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Deciphering mechanical properties of 2D materials from the size distribution of exfoliated fragments

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Two-dimensional(2D) materials have been attracting numerous research attention due to their distinctive physical properties and boundless application potential in various fields. Among diverse physical properties, the mechanical property is the most basic one and plays a crucial role in ensuring the high reliability of 2D material-based devices and products. However, characterizing the mechanical properties of 2D materials is always a challenge due to their atomic thickness. Here, we propose a facile method to decipher the mechanical property of 2D materials from the statistical distribution of the size of the fragments acquired via mechanical exfoliation. This method is essentially based on a probabilistic mechanics model correlating the distribution pattern of fragment size and the intrinsic mechanical properties of 2D materials. The ensuing experimental verifications on both graphene and 2H-MoS_2 show good agreement between our measurements and the results reported in literature. This work not only provides a facile method for characterizing the mechanical properties of 2D materials, but also implies approaches to attaining 2D material fragments with controllable size via mechanical exfoliation.

Mechanical property is a prior issue of concern when exploring applications for 2D materials in various fields[1, 2, 3, 4] as it not only affects the structural integrity and reliability of the ultimate devices but also plays a pivotal role in determining the performance of the devices by coupling with other physical properties (e.g., optical properties, electrical properties, *etc.*)[4]. Over the past decade, enormous efforts have been devoted to the characterization of the mechanical properties of 2D materials such as strength, stiffness and toughness[4, 5, 6]. For instance, indentation on a suspended monolayer graphene with an atomic force microscope (AFM) tip was used to measure the intrinsic strength of graphene[7]. This approach was subsequently extended to the characterization of the fracture behavior of graphene and other 2D materials[6, 8, 9, 10]. Recently, micro-electro-mechanical system devices were developed, whereby the

in-situ measurements of the mechanical properties of 2D materials were carried out with the aid of scanning electron microscope or transmission electron microscope[11, 12, 13, 14]. However, these characterization methods involve sophisticated devices and facilities, and additionally are destructive to samples[4, 15]. As an alternative approach, atomistic simulation such as molecular dynamics simulation and density functional theory-based calculation has been widely applied to investigate mechanical properties like strength[16, 17, 18, 19, 20], stiffness[9, 21, 22, 23] and toughness[24, 25, 26] of 2D materials. Nevertheless, simulation results highly rely on the selection of parameters. Inappropriate selection of parameters may result in inaccurate or even non-physical results.

As a matter of fact, the mechanical properties such as strength of a given 2D material is not a deterministic value[4, 5]. Instead, their values are distributed over ranges depending on the defects, vacancies, grain boundaries and grafted functional groups in the 2D materials[8, 9, 10, 16, 25, 26, 27, 28, 29]. Such uncertainty of the mechanical properties of 2D materials largely affects the performance and reliability of the related structures and devices[30] and has attracted more and more research attention recently. For example, 'weakest-link' theory and Weibull distribution[31] have been adopted to explain the statistical distributions of strength and toughness of graphene[25, 32] and MoS_2[27] observed in simulations. In spite of these progresses, quantitative characterization of the uncertainty in the mechanical properties of 2D materials is still deficient[32]. As a facile manufacturing approach, mechanical exfoliation (see Fig. 1a) has been widely applied to produce 2D materials. However, acquisition of an exfoliated fragment with desirable size requires not only a master hand but also good fortune sometimes. Figs. 1b-c show the distributions of the size of graphene and MoS_2 fragments obtained by hundreds of exfoliations using Scotch tape. Clearly, the exfoliated fragments of graphene and MoS_2 exhibit size distributions different in both mean value and standard deviation. Such difference is attributed to their distinct mechanical properties, implying that the mechanical properties of 2D materials can be deciphered from the distribution patterns of the size of the exfoliated fragments if the correlation between them is unveiled. For this purpose, a theoretical model is constructed in the framework of probabilistic fracture mechanics.

