Intercalator-assisted plasma-liquid technology: an efficient exfoliation method for few-layer two-dimensional materials

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Few-layer two-dimensional (2D) materials have attracted enormous interest since 2004 when graphene was successfully exfoliated from graphite by mechanical exfoliation [1]. In recent years, the burgeoning 2D family with unique and novel properties have found new applications in a myriad of fields including electronics, optoelectronics, catalysis, energy, and biomedicine [2–4]. However, in order to be commercially viable, a mass fabrication technique to synthesize few-layer 2D materials with high quality is critical. The typical large-scale fabrication strategy for few-layer 2D materials is liquid exfoliation from the bulk counterparts with a layered structure [5]. Although the exfoliation by chemical oxidation can produce graphene oxide from graphite on a large scale [6], functional groups are generally introduced and defects are produced [7]. Ultrasonic/shear liquid exfoliation applies to most 2D materials but requires a long processing time (generally several hours) and often obtains small and thick sheets. Modified physical exfoliation methods such as ultrasonic-ball milling and intermediate-assisted grinding exfoliation improve the yield and quality of the exfoliated materials; however, a further purification is needed [8,9]. Recently, methods based on electrochemical technology (ET) have been proposed [10–18]. Generally, intercalator-assisted electrochemical technique (IA-ET) with intercalators such as quaternary ammonium salts or Li^+ complexes enables the intercalation of ions/molecules into the interlayer, leading to the expansion of the bulk layered materials, thus facilitating subsequent exfoliation into few-layer sheets [11,13,15].

However, IA-ET exfoliation often damages the structure and alters the properties of the materials due to the residual intercalators [16]. Considering the complexity and variety of 2D materials, it is highly desired to develop a new exfoliation method for mass production of different types of few-layer 2D materials.

Plasma processing is a common method in surface modification, surface etching, and thin film deposition due to the production of abundant active species [19–23]. The plasma-liquid technology (PLT) has been adopted for nanomaterials synthesis and doping because it can effectively transfer the active species to the liquid through the plasma-liquid interface for the chemical reactions [20,24,25]. Compared with ET, PLT can produce more active species and trigger various charge transfer reactions such as quick decomposition and polymerization of organic molecules. Furthermore, PLT only requires a simple and economical atmospheric-pressure plasma system operated under ambient conditions. Our previous work has demonstrated that PLT enables scalable production of phosphorene without additional intercalators [20]. However, it is difficult to extend the use of PLT without intercalators to other types of 2D materials.

Herein, an intercalator-assisted PLT (IA-PLT) was designed and applied for the synthesis of 2D materials. LiCl served as the intercalator to rapidly expand graphite paper, leading to the formation of graphene with few defects on a large scale. The IA-PLT exfoliation process and the corresponding intercalation mechanism were investigated and compared with that of the traditional IA-ET method.

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The IA-PLT which can be readily extended to the production of other types of 2D material such as few-layer 2H-MoS₂ possesses a number of merits such as rapid expansion, high-quality few-layer products without introducing intermediates or functional groups, and outstanding expandability and controllability.

The IA-PLT-induced expansion process with graphite paper was first studied. The IA-PLT system comprises a high-voltage power supply, two electrodes and a cell filled with exfoliation solution [20]. The plasma generated by the high-voltage from a stainless tube upon the solution served as the anode and the cathode was the immersed graphite paper. The exfoliation solution was prepared by dissolving LiCl in dimethylformamide (DMF, 4 mg mL⁻¹) and the expansion process completed in 4 min (Video S1). During the exfoliation, a large amount of bubbles can be observed on the graphite paper (Fig. S1). Fig. 1a displays the photographs of the graphene-DMF dispersions produced by IA-PLT. Owing to the rapid IA-PLT reaction, four bottles (total of 3.6 L) of the graphene-DMF dispersions (0.8 mg mL⁻¹) can be produced in 12 h. The graphene product was characterized by atomic force microscopy (AFM), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and high-resolution TEM (HR-TEM). Fig. 1b and c show that the formation of few-layer graphene with a thickness dis-

