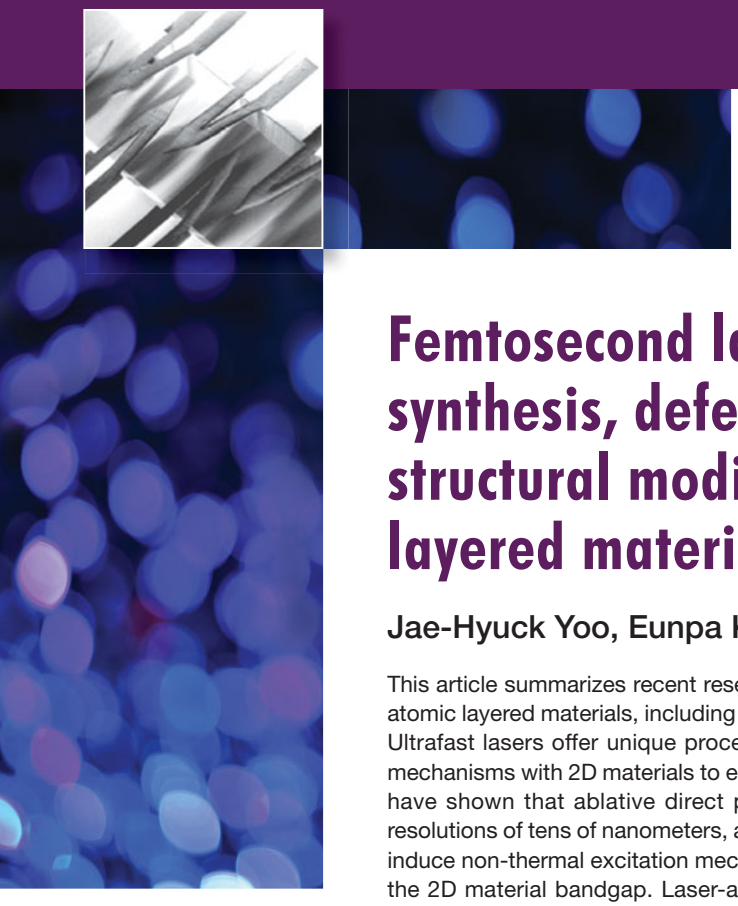


Femtosecond laser patterning, synthesis, defect formation, and structural modification of atomic layered materials

Jae-Hyuck Yoo, Eunpa Kim, and David J. Hwang

This article summarizes recent research on laser-based processing of two-dimensional (2D) atomic layered materials, including graphene and transition-metal dichalcogenides (TMDCs). Ultrafast lasers offer unique processing routes that take advantage of distinct interaction mechanisms with 2D materials to enable extremely localized energy deposition. Experiments have shown that ablative direct patterning of graphene by ultrafast lasers can achieve resolutions of tens of nanometers, as well as single-step pattern transfer. Ultrafast lasers also induce non-thermal excitation mechanisms that are useful for the thinning of TMDCs to tune the 2D material bandgap. Laser-assisted site-specific doping was recently demonstrated and ultrafast laser radiation under ambient air environment could be used for the direct writing of high-quality graphene patterns on insulating substrates. This article concludes with an outlook on developing further advanced laser processing with scalability, *in situ* monitoring strategies, and potential applications.

Introduction

The electronic and mechanical properties of two-dimensional (2D) materials have showed potential to overcome the drawbacks of conventional electronics in moving toward mechanically flexible/stretchable applications for lightweight, portable, and conformable products such as flexible displays, solar cells and batteries, and wearable health patches.[1] In spite of the very good properties of graphene, the zero-energy bandgap in its electronic structure makes it unsuitable for switching devices in electronics. Recently, 2D layered semiconductors, in particular, the series of transition-metal dichalcogenides (TMDCs) such as MoS_2 and WSe_2, have shown reliable performance as novel semiconductor materials that can provide mechanical flexibility and high mobility.[2] While the potential of 2D layered materials is exciting, major roadblocks will have to be resolved for successful transition to commercial applications by developing reliable technologies for the controlled fabrication of high-performance devices (e.g., gate width of ~10 nm for TMDC-based field-effect transistors with high ON-OFF current ratios,[3] reliable means to control the number of TMDC layers for bandgap tuning,[4] on-demand tuning of electronic properties by defect engineering,[5] and maskless patterning of 2D layered materials).