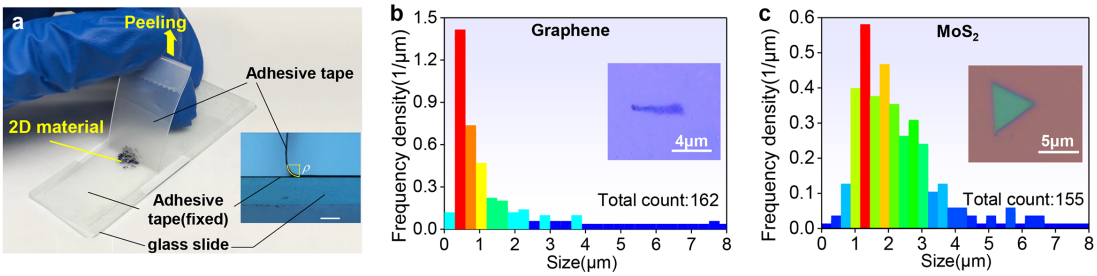

Fig.1| a, Image showing the mechanical exfoliation process of 2D material. The inset shows the side view of the peeling process. Scale bar, 500 μm. Frequency density of fragments size of **b**, mechanically exfoliated monolayer graphene, and **c**, mechanically exfoliated 2H-MoS_2. The insets in **b** and **c** show the optical microscope images of typical monolayer graphene and 2H-MoS_2 fragments, respectively. Here, fragment size is defined as the fragment length along the peeling direction.

Theoretical modeling

Fig. 2a schematically depicts the exfoliation process of a piece of flake (quasi-2D material) sandwiched by two adhesive tapes, as illustrated in Fig. 1a. With the separation of the adhesive tapes, the flake is split into two thinner ones. During this peeling process, the adhesive tape is bent at the frontier of peeling, resulting in tensile strain on the adhesive side given by (see Fig. 2a)[33]

$$\varepsilon_0 = t_{\mathrm{TP}}\sqrt{\frac{\gamma b}{2E_{\mathrm{TP}}I}} \tag{1}$$

where γ represents the adhesion energy of the tape adhesive, b is the width of the tape, E_{TP} is Young's modulus of the backing layer of the tape, I and t_{TP} are the cross-sectional moment of inertia and thickness of the backing layer of the tape, respectively. Due to such bending-induced strain of the adhesive tape, the peeled 2D material, which is firmly attached on the tape, gets stressed when entering the bending zone. The resulting tensile stress in the 2D material, as a function of the distance from the free end, is given by[34]

$$\sigma(x) = E\varepsilon_0\left[1-\exp(-\beta x)\right],\quad \beta = \sqrt{G_A / Et_A t} \tag{2}$$

where E and t are the Young's modulus and thickness of the 2D material, and G_A and t_A stand for the shear modulus and thickness of the adhesive layer of the tape, respectively. The tensile stress in Eq. (2) is a monotonic function of x, increasing from zero to an asymptotical value of $E\varepsilon_0$, as plotted in Fig. 2b. Under such tension,

fracture of the 2D material may take place as a random event. Once a fracture event happens, a new fragment is obtained, and the fractured point becomes the new free end of the remaining unfractured portion. As peeling proceeds, 2D material continuously enters the bending zone and fracture events happen repeatedly, giving rise to fragments of different sizes. At each fracture event, the distance of the fractured point from the free end determines the size of the obtained fragment of the 2D material.

For a material subject to uniform tensile stress σ, traditional probabilistic mechanics indicates that the failure probability of the material depends on its size. If the strength of the material follows the Weibull distribution, such dependence can be described by[25, 31, 35]

$$P_F\left(\Delta x,\sigma\right)=1-\exp\left(-\frac{\Delta x}{l_0}\left(\frac{\sigma}{\sigma_0}\right)^{\alpha}\right) \tag{3}$$

where Δx is the size of the material, l_0 is the characteristic size, σ_0 is the Weibull scale parameter (or characteristic strength), and α is the Weibull modulus, which determines the distribution width of the material's strength. The higher the α, the narrower the distribution width of the strength. The failure probability P_F for a given material should be independent of the selection of the characteristic parameters, implying that $l_0\sigma_0^{\alpha}$ in Eq. (3) must be a determinate constant[36]. In our study, l_0 is taken as 1 μm and σ_0 is to be determined.

