Movable-Type Transfer and Stacking of van der Waals Heterostructures for Spintronics

YUAN CAO¹,², XINHE WANG¹, XIAOYANG LIN¹,³,⁴, WEI YANG¹, YUAN LU², YOUGUANG ZHANG¹, (Member, IEEE), AND WEISHENG ZHAO¹,³,⁴, (Fellow, IEEE)

¹Fert Beijing Research Institute, School of Microelectronics and Beijing Advanced Innovation Centre for Big Data and Brain Computing (BDBC), Beihang University, Beijing 100191, China
²Institut Jean Lamour, UMR 7198, CNRS-Université de Lorraine, 54000 Nancy, France
³Beihang-Geotek Joint Microelectronics Institute, Qingdao Research Institute, Beihang University, Qingdao 266000, China
⁴Hefei Innovation Research Institute, Beihang University, Anhui 230033, China

Corresponding authors: Xinhe Wang (xinhe@buaa.edu.cn) and Xiaoyang Lin (XYLin@buaa.edu.cn)

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ABSTRACT

The key to achieving high-quality and practical van der Waals heterostructure devices made from various two-dimensional (2D) materials lies in the efficient control over clean and flexible interfaces. Inspired by the “movable-type printing”, one of the four great inventions of ancient China, we demonstrate the “movable-type” transfer and stacking of 2D materials, which utilizes prefabricated polyvinyl alcohol (PVA) film to engineer the interfacial adhesion to 2D materials, and provides a flexible, efficient and batchable transfer scheme for 2D materials. The experiments also verify the “movable-type” transfer can precisely control the position and orientation of 2D materials, which meets the burgeoning requirements such as the preparation of twisted graphene and other heterostructures. Importantly, water-solubility of PVA film ensures an ideal interface of the materials without introducing contamination. We illustrate the superiority of this method with a WSe₂ vertical spin valve device, whose performance verifies the applicability and advantages of such a method for spintronics. Our PVA-assisted “movable-type” transfer process may promote the development of high-performance 2D-material-based devices.

INDEX TERMS

movable-type, PVA transfer, two-dimensional materials, spin valve, spintronics.

I. INTRODUCTION

Two-dimensional materials (2D) with their heterostructure interface manipulating bring infinite possibilities and many unprecedented devices in electronics [1], [2]. For example, the various 2D materials have been the fertile ground of spintronics for low-power electronics [2], including efficient spin transport [3], spin relaxation [4], [5] and spin logic devices [6], [7] based on graphene and transition metal dichalcogenides (TMDs) [2], [8]–[10]. The 2D nature of 2D materials further endows these 2D electronic effects with extraordinary abilities of interface engineering. As a result, the interface control during the transfer process of 2D materials becomes a key issue of 2D electronics. One example is 2D van der Waals heterostructures, which breaks the limitation of lattice matching by freely stacking 2D crystals on top of each other, and produces many novel physical effects as well as innovative device applications [1], [11]–[14]. Another example is contact engineering between 2D materials and bulk materials (such as electrodes), which will also have a significant impact on 2D-material-based device characteristics [15]. In this sense, a strategy to realize efficient transfer of 2D materials with flexible control of interface properties is highly desired.

Inspired by Chinese traditional “movable-type printing”, one of the four great inventions of ancient China, as a great technological revolution in the history of printing, we introduce a “movable-type” process, which achieves a flexible,
efficient and modularized transfer and stacking scheme for 2D materials. Taking the polydimethylsiloxane (PDMS) film as the support piece, the prefabricated PVA films are manipulated to engineer the interfacial adhesion, and selective to pick up the 2D materials or their heterostructures precisely. Special devices, such as twist-angle graphene and hybrid 2D/3D heterostructures, can be constructed. Moreover, the cleanliness of the interface after transfer is guaranteed by the water solubility of PVA. The incomplete removals of mediator and impurities from the solvents are the main sources of pollution in the transfer process [16], which can be largely avoided by the good removal property of PVA in the purified water. As an illustration, the WSe₂-based vertical spin valves are fabricated and achieve a new record of magnetoresistance ratio. The universality and reliability of our “movable-type” transfer and stacking for van der Waals heterostructures could promote the development of emerging spintronic devices.