Ultrafast lasers offer unique processing routes that take advantage of distinct interaction mechanisms with 2D materials to enable extremely localized energy deposition. Both thermal and non-thermal mechanisms can contribute to the processing of layered materials, depending on the amount of energy absorbed.[6] For femtosecond laser processing of graphite, time-of-flight measurements have confirmed that femtosecond laser-driven Coulomb explosion (i.e., ionization assisted non-thermal ablation mechanism) is responsible for atomic-scale material removal from graphite, suppressing subsequent thermal material ejection.[7,8] It is also noted that atomic-scale material removal from layered materials by ultrafast lasers is frequently coupled with chemical reactions such as oxidation or burning, as in the case of graphene.[9] It is believed that such a disturbance can lead to non-thermal bond breaking and even stochastic extraction of atoms from layered materials. The reduced in-plane thermal conductivity tends to accelerate the process thus partly contributing to high spatial confinement of the process.[9]

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Similar bond-breaking behavior upon interaction with ultrafast lasers has been observed for MoS_2 layers, where precise layer removal or thinning can actively adjust its bandgap. In this case, a lattice sublimation mechanism (i.e., laser-induced bond breaking and subsequent atom removal) was proposed, possibly assisted by a non-thermal ionization state rather than by oxidation.[10] It is interesting to note that atomic-scale vacancies formed in MoS_2 by high-energy electrons (in a high-resolution transmission electron microscope environment) are easily refilled with impurity atoms,[11] suggesting a new method for defect engineering or doping of TMDC materials. This study indicates that further investigation of the effect of ultrafast lasers in terms of atomic-scale defect formation is necessary with advanced *in situ* diagnostics tools, as will be briefly discussed in later sections. It is also noted that a wider range of ultrafast laser- (typically a femtosecond or picosecond laser that can selectively induce non-thermal or thermal mechanisms) based modification processes such as ablative, additive, and defect engineering processes, are governed by closely linked mechanisms (e.g., non-thermal bond-breaking possibly accompanied by defect engineering or doping).

The nonlinear ultrafast optical properties of 2D layered materials have been actively investigated for photonic device applications.[12] Accordingly, the nonlinear absorption behavior has been examined during ablative processing of layered materials; two-photon absorption was confirmed during ablation of MoS_2.[9] Since the heat dissipation of 2D layered materials is dominantly influenced by thermal coupling to the surrounding environment, a dependence of the processing threshold in terms of laser fluence or energy density on the substrate material was also found. The underlying thin-film or substrate material can be incorporated into the layered materials, as shown in experimental work on localized laser synthesis of graphene.[6,13] Considering the precise and unique energy coupling mechanisms with 2D layered materials, more accurate thickness control is anticipated by using ultrafast lasers.

The following sections summarize research on ultrafast laser-based processing of 2D atomic layered materials, including ablative patterning and thinning, substitutional doping, and additive direct writing. The article concludes with an outlook toward advanced processing and *in situ* diagnostics.

Ablative patterning and pattern transfer of graphene by ultrafast lasers

In graphene device fabrication, typically a synthesized graphene layer on a metal foil is transferred to a target substrate, followed by patterning. However, these processes involve wet chemical steps and can unintentionally modify the graphene properties.[14,15] Furthermore, most of the transferred graphene area is etched away during the patterning step, so little area is used. Hence there is room for improvement in current graphene fabrication methods.

