Tailoring phonon modes of few-layered MoS₂ by in-plane electric field

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We discuss the effect of the in-plane electric field on the Raman spectroscopy for few-layered MoS₂. The characteristic Raman modes of MoS₂ show gradual red shift, while the intensity increases by 45–50% as the electric field is increased, showing a large electro-optical effect. Structural analysis suggests that our few-layered MoS₂ belongs to P6/m2 space group with broken inversion symmetry. We attribute this gradual red shift to this broken symmetry-driven piezoelectricity in MoS₂, which generates tensile strain along the perpendicular direction when the electric field is applied. The enhancement of the effect upon reversing the electric field direction adds credence to our interpretation. Our first principal density-functional theory calculation further substantiates the claim. This optical probing of the electromechanical coupling may lead to applications as a nondestructive technique for electric field/strain sensors in the nanoelectronics devices.

INTRODUCTION

Tunability of two-dimensional quantum materials (2DQM) through an external perturbation has strongly excited condensed matter research in recent years due to its possibility of applications, if not by the rich physics they offer. Graphene and other 2DQMs exhibit a wealth of unusual and fascinating properties, such as Dirac or Weyl semimetal, topological insulator, charge-density wave, and superconductivity. Among the vast library of 2DQMs, atomically thin semiconducting transition metal dichalcogenides (TMDCs) have attracted particular interest in optoelectronic applications due to their strong light-matter interaction, since it has shown several appealing applications, if not by the rich physics they offer.

Quasi-2D molybdenum disulfide (MoS₂) is a prototype material to study the light-matter interaction, since it has shown promising optoelectronic applications. MoS₂ crystallizes in hexagonal structure and belongs to the space group P3m1 (with inversion symmetry). Similar to other vdW crystals, e.g., graphene, an atomically thin version of this naturally occurring MoS₂ has been successfully obtained by mechanical exfoliation or liquid-phase exfoliation. The anisotropic crystal structure, strong in-plane Coulomb interaction, and weak out-of-plane van der Waal's interaction of MoS₂ leads to several interesting anisotropic optical and electronic behavior. Both monolayer and few-layered MoS₂ exhibit a number of intriguing physical properties, including a direct optical bandgap of about 1.8 eV, strong photoluminescence (PL), electroluminescence, and reasonably high mobility of the order of 1.8 eV/Vs.

In this paper, we describe the effect of moderate in-plane electric field on the phonon modes of few-layered MoS₂ probed by Raman spectroscopy at room temperature. We have been able to discuss the effect of the in-plane electric field on the Raman spectroscopy for few-layered MoS₂. The characteristic Raman modes of MoS₂ show gradual red shift, while the intensity increases by 45–50% as the electric field is increased, showing a large electro-optical effect. Structural analysis suggests that our few-layered MoS₂ belongs to P6/m2 space group with broken inversion symmetry. We attribute this gradual red shift to this broken symmetry-driven piezoelectricity in MoS₂, which generates tensile strain along the perpendicular direction when the electric field is applied. The enhancement of the effect upon reversing the electric field direction adds credence to our interpretation. Our first principal density-functional theory calculation further substantiates the claim. This optical probing of the electromechanical coupling may lead to applications as a nondestructive technique for electric field/strain sensors in the nanoelectronics devices.

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to correlate the effect of the applied electric field to the strain generated in MoS2, by density-functional theory (DFT)-based first-principal calculations of its phonon modes.

