

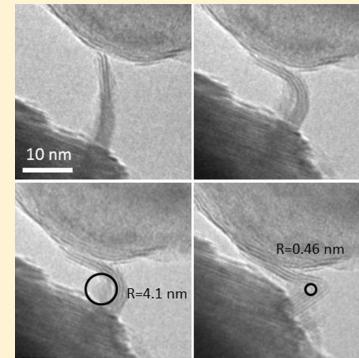
# Elasticity of MoS<sub>2</sub> Sheets by Mechanical Deformation Observed by in Situ Electron Microscopy

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## S Supporting Information

**ABSTRACT:** MoS<sub>2</sub> has been the focus of extensive research due to its potential applications. More recently, the mechanical properties of MoS<sub>2</sub> layers have raised interest due to applications in flexible electronics. In this article, we show in situ transmission electron microscopy (TEM) observation of the mechanical response of a few layers of MoS<sub>2</sub> to an external load. We used a scanning tunneling microscope (STM) tip mounted on a TEM stage to induce deformation on nanosheets of MoS<sub>2</sub> containing few layers. The results confirm the outstanding mechanical properties on the MoS<sub>2</sub>. The layers can be bent close to 180°. However, when the tip is retrieved the initial structure is recovered. Evidence indicates that there is a significant bond reconstruction during the bending with an outstanding capability to recover the initial bond structure. The results show that flexibility of three layers of MoS<sub>2</sub> remains the same as a single layer while increasing the bending modulus by 3 orders of magnitude. Our findings are consistent with theoretical calculations and confirm the great potential of MoS<sub>2</sub> for applications.



## INTRODUCTION

For more than 100 years transition metal sulfides (TMS) have been used as important catalysts in many petroleum-refining processes to remove sulfur-containing compounds. Probably the most important process is, for instance, hydrodesulphurization (HDS).<sup>1,2</sup> In recent years, a new wave of interest has arisen in the electronics field due to its transition from indirect band gap semiconductor ( $E_g = 1.29$  eV) to direct band gap semiconductor ( $E_g = 1.90$  eV), when the thin film is reduced to a monolayer, due to quantum confinement.<sup>3</sup> This transition makes monolayer MoS<sub>2</sub> a good candidate for electronic applications, such as transistors, photodetectors, and spintronics.<sup>4</sup> However, on the down side photoluminescence (PL) quantum efficiency of monolayer MoS<sub>2</sub> ( $\sim 4 \times 10^{-3}$ ) is low compared with other direct band gap semiconductors (typically >50%), e.g., GaAs. The reasons are not fully understood, although this is likely due to the high defect density on MoS<sub>2</sub>.<sup>5</sup> Further studies have revealed that transition metal dichalcogenides are highly flexible under tensile strain and could be used together with elastic-polymer substrates for development of flexible electronic devices.<sup>6</sup> Raman studies showed a red shift in the in-plane and out-of-plane modes with increased temperature,<sup>7</sup> as well as a broadening of the peaks by reducing the size of MoS<sub>2</sub> nanocrystals.<sup>8</sup>

There is a great similarity between carbon and MoS<sub>2</sub> and both can produce nanotubes, fullerenes, or single layers (in the case of MoS<sub>2</sub> we define single layer as the S-M-S unit) with properties that can be tuned with the diameter and with strain.<sup>9,10</sup> The mechanical properties of this material are very important for novel applications. There are several reports

regarding the mechanical properties of single layer MoS<sub>2</sub>, both experimentally and theoretically.<sup>9–14</sup> Most of them only considered the linear elastic properties. Nonlinear elastic behavior in MoS<sub>2</sub> monolayers was recently studied from first-principles calculations<sup>15</sup> finding good correlation between experimental and theoretical data.

