

Strong Non-Epitaxial Interactions: Crystallographically Aligned PbSe on VSe₂

Dmitri L. M. Cordova, Taryn M. Kam, Shannon S. Fender, Yu Hsin Tsai, and David C. Johnson*

The interaction between a rock salt compound, PbSe, and the surface of a dichalcogenide (VSe₂) is probed by making PbSe, VSe₂, [(PbSe)_{1.11}]₁(VSe₂)₁ and PbSe on VSe₂ films. PbSe precursors deposited on SiO₂ form rough films with randomly oriented PbSe crystallites. VSe₂ precursors deposited on a SiO₂ surface form crystallographically aligned films. The precursor to the metastable misfit layer compound [(PbSe)_{1.11}]₁(VSe₂)₁ deposited on SiO₂ forms a crystallographically aligned film. PbSe precursors deposited on VSe₂ are very crystallographically aligned relative to PbSe deposited on SiO₂. This reflects the strong interaction between PbSe and VSe₂ at the interface. The results suggest that comparing the degree of crystallographic alignment of films of precursors of prospective constituents on SiO₂ relative to depositing them on each other may be a simple test to show if a misfit layer compound will form between the two constituents.

1. Introduction

The number of known ternary and multinary compounds is much less than expected.^[1] There are many ternary systems that do not contain any known ternary compounds. Traditional solid-state synthesis techniques that involve directly reacting elements or mixes of elements and binary compounds evolve through complex mixtures of elements, binary compounds and/or ternary compounds as diffusion occurs. The search for new compounds is often difficult, because only trace amounts of a new compound are formed using the initial reaction conditions. Potential new compounds are found by identifying reflections in diffraction patterns of mixtures that cannot be explained by known phases. Reaction conditions are then modified to increase the amount of the new compound. For a new ternary or multinary compound to be the primary product in this reaction after long times at high temperatures, they need to be more stable than any of the intermediate mixtures. Other approaches have been developed that use a fluid phase to

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increase diffusion rates and make nucleation the rate-limiting step in the synthesis (flux-based growth techniques, vapor phase transport, molecular beam epitaxy, etc.). While there has been recent progress in understanding the speciation in fluids that control product formation, serendipity controls what forms.^[2] The reacting systems are more complicated and there is little fundamental understanding of how to adjust the composition of the system to change speciation and impact which compound nucleates.

In an attempt to increase the rate of discovery of new materials, a variety of high throughput methods have been developed in projects such as the Materials Genome Initiative.^[3–5] Typically these methods combine information in databases and

computational approaches to predict the stability and properties of materials that have yet to be synthesized in the lab. Unfortunately, attempted syntheses of many of the new compounds that have been predicted to be stable have failed. For example, a recent paper predicted 24 likely new compounds in phase diagrams that did not contain any known ternary compounds.^[6] The authors tried to prepare these predicted compounds, containing a cation, a transition metal and a chalcogen, using several synthesis approaches. The products of the reactions were mixes of known binary compounds and the elements. While the predicted compounds are potential local free energy minima in the energy landscape based on the calculations, the synthetic protocols used could not prevent the formation of binary compounds as reaction intermediates. In three systems, a compound forms with a stoichiometry close to that of a predicted compound, but the structure that forms is a misfit layer compound rather than the predicted structure. Calculating the formation energy of misfit layer compounds is challenging due to the incommensurate interface between the constituents.

The surprising thermodynamic stability of misfit layer structures has long puzzled researchers.^[7] Misfit layer structures consist of two interwoven lattices that are commensurately stacked along the crystallographic *c* axis. A common pairing of structures is a transition metal dichalcogenide such as NbSe₂ with a rock salt-structured layer such as PbSe.^[8] In the *a*-*b* plane, there is a size mismatch between the constituents and often a symmetry difference that results in an incommensurate relationship between the constituent layers. The atoms at the

interface in each layer are displaced from their average position due to the interaction with the atoms of the other layer. This results in a structurally incoherent interface between the two layers. Given the varying and irregular local coordination environment between the atoms at the interface that results from the structural incoherence, one would expect that a mix of the binary compounds (for example $\text{NbSe}_2 + \text{PbSe}$) to be more stable than the misfit layered compound $(\text{PbSe})_{1.14}\text{NbSe}_2$, which contains alternating planes of the dichalcogenide trilayer (Se-Nb-Se) with bilayers of PbSe . The misfit compound $(\text{PbSe})_{1.14}\text{NbSe}_2$, however, is the product that is formed on heating a stoichiometric mix of the elements at 1000°C for an extended time.^[9] This indicates that the interaction between the PbSe and NbSe_2 at the interface must be stronger than the sum of the interactions between NbSe_2 layers in NbSe_2 and between (001) planes of PbSe .

