealed microcavity. AFM can be used to measure the geometry of blister, including maximum deflection δ and radius a for different values of pressure. We can obtain the adhesive energy $W = V(a)\delta$, where $V(a)$ is a function of radius a (Koenig et al., 2011). The adhesive energy of monolayer graphene and SiO_2 substrate was measured with pressure-loaded blister test, which was 0.24–0.45 J m^{-2} (Koenig et al., 2011; Boddeti et al., 2013). However, the adhesive energy of multilayer graphene and SiO_2 was 0.31 ± 0.03 J m^{-2}. By applying a pressure difference across the membrane, Bunch et al. (2008) measured both the elastic constants and the mass of a single layer of graphene. Wang et al. (2016b) presented a continuum model for vdW interactions between graphene and the corresponding substrate. What is more, Cao et al. (2014) measured the adhesive energy of the graphene/copper interface. In addition, Wang et al. (2017), Wang et al. (2019)

reported that the blister test can be applied to measure the interlayer shear and bending properties of 2D materials. Recently, more and more researchers have employed the nanoblister approach to identify interfacial features of 2D heterostructures (Wang et al., 2016a; Sanchez et al., 2018; Wang et al., 2022).

Different from the pressure-loaded blister test, the shaft-loaded blister (SLBT) test is another traditional measuring method of adhesive energy (Na et al., 2012). For the shaft-loaded blister test, a substrate with a pre-existing hole should be fabricated first, and then a film is deposited on or adhered to it. During the test, an external load is applied on the shaft to delaminate the upper film through the pre-fabricated hole, as shown in **Figure 2C**. We can get the adhesive energy W as,

$$W = \frac{Et}{16}\left(\frac{w_0}{a}\right)^4 \tag{2}$$

where the w_0 is the height of the blister, E is the Young's modulus and t is the thickness of film.

By using the shaft-load blister test, the adhesive energies of Pt/Al_2O_3, Cu/Al_2O_3, and Cu/Cr/Al_2O_3 interfaces were quantitatively evaluated (Berdova et al., 2013). In addition, Lii-Rosales et al. (2020) measured the adhesive energy between Fe and graphene or graphite. With the help of MD simulation, Lou et al. (2020) measured the adhesive energy of graphene/MoS_2 heterostructures.

3.2 The Buckle-Based Test
The blister test can be used to measure the adhesive energy between 2D materials and rigid substrates, but for the adhesive energy between 2D materials and elastic substrate, the buckle-based test is more suitable (Yang et al., 2017a). Note that the buckle-based test can be used not only for cases with elastic substrates but also for cases with rigid substrates.

For the buckle-based test, an elastic substrate is first covered with 2D materials, and then the film-substrate system is pressed by finger indentation. When the pressure is released, wrinkles or buckle delamination occurs as shown in **Figure 2D**. According to linear elastic fracture mechanics, a simple formula can be used to calculate the adhesive energy (Vella et al., 2009),

$$\Gamma = 2\pi^4 \frac{B\delta^2}{\lambda^4}, \tag{3}$$

where $B = \frac{\bar{E}h^3}{12}$ is the bending stiffness of the 2D material, λ and δ are the corresponding width and height, and $\bar{E} = \frac{E}{(1-\nu^2)}$.

The adhesive energy between MoS_2 and polydimethylsiloxane (PDMS) was measured by the buckle-based test, which was 18 ± 2mJ/m^2. Scharfenberg et al. (2011), Scharfenberg et al. (2012) studied the bending rigidity, adhesion, critical stress for graphene on elastic substrate. Deng et al. (2017) measured the adhesive energy of MoS_2 sheets on silicon-based substrates, i.e., 170 ± 33mJ/m^2. Besides the measurement of the adhesion performance, the buckle-based method can be also used to measure the Young's modulus of 2D materials (Iguiniz et al., 2019).

4 EVALUATION OF TRIBOLOGICAL PROPERTIES

Reducing the wear of mechanical components is an important topic in the field of engineering. In recent years, it has been found that 2D materials are of lowest friction coefficient, and can be used as lubricants to reduce mechanical wear. Thus, the measurement of tribological behavior of 2D materials, such as surface friction and interlayer friction, is becoming main task of contact probing methods.

Due to its unique properties, the tribological behavior of 2D materials cannot be measured by traditional methods. As an important development of AFM, FFM is designed and employed to measure the frictional behavior of various 2D materials. During the FFM test, the tip of AFM cantilever moves at a fixed frequency on the material surface, and friction diagram, friction forces are measured. The surface of FFM sample should be flat enough, or else the tip may be damaged during the fast scratching. Note that the measurement of FFM is independent of the material of the tip, the moving speed, and the force exerted by the tip (Zhang et al., 2019). Li et al. (2016) studied the surface friction of graphene, MoS_2, h-BN and $NbSe_2$ by FFM. It is found that the friction force decreased when the film number of layers increases due to the wrinkles under the compression of the tip. Fang et al. (2017) studied of the influence of tip shape on tribological behavior, and it is found that the friction is not sensitive to the film thickness under planar tip. With the contact probing method, Filleter et al. (2009) analyzed the friction behavior of monolayer and bilayer graphene films grown on SiC substrate. Quereda et al. (2014) experimentally studied the surface roughness and the lateral friction of single-layer MoS_2 deposited on SiO_2, mica, and h-BN.