Femtosecond laser-ablative patterning of suspended and supported graphene under ambient conditions is summarized in **Figure 1**.[16] Direct ablation was observed upon single pulse irradiation of suspended graphene (Figure 1a), wherein graphene folds were formed in the periphery of ablated supported graphene spots (Figure 1b). Multiscale graphene patterns have been also transferred to arbitrary substrates by applying femtosecond laser pulses on a graphene/metal foil prior to the transfer step (Figure 1c–d). The diminished thermal transport accompanying femtosecond laser processing enables formation of high-resolution graphene patterns, even on thermally conductive metal foil.[17] Furthermore, a laser-induced pattern transfer (LIPT) method was developed. Femtosecond laser patterning is performed on a graphene/poly(methyl methacrylate) (PMMA) donor layer, and the patterned graphene is simultaneously transferred to a target substrate. This results in the simultaneous patterning and transfer of graphene in a single processing step. The LIPT method simplifies the complex graphene fabrication process while augmenting flexibility in graphene device design. Graphene/PMMA microscale patterns on a flexible substrate (Figure 1e) and suspended graphene cantilever structures were fabricated via the developed single-step method (Figure 1f).[18]

Direct nanoscale patterning of suspended and supported graphene has been demonstrated, where a vortex phase plate beam shaping technique was applied to achieve resolution down to 20 nm.[9] When graphene is patterned into nanoribbon shapes, an energy gap is created due to quasi-one-dimensional carrier confinement.[19] By employing high repetition rate ultrafast lasers, precise graphene patterning was achieved due to local oxidation and conversion into CO_2. Based on Raman spectroscopy, the defect-induced D-line was not detected after the patterning, indicating that high-atomic-structure quality was maintained in the atomic scale even at the cut edges. Hence direct patterning of graphene using ultrafast pulse lasers is a promising technique for nanoscale graphene fabrication, enabling defect engineering at the nanoribbon edges, which could be free from unintended contamination.

Laser thinning of 2D layered materials

TMDCs are attractive 2D layered semiconductor materials that have unique mechanical, electrical, and optical properties.[20] For example, monolayer MoS_2 has a direct bandgap of ~1.85 eV instead of the indirect bandgap of 1.2 eV for the bulk counterpart. A tightly focused continuous-wave laser beam was utilized to thin MoS_2 down to a single layer.[4] A similar approach was also applied to process multilayer graphene for obtaining single-layer graphene.[21] Although laser light may be absorbed in both mono- and multilayered materials, the thinning process is self-regulated due to the variation of the thermal conductivity with the number of layers in conjunction with the heatsink effect of the substrate. The MoS_2 thinning process can be further controlled by choosing the appropriate laser wavelength and by using ultrafast pulses, shorter than ~1 ps. Multilayer MoS_2 is opaque to near-infrared Ti:Sapphire femtosecond laser radiation. In contrast, single-layer MoS_2 is transparent, implying potential for selective-layer processing. A two-photon