There have been several explanations presented in the literature for the surprising stability of misfit layer compounds. While van der Waals forces certainly exist between the two structures, the interaction between the layers needs to be much stronger. For misfit layer compounds containing trivalent cations, such as rare earth metals in the rock salt layer, there is abundant evidence that there is electron transfer from the rock salt layer to the dichalcogenide. Such charge transfer would result in a strong enough ionic interaction between the layers $(\text{LnX})_{1+d}^{\delta+}(\text{TX}_2)_d^{\delta-}$ to stabilize the misfit layer structure.^[8,10] There is significant controversy in the literature, however, about whether charge transfer occurs in misfit layer compounds containing a semiconducting divalent rock salt structure (such as SnX or PbX where $\text{X}=\text{S}$ or Se) paired with a transition metal dichalcogenide. Ohno^[11] performed X-ray photoemission, X-ray absorption and reflection electron energy loss spectroscopy on misfit layer compounds containing alternating layers of PbS and TiS_2 and SnS and NbS_2 . He concluded that a small amount of charge transfer occurs from the rock salt layer to the dichalcogenide. A subsequent photoelectron spectroscopy study of the misfit compounds $(\text{MS})_{1+d}(\text{TS}_2)$ where $\text{M}=\text{Sn}$ and Pb and $\text{T}=\text{Ti}$, Nb , and Ta indicated that there was no, or only a very small amount of charge transfer and that the Pb and Sn were divalent.^[12] Band structure calculations also suggest that there is no charge transfer.^[13] If only a very small amount of charge transfer occurs, then the amount of ionic stabilization will be small. A subsequent study of several misfit layer compounds indicated that there was a systematic excess of the transition metal dichalcogenide cation (T) and a systematic shortfall in the rock salt cation (M). The authors suggested that T cations were substituting for M cations in the rock salt structure, resulting in layers that have high enough charges to stabilize the misfit layer structure via an ionic interaction between the constituents.^[14] They also suggested that these deviations from stoichiometry are necessary to stabilize the misfit layer structure. The cross substitution of M and T cations was supported by a subsequent photoelectron spectroscopy investigation.^[7]

In this work we probe the interaction between a rock salt compound, PbSe , and the surface of a dichalcogenide (VSe_2). We examine the formation and structure of PbSe , VSe_2 , $(\text{PbSe})_{1.11}\text{VSe}_2$ and PbSe on a layer of VSe_2 from precursors prepared by depositing sequences of ultrathin layers of the respective elements to form films of varying thicknesses. The

ultrathin layer thicknesses eliminate diffusion as a rate limiting step, while constraining the extent of long range diffusion because low reaction temperatures can be used. In all of the precursors, the respective crystalline compounds nucleated and grew during the deposition process. Post-deposition annealing increased both crystallite size and the amount of material crystallized in all films. The PbSe precursors deposited on a SiO_2 surface formed rough films with randomly oriented PbSe crystals. The VSe_2 precursors deposited on a SiO_2 surface formed crystallographically aligned films, with the basal plane of VSe_2 parallel to the substrate. In plane diffraction scans indicated a random rotational orientation of the VSe_2 crystallites. The precursor to the metastable misfit layer compound $[(\text{PbSe})_{1.11}]_1(\text{VSe}_2)_1$ deposited on SiO_2 formed a crystallographically aligned film, with the basal plane of VSe_2 parallel to the substrate. In plane diffraction scans indicated a random rotational in plane orientation of both the PbSe and VSe_2 . The PbSe precursor deposited on a thin film of VSe_2 was also crystallographically aligned, with the (001) surface of the PbSe crystallites parallel with the basal plane of the VSe_2 substrate. This is an indication of a strong interaction between PbSe and VSe_2 at the interface. At the low reaction temperatures, it is unlikely that there is sufficient energy to exchange Pb with V cations from the VSe_2 substrate. This suggests a simple test to see if it is likely that a misfit layer compound can be formed between a new constituent and a dichalcogenide. If there is not crystallographic alignment of a proposed new constituent on a dichalcogenide, a new misfit layer compound is unlikely. If there is crystallographic alignment of a proposed new constituent on the dichalcogenide, it is much more likely that a new misfit layer compound can be formed.

