

Article

Scalable and Contamination-Free Selenium-Assisted Exfoliation of Transition Metal Dichalcogenides WSe₂ and MoSe₂

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Abstract: In two-dimensional (2D) materials research, exfoliating 2D transition metal dichalcogenides (TMDs) from their growth substrates for device fabrication remains a significant challenge. Current methods, such as those involving polymers, metals, or chemical etchants, suffer from limitations like contamination, defect introduction, and a lack of scalability. Here, we demonstrate a selenium capping-based exfoliation technique. Its advantage lies in its ability to enable the clean, contamination-free exfoliation and transfer of TMD films. We successfully exfoliated and transferred monolayer and multilayer TMD films, including WSe₂ and MoSe₂. The selenium capping layer not only enables seamless exfoliation but also protects the film from oxidation, as confirmed by X-ray photoelectron spectroscopy and Raman spectroscopy. This approach is versatile and applicable to a range of TMDs and thicknesses, paving the way for the high-quality, scalable integration of 2D materials into nanoelectronic devices.

Keywords: two-dimensional materials; transition metal dichalcogenides; nanoelectronics; molecular beam epitaxy; chemical vapor deposition

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1. Introduction

Since the discovery of graphene, the library of two-dimensional (2D) materials has continued to expand rapidly, offering diverse functionalities and properties [1–11]. Among these, transition metal dichalcogenides (TMDs) have gained significant attention due to their atomic-scale thickness, tunable bandgap, and unique proximity effects [12–21]. TMDs with a direct bandgap in the monolayer limit or those exhibiting spin/valley polarization are particularly promising for applications in optoelectronics and spintronics [22–25]. Furthermore, the weak van der Waals (vdW) forces between monolayers allow for the stacking of exfoliated films at desired twist angles, enabling the exploration of perfect light absorption and superconductivity [26–30].

Despite these exciting opportunities, research on these materials still heavily relies on the traditional method of the tape exfoliation of bulk crystals [31–34]. While simple, this method suffers from several limitations, including small flake sizes, low yield, and contamination from adhesive residues, which often require prolonged solvent or plasma treatments [35–39]. Most importantly, tape exfoliation is not scalable, posing a significant bottleneck for the development of large-area devices.

To address these limitations, thin film deposition techniques such as molecular beam epitaxy (MBE) and chemical vapor deposition (CVD) have emerged as promising alternatives for producing large-area 2D films [40–43]. MBE, in particular, offers several advantages, including exceptional film quality, wafer-scale uniformity, and low defect density. Its low growth temperature makes it compatible with a wide range of commercial substrates,

supporting emerging applications like monolithic 3D integration (M3D) for next-generation logic and memory devices. Additionally, MBE is well suited for directly growing heterostructures and superlattices. However, the strong interactions between as-grown films and the growth substrate often prevent their mechanical exfoliation, complicating the transfer process required for further characterization and device fabrication.

Current wafer-scale film transfer techniques involve the use of polymer layers, such as polymethyl methacrylate (PMMA) or polydimethylsiloxane (PDMS), metal layers, or chemical etchants [44–57]. These methods, while effective to some extent, bring the 2D films into direct contact with external agents, raising concerns about contamination, oxidation, unintentional doping, and potential degradation [58–63]. See Table 1 for a comparison.

Table 1. Comparison of different exfoliation methods regarding defect density, yield, contamination levels, advantages, and disadvantages.

	Polymer-Assisted Transfer	Metal-Assisted Transfer	Liquid-Phase Transfer	Se-Assisted Transfer
Defect density	High [39]	Low [64]	Low [36]	Low
Yield	95% [45]	70–99.5% [54,64]	90% [65]	100%
Contamination level	High	Low	Low	Very low
Advantage	Low cost	Easily scalable	Low cost	Easily scalable; best for clean transfer
Disadvantage	Small flake size; carbon contamination	Metal contamination	Complex process; challenging scalability	Selenide preferred

In this work, we demonstrate a novel approach to addressing these challenges by capping TMD films with selenium. This capping layer enables the efficient exfoliation of TMD layers, taking advantage of the interaction between selenium and the transition metal selenide interface. Additionally, since these TMDs are selenides, the Se capping layer ensures the ultra-clean transfer of the TMD layers without the introduction of new elements. Unlike conventional methods, our process avoids the use of polymers, metal layers, or chemical etchants, preserving the purity and quality of the films. Additionally, the selenium capping layer protects the films from oxidation during the transfer process. The transferred films maintain their quality, as confirmed by X-ray photoelectron spectroscopy (XPS), Raman spectroscopy, and diffraction. This method is highly versatile, compatible with a wide range of TMD materials and thicknesses, and paves the way for scalable, high-quality 2D material integration in advanced nanoelectronic applications.