II. EXPERIMENTAL SECTION
A. FROM “MOVABLE-TYPE PRINTING” TO “MOVABLE-TYPE” TRANSFER
The essence of Chinese “movable-type printing” is to replace traditional manual transcription or non-reusable printing plates by using movable metal or clay stencils (as shown in Fig.1a). The movable single-character stencils are selected and arranged on a supporting platform, then with brushing ink on them and squeezing with a paper together, the characters information is transferred to the paper. “Movable-type printing” is flexible, selective and modularized. From the “movable-type printing” to “movable-type” transfer and stacking 2D materials, we take advantage of free-standing characteristic of PVA to achieve that. The PVA flakes can be arranged onto the PDMS pieces, acts as the movable stencils, as shown in Fig.1b. Due to the difference of the surface tension (see below), the 2D materials contacting PVA flakes can be picked up while those contact the bare PDMS cannot be, so the deterministic transfer of 2D materials can be achieved. Analogy to the “movable-type printing”, our “movable-type” transfer has the advantages of high efficiency, flexible interface control, batchable preparation of arrayed same-type devices or complex hybrid devices.

Conventional transfer methods employ polymethyl methacrylate (PMMA), poly propylene carbonate (PPC), polycarbonate (PC), polyvinyl pyrrolidone (PVP), PDMS [17]–[25] as the transfer mediators. Among them, the kind of methods represented by PMMA [17] relies on spin coating the transfer mediator on sample surface, which always causes disturbance and inevitable material waste whereby the entire 2D material on the substrate is transferred at one time. Furthermore, as the macromolecular polymer, the transfer mediator requires removal in volatile toxic solvents such as acetone, which necessitates special handling and disposal precautions [11], and it would cause polymer residuals with the resulting of sample inevitably polluting and damaging due to incomplete dissolution and absorption of impurities from the solvents. Another kind of transfer methods represented by pre-fabricated PDMS film has been shown to reduce the contamination [26], but it is difficult to pick up 2D atomic layers [27] to construct heterostructures, especially the stacking of more layers by a flexible and modularized way.

As a small-molecule, water-soluble and environment-friendly mediator, PVA is able to be prefabricated into centimetre-scale free-standing film [28]. PVA film has a similar surface tension (∼37.0 mN/m, see Table 1 & Ref. [29], [30]) to that of many van der Waals bonded surfaces [31], and its strong adhesion and good wetting capability to the 2D materials can be restored by heating.
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**FIGURE 2.** 2D materials transfer and heterostructure stacking by PVA/PDMS polymer structure. (a) A free-standing PVA film handled with tweezers. (b) A WSe\(_2\) film grown by chemical vapor deposition (CVD) is transferred onto a marked SiO\(_2\)/Si substrate. (c) A schematic illustration of the transfer process. (d) & (e) Mechanically exfoliated graphene and h-BN. (f) An optical microscopy image of the PVA film/h-BN/graphene heterostructure. (g) An optical microscopy image of the as-fabricated device with the 2D heterostructure of h-BN/graphene placed on the pre-patterned electrodes. The scale bar of (d-g) is 10 µm.

at 90°C. The sample could be a 2D material prepared by chemical vapor deposition (CVD) growth or mechanical exfoliated, even a 2D material covered by the deposited metal (see below, Fig. 3h) on a wide range of substrates, such as a silicon wafer, copper foil and quartz. Importantly, it’s shown that PVA film can be removed by the wildly-available purified water with convenient process, the sample’s intrinsic properties can maintain much better than ordinary transfer methods [28]. Utilizing the prefabricated PVA film, we demonstrate a “movable-type” transfer and stacking technique for diversified 2D materials.

