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Author(s): Turunen, Mikko T.; Hulkko, Eero; Mentel, Kamila K.; Bai, Xueyin; Akkanen, Suvi-Tuuli; Amini, Mohammad; Li, Shisheng; Lipsanen, Harri; Pettersson, Mika; Sun, Zhipei

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Deterministic modification of CVD grown monolayer MoS₂ with optical pulses

Mikko T. Turunen, Eero Hulkko, Kamila K. Mentel, Xueyin Bai, Suvu-Tuuli Akkanen, Mohammad Amini, Shisheng Li, Harri Lipsanen, Mika Pettersson and Zhipei Sun**

M. T. Turunen, X. Bai, S-T. Akkanen, Prof. H. Lipsanen, Prof. Z. Sun

Department of Electronics and Nanoengineering, Aalto University, P. O. Box 13500, FI-00076 Aalto, Finland

Email: zhipei.sun@aalto.fi

Dr. E. Hulkko, Dr. K. K. Mentel, Prof. M. Pettersson

Nanoscience Center, Department of Chemistry, University of Jyväskylä, P.O. Box 35, FI-40014 University of Jyväskylä, Finland

Email: mika.j.pettersson@jyu.fi

M. Amini

Department of Applied Physics, Aalto University, Espoo, 00076 Aalto, Finland

Dr. S. Li

International Center for Young Scientists (ICYS), National Institute for Materials Science (NIMS), Tsukuba, Japan

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Transition metal dichalcogenide monolayers have demonstrated a number of exquisite optical and electrical properties. Here, we report the optical modification of topographical and optical properties of monolayer MoS₂ with femtosecond pulses under an inert atmosphere. We demonstrate a formation of three-dimensional structures on monolayer MoS₂ with tunable height up to ~20 nm. In contrast to unmodified monolayer MoS₂, these optically modified structures show significantly different optical properties, such as lower photoluminescence intensity and longer fluorescence lifetime. Our results suggest a novel way to modify transition metal dichalcogenide materials for mechanic, electronic and photonic applications.

1. Introduction

Throughout the last decade, transition metal dichalcogenides (TMDs) have proven to be a fine addition to the library of two-dimensional (2D) layered materials due to their unique physical properties.^[1,2] In monolayer form, they exhibit strong photoluminescence (PL) owing to their

direct bandgap^[3,4] and they also show substantial second-order optical nonlinearity as a relatively high second harmonic generation (SHG) due to their centrosymmetry breaking structure.^[5] Currently, there is a great interest to tune their physical properties for applications by numerous methods (e.g., defect,^[6–8] strain,^[9–11] phase engineering,^[12,13] doping,^[14,15] electric field,^[16,17] plasmonics^[18,19], optical pumping^[20–23] and optical modification^[24–27]). Among them, direct optical modification is one of the most promising approaches due to its advantages, such as cost-effectiveness and spatial controllability. In addition, it does not rely on lithographic processes and, hence, no contaminating chemicals are required. Thus far, optical modification has been mainly studied on graphene (e.g., topographical modification,^[28,29] inducing luminescence^[30] and tuning its electrical and optical properties^[30]). Recently, few studies have been reported on optical modification of mechanically exfoliated mono- and few-layer TMDs for applications, such as PL enhancement,^[24] electrical contacts,^[25] photodetectors^[26] and sensors.^[27]

Here, we report a relatively large-scale direct optical modification of the topographical and optical properties of chemical vapour deposition (CVD) grown monolayer MoS₂. Our results show that laser modification induces tunable topographical changes, PL quenching, and fluorescence lifetime change. This method would allow deterministic local tuning of physical properties (e.g., mechanical and optical properties) of TMDs for future applications.