2. Materials and Methods

Sample growth in this study was performed in a ScientaOmicron (Uppsala, Sweden) MBE system. MBE operates in an ultrahigh vacuum environment of 10^{-10} mbar, effectively minimizing background gas molecules that could introduce contamination or alter the deposition trajectory of evaporated atoms. WSe₂ growth was performed on a single-side polished c-plane (0001) sapphire wafer through a co-deposition method, where elemental W and Se were evaporated using an e-beam evaporator for W and a valved effusion cell with a cracker for Se. A substrate temperature of 600 °C was used. A constant Se-to-W flux ratio of 200:1 was maintained, as calibrated by the beam flux monitor within the MBE chamber. A W flux of approximately 5×10^{-9} mbar was achieved with e-beam settings of 200 mA and 6 kV. A Se flux of approximately 1×10^{-6} mbar was used by setting the Se effusion cell's reservoir temperature to 130 °C and the cracker tip to 1100 °C. Before initiating the growth, the source shutters were left open for 30 min to stabilize the fluxes. Subsequently, the substrate shutter was opened to expose the substrate to the evaporated source materials, initiating the growth. MoSe₂ growth was performed on a double-side

polished c-plane sapphire wafer at a growth temperature of 500 °C and a Se-to-Mo ratio of 200:1. A Mo flux of approximately 5×10^{-9} mbar was used, which was achieved with e-beam settings of 100 mA and 6 kV.

For selenium layer deposition, the substrate temperature was kept at 20 °C, and the sample was exposed to a pure selenium flux of approximately 5×10^{-6} mbar, evaporated using a standard Knudsen cell with a selenium deposition rate measured to be 35 nm/min. For all samples, selenium was deposited for 30 min, resulting in a 1050 nm thick amorphous selenium film.

Exfoliation and transfer were conducted in a Class 10,000 cleanroom to maintain high cleanliness standards. After Se capping, the sample was taken out from the chamber, and a quarter-section of the 2-inch wafer was cut using a diamond scribe pen. A thermal release tape (Revalpha 3195 V, sourced from Semiconductor Equipment Corp., Moorpark, CA, USA) was then applied to the TMD/Se sample. To ensure uniform adhesion, the tape was gently pressed onto the wafer using a cleanroom wipe as an interface between the TMD/Se/tape stack and the operator's gloved hand. The tape was then carefully peeled off by hand at no specific peeling angle, effectively exfoliating the TMD/Se layer while leaving the sapphire substrate clean. The exfoliated TMD/Se/tape stack was subsequently transferred onto a SiO₂/Si substrate by gently pressing it with a cleanroom wipe to ensure proper contact. The sample was then heated at 170 °C for 10 s on a hot plate, enabling the detachment of the thermal release tape and leaving the TMD/Se layer securely on the SiO₂/Si substrate. To eliminate any residual tape contaminants, the sample was sequentially immersed in acetone and isopropyl alcohol (IPA) for 5 min each. Following the cleaning process, the sample was loaded into the MBE loading port and pumped down overnight under a vacuum pressure of $\sim 10^{-5}$ mbar to desorb any residual surface adsorbates, such as H₂O, O₂, and carbon-based contaminants. The sample was then transferred to the MBE deposition chamber, where it was annealed at 250 °C for 1 h. The selenium desorption process was monitored in real time using reflection high-energy electron diffraction (RHEED) to ensure the complete removal of the Se capping layer, leaving a clean TMD film on the SiO₂/Si substrate.

A total of eight samples were prepared and tested. The exfoliation success rate was 75% (six out of eight samples). For the successfully exfoliated samples, 100% of the film was successfully peeled from the sapphire substrate, leaving no film behind on the growth substrate. For the remaining two samples, exfoliation did not occur at all, indicating a binary success/failure outcome rather than partial exfoliation. Further experiments are underway to understand the underlying mechanisms.

