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First attempts of WSe₂ growth on GaP(111)B substrates by MBE

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Abstract — Few layers WSe₂ are grown on GaP(111)B surfaces passivated by selenium. The layers remains 2D at low growth temperature ($T_g = 200-250^\circ\text{C}$) while exhibiting the formation of the metastable 1T' phase. Medium growth temperatures (400-550°C) lead to a poor crystalline surface quality. In both cases, annealing over 600°C allows recovering the stable 1H/2H phase and a 2D morphology. At high temperatures (650-700°C), the formation of WSe₂ is accompanied by excess metallic W deposition. Raman features are only observed on the 1H/2H phase for which the A₁' excitation line is detected for a layer thickness above 2 MLs.

Keywords—transition metal dichalcogenides; WSe₂; GaP(111)B substrates

Over the past few years, 2D-Transition Metal Dichalcogenides (TMDs) have revealed great potential for optoelectronics and nanoelectronics devices, thanks to their electronic and optical properties, not encountered in other materials. They can be grown by van der Waals epitaxy allowing the use of materials with significant mismatch. Van der Waals epitaxy is usually performed on 2D substrates such as graphene, hBN or mica but 3D substrates can also be used after a proper surface passivation treatment, which for III-V semiconductors, has been applied successfully to GaAs(111) surfaces¹. Since the crystalline quality of the TMD layers improves with increasing growth temperature², GaP might represent an interesting alternative to GaAs considering its higher thermal stability. In this study, we present results relative to the growth of WSe₂ on Se terminated GaP(111)B surfaces.

Few layers (1-3) WSe₂ are grown in a Vinci technologies MBE reactor equipped with a Telemark linear electron gun evaporator as W source and a Riber VCOR valve cracker cell for Se. The substrate temperature is varied from 200°C to 700°C using very low W rate between 1 and 2 10^{-3} Å/s and under a Se beam equivalent pressure in the 10^{-6} Torr range. The growing surface is monitored by RHEED whereas after annealing, the layers are characterized *in-situ* by X-ray and UV photoemission (XPS/UPS) and *ex-situ* by atomic force microscopy (AFM) and Raman spectroscopy.

At low growth temperatures ($T_g = 200-250^\circ\text{C}$), starting from the 1x1 reconstruction characteristic of the GaP(111)B-Se surface, extra streaks appear in the RHEED pattern in-between the main ones while the 2D character is preserved (figure 1). These extra streaks are assigned to the formation of the metastable 1T' phase³ whose metallic character is further confirmed by UPS measurements (figure 2). They progressively vanish upon annealing and disappear at temperature higher than 600°C under a Se flux. For medium T_g values (400-550°C), the extra streaks are hardly distinguishable but the pattern exhibits numerous extra spots and even turn to a polycrystalline one when the thickness reaches 3 MLs. Annealing up to 650°C allows recovering a 2D pattern. Such layers exhibit a semiconducting character as expected for the 1H/2H stable phase (figure 2). Finally, for high temperatures (650°C-700°C), the pattern rapidly turns to a low intensity polycrystalline one. In this case, XPS clearly shows the presence of a large amount of metallic W on the surface, revealing an insufficient Se/W flux ratio (figure 3). For $T_g \leq 550^\circ\text{C}$, after annealing to 650°C, the mean surface roughness only slightly increases with respect to the starting surface with a morphology characterized by small grains with typical dimensions in the 20-50 nm range (figure 4). Raman peaks characteristic of WSe₂ have never been detected on the 1T' phase, nor on layers thinner than 2MLs. On the contrary, for 3ML thick 2H phase layer, the Raman A₁' mode is clearly observed (figure 5).

¹ A. Ohtake et al., Cryst. Growth Des. 17, 363 (2017)

² C. Vergnaud et al., Nanotechnology 31, 255602 (2020)

³ M.M. Ugeda et al., Nature Communications 9, 3401 (2018)

Transmission Electron Microscopy observations are currently under progress and will be available at the conference.



Figure 1: RHEED pattern observed on 3 MLs WSe₂ deposited on GaP(111)B at 250°C.