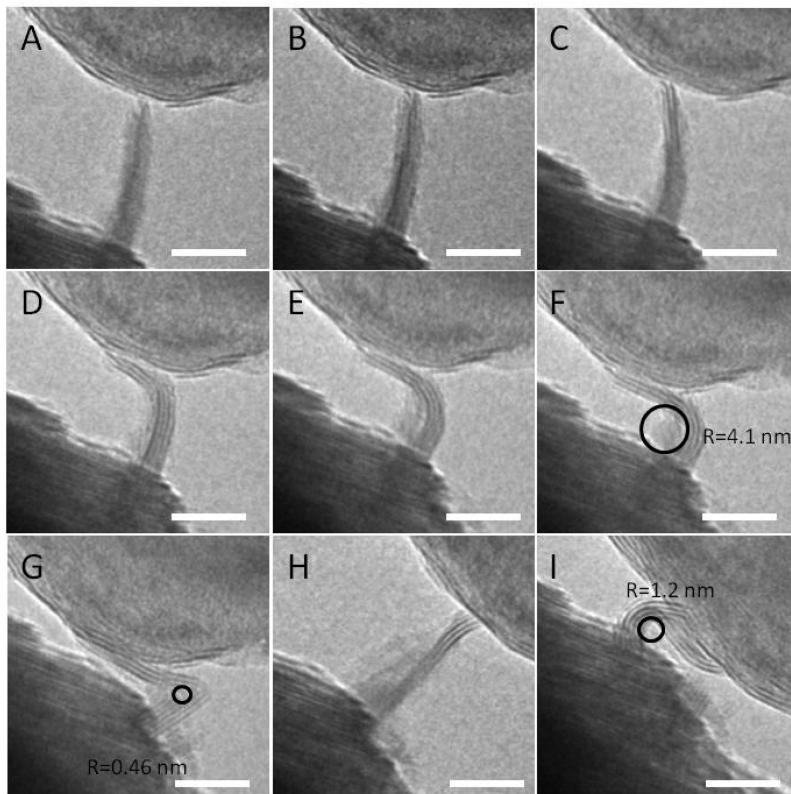
Experimental studies of MoS<sub>2</sub> monolayers are very limited. Bertolazzi et al.<sup>14</sup> performed experimental measurements on the stiffness and breaking strength of monolayer MoS<sub>2</sub> by using an atomic force microscope tip to deform the monolayer MoS<sub>2</sub> placed on a prepatterned SiO<sub>2</sub> substrate. They found that the effective Young's modulus and average breaking strength of monolayer MoS<sub>2</sub> are  $270 \pm 100$  and 23 GPa, respectively. In a different work, Castellanos Gomez et al.<sup>16</sup> in a similar experiment reported a mean Young's modulus of  $330 \pm 70$  GPa. These works clearly suggest that single layer MoS<sub>2</sub> is an ultrastrength material able to withstand large elastic deformation.

In the present work, we report direct observation of the mechanical response of MoS<sub>2</sub> layers to an applied by in situ HRTEM experiments. By using an in situ holder, we can observe the exact number of layers while measuring the deformation of the layers with sub-nanometer precision. Our results reveal that three layers of MoS<sub>2</sub> can be as flexible as single layers without sustaining plastic deformation, as suggested by the amount of stress applied to them. The

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**Figure 1.** Sequence of TEM images showing the flexibility of  $\text{MoS}_2$  sheets (see Supporting Information Video 1). Scale bars are 10 nm.

substantial increase in the bending modulus of three layers allows further engineering of flexible devices.

## ■ EXPERIMENTAL METHODS

**Experimental Setup.** TEM experiments were recorded in a JEOL 2010 equipped with a field emission gun operated at 200 kV. Minimum exposure was ensured to minimize beam damage to the  $\text{MoS}_2$  sheets.  $\text{MoS}_2$  were synthesized as described in ref 17, diluted in ethanol, and drop casted onto a 0.25 mm gold wire of 1 cm in length, which was then mounted on to a brass cap, to finally mount it in an AFM-TEM holder from Nanofactory AB as shown in Figure S1, Supporting Information. This holder features a Si cantilever with a sharp tip used to apply a load to the sample, while the process is recorded live in the microscope. The brass cap sits in a piezoelectric used to move the desired region close to the Si tip and perform the experiments. Micrographs were recorded in a bottom-mounted AMT CCD with an exposure time of 0.1 s. The load applied to the sample is estimated by measuring the deflection and multiplying it by the spring constant of the cantilever (2.3 N/m), then the stress can be computed by estimating the area of contact from the micrographs and dividing the applied load by the area of contact.