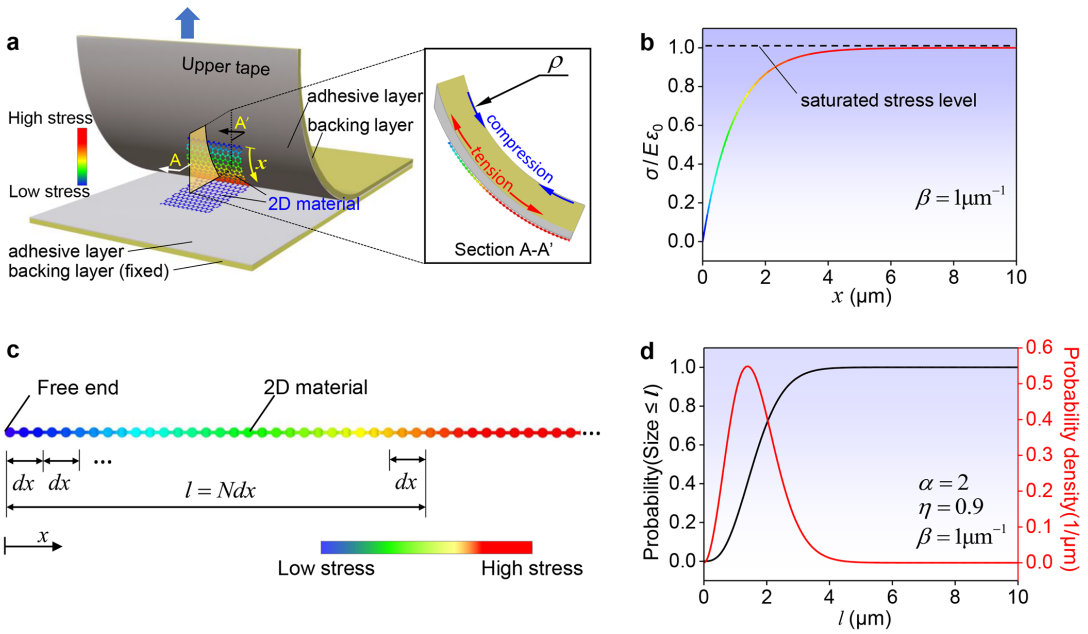

Fig.2 | Theoretical analysis. **a**, Schematic showing the peeling process of mechanical exfoliation of 2D material using adhesive tape. The lower adhesive tape is fixed to a rigid substrate. The *x*-direction is defined as the peeling direction. The inset shows the zoom in of cross-sectional view of section A-A'. ρ denotes the curvature radius of the bending tape. **b**, Distribution of in-plane tensile stress in 2D material attached to the peeling tape. **c**, Schematic showing the theoretical model for analyzing the sequential fracture of 2D material and the size of the resulting fragments. **d,** Typical cumulative probability distribution (black solid curve) and probability density distribution (red solid curve) of the fragments size.

For a 2D material subject to nonuniform tensile stress given by Eq. (2), we assume it is composed of many infinitesimal segments with size *dx* for each, as shown in Fig. 2c. The tensile stress in each segment can be treated as constant and therefor Eq. (3) still applies. The acquirement of fragments with size larger than l requires the survival of at least N consecutive segments from the free end, where $N = l/dx$. Then, the probability of obtaining a fragment with size less than l, which is the complementary event of $\text{size} \geq l$, is given by (Supplementary information Note 1):

$$P(\text{size} \leq l) = 1 - P(\text{size} \geq l) = 1 - \exp\left(-\frac{\eta^{\alpha}}{\beta l_0} \int_0^{\beta l} \left[1 - \exp(-x) \right]^{\alpha} dx \right) \tag{4}$$

where $\eta = E\varepsilon_0 / \sigma_0$. Eq. (4) is actually the cumulative probability distribution function of the fragment size l, as shown in Fig. 2d. Taking the derivative of Eq. (4) with respect to l gives rise to the probability density function of the fragment size as