Raman spectra were acquired at room temperature using a micro-Raman spectrometer, specifically the NRS-5100 model by Jasco (Tokyo, Japan). A 532 nm excitation wavelength with a power of 4.5 mW/μm² was used. The backscattered signal was dispersed using a 1800 g/mm grating. The scans were conducted with an integration time of 10 s, averaged over five integrations. The spectra were calibrated with respect to the Raman peak of silicon at 520.7 cm⁻¹.

XPS spectra were acquired by a Scientia Omicron (Uppsala, Sweden) XPS system with an Argus CU analyzer and using a monochromated Al Kα source (Energy = 1486.7 eV). A takeoff angle of 90° and pass energy of 15 eV were used. The high-resolution spectra were acquired with an energy interval of 0.05 eV and averaged over 15 scans. A charge neutralizer was used to eliminate charging, and spectra were calibrated with respect to the W 4f_{7/2} peak at a binding energy value of 32.7 eV.

3. Results

We begin our discussion with the exfoliation of monolayer (ML) tungsten diselenide (WSe₂). WSe₂ growth was performed on a 2-inch sapphire wafer using the parameters

described in the Materials and Methods Section. Growth was monitored in real time by in situ reflection high-energy electron diffraction (RHEED), and ML growth was confirmed by the disappearance of the RHEED pattern of the sapphire substrate and the appearance of the WSe₂ pattern, as shown in Figure 1a,b, as well as ex situ Raman measurements.

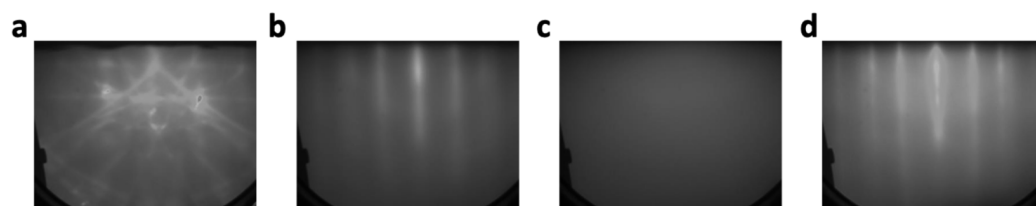


Figure 1. The RHEED pattern of (a) the c-plane (0001) sapphire wafer, (b) after the growth of WSe₂, (c) after the Se capping of WSe₂, and (d) after WSe₂ recovery post Se desorption. The streaky RHEED pattern in (b) is an indication of crystalline WSe₂, while the diffused RHEED pattern in (c) confirms the amorphous nature of the Se capping layer.

After the growth of WSe₂, the W and Se sources were turned off, and the substrate temperature was lowered to room temperature (20 °C). The sample was then transferred to the adjacent XPS chamber to collect in situ XPS data on this as-grown WSe₂. Subsequently, the sample was returned to the MBE deposition chamber, where it was exposed to a pure selenium flux of approximately 5×10^{-6} mbar at room temperature. Selenium was deposited for 30 min, which resulted in an approximately 1 µm thick amorphous film of selenium. The amorphous nature of the capping layer was confirmed by the appearance of a diffused RHEED pattern, as shown in Figure 1c. After selenium capping, the sample was taken out of the MBE chamber, and thermal release tape (TRT) was used to exfoliate the Se/WSe₂ film stack from the sapphire substrate. Note that, as schematically illustrated in Figure 2, the tape only comes into contact with the top Se cap and not the WSe₂ layer.