**Molecular Dynamics Simulations.** On the basis of the experimental evidence, we create a theoretical model to get a better insight about the underlying physics when a deformation is induced on the  $\text{MoS}_2$ . In this manner we can build a bridge between the experimental results with an *idealistic* model. Therefore, a set of molecular dynamics (MD) simulations of the deformation process on different  $\text{MoS}_2$  nanosheets was performed. In the case of ab initio or DFT based schemes, the study of these systems are limited by the size; therefore, the most accurate level of theory in this case is the use of a classical

approach. MD simulations can be very helpful in this respect because they are able to model discrete phenomena and systems that can be comparable to modern experimental sizes. The bend model is similar to the nanosheet with 460 Mo atoms and curved along the y axis. The width is 60 Å with a length of 66 Å (see Figure S2, Supporting Information). All the structures were relaxed prior to the simulation of the structural deformation. The tensile tests were performed using the LAMMPS code,<sup>18</sup> with the reaxFF<sup>19</sup> interatomic potential that describes bond formation and charge transfer between Mo and S atoms. An adiabatic process within the microcanonical ensemble (NVE) was chosen to equilibrate the structures during the deformation process (time step of 0.1 ps). In order to validate our method and therefore the potentials and parameters used in this work, several tests were performed obtaining similar results in comparison with previous theoretical and experimental work (see Figures S4 and S5, Supporting Information). For instance we obtained the lattice values of  $a = 3.182 \text{ \AA}$  and  $c = 12.380 \text{ \AA}$  from a single cell unit of  $\text{MoS}_2$ . Those values are in a good agreement with the work performed by Kaplan-Ashiri et al.<sup>20</sup> and Young.<sup>21</sup>

## ■ RESULTS AND DISCUSSION

Our experimental set up started with thin nanosheets of  $\text{MoS}_2$  containing a few layers, from 2 to 5. The sheets were prepared by methods described before.<sup>17</sup> We look for sheets in a vertical position, and then we proceed to apply a load with a Si tip to bend the sheets. As Figure 1 shows, the three layer sheet is bent from side to side without breaking. In Figure 1B, a 2 GPa stress (using a nominal thickness of 0.65 nm per layer) is applied parallel to the sheets without causing any significant bending, until they start to bend, changing the angle of the applied load (Figure 1C) resulting in further bending of the sheets. In Figure

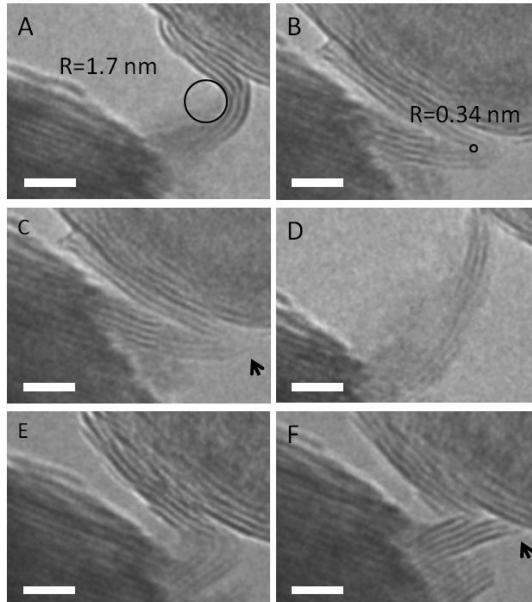
1D the stress immediately falls down due to the increase in contact area. Increasing the applied load leads to further bending reaching curvature radius of 4 nm and down to 0.5 nm (Figure 1F,G) without breaking bonds in the sheet. By moving the MoS<sub>2</sub> sheets to the left, the sheets roll back to a straight position, and at this state, the applied load was close to 8.31 GPa, which did not cause plastic deformation to the sheets (Figure 1H). This amount of stress is close to the previously found theoretical (and practical) elastic limit,<sup>14,16,22</sup> which implies that no defects (e.g., dislocations) were introduced to the sheets during the extreme bending conditions they were subjected to moments before. Moreover, as we keep moving the sheets to the left, it bends down to form a radius of just 1.2 nm in the opposite direction than before (Figure 1I) (see Supporting Information Video 1).