$$p = l_0^{-1} \eta^{\alpha} \left[1 - \exp(-\bar{l}) \right]^{\alpha} \exp\left(-(\beta l_0)^{-1} \eta^{\alpha} \int_0^{\bar{l}} \left[1 - \exp(-x) \right]^{\alpha} dx \right) \tag{5}$$

where $\bar{l} = l / l_0$. Eq. (5) correlates the distribution of the segment size and the mechanical properties of the 2D materials characterized by parameters α, l_0 and σ_0. Given the distribution of the size of exfoliated segments, the characteristic parameters of the 2D material α, l_0 and σ_0 thus can be determined via regression (see Methods).

Experimental verification

To verify the approach proposed above, mechanical exfoliation on graphene is conducted using three different tapes (Scotch tape, Scotch tape with plastic backing and Nitto tape with plastic backing, see Supplementary information Note 2 for detailed properties of the tapes) (see Methods). Monolayer graphene fragments are obtained, followed by the measurement and counting of their sizes. Histograms in Figs. 3a-c show the statistical distributions of the sizes of monolayer graphene fragments exfoliated with these three tapes. The black solid curves in Figs. 3a-c show the regression with Eq. (5) based on the least square method. The high values of R^2 (0.93, 0.98 and 0.94) imply the good consistency between the theoretical and experimental statistics. This indicates that our theoretical model describes the fracture behavior of graphene during exfoliation very well. Although the size distributions obtained with different tapes are not the same, the deduced Weibull modulus α and characteristic strength σ_0 of the 2D material (graphene) are quite close, implying the tape-independence and robustness of our approach.

