Oxidation dynamics of ultrathin GaSe probed through Raman spectroscopy

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Gallium selenide (GaSe) is a 2D material with a thickness-dependent gap, strong non-linear optical coefficients and uncommon interband optical selection rules, making it interesting for optoelectronic and spintronic applications. In this work, we monitor the oxidation dynamics of GaSe with thicknesses ranging from 10 to 200 nm using Raman spectroscopy. In ambient temperature and humidity conditions, the intensity of all Raman modes and the luminescence decrease rapidly with moderate exposure to above-gap illumination. Concurrently, several oxidation products appear in the Raman spectra: Ga$_2$Se$_3$, Ga$_2$O$_3$ and amorphous and crystalline selenium. We find that no safe measurement power exists for optical measurements on ultrathin GaSe in ambient conditions. We demonstrate that the simultaneous presence of oxygen, humidity, and above-gap illumination is required to activate this photo-oxidation process, which is attributed to the transfer of photo-generated charge carriers towards aqueous oxygen at the sample surface, generating highly reactive superoxide anions that rapidly degrade the sample and quench the optical response of the material.

Gallium selenide (GaSe) is a layered pseudo-direct gap semiconductor with strong non-linear properties in the visible, IR and THz ranges, a high photore- sponse from 2 to 5 eV, a highly anisotropic band structure exhibiting uncommon selection rules, and interesting spin physics. Atomically thin GaSe can be mechanically exfoliated from bulk samples or grown by chemical vapor transport. Its bulk band gap of 2.12 eV is predicted to increase to more than 3 eV for the monolayer. Atomically thin flakes and devices have shown a high photoresponse, a composition tunable band gap, near-unity optical polarization, the strongest second-harmonic generation observed in a monolayer 2D material, and transistors with high ON/OFF ratios.

Like most 2D materials, the properties of GaSe are expected to sensitively depend on the interaction of its surface with its chemical environment. In bulk form, GaSe is generally considered as a stable material and is known to have a high laser damage threshold suitable for non-linear optics applications. Nonetheless, bulk GaSe is also known to naturally form a native oxide and thermally- and photo-induced oxidation has been reported. Recently, exposure to intense laser light was found to degrade optical properties and lead to chemical transformations. Given the interest for few-layer GaSe for electronic and optoelectronic applications, it appears critical to examine the oxidation dynamics of ultrathin GaSe and determine the conditions in which this oxidation can be suppressed or at least minimized.

In this paper, we monitor the complex oxidation dynamics of ultrathin GaSe and identify the emergence of several oxidation products using Raman spectroscopy. This oxidation, highly detrimental to all optical and electronic processes, requires the simultaneous presence of oxygen, humidity, and above-gap illumination and is adequately described in the framework of the Marcus-Gerischer theory. We demonstrate that removing any of these three components greatly reduces the oxidation rate.

GaSe layers were mechanically exfoliated from Bridgeman-grown bulk crystals using PDMS stamps onto silicon (Si) substrates covered with a 100 nm thermal oxide (SiO$_2$). Flakes with thicknesses ranging from 10 to 200 nm were identified by optical contrast using an optical microscope and their thickness was established by spectral resolution and at 246 cm$^{-1}$.

The spatial and spectral resolution were 0.6 μm and 0.2 cm$^{-1}$, respectively. For sample in vacuum experiments, samples were exfoliated in a dry N$_2$-flushed environment and transferred to an optical cell evacuated to a pressure of 1.5 × 10$^{-6}$ mTorr. Deoxygenated water vapor was obtained from deionized water subjected to several freeze-thaw cycles under vacuum. Experiments in oxygen were done using 99.999% O$_2$ gas (<3 ppm H$_2$O) at a partial pressure of 250 Torr. Finally, measurements in ambient air were done at a temperature of 23 °C and under a 48% relative humidity.

Raman spectra are used to monitor the integrity of thin GaSe layers and the appearance of photo-induced oxidation products. In this first experiment, samples were exposed to 6 mW μm$^{-2}$ of radiation at 532 nm, which corresponds to about 6% of the established laser damage threshold for nanosecond pulses at 620 nm. This illumination is about 230 meV above the bandgap of GaSe. Fig. presents the evolution of the Raman spectrum as a function of exposure time for a 45 nm thick GaSe sample in air. Freshly exfoliated GaSe measured in vacuum (pristine GaSe) presents the usual dominant Raman modes expected from bulk GaSe: $A_1^\text{g}$ at 134 cm$^{-1}$, $E_1^\text{g}(TO)$ at 214 cm$^{-1}$, and $A_1^\text{g}$ at 308 cm$^{-1}$. Two other vibrational modes located at 236 cm$^{-1}$ and at 246 cm$^{-1}$ were often observed from pristine GaSe. These Raman
modes are also associated to bulk GaSe, but they will not be considered in this work due to their much weaker intensity compared to $A_1$, $E'_1(TO)$ and $A_1'$. The Raman spectrum evolves significantly as a function of exposure time.