The curvature radius in Figure 1G confirms previous experimental observations of MoS<sub>2</sub> synthesized nanotubes with a diameter close to 0.9 nm.<sup>23</sup> The work of Remskar et al. did not provide a full stoichiometry for the nanotubes, but on the basis of this observation, the well-known 2H structure is a viable option to form this type of nanotubes. This proves the extreme flexibility that MoS<sub>2</sub> sheets possess and that prove extremely valuable in flexible electronics. Even though actual devices have already been built,<sup>24–26</sup> the limits in flexibility of MoS<sub>2</sub> sheets had not been examined before, let alone down to the atomic level.

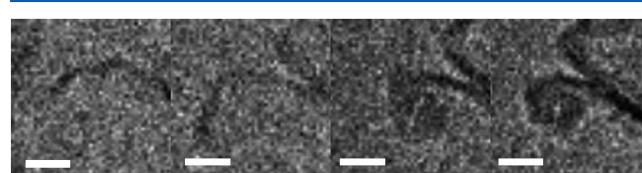
The same process was repeated several times by applying normal forces in the direction of the tip in the same sheets as is shown in Figure 2. Figure 2A shows the nanosheets being bent

bonded to the MoS<sub>2</sub> layer on the Si tip; however, upon loading release, the sheets reconstructed the bonds with the original sheets returning to the original configuration (Figure 2D). After the repeated bending, the sheets sustained a tensile stress of 5 GPa (Figure S3, Supporting Information). This suggests that any defects formed during the bending process disappear after the stress is removed. Finally, a shear stress was applied in order to finally break the sheets as can be seen in Figure 2E,F with a normal stress of 0.3 GPa (see Supporting Information Video 2). The fact that the sheets can be bent repeatedly without introducing defects in the structure proves that MoS<sub>2</sub> sheets can be readily used for flexible electronic devices, as the performance will not significantly change with elastic deformation. Performance of flexible transistors has already been tested at limited bending states,<sup>26</sup> but this work suggests that these devices may work at more extreme bending angles. More importantly, even if bond breakage occurs in the sheet, they may bond again upon releasing the applied load and allowing the sheets to return to a more relaxed state.

Interestingly, we observed the formation of a MoS<sub>2</sub> nanoscroll during our experiments. This morphology is of potential interest since some studies have shown potential applications for graphene nanoscrolls;<sup>27,28</sup> however, up to our knowledge; there has not been a report of MoS<sub>2</sub> forming such nanoscrolls. Because of the different nature of the MoS<sub>2</sub> sheets, it may be harder to form a MoS<sub>2</sub> scroll than a graphene scroll, but this work provides proof that it can be formed as shown in Figure 3. This scroll suggests that it might be possible to synthesize scroll-type structures different from the inorganic fullerenes.