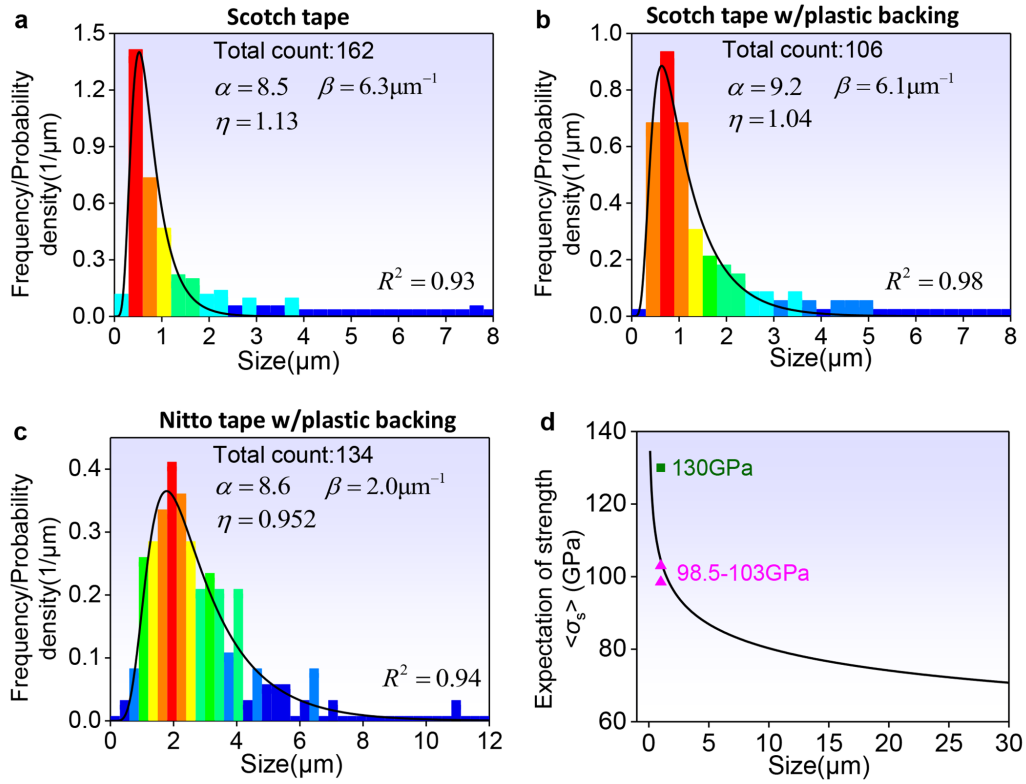

Fig.3 | Experimental verification results of graphene. Curve fitting of the size distributions of monolayer graphene fragments obtained from mechanical exfoliation using different types of tape: **a,** pristine Scotch tape, **b**, Scotch tape with plastic backing, and **c**, Nitto tape with plastic backing. During the curve fitting, α, β and η are taken as fitting parameters, the characteristic strength σ_0 of graphene sample is further derived from η (see Method). The results of σ_0 for graphene samples obtained using tapes shown in **a-c** are 114.7 GPa, 107.0 GPa and 109.9 GPa, respectively. **d**, Expectation of the strength of graphene as a function of fragment size. The scatter points indicate the results measured in literature.

Based on the Weibull modulus and characteristic strength, the expectation of the strength of a 2D material sample can be further obtained:

$$\langle \sigma_s \rangle = \left(\frac{l}{l_0} \right)^{-\frac{1}{\alpha}} \sigma_0 \Gamma\left(1 + \frac{1}{\alpha} \right) \tag{6}$$

where l stands for the size of the sample; $\Gamma(\cdot)$ is the gamma function. Taking the mean value of α and σ_0 deduced above, the expectation of the strength of graphene given by Eq. (6) is plotted in Fig. 3d as a function of sample size. The scatter points in Fig. 3d represent the results reported in literature, which are measured by the indentation of an AFM tip into suspended graphene membranes with diameter around 1μm. Results of the strength of graphene obtained by the proposed method agree well with those reported in literature (98.5-103 GPa[37], 130 GPa[7]), as shown in Fig. 3d.

This proves the good validity of the measurement by the proposed approach.

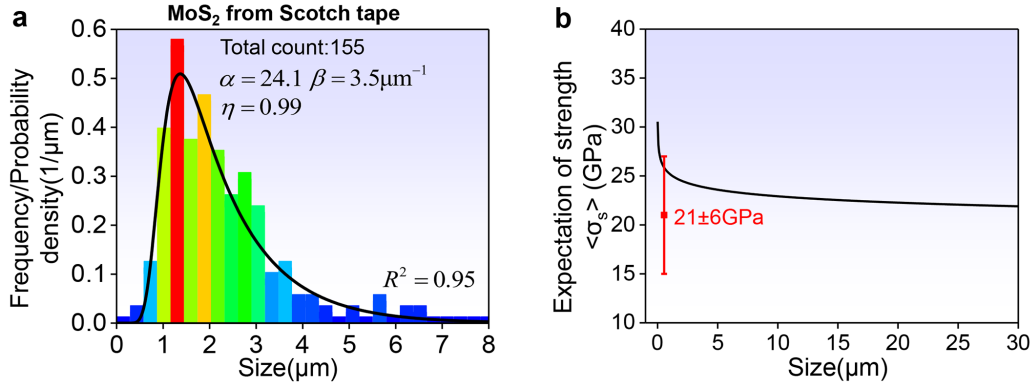

Fig. 4 | Experimental verification results of MoS_2. a, Curve fitting of the size distributions of MoS_2 fragments obtained from mechanical exfoliation using Scotch tape. The same curve fitting method as in the case of graphene are adopted. The characteristic strength σ_0 of MoS_2 derived here is 25.8 GPa. **b**, Expectation of the strength of MoS_2 as a function of fragment size. The red point with error bar shows the literature result of MoS_2 strength, the corresponding sample size is 0.55 μm[38].