2. Results and Discussion

Monolayer MoS₂ samples are prepared using CVD technique based on vapour-liquid-solid growth.^[31,32] The samples are grown directly on a silicon substrate with a 280 nm silicon oxide layer. The size of the resulting flakes is ~20 μm. Then, the sample is put in a nitrogen environment where it is exposed to femtosecond laser pulses at 515 nm with 600 kHz repetition rate and ~250 fs pulse duration for optical modification. The nitrogen atmosphere is used to prevent oxidation of the sample. The spot size of the laser is ~500 nm. The process parameters are selected based on our previous optical modification results on graphene^[28–30]: We choose the 532 nm laser wavelength because of the possibility of forming smaller patterns and the high laser power stability; We select 600 KHz repetition rate, mainly because higher repetition rate will decrease the modification threshold fluence of the material and speed up the modification process. However, we also notice that too high repetition rate (>1MHz) may lead to faster material heating, which is not desirable; We use ~250 fs pulse duration for modification due to that longer pulse duration (above ~1ps) will increase heating of the sample. Therefore, in this experiments, we focus on the influence of the irradiation time, while keeping constant

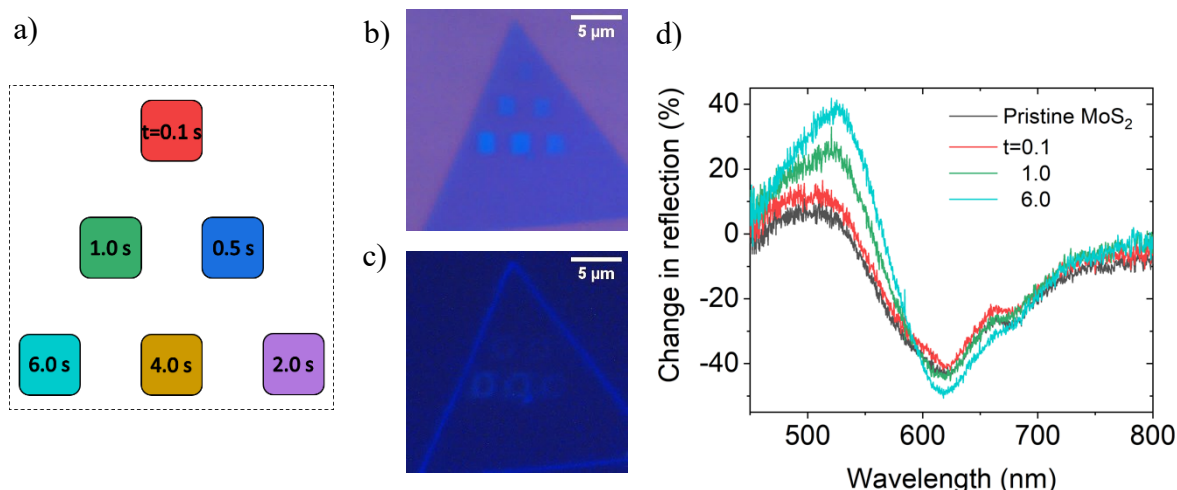


Figure 1. Optical modification pattern and the optical images of the modified MoS₂ flake. a) Different irradiation times (t) used to create deterministic rectangular patterns for optical modification. Higher irradiation time per spot equals a higher optical dose. b) Bright-field and c) dark-field optical microscope images of the modified flake. d) Reflection curves of pristine MoS₂ and modified areas relative to the SiO₂/Si substrate.

excitation wavelength, repetition rate and pulse duration. The optical modification experiment is carried out to pattern rectangular shapes illustrated in **Figure 1a**. We also prepare a sample with a spot-like modification pattern, the results of which are similar to the results shown below (**Figure S1** in Supporting information). The rectangular shape pattern is prepared by irradiating multiple spots separated by 100 nm. We fix the pulse energy at ~ 60 pJ but change the irradiation time for a spot (t) from 0.1 to 6.0 s, as indicated in Figure 1a.