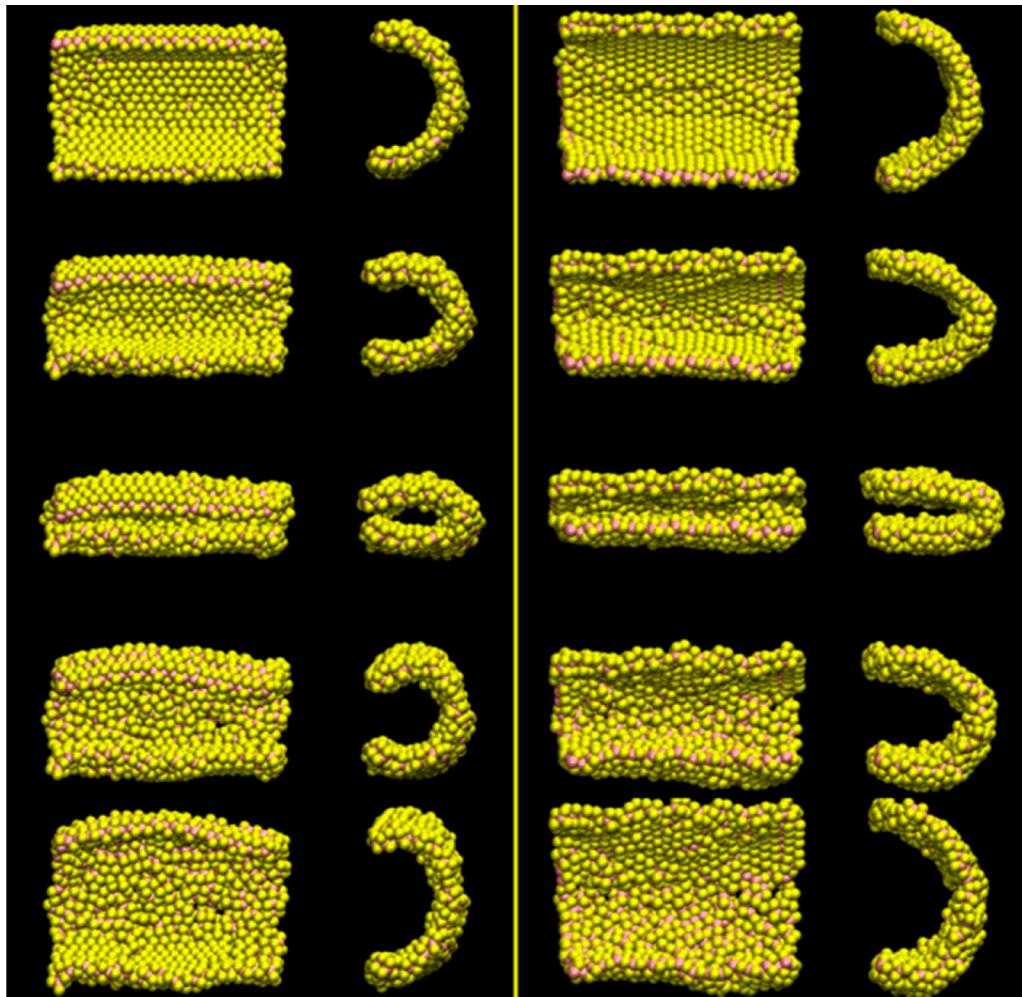
**Figure 2.** Sequence of TEM images showing the maximum bending limits of MoS<sub>2</sub> sheets (see Supporting Information Video 2). Scale bars are 5 nm.



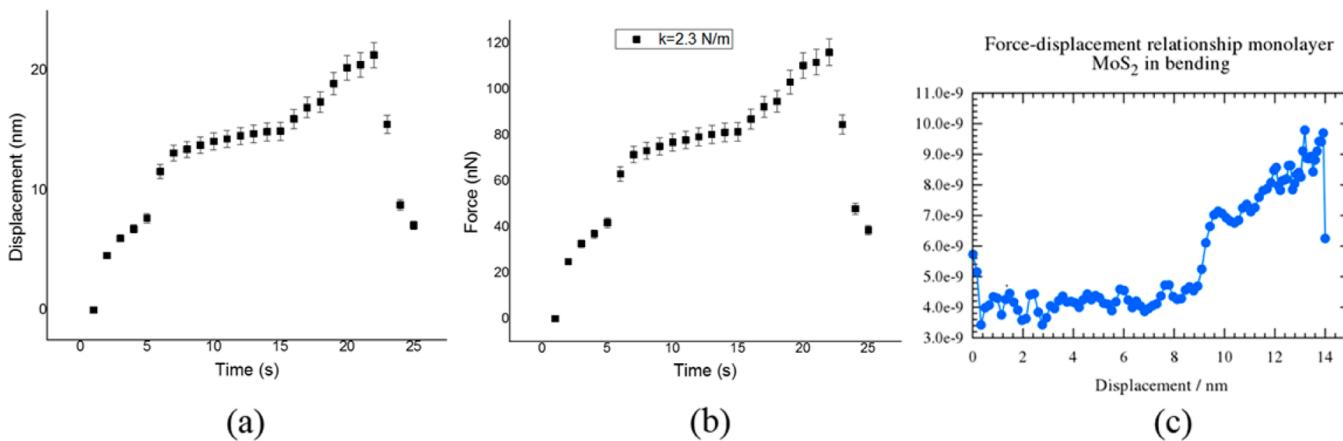
**Figure 3.** TEM sequence of the formation of a MoS<sub>2</sub> scroll. Scale bars are 2 nm.