RESULTS
Structural characterization
The morphology of the as-prepared MoS2 was observed in transmission electron microscopy (TEM) and shown in Fig. 1a. It possesses nanosheet-like structure. In the inset of Fig. 1a we show the thickness map by Energy Filter TEM imaging recorded at 300 keV. The higher magnification image of the yellow-colored highlighted area in Fig. 1a is shown in Fig. 1b. We calculate the interplanar spacing as 0.63 nm, which corresponds to (002) plane of MoS2 from the high-resolution TEM image (Fig. 1b). The selected area electron diffraction (inset of Fig. 1b) shows the hexagonal lattice arrangement of MoS2. The line profile along the blue rectangular box (Fig. 1a, inset) gives a numerical value of the relative thickness (\(t_\lambda\)) of the specimen (Fig. 1c), where \(t\) is the thickness and \(\lambda\) is the inelastic mean-free path of the electron.\(^{40,41}\) The thickness of the material can be determined by knowing \(\lambda\), which is 119 nm\(^{40}\) at 300 keV. This implies that the average thickness of one of the segments of the MoS2 flake (highlighted in Fig. 1a, inset) is \(\approx 1.8\) nm, which is close to the thickness of a three-layered MoS2. A typical thickness distribution histogram, measured in a similar manner, is shown in Fig. 1d. The crystalline structure and phase of MoS2 nanosheets were further confirmed by XRD as shown in Fig. 1e, with the respective planes shown in parenthesis.\(^{42,43}\) In Fig. 1e, the dots represent the experimental points, while the blue line is the Rietveld refinement fit\(^{44,45}\) and the red line is the difference between the two. The structural refinement suggests that our MoS2 sample is of pure hexagonal structure (space group P6\(_3\)/mmc) with the lattice parameters \(a = b = 0.3117\) nm and \(c = 0.127\) nm. We observe a shrinkage along the \(a\) and \(b\) axes and an elongation along the \(c\) axis. Compared with the standard relative intensities of different peaks, it is found that (103) and (105) peaks are suppressed, indicating an irregular stacking arrangement of the atomic layers.\(^{46}\) Since \((h-k=0)\) reflections are unaffected by the stacking fault broadening, the (110) reflection has a larger relative intensity than (103) and (105) peaks. Furthermore, it can be seen from the fitting parameters in Supplementary Table 1 (see supplementary information) that the fault probability is \(\approx 14\%\), suggesting a disordered stacked system.

Raman spectra characterization of MoS2
The Raman spectra of our few-layered MoS2 at various in-plane electric fields are shown in Fig. 2. For zero field, the major characteristic Raman modes are observed at 382.5 and 406.7 cm\(^{-1}\) that belong to the in-plane E\(_{1}\)\(^{2}\)g and out-of-plane A\(_{1}\)g vibrations,
respectively. As an indirect measure, the position and the difference between the $E_{2g}$ and $A_{1g}$ evolves with the increase in the number of layers in MoS$_2$ until six layers before it gets saturated. At zero electric field, we find that the difference between these two Raman modes is 24.2 cm$^{-1}$, which is close to what is observed earlier for the five-layered MoS$_2$. Furthermore, the presence of a strong direct PL (and subsequent absence of an indirect PL) (see supplementary information) suggests that our MoS$_2$ is indeed a few-layered thick, as the bulk MoS$_2$ shows only indirect electronic transition.

Electric field effect on Raman spectra of MoS$_2$

As the in-plane electric field is increased, both the Raman characteristic peaks showed gradual red shifts in frequency (Fig. 2). This can be seen more clearly in Fig. 3, where we show the (a) $E_{2g}$ and (b) $A_{1g}$ spectra measured at different electric fields. A clear change in the peak position for both the peaks with the changing electric field can be observed. Alongside the shift in the peak positions to a lower value, the peak intensity increases with the increase in the electric field (Fig. 3d), while the full-width-half-maxima (FWHM) remain almost unchanged (Fig. 3c). Moreover, the difference between the $E_{2g}$ and $A_{1g}$ peak remains almost unaffected over the window of the applied electric field (see supplementary information). The intensity of the phonon modes increased substantially by 45–50% as the electric field is increased (Fig. 3d), implying a large electro-optical coupling. This gradual increase can be attributed to the manipulation of the MoS$_2$ electrons under the electric field as it in turn affects the Raman phonon intensity. A more careful look at the Raman spectra reveals the presence of an asymmetry near 377 cm$^{-1}$, which appears due to the transverse optical phonon mode of MoS$_2$. The presence of this asymmetry in the zero electric field spectra allows us to infer that this arises due to the quality of the sample and is not related to the effect of the electric field.