**B. “MOVABLE-TYPE” TRANSFER AND STACKING PROCESSES OF 2D MATERIALS**

The focus of the “movable-type” transfer is to prepare the PVA film of appropriate thickness (~50 µm, see Fig. 2a), which acts as the movable stencils. The much thinner one is easily curled which is not convenient for the transfer operation while the much thicker one will be difficult to be completely removed. In our preparation, dissolve the PVA powder (Alfa Aesar, 98-99% hydrolyzed, high molecular weight) in deionized water with a mass ratio of 4%~6%. Decant the solution into a Petri dish with the liquid height being about 1 mm. Place the dish in the fume hood to accelerate the evaporation of the solvent. The PVA film could be formed after a few hours. Subsequently, it could be cut and peeled off as free-standing flakes, which serve as the movable stencils.

A variety of 2D materials have been transferred in our trials. Firstly, a full-coverage PVA film is used for the overall transfer of the continuous monolayer WSe\(_2\) on SiO\(_2\)/Si substrate. The transfer process refers to the description in the following paragraphs. The result is shown in Fig. 2b, the whole film of CVD-grown WSe\(_2\) is transferred at one time to the substrate with pre-patterned electrodes. This method can also be scaled up for the transfer of large-area 2D materials, provided the PVA film is large enough.

Further, the heterostructures of h-BN/graphene is prepared with our transfer method (The transfer setup is shown in supplementary material S1.). The process is schematically illustrated in Fig. 2c. After mechanical exfoliation, the h-BN and graphene crystals on SiO\(_2\)/Si substrate are selected for stacking under an optical microscope (Fig. 2d-e). Then, take a glass slide with a piece of PDMS (10*10*2 mm) on it, to act as a releasable supporting platform for PVA. Next, a PVA film is taken and stuck onto the PDMS, whereby the PVA film could be easily bent and adhered tightly to the PDMS due to the flexibility of PVA and the appreciable adhesion.

Under the microscope and with the help of a home-built transfer set with micromanipulation stage, the glass slide/PDMS/PVA is aligned and made to contact tightly with the as-selected h-BN flake. And heat the stage at 90°C for 2 minutes to guarantee the PVA film adheres to both the silicon wafer and h-BN flake firmly. The binding between PVA and PDMS is relatively weaker (since the surface tension of PVA~37.0 mN/m mismatches with
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FIGURE 3. The features of “movable-type” transfer and stacking. (a) A schematic illustration of a selective “movable-type” transfer process. (b) & (c) Exfoliated graphene fabricated by transferring the undesired part through the “movable-type” transfer method. The scale bar is 10 µm. (d) Schematic illustration of the bilayer graphene with a twist that realized by “movable-type” transfer from monolayer graphene using the PVA film edge. (e-g) Optical images corresponding to the stacking process of (d). The scale bar is 10 µm. (h) A schematic illustration of a 2D material with a pre-deposited metal film transferred to another substrate. (i-k) Optical images corresponding to the transferring process in (h). Exfoliated graphene with deposited 30 nm Co and 5 nm Au films is transferred to a sputtered LSMO substrate. The scale bar is 15 µm.

III. RESULTS AND DISCUSSION

A. SELECTIVE FEATURE OF “MOVABLE-TYPE” TRANSFER WITH SPATIAL RESOLUTION OF ONE MICRON

With the pre-arranged PVA flakes, our “movable-type” transfer method has a spatial resolution of approximate one micron and can be used to select 2D materials at a specific position. In practice, the grown or mechanically exfoliated 2D materials always appear in groups, acquiring independent thin or single layer samples is challenging, which greatly complicates the fabrication process, especially the wire layout. Here, by manipulating the PVA flake to contact a specific area of 2D samples, the transfer of the selected sample can be achieved, as shown in Fig. 3a-c. The positioning accuracy can reach one micron under an optical microscope. Here the key point is that though the exposed PDMS also contacts other 2D materials elsewhere during the transfer process, the adhesion of PDMS is not as strong as that of the PVA due to the mismatch of surface tension between PDMS and van der Waals bonded surfaces[31]. When separating the PVA with the selected sample from the substrate, the samples that contact the PDMS will not be taken away. Therefore, the “movable-type” transfer is able to precisely selective transfer the sample in a specific area without affecting surroundings.