Figure 1b shows an optical microscope image of a MoS₂ flake after optical modification, and some of the intended rectangular patterns shown in Figure 1a (e.g., the bottom rectangles with $t = 2, 4, 6$ s) are clearly visible on the flake. At the patterned areas, the flake appears a lighter shade of blue than the unmodified pristine flake, and this effect gets stronger when the irradiation time increases. **Figure 1c** shows a dark field microscope image, and again the patterned structures can be observed with the flake edge. Here, it can be noticed that the edges of the patterned areas are brighter, indicating a scattering edge. However, inside the modified areas, such scatter signal is not found in the dark-field image (Figure 1c). These observations imply that non-scattering 3D rectangular structures are formed at the modified positions. We then measure the reflection spectra of the optically modified areas (the measurement spot size is ~ 5 μm), and **Figure 1d** shows the relative reflection of those areas compared to the SiO₂/Si substrate. As seen in the microscope image (Figure 1b), the reflection increases in cyan wavelengths (~ 500 nm) with the irradiation time. More interestingly, with longer irradiation

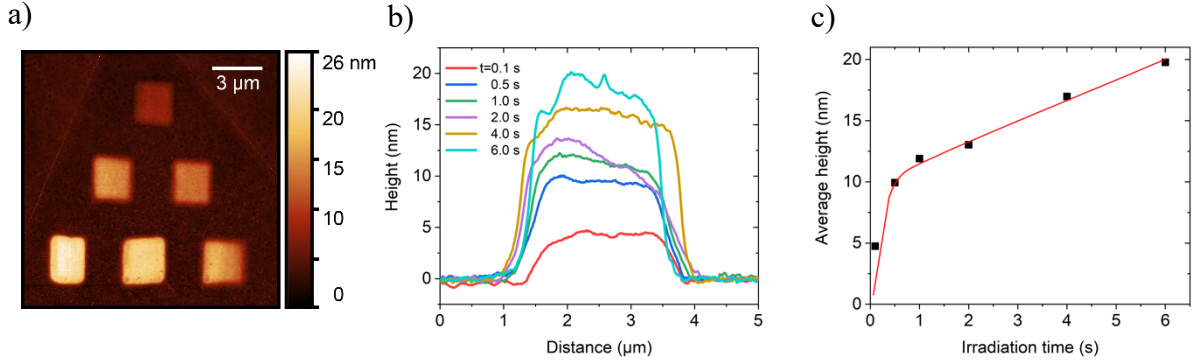


Figure 2. Tunable topography with optical modification. a) An AFM scan of the patterned sample. b) Height profiles of the irradiated areas. c) Average height as a function of irradiation time. The red line is a guide to an eye.

time, the reflection slightly decreases for the wavelengths around 600–700 nm where the excitons are situated. In addition, the local maximum (at ~ 660 nm) between A and B excitons gets more attenuated with the longer irradiation times.

To investigate the topography of the patterned areas, we perform an atomic force microscopy (AFM) scan, shown in **Figure 2a**. The scan result confirms that the sample is indeed more elevated in the irradiated areas. The height profiles are depicted in **Figure 2b**, and they indicate that the created 3D structures can reach ~ 20 nm height and have nearly a flat rectangular shape identical to our design (Fig. 1a). More interestingly, as the irradiation time of modified areas is increased, the 3D structures get higher. This is clearly shown in **Figure 2c**. It appears that the largest change in the height is observed for the irradiation time below 0.5 s/spot (i.e., ~ 20 nm height change per second of optical modification) after which, it follows a quite linear flatter curve (~ 2 nm height change per second of optical modification). The tunable 3D topography results correspond to what we observed before with graphene,^[28] possibly due to their similar 2D crystalline structure. We assign the process as the optical forging, which could be very useful for fabricating different 3D structures (e.g., suspended devices) with 2D materials.