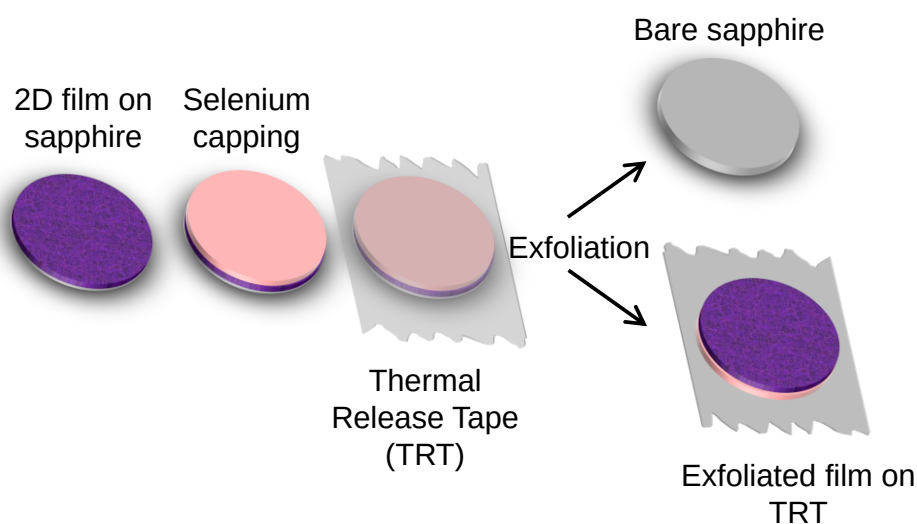


Figure 2. A schematic illustration of the selenium-assisted exfoliation of a 2D film grown on sapphire, where the film is never brought into direct contact with any metal, polymer, or etchant.

The exfoliated Se/WSe₂ film stack was then transferred onto the desired substrate, consisting of 270 nm of thermally grown SiO₂ on silicon, and heated at 170 °C on a hot plate for 10 s to release the tape. The Se/WSe₂/SiO₂ sample was later cleaned with acetone and isopropyl alcohol for 5 min each to remove any tape residue from the surface of the capping layer and then loaded back into the MBE chamber. Lastly, the Se/WSe₂/SiO₂ stack was annealed at 250 °C for approximately an hour in MBE to desorb the selenium capping layer and recover the transferred WSe₂ layer. Se desorption and WSe₂ recovery were monitored

in real time with RHEED. Figure 1d shows a representative image of the recovered WSe₂ film post Se desorption. Finally, the sample was moved to the XPS chamber to collect data on this exfoliated WSe₂. Figure 3 shows the film transfer process.

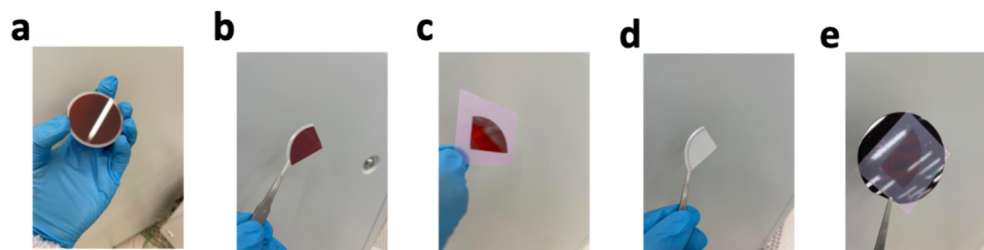


Figure 3. (a) Selenium-capped WSe₂. (b) Small piece used for exfoliation. (c) Se/2D stack exfoliated by thermal release tape. (d) Sapphire substrate post exfoliation. (e) Se/2D stack transferred onto SiO₂ substrate.

To confirm whether the exfoliation process worked successfully, we first employed Raman spectroscopy. Figure 4a shows a comparison of the Raman spectra of a monolayer of WSe₂ grown on sapphire and later transferred onto a SiO₂ substrate. The first thing to note for the as-grown sample is the presence of the degenerate Raman mode E¹_{2g}/A_{1g} around 250 cm^{−1} and the 2 LA(M) mode at ~260 cm^{−1} that are signatures of the semiconducting H-phase of WSe₂ [66,67]. The ‘A’ mode refers to an out-of-plane vibrational mode, which involves the movement of atoms perpendicular to the plane of the film, while the ‘E’ mode is an in-plane vibrational mode. These modes are almost degenerate at around 250 cm^{−1} in monolayer WSe₂. The ‘2 LA(M)’ mode is a second-order scattering process which involves longitudinal acoustic phonons at the M point of the Brillouin zone. The absence of the interlayer breathing mode (B_{2g}) around 310 cm^{−1} confirms the monolayer thickness of the sample, as this mode, caused by interlayer interactions, only appears in multilayer films [68]. Compared with the Raman spectra of the same film transferred onto SiO₂, we saw similar Raman peaks with a slight blue shift of ~0.4 cm^{−1} in the peak position. This small shift, which is likely due to uniaxial strain caused by the SiO₂ substrate due to its larger surface roughness (4 Å–8 Å) than sapphire (1 Å–2 Å), has been reported previously [69]. Most importantly, no enhancement was observed in the 2 LA(M) mode of the transferred film which confirms that neither the transfer process nor the cap desorption process introduces any disorder or lattice defects in the transferred film. This observation is significant because an increase in the intensity of the 2 LA(M) mode has been ascribed to be a signature of defective WSe₂ films [70].