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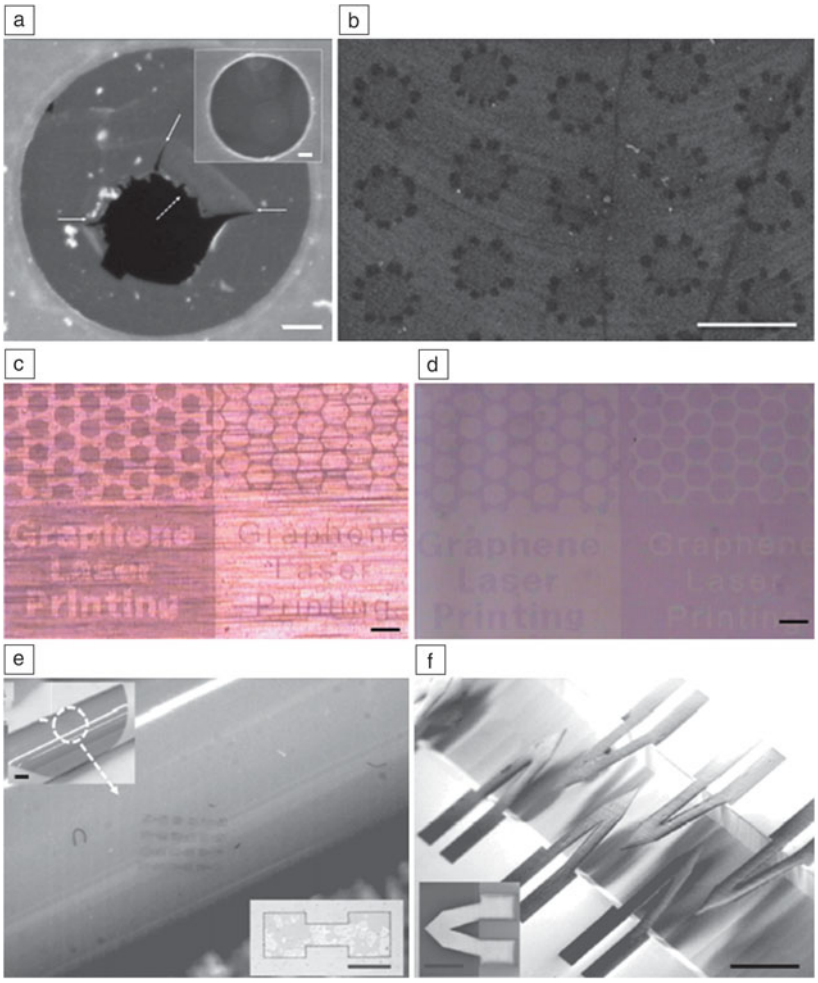


Figure 1. Direct laser patterning of graphene. (a) Scanning electron microscope (SEM) image of a single pulse irradiated suspended single-layer graphene on a transmission electron microscope grid. Arrows indicate cracks developed by ablation. The inset shows a pristine suspended graphene. The scale bar indicates 200 nm. (b) SEM image of folded graphene structures on a SiO_2/Si substrate.[16] The scale bar indicates 2 μm. Optical micrographs of (c) various graphene patterns on a graphene/copper foil after femtosecond laser patterning and (d) transferred graphene patterns on a SiO_2/Si substrate. Scale bars are 100 μm. (e) Optical microscope image of transferred graphene/poly(methyl methacrylate) (PMMA) patterns on a flexible polyimide film over a curved surface. The entire view is shown in the upper inset. The bottom inset shows a transferred pattern with higher magnification. (f) Tilted SEM image of graphene/PMMA cantilevers over a trench in a silicon substrate. The inset shows a SEM top view image of the cantilever pattern.[18] Scale bars in (e–f) are 200 μm.

absorption process was experimentally confirmed in the course of the ultrafast laser-based MoS_2 thinning process.[10]

Ultrafast laser direct writing of graphene patterns

Although pattern-transfer approaches have demonstrated high-quality graphene patterning capabilities, these methods involve multiple processing steps and may cause structural damage during graphene transfer. To resolve these issues, researchers must address these key challenges with the development of direct and transfer-free writing of graphene on desired substrates. To this end, laser direct-writing methods such as laser-induced thermal reduction of graphene oxide (GO) films,[22,23] or laser-assisted chemical vapor deposition (CVD),[24,25] have been examined. However, the laser-induced thermal reduction of GO films yields a substantial number of defects, significantly deteriorating electrical conductivity. The quality of the graphene pattern by laser-assisted CVD on Ni foils was significantly improved over that of thermally reduced GO,[25] yet a postgrowth graphene-transfer process (i.e., etched removal from Ni foil, then transferred to the target substrate) was necessary.