tribution ranging from 2 to 9 nm corresponding to 3–12 layers [26]. The lattice spacing of 0.21 nm shown in the HR-TEM image (inset in Fig. 1c), is consistent with the (100) plane of graphene. The SEM images (Fig. S2) show a clear surface of the graphene with a lateral size distribution from hundreds of nanometers to several micrometers. Fig. 1d depicts the high-resolution Raman spectrum of a single graphene sheet and the D band at 1350 cm⁻¹, G band at 1583 cm⁻¹, and 2D band at 2720 cm⁻¹ are observed [25–28]. The D band reflects the disorders within sp²-bonded carbon regions caused by defects and the intensity ratio I_D/I_G is generally used to indicate the degree of defects at the in-plane area of graphene [29]. Here, I_D/I_G of the IA-PLT-exfoliated graphene is only 0.02, which is much less than that (0.16–0.95) of graphene reported previously [26], indicating that the IA-PLT-exfoliated graphene has high quality.

X-ray photoelectron spectroscopy (XPS) was conducted to determine the chemical composition of the IA-PLT-exfoliated graphene. As shown in Fig. 1e, a weak O 1s peak is found and the atomic ratio of carbon and oxygen (C/O) is 31.5, which is larger than those (9.26–21.2) of graphene synthesized before [26]. The high-resolution C 1s peak indicates four types of carbon bonds by data fitting (Fig. 1f). The peaks at 284.5, 284.8, 285.6 and 287.1 eV can be assigned to C–C, C–H, C–N, and C=O

Figure 1 Characterization of the IA-PLT-exfoliated graphene: (a) photographs of the graphene-DMF dispersions; (b) AFM images and thickness distribution of graphene (at least 100 sheets were measured); (c) TEM image and HR-TEM image (inset); (d) Raman scattering spectrum; (e, f) XPS survey and C 1s spectra.

bonds, respectively [27,29] and that at 291.1 eV is related to $\pi\text{-}\pi^*$ which indicates the existence of a conjugated aromatic structure. The additional carbon bonds (such as C–H, C–N, C=O) are attributed to the residual solvent from the exfoliation and washing processes [27,29]. Hence, the large C/O ratio indicates negligible oxidation during the IA-PLT exfoliation process.

To explore the mechanism of the IA-PLT-induced exfoliation, graphite papers with different expansion degrees were examined by SEM and X-ray diffraction (XRD). It is known that the intercalation and functional groups cause the expansion during the ET-exfoliation, and the expansion degrees are related to the residual intermediates or functional groups, which result in the shift of XRD peaks [10–18,30,31]. Photographs inset in Fig. 2 clearly show the expanded graphite paper with small and large expansion degrees and Fig. 2a–c show the cross sections of the compact graphite paper and wrinkled expansion samples, especially the enlarged spaces, indicating that the van der Waals (vdW) force between the layers has been greatly weakened. As shown in Fig. 2d, two typical diffraction peaks originating from graphite at around 26.5° (002) and 54.7° (004) can be found from all the samples and the sharp diffraction peaks can be attributed to the restacking of the expanded layers. In traditional IA-ET exfoliation of graphite, the (002) peak

generally shifts to a small angle due to the intercalation and formation of intermediate compounds [30,31]. In contrast, during IA-PLT expansion, the (002) peak becomes slightly broadened and does not shift significantly, indicating that there is no or very little intercalator residue in the layer space. In addition, no peak belonging to Li compounds appears in the XRD spectra and no XPS signal of Cl or Li element can be found (Fig. S3), suggesting that no or few Li compounds are formed during the IA-PLT-induced exfoliation. In the control experiment in which DMF is exchanged with water or LiCl with HCl, no expansion can be observed. However, when Li^+ is replaced by other metal ions such as Na^+ , K^+ , Mg^{2+} , Ca^{2+} and Sr^{2+} , similar IA-PLT-induced expansion can be observed. Fig. S4 shows the graphene products synthesized with CaCl_2 and SrCl_2 .