B. FLEXIBLE FEATURE OF “MOVABLE-TYPE” TRANSFER WITH STACKING CONTROL OF BOTH ANGLE AND POSITION

The interfacial engineering of 2D heterostructures, for example, by controlling the stacking order and angle, has endowed 2D materials/heterostructures with fascinating properties, such as anisotropic spin relaxation [5] and tunable spin absorption [7]. On the basis of selective, “movable-type” transfer allows us easily realizing these structures by flexibly adjusting the angle and position of the “stencil”, that is, the PVA flake with 2D materials on it. Fig. 3d describes the fabrication process of an angularly controlled stacked sample consisting of two rotationally aligned monolayer graphene flakes. The corresponding optical micrographs are shown in Fig. 3e-g. The target graphene is partially picked up by setting the contact area with the PVA film edge, subsequently, the remaining graphene is rotated with a certain angle by a rotator and adhered to the graphene that had just been picked up, forming a twist-angle bilayer graphene with rotationally aligned. Compared with the requirement of making a hemispherical handle substrate [32] to transfer a sample with a specific area, in our method, inspired by the concept of “movable-type stencil”, it only needs to control the edge...
of the PVA to overlap with a designate area, and greatly simplifies the process.

C. DIVERSITY FEATURE OF “MOVABLE-TYPE” TRANSFER OF 2D MATERIALS WITH PRE-DEPOSITED METAL FILMS

Furthermore, contact engineering between 2D materials and bulk materials (such as metallic electrodes) has been a key issue of 2D electronics [15], [33]. Conventional techniques are problematic in terms of impurities (introduced in the lithography of electrode pattern before deposition), which give rise to a large contact resistance. Several approaches [34], [35] have been proposed but none has been fully developed. We suggest that the “movable-type” transfer may be able to improve this issue, especially in vertical spin valves. Fig. 3h describes the transfer process of a heterostructure consisting of 2D material with pre-deposited metal film, which can avoid lithography contamination on the material’s surface. The corresponding optical images are shown in Fig. 3i-k. After the graphene with a deposited Co film (60 nm) is peeled off from the original SiO$_2$/Si substrate and transferred onto a LSMO magnetic substrate, a LSMO/graphene/Co spin valve is successfully prepared. Since the binding force between graphene and Co is larger than that between graphene and substrate[36], the deposited Co film and graphene are picked up together. Our “movable-type” peeling off and transferring hybrid 3D/2D heterostructures technology supports simultaneous fabrication of arrayed same-type devices. It expands the 2D/ferromagnetic system and other van der Waals heterostructures and may open a new way for exploring various functional 2D relevant devices especially under ambient conditions.