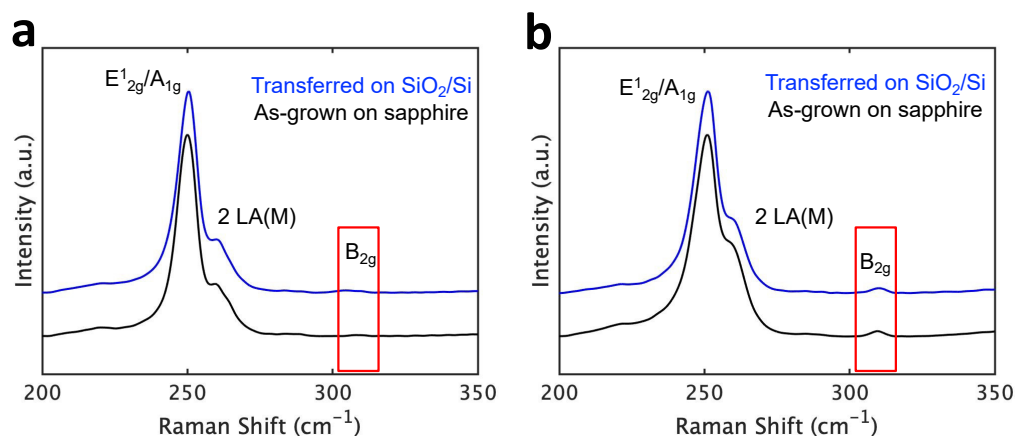


Figure 4. A Raman comparison of as-grown WSe₂ on sapphire (in black) versus the same film transferred on SiO₂ (in blue). (a) shows the plot for monolayer WSe₂, and (b) shows that for bilayer WSe₂. Three measurements were taken for all samples at random locations. Error is ±0.7 cm^{−1}.

To further check whether this method can be used to transfer more than one layer, a bilayer sample of WSe₂ was grown and later transferred to SiO₂ using the same process. Unlike in monolayer WSe₂, the energy degeneracy of the E_{2g}¹ and A_{1g} modes is lifted in bilayer WSe₂, resulting in a redshift in the E_{2g}¹ mode and a blueshift in the A_{1g} modes, as reported in Ref. [71]. Consequently, the Raman line shape of bilayer WSe₂ appears slightly broader than that of monolayer WSe₂, as the bilayer spectrum is now a convolution of two slightly shifted peaks. This is evident in the comparison in Figure 4a,b. This, combined with the observation of the interlayer breathing mode at 310 cm^{−1} in both the as-grown and transferred bilayer films, confirms that this method is equally effective in exfoliating thicker films.

A significant advantage of this exfoliation technique is that the TMD layer, protected by the selenium capping layer, remains shielded from oxidation. To confirm this, we performed XPS. Figure 5 presents a comparative XPS analysis of an as-grown WSe₂ monolayer on the sapphire substrate and the same layer transferred onto SiO₂, along with a Se capping layer. The plot includes XPS spectra for the W 4f, W 5p, and Se 3d regions, with the data normalized and aligned relative to the W 4f_{7/2} peak at 32.7 eV. In the as-grown sample, only the characteristic peaks corresponding to a high-intensity Se–W bond and a low-intensity Se–Se bond are observed, as shown by the peak deconvolution. The presence of Se–Se bonds is similar to that of As–As bonds in the As region of GaAs [72], alongside As–Ga bonds. Oxidized WSe₂ typically exhibits additional W–O and Se–O peaks at higher binding energies relative to the main W 4f and Se 3d peaks; however, such peaks were absent in our as-grown film due to the in situ transfer to the XPS chamber [73–75]. For the Se capping layer, we observed a single doublet representing the Se–Se bond. The peak position is the same as that of the Se–Se bond in WSe₂ but with a broader width, which is attributed to the amorphous structure. The XPS profile of the transferred film closely matched that of the as-grown film, with no W–O or Se–O peaks detected, confirming the oxidation-free nature of the transfer process, even though the capped sample was exposed to air for two days. These findings demonstrate the effectiveness of the transfer method in preventing oxidation, addressing a common challenge associated with alternative transfer techniques.