The possible IA-PLT exfoliation mechanism was proposed (Fig. 3). On account of the abundant electrons and active species generated by the plasma, chemical reactions initiate at the interface between the plasma and liquid phase simultaneously as a result of the transport processes [23,24]. As described previously, the DMF ($\text{C}_3\text{H}_7\text{NO}$) molecules can decompose into cations ($\text{C}_3\text{H}_8\text{N}^+$, $\text{C}_2\text{H}_8\text{N}^+$, $\text{C}_2\text{H}_6\text{N}^+$, $\text{C}_2\text{H}_4\text{N}^+$, and H^+) with plasma, which together with the solvated metal ion/DMF complexes move to the cathodic graphite paper under the electric field [20]. The

Figure 2 (a–c) Cross-section SEM images, inset photographs and (d) XRD spectra and enlarged specific region of the graphite paper in different expansion degrees.

Figure 3 Schematic of the IA-PLT exfoliation process in graphite paper.

metal ions quickly receive electrons from the graphite cathode and transfer to the surrounding decomposed DMF species. The interaction impacts the boundaries of graphite to weaken the vdW force between layers [18]. Subsequently, the DMF decomposed cationic species enter the open graphite layer to further weaken the vdW force and quick electron transfer leads to efficient conversion of the cations to gases including C_3H_9N , C_2H_7N , and H_2 , which expand the graphite, resulting in exfoliation.

The IA-PLT exfoliation method was compared with the conventional IA-ET method which involves an electric field and intercalator. Control experiments were performed by changing the plasma anode to a platinum wire anode generally used in ET exfoliation. Only weak expansion of graphite paper can be observed for IA-ET with LiCl or other metal chlorides as the intercalator. In fact, ET cannot decompose DMF efficiently (Fig. S5). Moreover, metal ions such as Li^+ and Ca^{2+} in the IA-ET exfoliation tend to react with graphite to form complexes on the surface [30], while this phenomenon is not observed from IA-PLT (Fig. S6). Metal ion intercalation in IA-ET exfoliation is described as an ion-electron transfer reaction and water is requisite in the hydration process. Therefore, highly active pure Li foil or Li compounds such as $LiClO_4$, $LiBF_4$ and C_4H_9Li , are required to enable exfoliation, but the use of Li foils or Li compounds is dangerous and Li intermediates are often formed [32]. In contrast, owing to the mass of the active species and quick

electron transfer induced by the plasma, IA-PLT is more efficient than IA-ET because the former technique requires safe metal ion intercalators only. In addition, IA-PLT involves no residual metal complexes, thus eliminating the possible influence of intercalators on the properties of the final materials.

To investigate the versatility, IA-PLT was applied to exfoliate another type of 2D materials, molybdenum disulfide (MoS_2). As a typical transition-metal dichalcogenide (TMD), MoS_2 crystal has the 2H-phase and the single layer is composed of a three-atom-thick hexagon of Mo–S–Mo (Fig. S7a). The traditional IA-ET method utilizing Li^+ intercalation generally leads to the phase transition of MoS_2 from 2H to 1T accompanied by the formation of the Li_xMoS_2 intermediate [33,34]. Although the quaternary ammonium (THAB) has been used for the production of 2H- MoS_2 , it needs a long-time intercalation process with the formation of the $(THA^+)_xMoS_2^{x-}$ intermediates [35]. As shown in Fig. 4a, IA-PLT with the LiCl/DMF solution enables full expansion of the MoS_2 crystal in 10 min (Video S2). The expanded sample is dispersed in isopropanol (IPA) by sonication to obtain the MoS_2 sheets-IPA dispersions with a dark green color. Fig. S7b shows the XRD spectra of the bulk MoS_2 with different expansion degrees. Similar to graphite, the (002) plane of MoS_2 shows no small angle shift and no peak belonging to Li compounds appears during expansion. Furthermore, MoS_2 maintains the 2H-phase during the IA-PLT-induced expansion.