2. Discussion

The precursors designed to self-assemble the PbSe , VSe_2 , $(\text{PbSe})_{1.11}\text{VSe}_2$, and PbSe on a few layers of VSe_2 films were made by sequentially depositing alternating layers of either Pb and Se or V and Se . The number of $\text{M}|\text{Se}$ pairs deposited was varied to obtain the desired total film thickness. The thickness of the Pb and Se layers in each $\text{Pb}|\text{Se}$ pair contained the number of atoms required to form a single rock salt structured PbSe bilayer. The thickness of the V and Se layers in each $\text{V}|\text{Se}$ pair contained the appropriate number of atoms to form a single trilayer of VSe_2 . The number of atoms of each element was calculated from the lattice parameters of PbSe ^[15] and VSe_2 .^[16] For example, the number of Pb atoms per unit area in a PbSe bilayer is given by the product of the number of Pb atoms per unit cell divided by the basal plane area of the unit cell [$4 \text{ Pb atoms } (6.117 \text{ \AA})^{-2} = 0.107 \text{ Pb atoms } \text{ \AA}^{-2}$]. X-ray fluorescence analysis was used to determine the number of atoms per square Angstrom in the total film thickness.^[17] Dividing the number of atoms per square Angstrom in the total film thickness by the number of layers deposited yields the average number of atoms deposited in each bilayer. We investigated what structure is formed as the precursors are deposited and how the structure evolves as the film is annealed using X-ray diffraction.

Figure 1 contains specular X-ray diffraction data of as deposited and annealed PbSe films of various thicknesses (16,

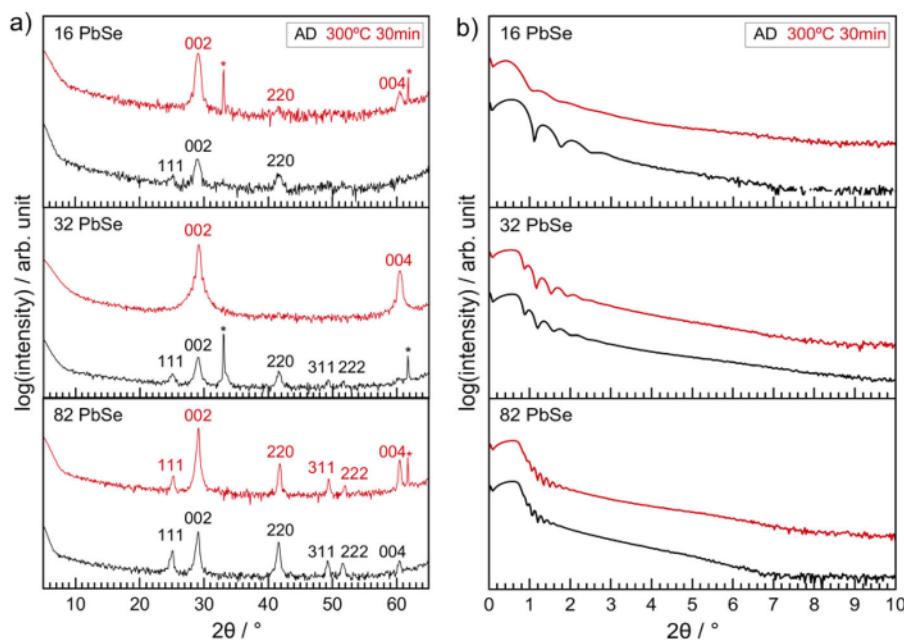


Figure 1. a) Specular X-ray diffraction and (b) X-ray reflectivity patterns of various thicknesses of PbSe films on SiO_2 as deposited (black) and annealed at 300°C for 30 min (red). The presence of non-00l reflections in the specular diffraction patterns indicates that PbSe is randomly oriented. Kiessig fringes that extend only up to $\approx 2^\circ$ suggest that the film is rough. *Substrate.