D. CHARACTERIZATION AND DEVICE PERFORMANCE

We use optical microscopic, atomic force microscopic (AFM), and spectroscopic approaches to evaluate the quality of the samples transferred by PVA film. Taking the mechanically exfoliated WSe$_2$ as an example, we first mechanically exfoliated WSe$_2$ flakes onto a SiO$_2$/Si substrate and then selectively transferred a portion of the samples to another marked substrate. After the transfer, the samples preserve their original morphology and relative position (Fig. 4a-b), and no breakages or wrinkles are found, which are further confirmed by AFM (inset of Fig. 4a-b, and larger area images are shown in supplementary material S2). The following is Raman and photoluminescence (PL) spectra characterization under 532nm excitation[37]. The exfoliated WSe$_2$ clearly presents two Raman peaks in the range of 250 and 260 cm$^{-1}$ (Fig. 4c), corresponding to the in-plane E$_{2g}$ mode and out-of-plane A$_{1g}$ mode of WSe$_2$, respectively. The Raman peaks are essentially coincident before and after the transfer process, indicating that no contamination is introduced to the transferred WSe$_2$. The positions of PL peaks before and after transfer display an identical band gap of 1.65 eV (Fig. 4d), indicating that the band structure of the WSe$_2$ is not affected during the transfer. Though the full width at half maximum (FWHM) shows a broadening from 29nm to 39nm, combined with substantially unchanged Raman and AFM data, this change can be attributed to the interaction between the sample and different substrates [38].

The interface quality significantly affects the electronic structure and the transport performance of 2D materials devices [39]. Especially for spintronic applications, the interface is a key issue [40]. To investigate the interface quality in our transfer method, we further fabricate a WSe$_2$ vertical spin valve, in which bilayer WSe$_2$ is sandwiched between two ferromagnetic (FM) electrodes (NiFe and Co) and served as a nonmagnetic spacer layer [41]. As shown in Fig. 5a, the resistance of junction is measured with four-probe method. And the coercivities of two FM electrodes are designed to be different, so that the magnetization alignments of them can be made semi-parallel or semi-antiparallel by sweeping the in-plane magnetic field ($H$). The two resistance states corresponding to the two magnetization alignments give us the magnetoresistance (MR) of the spin valve. The linear current-voltage ($I-V$) behaviors of the junction indicate the good Ohmic contacts of the NiFe/WSe$_2$/Co spin valve, and the resistance has a positive correlation with temperature, exhibiting a metallic behavior similar to what has been reported for other TMD spin valve device [42]–[44]. A strong bonding is expected between WSe$_2$ and NiFe, yielding a strong wave-function overlap between W and NiFe states that results in a metallic junction. This expected bonding confirms that our method guarantees a 2D material to maintain a clean interface after transfer.

The typical MR curves of our Co/WSe$_2$/NiFe spin valves at 300 K and 100 K are shown in Fig. 5b. There are two states, parallel alignment of the magnetization for low resistance ($R_P$) and antiparallel alignment for high resistance ($R_{AP}$). The MR ratio is then defined as $[R_{AP}−R_P]/R_P$, which is 1.2%
at 50 K and 0.5% at 300 K, as shown in Fig. 5c. For evaluating this result, we compare the MR ratios at various temperatures that obtained in different vertical spin valves of 2D materials, while their structures are uniformly FM/2D/ FM (FM=Co or NiFe). Those modified structures are excluded, such as adding a tunneling layer for enhancing the MR ratio). Our spin valves of PVA transferred bilayer WSe$_2$ show a relatively highest range of MR ratios (solid pentagram). For comparison, we also prepare the same device with conventional spin-coating-PMMA-assisted transfer (open pentagram), whose MR range is shown in the reported results from var-coating-PMMA-assisted transfer (open pentagram). The insets present the I-V curves from a four-probe measurement and the corresponding device diagram. The resistance shows a typical metallic behavior. The in-plane magnetic field H is applied at approximately 45$^\circ$ relative to the direction of the ferromagnetic electrodes. The scale bar is 10 $\mu$m. (b) The magnetoresistance (MR) ratio versus temperature of a WSe$_2$ spin valve at 100 K and 300 K. (c) The distribution of absolute MR ratios of 2D vertical spin valves prepared by different transfer methods at various temperatures. The results of this work are labeled by red symbols (solid/open pentagram and triangle).