Figure 4 Characterization of the IA-PLT-exfoliated MoS₂ sheets: (a) photographs showing the IA-PLT exfoliation process and the MoS₂ sheets-IPA dispersions; (b) TEM image and HR-TEM image (inset); (c) absorbance spectrum of the MoS₂ sheets-IPA dispersions; (d) PL spectrum of one representative MoS₂ sheet.

Fig. 4b and Figs S8, S9a show that the MoS₂ sheets have a typical thickness of about 3.2 nm with the lateral size between 1 and 30 μm. The HR-TEM image shows a lattice space of 0.26 nm ascribed to the (100) plane. The Raman spectrum in Fig. S9b reveals prominent E_{2g}¹ and A_{1g} phonons at 380 and 403 cm⁻¹, respectively, consistent with those of the previously reported few-layer MoS₂ [34]. The Mo 3d XPS spectrum in Fig. S9c shows peaks at around 229.0 and 232.1 eV associated with Mo⁴⁺ 3d_{5/2} and Mo⁴⁺ 3d_{3/2} of the 2H-MoS₂, respectively [33]. The S 2p doublets at 162.9 eV (S 2p_{1/2}) and 161.7 eV (S 2p_{3/2}) are observed from Fig. S9d. The XPS results thus confirm successful preparation of the 2H-phase few-layer MoS₂ sheets by IA-PLT. The optical properties of the MoS₂ sheets were determined. As shown in Fig. 4c, the A and B exciton peaks from the K point of the Brillouin zone appear at 676 and 615 nm, respectively, and the convoluted C and D excitonic peaks are located at around 425 nm. Photoluminescence (PL) is observed from a typical MoS₂ sheet deposited on the Si/SiO₂ substrate with a 532 nm laser and Fig. 4d shows the PL spectrum of the 2H-MoS₂ sheets which agrees well with the mechanically exfoliated samples reported previously [36]. The PL peak at 670 nm and a small shoulder at about 620 nm are

observed.

In summary, an efficient IA-PLT exfoliation method for the production of high-quality 2D materials such as graphene and MoS₂ is demonstrated. By using graphite paper as the raw materials and LiCl as the intercalator, large-scale production of few-defect graphene with I_D/I_G of 0.02 and C/O ratio of 31.5 is demonstrated and the efficiency is much higher than that of previously reported exfoliation techniques. This technique can be extended to the rapid production of few-layer metal sulfide (2H-MoS₂). Owing to the special plasma-liquid interface reactions created by the plasma, IA-PLT has many merits such as free choice of safe metal ion intercalators, almost no residual intercalator, high-quality (few defects) products, rapid expansion, high production yield, as well as outstanding controllability and expandability. These advantages cannot be matched by other current techniques and hence, IA-PLT has great potential in the production of different types of few-layer 2D materials.

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Conflict of interest The authors declare no conflict of interest.

Supplementary information Experimental details and supporting data are available in the online version of the paper.

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插层剂辅助等离子体液相技术用于高效制备少层二维材料

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摘要 本工作提出了一种插层剂辅助的等离子体液相技术用于高效地剥离高质量少层二维材料。采用氯化锂为插层剂可快速剥离石墨纸, 形成具有小D峰/G峰比(0.02)以及大碳氧比(31.5)的石墨烯产物。并且, 这种方法可以拓展至高质量少层2H相二硫化钼的制备。和传统的插层剂辅助电化学方法相比, 等离子体诱导产生的大量活性粒子以及快速的电子转移, 使得插层剂辅助的等离子体液相技术剥离的产物缺陷少且不会引入额外的基团。这种可控的快速剥离方法在制备其他各种类型的高质量二维材料方面都具有巨大潜力。