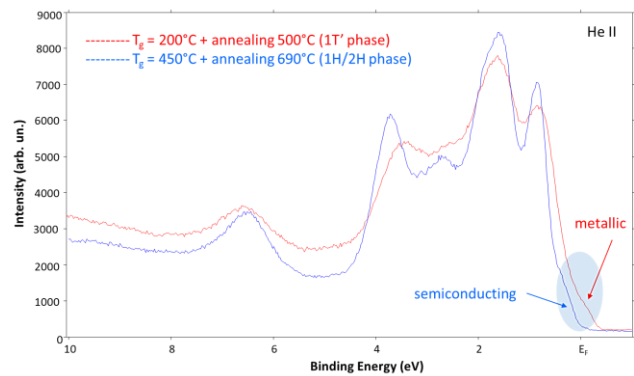


Figure 2: UPS spectra recorded on 3 MLs WSe₂ grown on GaP(111)B respectively at 200°C and annealed at 500°C (red curve) or at 450°C annealed at 650°C (blue curve) exhibiting the metallic character of the layers grown at low temperature.

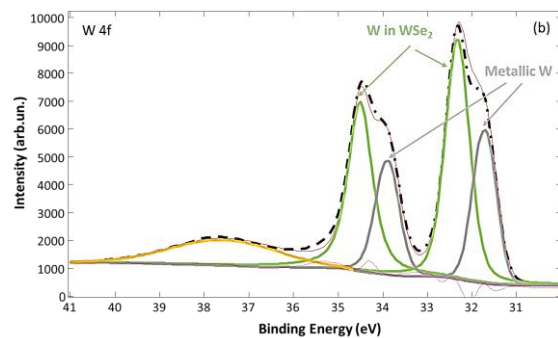
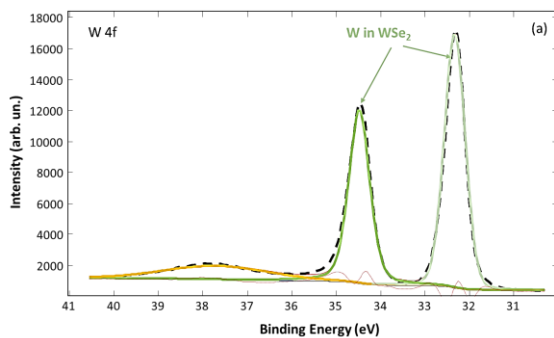


Figure 3: W 4f XPS spectra recorded on 3 MLs thick WSe₂ layers grown at 450°C (a) or 650°C (b).

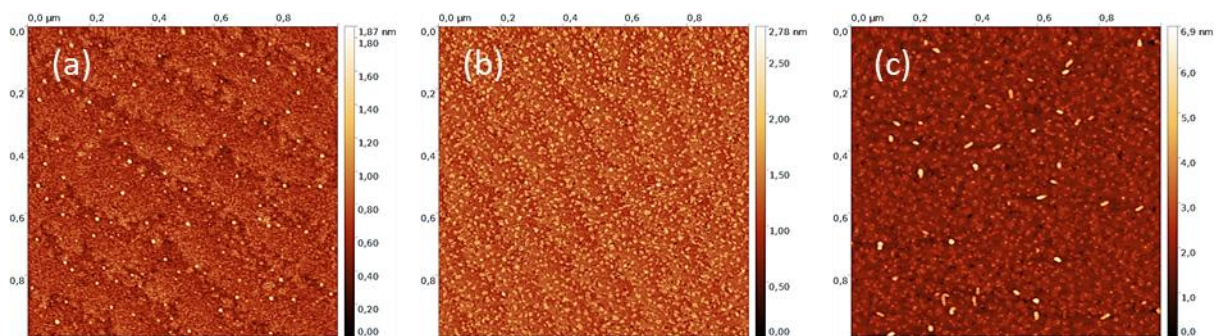


Figure 4: 1 μm x 1 μm AFM images of the GaP(111)B surface after deoxidation (a), growth of 3 MLs WSe₂ at 250°C and annealing at 650°C (b) and growth of 3 MLs WSe₂ at 550°C and annealing at 650°C (c).

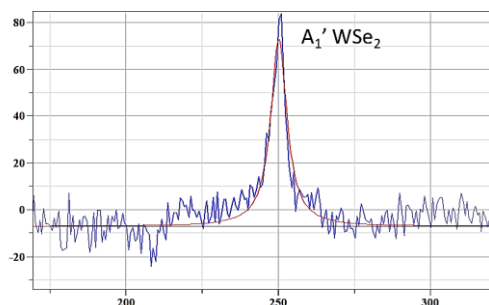


Figure 5: WSe₂ A₁' Raman line recorded on a 3 ML thick WSe₂ layer grown at 450°C and annealed at 650°C.