Monolayer MoS₂ is known to show relatively high PL intensity due to its direct bandgap.^[3,4] We measure the PL of the flake after optical modification. **Figure 3a** shows PL spectra measured from the centers of the modified areas. The intensity of these spectra is over a magnitude lower than that of the unmodified flake area. **Figure 3b** shows a PL scan map of A exciton (a wavelength range 650–710 nm). The designed pattern can be seen clearly, as the intensity is severely quenched even for the least irradiated area, whose PL intensity is only

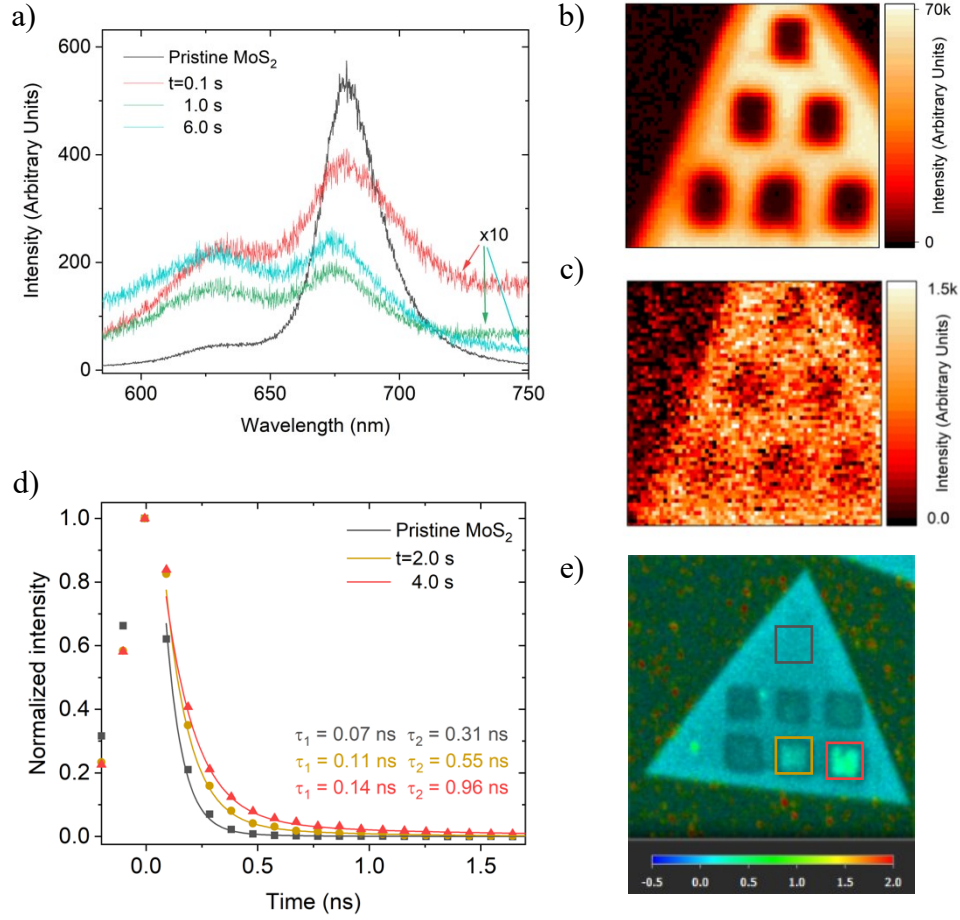


Figure 3. PL emission and dynamics. a) PL spectra of unmodified MoS₂ and modified regions with 60 pJ. PL spectra for optically modified areas are multiplied by ten for convenience. b) PL map of A and c) B exciton d) TRPL data of irradiated MoS₂ sample with 120 pJ. The lifetime values are acquired from the fit functions. e) Lifetime map of the sample. The color scale shows the lifetime in nanoseconds. The areas where the data in d) is acquired are marked with squares.

~5 % of that of the unmodified pristine area. We also plot the results for B exciton (615–645 nm), which is shown in **Figure 3c**. However, the PL quenching effect is not as pronounced as that observed for A exciton because its intensity is basically halved at its best.