The Raman spectrum of a 45 nm-thick GaSe sample in air as a function of exposure time to a 6 mW $\mu$m$^{-2}$ radiation at 532 nm. Pristine refers to a second sample measured in vacuum immediately after exfoliation; the topmost curve is a spectrum obtained after 60 min of 6 mW $\mu$m$^{-2}$ exposure in vacuum of the same sample. In the experiment, the temperature increase induced by the exposure light is estimated at less than 40 K.

The Raman spectrum of $\alpha$ GaSe rapidly decreases and several additional Raman modes appear at frequencies of 155 cm$^{-1}$, 257 cm$^{-1}$ and 282 cm$^{-1}$. All GaSe vibrational modes are already accounted for and none of these additional features can be related to any of the various known GaSe polytypes. They are attributed to a photo-induced chemical transformation of GaSe and corresponds to well-known thermal oxidation products: Ga$_2$Se$_3$, a-Se, $\beta$-Ga$_2$O$_3$ (see §SI in Supplementary Material for a description of the three main thermal oxidation pathways and their activation temperatures).

The frequency and width of the first mode at 155 cm$^{-1}$ corresponds to the vibrational mode $A_1$ of Ga$_2$Se$_3$. Although Ga$_2$Se$_3$ exhibits several Raman allowed modes, the one at 155 cm$^{-1}$ is the narrowest and most intense and thus the most likely to be observed. Ga$_2$Se$_3$ has a defect zinc-blende structure in which Ga vacancies may or may not be ordered. The polarization-resolved Raman measurements presented in Fig. S1(a) in Supplementary Material suggest that the photo-induced $\alpha$-Ga$_2$Se$_3$ phase is polycrystalline. Another feature related to Ga$_2$Se$_3$ is observed at 292 cm$^{-1}$ after a 10 min photoexposure. This broad ($\sim$10 cm$^{-1}$) feature actually corresponds to a group of at least three vibrational modes with various symmetry representations ($A_1$, $B_1$, $B_2$) that have been assigned to localized vibrational modes of vacancy-disordered Ga$_2$Se$_3$. For its connection with multiple vibrational modes, this feature is labeled Ga$_2$Se$_3$ (M). Its presence suggests a high level of disorder in the Ga$_2$Se$_3$ phase.

After exposure of 20 min and more, the Raman signal is dominated by two even broader features. The first at 130 cm$^{-1}$ likely corresponds to SeO$_2$ or SeO$_3$ Raman modes, but a quantitative analysis of this feature has proven difficult; it will not be further discussed. The other feature at 257 cm$^{-1}$ matches the characteristics of a mode associated to selenium and its width ($\sim$20 cm$^{-1}$) suggests an amorphous phase (a-Se). The narrower peak ($\sim$5 cm$^{-1}$) at 241 cm$^{-1}$ appears last and is associated to a selenium crystalline phase (c-Se). As it has been often demonstrated, a-Se easily photocrysallizes.

The feature observed at 161 cm$^{-1}$ does not correspond to any modes belonging to GaSe, $\alpha$-Ga$_2$Se$_3$, or Se and its oxides. However, its frequency and width match the characteristics of the $A_2$ vibrational mode of $\beta$-Ga$_2$O$_3$. According to Ref. 15, this feature should be the second most intense Raman feature from $\beta$-Ga$_2$O$_3$; the most intense one, found in single crystals at 199 cm$^{-1}$, has not been unambiguously observed in this work. Polarization-resolved data is presented in Fig. S1(b) in the Supporting Information.