Ultrafast laser direct writing has been employed for directly growing graphene patterns on various insulating substrates under ambient conditions through rapid heating of cosputtered Ni/C thin films (the detailed process sequence is depicted in **Figure 2**a).[26] The sheet resistance of the resulting graphene patterns is approximately two times lower than the bilayer graphene fabricated by the laser-assisted CVD and one order of magnitude lower than that for the reduced GOs, possibly enabled by the preservation of a high-quality graphene lattice by the transfer-free growth process, and a Ni doping effect.[27,28] The as-measured optical transmittance was 94.3% at 550 nm, supporting potential use as a transparent electrode.

The linewidth of the as-fabricated graphene patterns was close to the focal spot size of the femtosecond laser direct-writing system (submicrometer for the tested setup). By heating the cosputtered Ni/C thin film using the femtosecond laser, graphene growth was completed within an order of ms, enabled by the highly efficient energy delivery to the substrate maintained at room temperature. In the controlled comparison experiments, the conventional rapid thermal annealing process[29] required exposure for a few minutes at 1100°C under vacuum. As shown in Figure 2b, a distinct femtosecond laser processing window exists to achieve optimum bilayer graphene formation at ~24 mW laser average power (confirmed by the 2D/G band ratio in the Raman spectrum being close to 1; the G band indicates in-plane vibrational mode induced peak intensity, and the 2D band is induced by the second-order defect or disorder band). Lower laser power yields multiple graphene layers with defects, while higher laser power forms additional defects through lattice damage.

Under the optimized condition for high-quality graphene growth, large-scale graphene-based integrated-circuit patterns have been fabricated in a programmable manner (Figure 2c)

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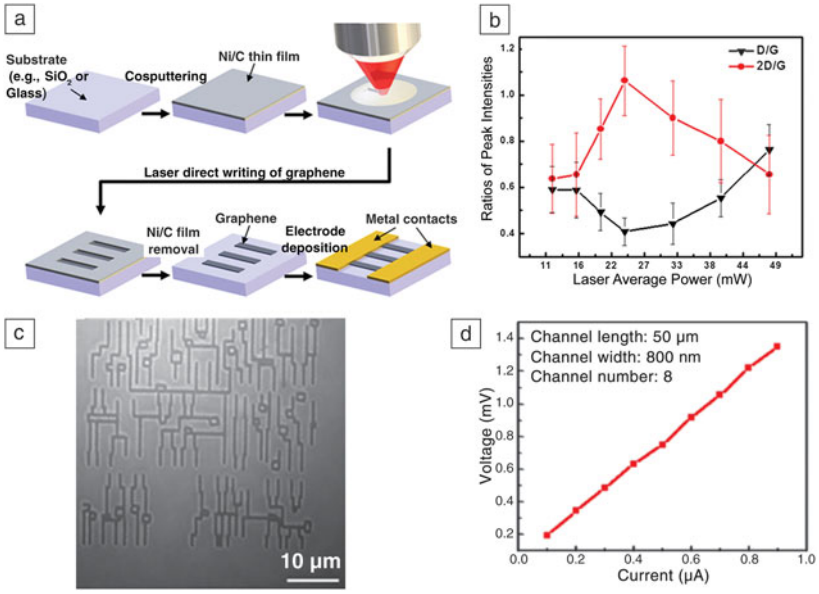


Figure 2. Direct writing of graphene patterns by a femtosecond laser. (a) Process sequence of femtosecond laser direct-writing process to fabricate graphene patterns under ambient conditions. (b) Dependence of the graphene quality (Raman D/G and 2D/G ratios; G band induced by in-plane vibrational mode, D band by defect or disorder, and 2D band by the second-order defect) on laser average powers. At an optimal laser power of 24 mW, a minimal D/G ratio indicates high-quality crystallinity, and a 2D/G ratio close to 1 confirms formation of bilayer graphene. (c) Optical micrograph of large-scale graphene patterns on a glass substrate fabricated by femtosecond laser direct writing, for integrated-circuit applications. (d) Electrical characterization of graphene devices fabricated by femtosecond laser direct writing, current–voltage curve of the four-terminal electrical device with eight graphene straight line channels. The linear trend and high electrical conductance of the current–voltage curve show metallic characteristics of the fabricated graphene patterns, indicating its potential as an electrode. Reproduced with permission from Reference 26. © 2014 Nature Publishing Group.