**DISCUSSION**

The intriguing red shift of principal phonon modes of MoS$_2$ under the electric field, can be attributed to the doping by source–drain bias due to the electric field or by an increase in temperature due to Joule heating. The effect of temperature, in this case, can be refuted as under increasing temperature, the Raman peak intensity decreases and the FWHM increases, while we observe the opposite trend in our system. The electrostatic doping, due to band bending, on the other hand, can lead to the softening of the phonon modes. However, the moderate applied electric field, to our understanding, will not be able to induce doping in the few-layered MoS$_2$ under consideration.

On the contrary, we believe that the red shifts in Raman spectra under the electric field can be due to the strain generated in the system. MoS$_2$, with an even number of layers, belongs to the P3m1 space group and with an odd number of layers belongs to the P6m2 space group (without inversion symmetry). Interestingly, this broken inversion symmetry induces piezoelectricity in MoS$_2$ with an odd number of layers. Notably, in a piezoelectric material, if an electric field is applied, a strain is generated along its perpendicular direction and vice versa. This piezoelectricity in MoS$_2$ develops and vanishes as the number of
layers change from odd to even due to the breaking and the recovery of the inversion symmetry. As our MoS\(_2\) belongs to the space group P\(6\text{m}2\), we believe that the applied in-plane electric field induces an out-of-plane tensile strain in it due to the broken inversion symmetry-driven piezoelectricity.

To substantiate our claim, we performed DFT-based first-principal calculations of the Raman active phonon modes of MoS\(_2\) for different percentages of out-of-plane tensile strains on it (for calculation details, see "Methods" section).

The DFT-calculated, Raman active modes of MoS\(_2\), for zero tensile strain (or zero electric field) occur at 385.3 cm\(^{-1}\) (\(E_{1g}^1\)) and 408.3 cm\(^{-1}\) (\(A_{1g}\)), which is in agreement with the experimental results. The plots for the calculated Raman modes are shown in the supplementary information.

In Fig. 4a, b, we show the experimental change in the Raman mode for \(E_{1g}^1\) and \(A_{1g}\) with the applied electric field, with respect to its zero electric field value. In the same figure, we show the corresponding change in Raman frequency with applied out-of-plane tensile strain with respect to its zero-strain value, calculated by first-principal calculation. With the applied electric field/tensile strain, both the \(E_{1g}^1\) and \(A_{1g}\) modes shift to lower frequencies.

The frequency shift (\(\Delta\omega\)) is defined as the difference between the frequencies of Raman active mode \(E_{1g}^1\) (or \(A_{1g}\)) at a finite electric field/tensile strain and at zero electric field/tensile strain. Mathematically, \(\Delta\omega_{ph} = \omega_{ph}(E) - \omega_{ph}(E = 0)\) where ‘ph’ stands for either \(E_{1g}^1\) or \(A_{1g}\). It is found that the frequencies of \(E_{1g}^1\) and \(A_{1g}\) decrease with increasing tensile strain along [001] direction in a similar manner as it changes with the applied in-plane electric field. Our first-principal calculation results are in agreement with the experimental data within an acceptable limit, and show similar features as observed previously on DFT-based calculation of Raman modes of MoS\(_2\) under tensile strain. The tensile strain...
increases the interlayer distance between S–Mo–S layers, which weaken the vdW interactions between adjacent layers. This results in a red shift in both $E_1^g$ and $A_{1g}$ modes. However, for a given tensile strain, a reduction in Raman shift of the $A_{1g}$ mode is larger than that of the $E_2g$ mode. In the $A_{1g}$ mode (arises from out-of-plane vibration of S atoms in the opposite direction along the z direction), the change in atomic vibration is dominated by interlayer force. While for $E_2g$ long-range coulombic interlayer force plays a key role and thus explains the observations.