In addition to graphene, MoS_2 is also applied to further verify the validity of the proposed approach. Fig. 4a shows the distribution of the size of MoS_2 fragments exfoliated by Scotch tape in comparison with the theoretical regression with Eq. (5). The deduced α of the MoS_2 sample reaches 24.1, suggesting a quite high uniformity of the mechanical property of the sample. Based on the deduced α and σ_0, the expectation of the tensile strength of MoS_2 is evaluated with Eq. (6) and plotted in Fig. 4b. It can be seen that the expectation of the strength of MoS_2 ranges from 20 to 30 GPa, depending on the sample size. With the increase of the sample size, the strength of MoS_2 decreases monotonically at a rate much lower than that of the graphene shown in Fig. 3d. The strength of MoS_2 we measured agrees with the value reported in literature[38], which is 21±6 GPa for samples with size of 0.55 μm, as shown in Fig. 4b. This reconfirms the applicability of our approach. It's worth noting that the strength of 2D material always decreases monotonically with the increase in sample size, but the decreasing rate is largely determined by α, as indicated in Eq. (6). Higher α gives rise to lower decreasing rate of strength and thus ensures high strength for large-scale samples. Foregoing experimental verifications prove the reliability and validity of the proposed approach in assessing the mechanical properties of 2D materials as well as their dependence on the sample size. These findings provide useful reference to the design and fabrication of 2D material-based nanodevices as

well as approaches to attaining 2D material fragments with controllable size via mechanical exfoliation.

Discussion

In summary, a nondestructive method for characterizing the mechanical properties of 2D material is proposed and verified. This method is essentially based on a probabilistic fracture mechanics-based theory that correlates the size distributions of exfoliated fragments and the mechanical properties of the 2D material. Statistical mechanical parameters such as Weibull modulus α and characteristic strength σ_0 can be obtained by fitting the experimental size distribution of 2D material fragments to the theoretical prediction. Based on these statistical parameters, the strength of 2D material and its size dependence could also be evaluated. The mechanical properties of graphene extracted from size distribution patterns of fragments exfoliated using three different tapes show good consistency. This substantiates the reliability of our method. The results of graphene and MoS_2 obtained by the proposed method also show reasonable agreement with results reported in the literature, which further verifies the validity and generality of our method. These findings on mechanical properties of 2D materials could provide useful guidance for the design of 2D material-based devices to achieve optimal mechanical strength and reliability. Our method also paves an avenue of deciphering the underlying physical properties of 2D materials from the statistics of their appearance features.

Methods

Mechanical exfoliation of graphene

Three different tapes were used for the mechanical exfoliation on graphene (see Supplementary information Note 2 for the details of the tapes). The graphene samples attached to these tapes were then transferred to the target substrates (SiO_2(300nm)/Si wafer) for optical observation. The target substrates were cleaned by sonicating in acetone and isopropyl alcohol (IPA). Optical observation and size measurement of the graphene fragments were performed with an optical microscope (Leica DM2700M).

Data fitting

Lease square method was used to fit the experimental size distributions with theoretical predictions. The mean of the experimental data can be calculated by

$$\overline{p}_{\text{exp}} = \frac{1}{n}\sum_{i=1}^{n} p_i^{\text{exp}} \tag{7}$$

where p_i^{exp} is the experimental frequency density of the 2D material size. The total sum of squares can be obtained as

$$SS_{\text{tot}} = \sum_i \left(p_i^{\text{exp}} - \overline{p}^{\text{exp}} \right)^2 \tag{8}$$

The sum of squares of residuals is given by

$$SS_{\text{res}} = \sum_i \left(p_i^{\text{exp}} - p_i^{\text{theory}} \right)^2 \tag{9}$$

where p_i^{theory} is the theoretical prediction of probability density of the 2D material size, which is given by Eq. (5). The coefficient of determination can be calculated as

$$R^2 \equiv 1 - \frac{SS_{\text{res}}}{SS_{\text{tot}}} \tag{10}$$

From Eq. (5), it is found that in order to obtain the theoretical probability density function, we need to determine the parameters α, β, and η. The best α, β, η are taken as such values that give rise to the maximum R^2. Adaptive Simulated Annealing algorithm in Isight optimization toolbox (Dassault Systèmes) was employed to search for such α, β, and η. Given the initial tensile strain ε_0 (Supplementary information Table S2) and Young's modulus E (*e.g.* for graphene, E=1 TPa[7]), the characteristic strength σ_0 can be readily derived using $\eta = E\varepsilon_0 / \sigma_0$.

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