FIG. 1. Evolution of the Raman spectrum of a 45 nm-thick GaSe sample in air as a function of exposure time to a 6 mW $\mu$m$^{-2}$ radiation at 532 nm. Pristine refers to a second sample measured in vacuum immediately after exfoliation; the topmost curve is a spectrum obtained after 60 min of 6 mW $\mu$m$^{-2}$ exposure in vacuum of the same sample. In the experiment, the temperature increase induced by the exposure light is estimated at less than 40 K.
FIG. 2. Integrated Raman intensity and luminescence as a function of exposure time of a 45 nm thick GaSe flake in air. (a) Relative intensity of GaSe Raman modes and photoluminescence. (b) Integrated intensity of Raman modes associated to the oxidation products and Silicon. (c) AFM surface profile (color scale in nm) of the laser damage on a 58 nm thick flake after 30 min of 6 mW laser exposure. A black arrow indicates the ∼12 nm deep, sharply defined hole caused by the laser induced oxidation.

mechanism due to the low temperatures involved. As detailed in §II of the supplementary material, the temperature increase occurring in the experiment presented in Figs 1 and 2 is less than 40 K.

The Raman intensity is proportional to the quantity of material in the probed volume, but it is also sensitive to its structural quality. It is therefore difficult to establish the concentrations of crystalline oxidation products, as, for example, the decrease of both Ga₂Se₃ modes could be related to its oxidation into Ga₂O₃ (see §SI of the Supplementary Material) or to the degradation of its crystalline structure as the oxidation progresses. During oxide formation, excess Se segregates at the surface. As seen from Fig. 2(b), its intensity quickly increases at short exposure times and mirrors the drop of intensity of GaSe Raman modes and luminescence signal. Because its response is much less sensitive to its structural quality, a-Se is believed to be the most reliable Raman marker of oxidation amongst the various products (see also §V in Supplementary Material).

The Raman signal from the Si substrate located underneath the GaSe flake steadily increases with time even though the excitation conditions are rigorously controlled. This is explained by the generation of a large-gap oxide and localized ablation. Oxidation leads to the formation of β-Ga₂O₃ with a gap of 4.9 eV, which is transparent at the excitation energy of 2.33 eV. Also, post-exposure AFM measurements indicate a layer-by-layer thinning induced by the laser exposure (see Fig. 2(c)). This is explained by the high volatility of Se and the high atomic mobility of the remaining metallic Ga. As a net result, the absorption from the GaSe flake decreases.

As suggested by the data presented in Fig. 1 (topmost curve), placing the sample in a vacuum environment protects GaSe samples from photo-induced oxidation. Curve A presented in Fig. 3 shows the evolution of the A₁ peak Raman intensity of a 10 nm thick flake as a function of time for an excitation power of 250 µW. For such thin layers, this power yields a weak but measurable Raman signal. The intensity of GaSe Raman modes is only marginally affected after an exposure of more than 60 min in vacuum. At the time indicated by the vertical dashed line, the sample was exposed to ambient air. After a few minutes, the Raman intensity has dropped by two orders of magnitude and the signal is completely lost after 10 min. This clearly shows that thin GaSe flakes are unstable in ambient conditions and that minimal laser illumination is sufficient to induce significant degradation, establishing that no safe above-gap excitation power exists for atomically-thin samples.

FIG. 3. Integrated Raman intensity of the A₁ peak of GaSe flakes as a function of exposure time to a 532 nm laser in vacuum and in different environments. Flake thickness and exposure power are indicated on the curves.

Curve B presented in Fig. 3 shows the Raman intensity of A₁ of a 150 nm flake exposed to 1000 µW of laser intensity. For the first data point, the sample is in vacuum. The sample is then exposed to ambient air without any illumination. After 12 hours, the sample is again measured under vacuum conditions and the Raman intensity is very similar to that initially measured, indicating that exposure to oxygen and water vapor alone does not induce significant chemical changes. After 60 min of laser illumination in vacuum, no appreciable change in intensity is
observed, even though residual H₂O is present in the sample chamber and on the sample. Then, dry oxygen (<3 ppm H₂O) is introduced in the optical cell and the sample is exposed to radiation for another 60 min. A slight change in Raman intensity is observed, but the overall signal loss is minor because of the limited amount of H₂O available at the sample. Finally, the sample is exposed to ambient air and continually illuminated for another 60 min. It takes about 15 min for the intensity to change significantly. This delay is attributed to a relatively slow nucleation and initial growth of degradation by-products for thicker samples. From that point on however, a rapid decline of intensity takes place, losing more than one order of magnitude within less than one hour. Curve C in Fig. 3 shows the Raman intensity from a 120 nm flake measured in vacuum and then in deoxygenated water vapor. The Raman intensity does not show any appreciable change after 1 hour in each environment. Introduction of oxygen in the chamber at the 120 min mark leads to a rapid decrease in the Raman intensity. These experiments establish that continuous above-gap illumination, water vapor and oxygen alone do not support the photo-oxidation process, but that the simultaneous presence of all three is required.