32, and 82 layers) deposited on SiO_2 . The measured number of Pb and Se atoms per bilayer in each film are given in Table 1. In the high angle specular diffraction scans of the as deposited precursors (Figure 1a), Bragg reflections consistent with the known rock salt structure of PbSe are observed in all of the films. This indicates that the Pb and Se atoms self-assemble during the deposition process forming PbSe. The a -axis lattice parameters are given in Table 2. The degree of crystallographic alignment in the as deposited films is similar, based on the ratios of the intensities of the (111), (200), and (220) reflections. Crystallographic alignment increases as the films are annealed, with significant growth of the intensity of the (002) and (004) reflections in all of the films. In the 16 layer film the only non-00l reflection visible after annealing is a weak (220) reflection. In the 82 layer film, the intensity of the (002) reflection increases fivefold after annealing, but other non-00l reflections remain prominent. The X-ray reflectivity patterns of these films (Figure 1b) suggest that the roughness increases as the film thickness increases, as the angle where Kiessig fringes are no longer resolvable decreases with increasing film thickness.^[18]

Table 1. The number of Pb and Se atoms per \AA^2 in the different PbSe precursors determined by X-ray fluorescence. The target composition to obtain a bilayer of PbSe is: Pb and Se: 0.107 atoms per \AA^2 .

# Layers Pb Se	Pb atoms per \AA^2		Se atoms per \AA^2		Composition ratio (Se/Pb)
	Total	Per layer	Total	Per layer	
16	1.62	0.101	1.63	0.102	1.01
32	3.15	0.098	3.10	0.097	0.98
82	8.44	0.103	8.14	0.099	0.96

Table 2 summarizes the measured lattice parameters of the three PbSe films both before and after annealing.

An annealing study was carried out on several V|Se precursors to investigate how the structure evolves as the temperature is increased. Table 3 summarizes the amount of each element in each of the as deposited films. The slight excess of Se in one as deposited film is lost through evaporation as the sample is annealed. The specular X-ray patterns of a representative sample collected after annealing at several temperatures is shown in Figure 2. The diffraction pattern of the as deposited precursor contains four 00l reflections indicating that VSe_2 self assembles during the deposition, crystallographically aligned to the SiO_2 substrate. The (001) reflection at around 15° , however, has a shoulder at low angles from the artificial layering of the precursor. This suggests that only part of the film has formed VSe_2 . As the annealing temperature increases, the intensity of this low angle shoulder decreases while the 00l reflections become more intense and have narrower line widths. The increased intensity suggests that more of the film becomes VSe_2 .

Table 2. Lattice parameters calculated from specular X-ray diffraction patterns of PbSe on SiO_2 . AD = as deposited, AN = annealed.

# Layers Pb Se	AD	a-Lattice parameter [\AA]
16	AD	6.08(1)
	AN	6.10(1)
32	AD	6.11(1)
	AN	6.11(1)
82	AD	6.08(1)
	AN	6.10(1)

Table 3. The number of V and Se atoms per \AA^2 in the different VSe_2 precursors determined by X-ray fluorescence. The target composition to obtain a trilayer of VSe_2 is: V=0.103 atoms per \AA^2 and Se=0.205 atoms per \AA^2 .

# Layers Pb Se	V atoms per \AA^2		Se atoms per \AA^2		Composition ratio (Se/Pb)
	Total	Per layer	Total	Per layer	
49	5.08	0.104	11.0	0.224	2.17
82	9.18	0.112	17.6	0.215	1.92

and/or the VSe_2 is becoming more crystallographically aligned to the substrate. The decreasing line widths indicate that the structural coherence of the VSe_2 perpendicular to the substrate is increasing. At 300°C Laue fringes were observed on either side of the (001) reflection of the thinner sample due to the finite number of unit cells, suggesting a uniform thickness exists over a significant portion of the film. In plane diffraction patterns, done on a subset of samples, contained only $hk0$ reflections, which gives further support that the VSe_2 film is crystallographically aligned to the substrate. Table 4 contains a summary of the structural parameters derived from the diffraction studies of the VSe_2 films.