As it is expected for any material subject to a bending load, the thicker the material is, the harder it is to bend, usually reducing the flexibility of the material. Bending modulus can be estimated by using the well-known formula  $D = Eh^3/12(1 - \nu^2)$  where  $E$  is the young modulus of the material,  $h$  is the thickness, and  $\nu$  is the Poisson's ratio. While for very thin materials the modified formula  $D = E^{2D}h^2/12(1 - \nu^2)$ , where  $E^{2D}$  is the two-dimensional stiffness, should be used to consider the finite thickness of the materials; however, in the case of MoS<sub>2</sub> layer, the general formula gives a good approximation of the bending modulus. Bertolazzi et al. calculated experimentally the  $E^{2D}$  for one and two layers to be in the range of 120 and 240 N m<sup>-1</sup>, and 190 and 330 N m<sup>-1</sup>.<sup>14</sup> Using the modified formula for thin materials, we can estimate the bending modulus for one and two MoS<sub>2</sub> layers. For one layer, the bending modulus falls in the range of 6.62 and 13.24 eV, while for two layers in the range of 183 to 319 eV. Now, using the general formula for bulk materials, the bending modulus of one and two layers is 4.1 eV (with a thickness of 0.312 nm) and 297 eV (with a thickness of 1.3 nm), respectively, which are still in the same order of magnitude as the values computed with the formula for thin materials. Therefore, it is a fair approximation

down to 90°, forming a curvature radius of 1.7 nm without breaking. We pushed the limits of the nanosheets by bending the sheets down to a radius of curvature of 0.34 nm as seen in Figure 2B, and the three layers seem to be intact. This exceeds the value of the smallest MoS<sub>2</sub> nanotube diameter of 0.961 nm.<sup>23</sup> We did observe bond breaking of the sheets moments afterward (Figure 2C, marked with an arrow) as the top layer



**Figure 4.** Deformation process of both bent MoS<sub>2</sub> sheets. In the left panel, the process for the armchair sheet is shown. In the right panel the deformation for the zigzag sheet can be seen.



**Figure 5.** Force and displacement forces have been plotted with the experimental data: (a) displacement vs time and (b) force vs time. (c) Total force versus displacement for a monolayer of MoS<sub>2</sub> during the bending process. The linearity between the force and displacement is “broken” for a critical displacement value. This suggests that the stiffness is not linear.

to use the bulk formula and estimate the bending modulus of three layers of MoS<sub>2</sub>, which is the case in our experiment. Using a thickness of 1.95 nm, we get a bending modulus of 1002 eV. This is 3 orders of magnitude higher than that of a single layer, which implies that three layers are stiffer than a single layer with the same flexibility. This means that real devices can be

engineered with different stiffness while retaining their flexural nature.

In order for the outer layer in the bent sheets to sustain a similar bending state as the inner one there must be sliding between layers so they do not exceed the elastic limit and break. While bonding rotation and elastic strain would also play a role,

we consider that sliding is the one accommodating such a deformed state without breaking, which is consistent with previous results.<sup>29</sup>

Different atomistic models of MoS<sub>2</sub> were deformed using classical MD approach focusing on the behavior of the nanosheets during the bending. The models were built up based on the experimental evidence. Those models exhibit armchair and zigzag edges. The bent sheets were built from a rectangular MoS<sub>2</sub> slab and given a "C" shape starting configuration to facilitate the simulations (Figure S2, Supporting Information). They experience a perpendicular mechanical stress in both armchair and zigzag structures. The deformation was applied along the *y* axis, stopping until the sheets achieve a maximum deformation, i.e., when the "C" shape of each sheet is totally closed. After this condition is reached, the stress is applied in opposite direction until the structures obtain their initial shape as can be observed in Figure 4. During this process, the atomic structure undergoes small rearrangements. To quantify this, we calculated the radial distribution function (RDF) for each case. In Figure S6, Supporting Information, it can be observed that the first and second neighbor distances are kept. The resulting structure can be clearly associated with a crystalline structure rather than an amorphous one. The RDF relevant feature is the appearance of an extra peak among the crystalline first and second neighbors, at 4.65 Å. This peak indicates the transition, in some regions, from a molybdenum ideal planar lattice into a nonplanar one, i.e., lattice distortions appear due to mechanical deformation. No significant difference is observed between armchair and zigzag configuration.