To study the dynamics of the PL, we perform time-resolved PL (TRPL) measurements for another similarly irradiated sample. For this sample, we use a higher pulse energy of ~120 pJ during the optical modification. MoS₂ is known to have bi-exponential decay in PL. For example, it has been reported that A exciton typically has a fast decay component with ~few ps lifetime and a slow decay component with ~ 100 ps lifetime.^[33,34] Here, we observe that the PL emitted from the irradiated areas has longer lifetime components compared to those of the unmodified MoS₂. This is depicted in **Figure 3d**, which shows the PL intensity as a function

of time after the optical excitation of three different regions on the sample (**Figure 3e**). The fit curves and lifetimes, which are acquired from fitting, are also presented in Figure 3d. The time resolution of our instrument is ~ 250 ps, thus the faster components (marked with τ_1) reaching ~ 100 ps are not as reliable. Nevertheless, it is observed that the slower components (τ_2) tend to get longer (reaching nearly 1 ns for the most irradiated area) as the irradiation time increases. This can also be seen in Figure 3e, which shows a lifetime map of the sample, as the areas highlighted with yellow and red squares can be distinguished with longer lifetimes. However, when filtering the PL signal for energies above A exciton, these areas also show a lot higher PL intensities compared to unmodified MoS₂. The broad band of these PL signals might be caused by impurities resulting from high irradiation energy and time (**Figure S2** in Supporting information).

To understand the crystalline structure of the irradiated areas, we measure Raman spectra with a 532-nm laser (**Figure 4a**). The studies reveal that, after optical modification, typical E^{1}_{2g} and A_{1g} Raman modes of monolayer MoS₂ (illustrated in the inset of Figure 4a) are still observed, and there is only a small decrease in their intensity (**Figure 4b**). However, as the spectral position of the A_{1g} peak remains constant after modification, a clear blueshift (~ 2.2 cm⁻¹ for the sample with $t = 6.0$ s) and broadening can be observed for the E^{1}_{2g} peak (**Figure 4c** and **4d**). The shifting and broadening of the E^{1}_{2g} peak might be caused by strain, but similar changes should also be observed for the A_{1g} peak.^[35] More likely, the results suggest in-plane crystalline structure change, but not the out-of-plane direction. In addition to the slight changes in these two peaks, there are smaller satellite peaks appearing around them (marked with arrows in Figure 4a). These satellite peaks are getting more pronounced as the irradiation time is increased. Similar observations have been reported before for defect induced MoS₂.^[36,37]

A possible reason for the PL decrease is a phase change of MoS₂. Our sample is at 2H phase. However, MoS₂ also has a semi-stable 1T phase, which, in contrast to 2H, is metallic and, hence, would not emit any PL. To exclude the phase change during the optical modification, we carry out SHG measurement, as 1T phase monolayer MoS₂ has a relatively weak second harmonic response due to its centrosymmetric structure. Our SHG results show a slight decrease in the SHG signal presented in SI, which excludes the total phase change (**Figure S3** in Supporting information). Note that there are studies showing different approaches to induce a partial phase change in 2D TMD materials.^[13,38–40] However, even if our sample would be in a mixed-phase (partly 2H and partly 1T), the Raman studies should reveal some peaks that can