Although the observed oxidation byproducts are those observed from thermally-activated oxidation (Eqs. (S1a)-(S1c) in the Supplementary Material), the photo-oxidation process observed in this work is not temperature driven. The Raman shifts observed correspond to a temperature variation of at most 43 K at the studied exposure powers (see §SII in the Supplementary Material for the detailed analysis). This result confirms that the above gap illumination is not contributing significant heat, but rather generating an carrier density δη in the conduction band.

The results presented thus far suggest that the photo-oxidation involves these three steps: optical excitation of carriers, electron transfer towards aqueous (aq) oxygen molecules at the surface of the sample, and spontaneous reaction between aqueous oxygen superoxide anions (O₂aq) with the p-doped semiconductor. This process can be modeled using the following equations:

\[ \text{GaSe} + h\nu \rightarrow \text{GaSe}^* \]  
(1a)

\[ \text{GaSe}^* + O_{2(aq)} \rightarrow O_{2(aq)}^- + \text{GaSe} + h^+ \]  
(1b)

\[ O_{2(aq)}^- + \text{GaSe} + h^+ \rightarrow \{\text{GaSe}\}_\text{ox}, \]  
(1c)

where GaSe* represents GaSe with photoexcited electron hole pairs, and \{GaSe\}_ox represents GaSe oxidation byproducts. Although the photo-driven dynamics are expected to be quite different from the thermal dynamics discussed in Ref. 46, our results confirm that the oxidation byproducts are similar.

The charge transfer between the photo-excited carriers and aqueous oxygen (Eq. 1b) can be understood in the framework of the Marcus-Gerischer theory, where the rate of charge transfer is described by:

\[ \frac{d[O_2^-]}{dt} \propto \delta\eta[O_2] \exp \left[ -\frac{\chi \text{GaSe} - E_{F,\text{redox}}^0 - \lambda}{4k_B T} \right]^2, \]  
(2)

where δη is the conduction band electron density, [O₂] and [O₂⁻] are the concentrations of neutral oxygen and superoxide in the water at the sample surface, χGaSe is the GaSe electron affinity, \( E_{F,\text{redox}}^0 \) is the Fermi level of the oxygen-water solution and λ is the solvent reorganization energy around oxygen molecules. Since GaSe has an electron affinity which positions the lower conduction band in line with the aqueous oxygen acceptor state, the exponential function in Eq. 2 enables charge transfer towards the aqueous O₂ (see §SVI in the Supplementary Material). The generated aqueous superoxide anions (O₂aq) at the surface then rapidly react with the p-doped GaSe (Eq. 1c) to produce the observed oxidation byproducts, which will in turn decrease the observed GaSe Raman intensity.

In the thickness regime studied here, the band gap variation is negligible 15,16 and the absorption coefficient is independent of thickness. The rate at which superoxide anions are generated is thus proportional to the exposure power, \( d[O_2^-]/dt \propto \delta\eta \propto P_{\text{exp}} \). Fig. 4 shows the intensity of \( A_1^\text{G} \) obtained after an exposure of 15 min at the indicated exposure power in ambient air for samples of varying thicknesses. As predicted by Eq. 2, the observed degradation rate increases linearly with exposure power and is independent of sample thickness, as evidenced from the very similar slopes measured. This confirms the role of the linear dependence of the photooxidation mechanism on above gap illumination and further rules out thermal processes.

![FIG. 4. Integrated Raman intensity of \( A_1^\text{G} \) as a function of exposure power for samples of varying thicknesses. Each data point represents a 15 min exposure at the specified power before a Raman spectrum was measured using a 500 µW laser excitation. Linear regression slopes are shown.](image)

In summary, GaSe exhibits a relatively complex oxidation dynamics compared to other 2D materials. It
involves several components: oxygen, humidity, and above-gap illumination, and results in the generation of several oxidation products, Ga$_2$Se$_3$, Ga$_2$O$_3$ and amorphous and crystalline selenium. Luminescence and Raman measurements in ambient conditions are found to severely degrade the optical and electronic characteristics of GaSe, indicating that no safe illumination threshold exists, especially for thin layers of GaSe. Hence, the study of the intrinsic characteristics of 2D GaSe requires a controlled environment at all stages of processing and characterization.

See supplementary material for more detail on the thermal oxidation process and thermal effects, for further analysis of the polarized Raman spectra of the oxide products and of sample photoluminescence, and for additional information regarding the role of water vapor and oxygen in the oxidation process.

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