Raman spectroscopy measurements by reversing the polarity of the electric field (Fig. 4c) add more credence to our claim of this electric field-controlled strain-mediated tailoring of the principal phonon modes of MoS2. Intuitively, we expect a similar outcome as before, and indeed observe similar red shifts of the phonon modes with respect to the electric field (Fig. 4c). However, the magnitude of the change in the phonon mode frequency under the electric field with respect to its zero-field value is different in two different directions of the electric fields (Fig. 4d). A simple diagrammatic approach (Fig. 4e) of the experimental situation can help us to understand this. Although it cannot be determined unambiguously, we consider that the left panel [(i) of Fig. 4e] denotes the negative electric field direction, whereas the right panel [(ii) in Fig. 4e] denotes the positive electric field direction in accordance with our previous analogy. For (i), the piezoelectric strain on the MoS2 flake is along the out-of-plane (positive z) direction; however, for (ii), it is along the negative z direction. Now for situation (i), the strain is applied along the free surface of MoS2 flake, whereas for case (ii), it is toward the substrate. The substrate suppresses the effect of strain, leading to a lesser change in the Raman shifts under the electric field.

Fig. 4  Comparison of the successive shifts of the Raman modes under an electric field (tensile strain). The plots of the successive shift of Raman mode with respect to its zero-field (or zero-strain) value. The symbols are the experimental data (in red, left and bottom axis), and the solid line is the theoretical calculation (in blue, right and top axis). $\Delta \omega_{ph} = \omega_{ph}(E) - \omega_{ph}(E = 0)$ where ‘ph’ stands for either $E_{2g}$ or $A_{1g}$. The variation of experimental (red, left axis) and theoretical (blue, right axis) $\Delta \omega_{ph}$ with the electric field (E) (red bottom axis) or tensile strain (blue, top axis) for $E_{1g}$ and $A_{1g}$ modes. $\Delta \omega$ for both $E_{1g}$ and $A_{1g}$ decreases linearly with the electric field or tensile strain. The errors in determining $\Delta \omega$ are obtained from the Lorentzian fit to the experimental data (see Fig. 2). C Raman spectra of few-layered MoS2 under two different directions of the applied electric field along with the zero-field data. For both the directions of the applied electric field (E), the principal Raman modes show a red shift in frequency with respect to their zero-field value (indicated by black dashed lines). D The plots of the successive shift of Raman mode under different directions of the applied electric field with respect to their zero-field value. The lines are to guide the eye. E Cartoon describing the experimental situation for observance of a different amount of shifts of the phonon mode frequency by reversing the direction of the applied electric field. In case (i) the strain is applied along the free surface of MoS2 flake, whereas for case (ii), it is toward the substrate. The substrate suppresses the effect of strain, leading to a lesser change in the Raman shifts under the electric field.
of-plane tensile strain shows a similar red shift and adds credence to our claim. Experimentally, our interpretation was further supported by the Raman spectroscopy measurements by reversing the polarity of the electric field. Such an optical probing of the electromechanical coupling may lead to diverse applications as a nonextensive technique for the electric field and (or) strain sensors in the nanoelectronic devices.

DATA AVAILABILITY
The data that support the findings of this study are available from the corresponding author on reasonable request to S.M. (email: sreemanta85@gmail.com) or A.S. (email: achintya@jcbose.ac.in).

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AUTHOR CONTRIBUTIONS

S.M. and A.S. designed and directed the project. S.S.S. prepared the sample. S.M. and S.S.S. performed the Raman spectroscopy experiments. S.M. analyzed the data and prepared the figures. D.S. performed the DFT calculations and analysis thereof with input from S.M., M.K. and A.S. S.D. performed the crystal structure analysis. B.S. performed the transmission electron microscopy on the sample. A.G. and M.K. contributed intellectually to the project. S.M. wrote the paper with input from all the authors. A.S. supervised the project. All the authors have gone through the paper and commented on it.

COMPETING INTERESTS

The authors declare no competing interests.

ADDITIONAL INFORMATION

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