A similar annealing study was carried out on Pb|Se|V|Se precursors to compare the formation of $(\text{PbSe})_{1.11}(\text{VSe}_2)_1$ relative to that of the individual constituents. Table 5 summarizes the amount of each element in each of the as deposited films. Figure 3 contains the diffraction patterns collected in the annealing study of a representative film. The appearance of higher order $00l$ reflections in the specular diffraction pattern, (Figure 3a), indicates that a superstructure forms during the deposition. Since only $00l$ reflections are observed, it is crystallographically aligned to the substrate. The c -axis lattice parameter determined from the $00l$ reflections (12.28 (1) \AA) is close to the previously reported value for

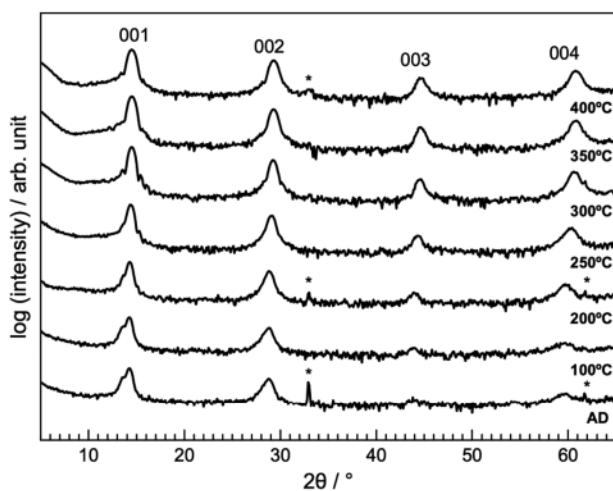


Figure 2. Specular X-ray diffraction of representative V|Se precursors after annealing at each of the indicated temperatures for 30 min. The prominent $00l$ reflections appearing in the as deposited sample indicates that crystallographically aligned VSe_2 forms upon deposition and becomes more ordered as the film is annealed.

Table 4. Structural parameters (c and a lattice constants) calculated from specular and in-plane X-ray diffraction patterns of VSe_2 on SiO_2 . AD = as deposited, AN = annealed 350°C for 30 min.

# Layers V Se	c-Lattice parameter [\AA]	a-Lattice parameter [\AA]
49	6.20(1)	3.37(1)
	6.07(1)	
82	6.10(1)	—
	6.03(1)	

$[(\text{PbSe})_{1.11}](\text{VSe}_2)_1$, 12.25(1) \AA .^[19] The in-plane diffraction pattern of the as deposited sample (Figure 3b) contains broad reflections of both PbSe and VSe_2 , further indicating that $[(\text{PbSe})_{1.11}](\text{VSe}_2)_1$ forms during the deposition. Only $hk0$ reflections are observed for PbSe and VSe_2 in the in plane diffraction pattern, indicating strong crystallographic alignment of each constituent with respect to the substrate. X-ray reflectivity of the as deposited sample indicates that the Pb|Se|V|Se precursor is much smoother than the films of PbSe or VSe_2 . The as deposited Pb|Se|V|Se precursor contains significantly more order than the corresponding films of the individual constituents. As the annealing temperature is increased, the $00l$ and $hk0$ reflections become more intense and narrow as atoms diffuse forming a more coherent structure perpendicular to the substrate and larger domains of PbSe and VSe_2 in the plane of the substrate. While the misfit layer compound $[(\text{PbSe})_{1.11}](\text{VSe}_2)_1$ has not been reported in the literature, the $[(\text{PbSe})_{1.11}](\text{VSe}_2)_1$ formed from the ordered precursor is at least kinetically stable at 400°C. Table 6 contains a summary of the structural parameters derived from the diffraction studies of Pb|Se|V|Se precursors. Precursors that have formed around the targeted layering thickness and composition also form $[(\text{PbSe})_{1.11}](\text{VSe}_2)_1$, although a variety of defects (extra PbSe or VSe_2 layers between $[(\text{PbSe})_{1.11}](\text{VSe}_2)_1$ crystals, partial replacement of a PbSe (or VSe_2) layer with VSe_2 (or PbSe), extra PbSe or VSe_2 on the surface) are present. This suggests that $[(\text{PbSe})_{1.11}](\text{VSe}_2)_1$ is at least a significant local free energy minimum in the free energy landscape. The large difference in the degree of order observed in Pb|Se|V|Se versus either Pb|Se or V|Se precursors in both as deposited and annealed states indicates a strong interaction must be present between bilayers of PbSe and trilayers of VSe_2 .