with high-quality electrical performance (measured *I–V* curve shown in Figure 2d). A similar approach was recently applied to fabricate a flexible solid-state microsupercapacitor based on porous carbon formation in polyimide.[30] Considering the high degree of simplicity, flexibility, and reliability in producing graphene patterns, the ultrafast laser direct-writing method could open the door for fabricating a wide range of advanced graphene-based devices.

Laser doping of 2D layered materials

Laser-assisted doping has reportedly modified the semiconductor device characteristics in many 2D materials.[31–34] Since doping is facilitated by defect sites, GO was typically subjected to laser illumination under a controlled dopant precursor gas environment for the simultaneous reduction of GO into doped graphene. When the doping process is carried out using nanosecond lasers, ultraviolet wavelengths are used since they are absorbed by the GO material.[32] A similar process using a femtosecond laser (800 nm wavelength) in a controlled ammonia environment showed efficient reduction into *n*-doped graphene.[33]

Two-dimensional TMDC layered materials with the formula MX_2 (M = Mo, W; X = S, Se, Te) have direct bandgaps ranging from 1.5 to 2.0 eV, therefore, a visible range laser is an effective choice for processing. Laser-assisted phosphorus doping in ultrathin (monolayer and bilayer) TMDCs, including *n*-type MoS_2 and *p*-type WSe_2, was recently demonstrated using a 532-nm-wavelength laser, which is a versatile, air-stable, and tunable method, carried out by varying the duration and intensity of a focused laser irradiating in a phosphine environment to accomplish site-specific doping (**Figure 3**).[34] Extending the laser-doping method to emerging 2D layered materials will not only allow site-specific selectivity, but also prevent the redistribution of the impurity profile via a pulsed laser with short irradiation duration. As mentioned earlier, ultrafast lasers may provide a new pathway to TMDC layer doping through atomic-scale control of vacancy generation and the subsequent refilling with impurity atoms via non-thermal lattice disturbance processes.[11]

Further ultrafast laser processing of 2D layered materials and *in situ* diagnostics

Considering that engineering of imperfections or defects in 2D layered materials (e.g., vacancies, edges, dopants, adsorbates, or substrates) can play a critical role on the properties (e.g., electrical, optical, thermal, or magnetic) of 2D layered materials owing to their low dimensionality, atomic-scale subtractive and additive control over the 2D layered materials can be a powerful processing tool. To this end, optical near-field technology can be used to improve lateral processing resolution, as demonstrated at the nanoscale by various processing examples,[35–37] or laser-assisted atomic layer deposition or etching to achieve atomic thickness level control toward 2D layered heterostructure fabrication.[36,38]

For achieving high throughput and precision, various schemes have been proposed for scalable and programmable processing on the basis of multiple laser light confining elements in conjunction with *in situ* diagnostics and metrology.[39] Integrating *in situ* process monitoring tools, such as both optical far- and near-field laser illumination arrangements, into scanning and transmission electron microscopes will enable investigation of the fundamental interaction mechanisms of ultrafast lasers with 2D layered materials.[40–42] Further, the structural modification signature measured by the imaging and analytical diagnostic functions of electron microscopes can be correlated with *in situ* measured optical spectroscopic signals (e.g., *in situ* Raman spectroscopy[43,44]).