IV. CONCLUSION

In conclusion, we develop a “movable-type” transfer and stacking for 2D materials and their van der Waals heterostructures based on the principle of engineering different surface tension between PVA, PDMS and 2D van der Waals surface, in which the prefabricated and water-soluble PVA flakes arranged on the PDMS pieces are wielded for selective and modularized manipulation. Such as the selective transfer with a one-micron spatial resolution, flexible stacking control of both angle and position, and transfer of 2D materials with pre-deposited metal films are realized via our method. The cleanliness of the interface after the transfer is verified by AFM. Raman and PL characterizations, which indicate our method guarantees the sample’s intrinsic properties. Furthermore, we construct the vertical spin valves based on TMD materials, which show superior performances. With efficient, flexible and batchable achieving the interface and contact engineering of 2D materials, such a “movable-type” transfer method can promote the development of future high-quality spintronic devices.

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YUAN CAO received the B.S. degree from the School of Information Engineering of Zhengzhou University, Henan, China, in 2015. He is currently pursuing the Ph.D. degree with the School of Microelectronics, Beihang University, Beijing, China. His current research interests include the two-dimensional materials and heterostructures spin valve, from vertical to lateral.

XINHE WANG received the Ph.D. degree from the Department of Physics, Tsinghua University, Beijing, China, in 2018. He is currently a Postdoctoral Researcher with BDBC, Beihang University, Beijing, China. His current research interests include spin current transport in low dimensional materials and novel spin logic devices.

XIAOYANG LIN received the Ph.D. degree in physics from Tsinghua University, China, in 2015. He is currently an Associate Professor with Beihang University. He has published more than 30 articles in SCI journals, such as Nature Electronics, Nature Communications, Physical Review Applied, and so on, with more than 700 total citations; >30 USA invention patents. He has been working on emerging materials and devices of electronics, especially 2D spintronics and optically controlled spintronics. He also leads the Group of Two-dimensional Opto-spintronics (TOS) in the institute.

WEI YANG received the B.S. degree from the School of Electronic and Information Engineering, Beihang University, Beijing, China, in 2017. He is currently pursuing the Ph.D. degree with the School of Microelectronics, Beihang University. His current research interests mainly include the magnetoresistance junction and lateral spin valve that based on the two-dimensional materials.

CHEN LV received the B.S. degree from the School of Physics and Electronics, University of Electronic Science and Technology of China, Chengdu, China, in 2018. He is currently pursuing the Ph.D. degree with the School of Microelectronics, Beihang University, Beijing, China. His current research interests mainly include the light controlled magnetoresistance and magnetoresistance junction.

YUAN LU received the B.S. degree from the Department of Physics, Tsinghua University, Beijing, China, in 1999, and the Ph.D. degree in Institute of Semiconductors, Beijing, in 2004. Since 2008, he has been a Research Scientist with the CNRS, Institut Jean Lamor, Nancy, France. He has authored or coauthored more than 60 scientific articles. His current research interests include the two dimensional based and organic based spintronics.

YOUGuANG ZHANG (Member, IEEE) received the M.S. degree in mathematics from Peking University, Beijing, China, in 1987, and the Ph.D. degree in communication and electronic systems from Beihang University, Beijing, in 1990. He is currently a Professor with the BDBC, Fert Beijing Institute, and the School of Electrical and Information Engineering, Beihang University. His current research interests mainly include microelectronics and computer architectures.

WEISHENG ZHAO (Fellow, IEEE) received the M.S. degree in electrical engineering from the ENSEEIHT Engineering School, Toulouse, France, in 2004, and the Ph.D. degree in physics from the University of Paris-Sud, Orsay, France, in 2007. From 2008 to 2009, he was with the Embedded Computing Laboratory, CEA. From 2009 to 2013, he was with the CNRS as a Tenured Research Scientist. He is currently a Professor with the BDBC, Fert Beijing Institute, and the School of Microelectronics, Beihang University, Beijing, China. He has authored or coauthored more than 120 scientific articles. His current research interests include the spintronics and its related device, circuit, and architecture investigations. He is an Associated Editor of the IEEE TRANSACTIONS ON NANOTECHNOLOGY and IET Electronics Letters.