We prepared precursor films with different thicknesses of PbSe on a thin layer of VSe_2 to determine if the PbSe layer was different than that found when PbSe was deposited on SiO_2 -coated silicon described earlier. Table 7 summarizes the

Table 5. The total number of Pb, V, and Se atoms per \AA^2 in the different Pb|Se|V|Se precursors determined by X-ray fluorescence.

# Layers Pb Se V Se	Pb atoms per \AA^2		Se atoms per \AA^2		V atoms per \AA^2	
	Total	Per layer	Total	Per layer	Total	Per layer
41*	4.39	0.107	12.6	0.308	4.67	0.114
24	2.66	0.111	8.21	0.342	2.09	0.087

*Sample used in annealing study.

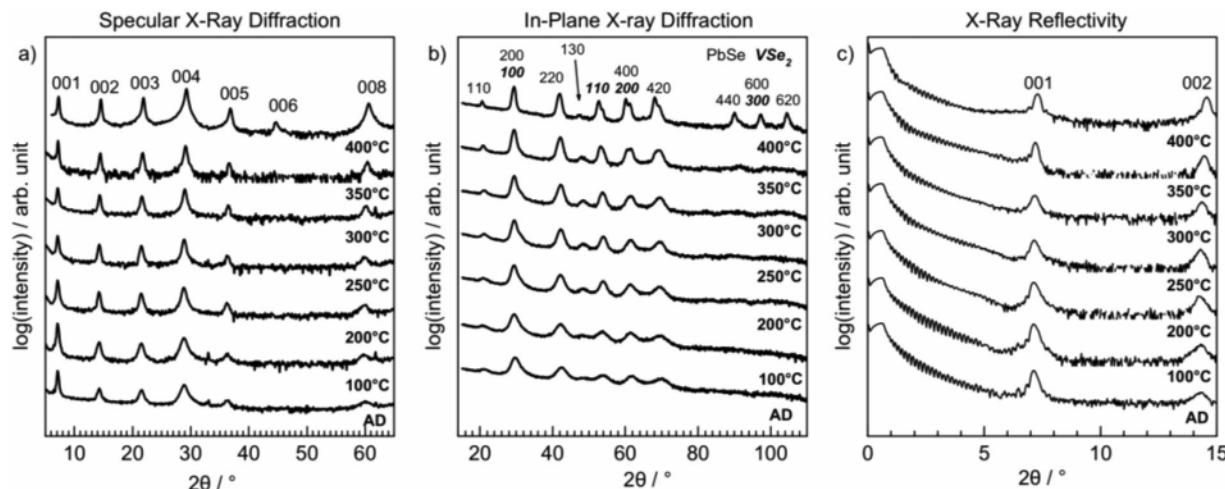


Figure 3. a) Specular X-ray diffraction of $[(\text{PbSe})_{1.11}]_1(\text{VSe}_2)_1$, (b) in-plane diffraction of $[(\text{PbSe})_{1.11}]_1(\text{VSe}_2)_1$, and (c) X-ray reflectivity patterns of $[(\text{PbSe})_{1.11}]_1(\text{VSe}_2)_1$ after annealing at the indicated temperatures for 30 min. Since the film is crystallographically aligned to the substrate, only 00 l reflections are observed in the specular scans. The higher order 00 l reflections observed in the as deposited film suggest crystallization of the superlattice taking place upon deposition. Reflections for independent lattices of PbSe and VSe₂ are observed in the in-plane diffraction pattern and show that both constituents are present starting at the as deposited state. Kiessig fringes are retained in the X-ray reflectivity pattern even after multiple steps of annealing, suggesting that the film remains smooth throughout the self-assembly process.