In addition to the RDF, we also calculated and plotted the total force versus the displacement during the simulation (Figure 5). For each simulation the applied force increased linearly with displacement until a critical point at which the maximum in the curvature occurs, resulting in a significant increase in force. This suggests that the stiffness is not constant and may depend on the number of layers. The experimental data has been now plotted in Figure 5a,b, as well as the simulation obtained with our model. The range from the initial conditions up to about 9 nm in the simulations agrees with the regions in which the displacement is shown in the experimental graphs. After 10 nm the curve has a different slope in agreement with the experiment. Experimental graphs of displacement versus time and force versus time have been plotted separately since the representation of them using elastic conditions will show linear tend and will not represent the behavior of the experiment and simulations.

We consider only one MoS<sub>2</sub> layer in the simulations because we assume that, during multilayer bending, sliding of the outer layers with respect to the inner layers occur, accommodating the elastic deformation as discussed before, while the overall appearance does not change significantly, as seen by the RDF (Figure S6, Supporting Information). As expected, the force needed for bending the sheets is not homogeneous across the different orientations. For the armchair system, the sheet bends on a (10̄10) plane, whereas for the zigzag system, the sheet bends on a (11̄20) plane. Since the density of these two different planes is different, the overall deformation process will depend strongly on the plane geometry, which is being bent; nonetheless, both showed the same range of bending without any plastic deformation.

It is important to point out that even though the atomistic models are not the same as the experimental one, we found a

similar behavior when a deformation is induced. For instance, we were able to observe in both, experiment and simulation, how the MoS<sub>2</sub> layers can be as flexible as single layers without sustaining plastic deformation. That means we are obtaining similar results with two different approaches.

## CONCLUSIONS

We have performed *in situ* TEM deformation experiments in thin MoS<sub>2</sub> sheets. The sheets proved to be very flexible while remaining as strong as pristine ones, which further confirms the potential to use this material in flexible devices.

Defects in the MoS<sub>2</sub> lattice would decrease the maximum stress applied to the sheets (but no plastic deformation is expected); however, the high stresses observed here suggest there are no significant number of defects present in the deformed sheets. Despite the bending modulus being 3 orders of magnitude higher than that of a single layer, the flexibility of the three layered MoS<sub>2</sub> remain the same as a single layer sheet. It may be said that the addition of layers increases the stiffness of the material without losing flexibility, which could prove useful when engineering new devices. MD simulations corroborated the experimental observations. It is observed that during the bending processes a significant bond reconstruction is produced. A remarkable fact is that this process is reversible. These results are in agreement with the results by Tang et al., which observed that for less than 23 layers the bending of MoS<sub>2</sub> is smooth without kinks.<sup>29</sup>

A difference in bending zigzag and armchair sheets was observed; however, none of the sheets showed Mo–Mo bond breaking. A more detailed study may be necessary to elucidate the nature of the different bending conditions for zigzag and armchair sheets and their mechanical response. The observed super elasticity of MoS<sub>2</sub> sheets is a step forward in finding new applications of this material. It, however, should be noted that there are practical limitations on the state of the art capabilities of the mechanical deformation stage that make a fully quantitative study possible. Nevertheless, this study shows the remarkable mechanical behavior of layered materials such as MoS<sub>2</sub>.

## ASSOCIATED CONTENT

### Supporting Information

Supplemental figures and videos. This material is available free of charge via the Internet at <http://pubs.acs.org>.

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### Notes

The authors declare no competing financial interest.

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