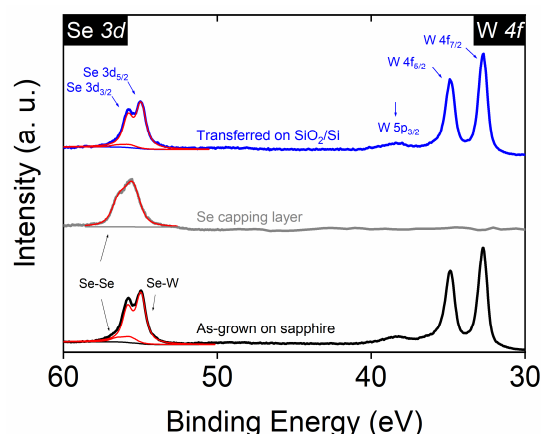


Figure 5. An XPS comparison of an as-grown WSe₂ monolayer and one transferred to SiO₂, together with a Se capping layer. The Se regions are fitted in red. For WSe₂ samples, two doublets corresponding to Se–W and Se–Se bonds are used, while the Se capping layer is fitted with a single Se–Se doublet. The identical line shapes of WSe₂ indicate no oxidation and no residual Se from the cap or any other chemistry changes after film transfer.

Another notable advantage of this transfer method is its versatility, as it is not limited to a specific TMD but can be applied to a wide range of other 2D chalcogenides. For instance, we successfully transferred molybdenum diselenide (MoSe₂) as well. Figure 6 presents

a Raman analysis comparing a thick MoSe₂ film grown on sapphire with the same film after being transferred onto SiO₂. The characteristic Raman peaks of MoSe₂, corresponding to the A_{1g} and E_{12g} modes, were observed near 242 cm^{−1} and 287 cm^{−1}, respectively, consistent with the values reported in the literature [76]. Similar to WSe₂, few-layer-thick MoSe₂ exhibits an interlayer breathing mode near 353 cm^{−1} [77]. This mode is the strongest for bilayers and gradually diminishes with increasing layer thickness, becoming absent in bulk MoSe₂ due to translational symmetry [76]. In our case, we observed a very weak B_{2g} mode, consistent with the target thickness of approximately 5 MLs. Lastly, similar to the results obtained for WSe₂, the transferred MoSe₂ film exhibited no broadening, peak shifts, or intensity degradation, thereby confirming both the successful transfer and the general applicability of this method.

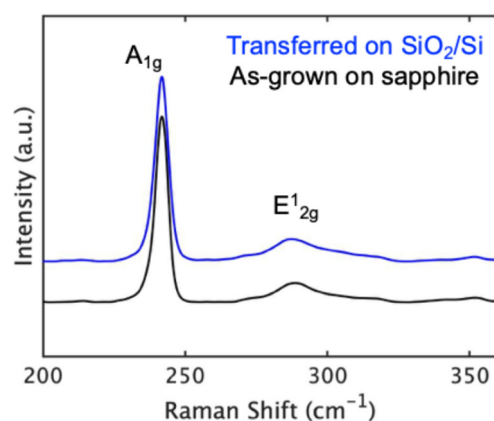


Figure 6. A Raman comparison of a thick MoSe₂ film grown on sapphire (in black) versus the same film transferred on SiO₂ (in blue).

Multiple experimental trials showed that the overall peeling efficiency of selenium-assisted exfoliation was 100% and that the entire film, including both monolayers and thick films, was successfully exfoliated from the sapphire growth substrate, leaving no residual material behind. The transfer success rate onto the SiO₂/Si target substrate exceeded 95% of the overall film. The minor material loss (~5%) can be avoided through refined handling techniques or automation. The exfoliation and transfer technique proved effective for a wide range of film thicknesses, from monolayers and bilayers to thick films, as demonstrated in Figures 4 and 6.