For multilayer 2D materials such as vdW heterostructures, it is more important to study the corresponding interlayer friction properties (Filleter et al., 2009; Fang et al., 2017; Mak et al., 2019). Kobayashi et al. (2017) reported the preparation of WS_2/h-BN vdW heterostructures *via* CVD, and manipulated the motion of atomic layers with a tip. In recent years, with the development of MD and density functional theory (DFT), atomic simulations have been developed to study interlayer friction of 2D materials. So far, Xu et al. (2011) have studied the interlayer friction of layered graphene by MD method. It is found that the interlayer friction of two or three layers of graphene was almost zero, and the interlayer friction of graphene increases with the increase of the number of layers. As shown in **Figure 2E**. Ye et al. (2012) studied the role of wrinkle height in friction variation with number of graphene layers. Han et al. (2019) investigate the friction and wear properties of Ti_2AlN/TiAl composite, and found that the introduction of Ti_2AlN results in anti-friction and wear reduction. By using DFT calculation, Wang et al. (2012) showed that chemical modification had a significant effect on the interlayer friction of 2D materials. Sun et al. (2017) reported the frictional behavior of graphene and MoS_2 under high loads. Liang et al. (2008) investigated the atomic-scale energetic barriers during the sliding at MoS_2/MoO_3

interface. Cahangirov et al. (2012) determined the frictional merit figures of layered honeycomb nanostructures. By combining experiment and DFT, Yadav et al. (2021) studied the tribological behavior and interfacial interactions of metal-oxide/2D-material contacts.

5 THE MEASUREMENT OF ELECTRICAL PROPERTIES

In order to measure the electrical properties of 2D materials, some scanning probe microscopy technologies including conductive atomic force microscopy (CAFM), piezoresponse force microscopy (PFM), Kelvin probe force microscopy (KPFM) and Scanning tunneling microscopy (STM) have been developed. For conventional 2D materials, it is difficult to obtain their electrical properties by traditional methods due to the scale. CAFM provides a convenient and high-precision measurement equipment. For ferroelectrics, PFM is more suitable. PFM enables non-destructive visualization and control of ferroelectric nanodomains, as well as direct measurements of the local physical characteristics of ferroelectrics. KPFM is a scanning probe technique that is capable of measuring the local distribution of contact potential difference (CPD), which is a measure of the electrical surface potential or work function of the sample. STM can be used to study the electronic structure and electrical transport characteristics of samples (Hla, 2005).

By using contact probing methods, quantum transport in graphene/h-bN heterostructure superlattices (Zhao et al., 2017; Wu et al., 2018b), resonant tunneling in graphene/h-BN heterostructure (Mishchenko et al., 2014), etc. have been studied. The conductivity of MoS_2/graphene heterostructures is found to be directly related to the rotation angle (Liao et al., 2018). For graphene on silica substrate and bilayer graphene, the relationship between the rotation angle and the conductivity was uncovered (Wu et al., 2018c; Zhang et al., 2020). The contact quality improvement strategy between monolayer MoS_2 and graphite was given by Liao et al. (2019a). Zeng et al. (2021) reported an advanced PFM technology for measuring piezoelectric strain on nanoscale. Moores et al. (2010) studied KPFM in application to biomolecular films. A micro-four-point probe in STM for *in situ* electrical transport measurement was developed by Ge et al. (2015).

Besides the contact probing methods, there are some approaches widely used for property characterization of 2D materials and vdW heterostructures. For example, microelectromechanical system (MEMS) offers a number of merits for nanomechanical testing including controlled actuation, high-resolution force/displacement measurements (Luo et al., 2017). Raman spectroscopy is an important tool to study carbon nanotubes, graphene and other graphitic materials (Malekpour and Balandin, 2018). Due to high precision requirement and the difficulty of sample preparation, it is highly recommend to combine these tools to explore mechanical, interfacial, tribological, and electrical properties of 2D materials and vdW heterostructure through suitable strategies.

6 CONCLUSION AND PERSPECTIVES

In this mini review, we summarize some recent progress of contact probing methods of 2D materials and vdW heterostructures, including the measurement of mechanical properties, the measurement of interfacial properties, the measurement of tribological properties, as well as electrical properties. For the measurement of mechanical properties, the working mechanism of contact probing methods, the commonly used theoretical models are mainly discussed. For the measurement of interfacial properties, the blister test and buckle-based method are summarized. Generally, the blister test are suitable for measuring the adhesion energy between 2D materials and rigid substrates, while the buckle-based method can also measure the adhesion energy between 2D materials and elastic substrates. For measurement of frictional behavior, the progress of surface and interlayer friction of 2D materials are introduced. What is more, the appearance of CAFM provides a convenient and high-precision equipment to measure electrical properties.

From this mini review, it is found that the contact probing methods greatly improve the property measurement of 2D materials and vdW heterostructures. Although the contact probing methods have been well developed, it is not enough to characterize or measure properties of 2D materials and vdW heterostructures like bulk materials. For example, further development of *in situ* measurement for nano materials should be done to capture the evolution properties. Moreover, strain engineering of 2D materials requires more accurate manipulation of AFM and other scientific instruments.

Besides existing experimental studies and numerical simulations, more useful theoretical models which take the size effect and surface effect into consideration should be proposed. We believe that the contact probing methods will be more advanced to promote the blooming applications of 2D materials and vdW heterostructures.

AUTHOR CONTRIBUTIONS

JZ is responsible for conceptualization, and writing of the original draft; PC is in charge of conceptualization, project administration and investigation; JP takes charge methodology and revision; YZ contributes to the revision version of the work. All the authors have reviewed and edited the manuscript.

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