amounts of each element in the as deposited films. Figure 4 contains diffraction data collected on two samples. For both films, PbSe forms during the deposition process. For the 20 layer PbSe sample on 4 layers of VSe₂, only 00 l reflections of PbSe are present, indicating that the PbSe is crystallographically aligned. After annealing, there is a relatively small change in the intensity (about 1.5×) of the 002 reflection suggesting that the as deposited film was already mostly crystalline and/or that the extent of crystallographic alignment increases. Kiessig fringes extend up to 5°, suggesting that the film is smooth, with calculated roughness of ≈ 10 Å. In the as deposited 82-layer PbSe on eight layer VSe₂ film, weak non-00 l reflections of PbSe are observed indicating that some grains are randomly oriented. However, since the intensity ratio of these reflections are low compared to the expected values of a randomly oriented powder, the majority of the film is crystallographically aligned. After annealing, the non-00 l reflections disappear completely and the

intensity of the 00 l reflections increases. The amount of the intensity increase is significantly less than was observed in a similar thickness film of PbSe on SiO₂, suggesting that the as deposited film on VSe₂ is mostly crystalline and aligned as deposited. The (001) reflection of VSe₂ also appears after annealing. The thicker film on VSe₂ is significantly smoother than the film on SiO₂. The X-ray reflectivity scan of the thicker PbSe film on VSe₂ contains high frequency fringes coming from the thick PbSe layer and broader low frequency fringes due to the VSe₂ layers beneath the PbSe. Table 8 contains a summary of the structural parameters derived from the diffraction studies of Pb|Se precursors deposited on thin VSe₂ layers. The difference between the diffraction data of films of PbSe deposited on VSe₂ and on SiO₂ reflects the presence of a strong interaction between the PbSe and VSe₂.

3. Conclusion

There are a large number of potential misfit layer compounds, especially because there is not a requirement for lattice matching at the interface between different constituents. The incoherent interfaces and the lack of an understanding why the interactions

Table 6. Structural parameters (superlattice d-spacing and a-lattice constants) calculated from specular and in-plane X-ray diffraction patterns of Pb|Se|V|Se precursor annealed at various temperatures. AD = as deposited.

Temperature [°C]	d-Spacing [Å]	PbSe a-lattice parameter [Å]	VSe ₂ a-lattice parameter [Å]
AD	12.28(1)	–	–
100	12.38(1)	–	–
200	12.38(1)	–	–
250	12.36(1)	–	–
300	12.31(1)	6.03(1)	3.43(1)
350	12.25(1)	–	–
400	12.17(1)	–	–

Table 7. The total number of Pb, V, and Se atoms in the different PbSe precursors on VSe₂ determined by X-ray fluorescence. Exact composition ratio of Pb/Se cannot be determined because of the presence of Se in both constituents.

# Layers	PbSe:VSe ₂	Pb atoms per Å ²		Se atoms per Å ²		V atoms per Å ²	
		Total	Per layer	Total	Per layer	Total	Per layer
20:4		2.66	0.133	3.05	–	0.450	0.113
82:8		8.76	0.107	8.40	–	0.953	0.119

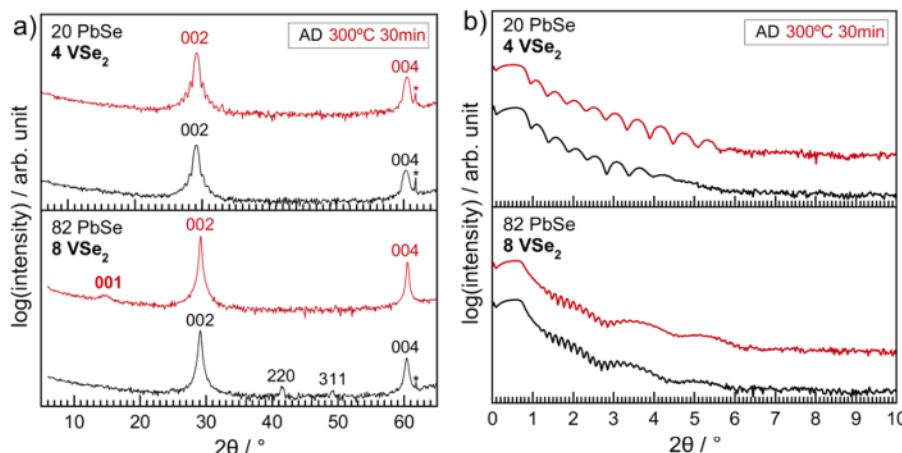


Figure 4. a) Specular X-ray diffraction and (b) X-ray reflectivity patterns of 20 layers of PbSe on 4 layers of VSe₂ (20:4) and 82 layers of PbSe on 8 layers of VSe₂ (82:8) films as deposited (black) and annealed at 300 °C for 30 min (red). The thicker (82:8) film has weak non-00l reflections implying that there is a small fraction of randomly oriented grains. Annealed film samples have very strong 00l reflections indicating crystallographic alignment to the substrate due to the presence of the intervening layers of smooth VSe₂. The films are exceptionally smooth compared to PbSe on SiO₂ because the Kiessig fringes extend to higher angles.