4. Discussions

The outstanding performance of selenium-assisted exfoliation provides insights into understanding the exfoliation mechanism. Studies on other methods, such as metal-assisted exfoliation, have demonstrated that the stamp layer exhibits higher adhesion energy with 2D materials compared to the substrate. For instance, the average adhesion energy [55] for 2D-2D interactions is 0.45 J/m², while for 2D sapphire, it is 0.26 J/m². In contrast, the adhesion energy [55] between nickel and 2D materials reaches 1.4 J/m². Although the adhesion energy for Se–WSe₂ has not yet been reported, considering the Se–W–Se sandwich structure and the bonding of the top Se layer with the selenium capping layer, the van der Waals adhesion is expected to be significantly weaker than that of metal/WSe₂ interactions. For example [78], the adhesion energy of Se on graphene has been reported to be only 0.34 J/m² which is still larger than the 0.26 J/m² of a TMD film on sapphire, indicating that transfer off the sapphire is still energetically favorable with a Se cap. However, other mechanisms beyond the adhesion energy should also be considered. It has been previously suggested [53] that when a semiconductor comes into contact with a metal or another

semiconductor, the built-in electric field exerts a force on both materials, influencing the structure and bonding characteristics, an effect particularly pronounced in 2D materials. Consequently, the interlayer distance between adjacent TMD layers could be altered, in this case, increased, and results in a smaller 2D-2D interaction which facilitates exfoliation [53].

As for the ability of a Se cap to prevent oxidation, selenium is known for its relative inertness to air. Studies have shown that a strong oxidizing agent is required to obtain selenium oxide, and its reaction with oxygen requires catalysts such as nitrogen oxides to facilitate oxidation [79]. Therefore, selenium has been widely used as a capping layer, preventing the thin film from oxidizing [80]. The Se capping layer can be easily evaporated at 200 °C, a low enough temperature to prevent degradation and chalcogen vacancy formation in most TMDs. Here, selenium-assisted exfoliation also benefits from this inert nature in protecting the underlying thin film. These unique physical and chemical properties make selenium a superior candidate for TMD transfer compared to other existing methods.

5. Conclusions

We introduced a selenium-assisted exfoliation technique for the transfer of 2D transition metal dichalcogenide films as a scalable and contamination-free solution to the challenges faced by conventional methods. By employing a selenium capping layer, we demonstrated that monolayer and multilayer TMD films could be efficiently exfoliated and transferred to target substrates while preserving their structural and electronic integrity. Characterization through Raman spectroscopy and X-ray photoelectron spectroscopy confirmed that the transferred films maintained their quality and remained free of defects and oxidation. This versatile approach not only overcomes the limitations of traditional exfoliation methods, such as contamination, oxidation, unintentional doping, damage, and scalability issues, but also offers a platform for integrating various 2D materials into nanoelectronic devices. Moreover, in practical industrial applications, selenium can be deposited using sputtering, e-beam evaporation, or thermal evaporation, offering good uniformity and excellent scalability for large-area applications, an advantage not achievable with polymer- or liquid-assisted methods. We also anticipate this technique to be extendable to other TMDs, such as MoS₂ and WS₂, with minimal parameter adjustments due to the weak interaction of the Se cap on inert 2D surfaces that enables Se desorption at low temperatures. Additionally, our results suggest a potential strategy for identifying materials suitable as an exfoliation stamp, following the guiding principle that materials with inert properties, shared elemental composition, and low-temperature evaporability are promising candidates. As the demand for wafer-scale, high-quality 2D materials grows, this selenium-assisted exfoliation process presents a significant step forward in advancing the fabrication and application of next-generation electronic and optoelectronic devices.

Author Contributions: R.Y. and C.L.H. conceived and designed this study; R.Y. conducted the experiments and collected data under the guidance of G.Z. and C.L.H. R.Y. and C.L.H. wrote the manuscript with the input from all the authors. All authors have read and agreed to the published version of the manuscript.

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Data Availability Statement: All data that support the findings of this study are included within the article.

Conflicts of Interest: The authors declare no conflicts of interests.

Abbreviations

The following abbreviations are used in this manuscript:

MBE	Molecular beam epitaxy;
MoSe ₂	Molybdenum diselenide;
RHEED	Reflection high-energy electron diffraction;
WSe ₂	Tungsten diselenide.

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