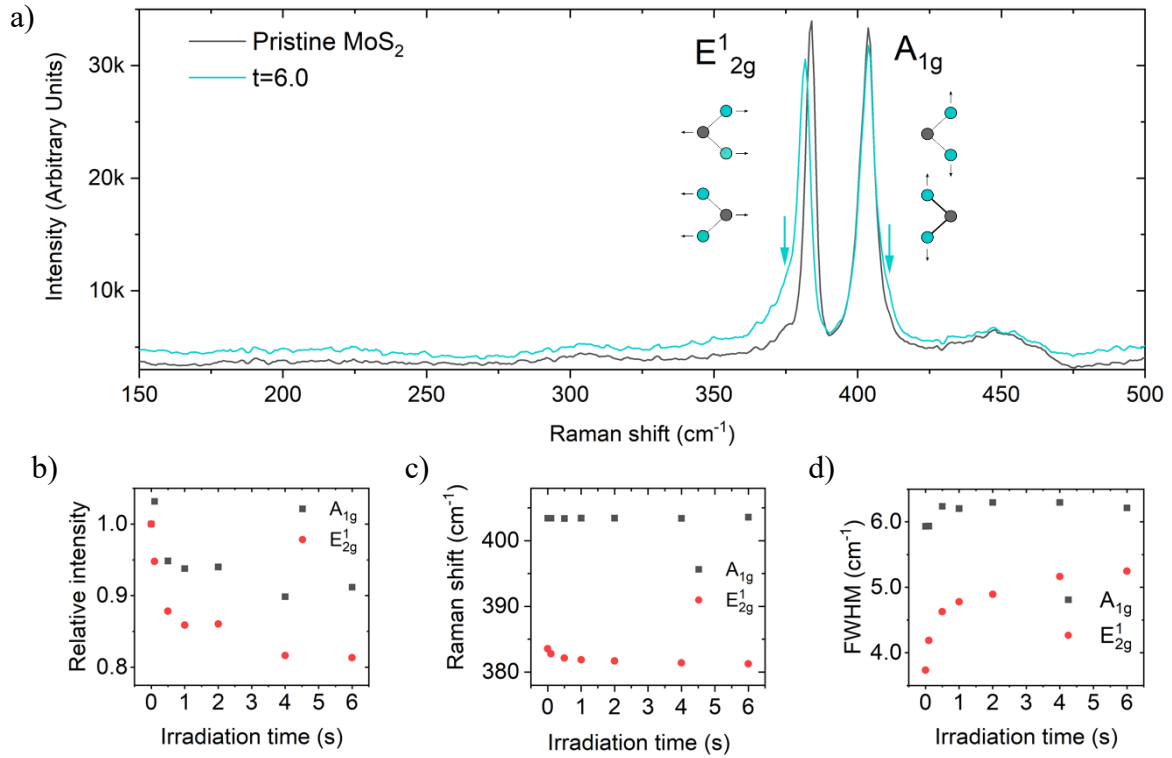


Figure 4. Raman signal of the optically modified sample a) Raman spectra of modified and unmodified areas. The E^1_{2g} and A_{1g} vibration modes (gray=Mo, cyan=S) are illustrated next to their respective peaks. The arrows highlight the satellite peaks b) Dose dependencies of intensity, c) Raman shift and d) spectral width as full-width at half maximum (FWHM) of the E^1_{2g} and A_{1g} peaks.

be assigned to the 1T phase. Such peaks, however, are not found, suggesting that this is not the case for our sample.

As discussed before, the small broadening and shifting of the Raman peaks and the decrease in the PL intensity possibly imply the existence of defects. These defects are most likely induced during the optical modification process and create energy levels within the bandgap of MoS₂.^[41,42] As the exciting photons are absorbed, the resulted electron-hole pairs will recombine via these defect levels most likely non-radiatively. The increase in fluorescence lifetimes also suggests the existence of defects.^[43,44] As the irradiation of optical modification is performed in an inert nitrogen environment, the defects should mainly be structural. Nevertheless, the type of these defects and the PL quenching mechanism are mainly unknown and deserve further study. For example, high-resolution transmission electron microscopy can be carried out to measure alterations in the crystalline structure; X-ray photoelectron spectroscopy can be utilized to show the potential existence of impurities on modified areas;

electrical measurements can be exploited to verify possible changes in the conductivity of the material.

3. Conclusions

In summary, we have successfully modified the topographical and optical properties of monolayer MoS₂ using laser irradiation in an inert environment. We noticed not only similar 3D structural formations as previously seen in similarly irradiated graphene but also changes in the optical properties of the MoS₂. The changes manifested as a pronounced decrease in PL intensity and a small increase in fluorescence lifetime. Our results indicate the deterministic local tuning of physical properties (e.g., mechanical and optical properties) of CVD TMDs with optical modification for applications (such as micro-electro-mechanical systems, and optoelectronics).

Supporting Information

Supporting Information is available from the Wiley Online Library or from the author.

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