Conclusion and outlook

Ultrafast lasers can drive a wide range of subtractive and additive processes for the patterning and functionalization of

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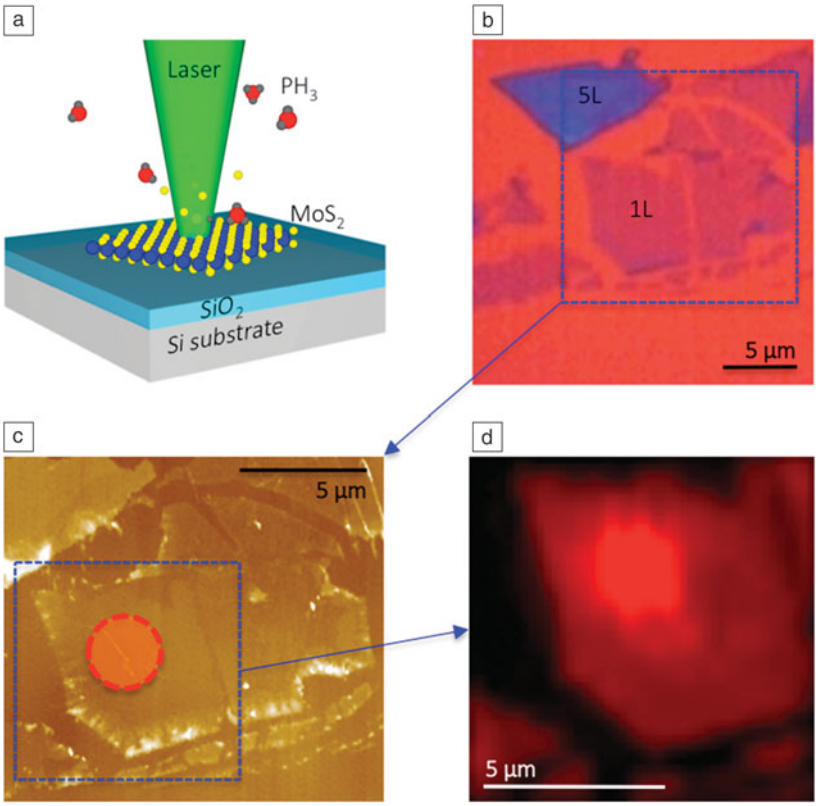


Figure 3. Laser-assisted doping of MoS_2. (a) Schematic diagram of laser-assisted doping. Focused laser locally illuminated onto the MoS_2 flakes (blue atoms, Mo; yellow atoms, S) located on a SiO_2/Si substrate under diluted phosphine (PH_3) dopant gas environment. The function of laser illumination is to create sulfur vacancies and simultaneously dissociate the dopant molecules in preparation for the subsequent doping process. (b) Optical image of as-prepared monolayer (1L) and five-layer (5L) MoS_2 on a SiO_2/Si substrate (blue color represents MoS_2). (c) Atomic force microscope image of the (b) zoomed area. Its thickness is approximately 0.7 nm, in good agreement with the thickness of monolayer MoS_2. The circle in (c) is the laser spot area in the laser doping. (d) Photoluminescence (PL) mapping of the zoomed area in (c) that clearly shows the PL intensity enhancement of the laser-assisted doped area.[34]

atomic layered materials. Mechanisms such as multiphoton absorption, minimized thermal effects, non-thermal interactions, as well as compatibility and integration with advanced diagnostics can be beneficial in this effort. Research at the fundamental level with the aid of *in situ* diagnostics tools is necessary to achieve atomic-scale manipulation. Furthermore, ultrafast lasers can be configured to scalable processing tools and therefore realize the next-generation high-performance portable, flexible, and wearable devices based on 2D materials.

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Washington, DC | September 17–22, 2017

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The **2017 International Conference on Silicon Carbide and Related Materials (ICSCRM 2017)** is the premier biennial meeting covering all aspects of the latest SiC research and development. Scientists, engineers, and students in the SiC field will gain insights into how challenges and advances in one subfield motivate research in other subfields through a combination of tutorials, industrial talks and presentations of the latest research and development from the SiC community.

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