between constituents at the interfaces is strong make it difficult to theoretically predict which misfit layer compounds might be thermodynamically stable or at least local free energy minima in the free energy landscape. Figure 5 compares grazing incidence in-plane diffraction patterns of PbSe on SiO₂, PbSe on VSe₂, and (PbSe)_{1.11}VSe₂ annealed at 300 °C for 30 min. The difference in the degree of crystallographic alignment reflects the strong interaction between the PbSe and VSe₂ layers. This suggests that comparing the degree of crystallographic alignment of films of A and B on SiO₂ or another convenient substrate with films of A on B (and/or B on A) may provide a way to quickly determine whether a misfit layer compound will form from a designed precursor with repeating layers of A and B. Since there are many possible potential combinations of constituents, such a fast screening test would save a significant amount of time.

4. Experimental Section

Thin film precursors consisting of elemental Pb (99.8%, Alfa Aesar), V (99.995%, Alfa Aesar), and Se (99.99%, Alfa Aesar) were deposited on silicon substrates with a native oxide layer using a low pressure ($<3 \times 10^{-7}$ torr) physical deposition chamber. The elements were evaporated by using Thermionics 6 kW electron beam guns or custom-built Knudsen effusion cells producing a plume of atoms that deposit on the substrate. The thickness of each element deposited is controlled by opening and closing a pneumatic shutter that blocks the plume for a

specific time. The deposition time is determined by the measured Angstrom thickness by a quartz crystal microbalance and custom-made LabView software. Crystallization of the films was accomplished by annealing the precursor in an inert N₂ atmosphere (O₂ < 0.8 ppm).

Out-of-plane specular diffraction and X-ray reflectivity were collected using a Bruker D8 Discover diffractometer to determine the lattice parameters and degree of crystallographic alignment of the film constituents. Grazing incidence in-plane diffraction was collected on a Rigaku Smartlab diffractometer to assess the crystallized constituents in the film. X-ray fluorescence measurements were taken using a Rigaku Primus II ZSX spectrometer to determine the elemental composition of the films.

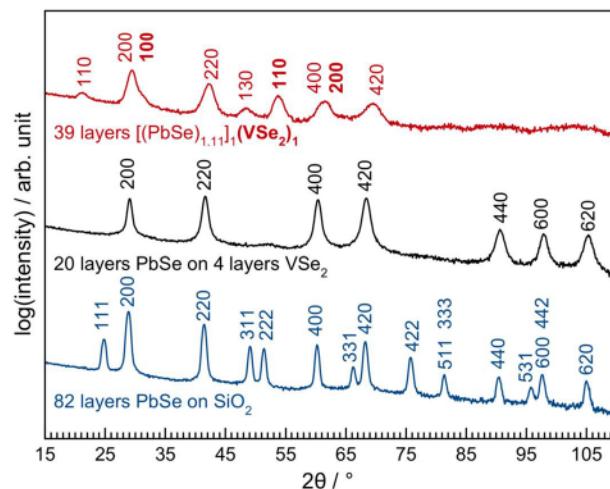


Figure 5. Grazing incidence in-plane diffraction patterns of 82 layers of PbSe on SiO₂, 20 layers of PbSe on 4 layers VSe₂, and [(PbSe)_{1.11}]₁(VSe₂)₁. The PbSe films have reflections that can be indexed to rock salt PbSe. All possible hkl reflections are observed in PbSe in SiO₂ film, indicating that the grains are randomly oriented. The absence of hkl reflections in the 20:4 film indicates that grains are parallel to the substrate.

Table 8. Lattice constants calculated from specular X-ray diffraction PbSe on VSe₂. AD = as deposited, AN = annealed.

# Layers PbSe:VSe ₂		a-Lattice parameter [Å]
20:4	AD	6.13 (1)
	AN	6.12 (1)
82:8	AD	6.12 (1)
	AN	6.11 (1)

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

crystallographic alignment, interfaces, misfit layer compound, PbSe

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