Atomically thin resonant tunnel diodes built from synthetic van der Waals heterostructures

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Vertical integration of two-dimensional van der Waals materials is predicted to lead to novel electronic and optical properties not found in the constituent layers. Here, we present the direct synthesis of two unique, atomically thin, multi-junction heterostructures by combining graphene with the monolayer transition-metal dichalcogenides: molybdenum disulfide (MoS2), molybdenum diselenide (MoSe2) and tungsten diselenide (WSe2). The realization of MoS2–WSe2–graphene and WSe2–MoS2–graphene heterostructures leads to resonant tunnelling in an atomically thin stack with spectrally narrow, room temperature negative differential resistance characteristics.
Resonant tunnelling of charge carriers between two spatially separated quantum layers can lead to a unique current transport phenomenon known as negative differential resistance (NDR)\(^1\,^2\). This is a key feature for novel nanoelectronic circuits that utilize bistability and positive feedback, such as novel memories, multi-valued logic, inductor-free compact oscillators and many other not-yet-realized electronic applications\(^3\,^4\). However, realizing spectrally narrow NDR in a resonant tunnelling diode (RTD) at room temperature has been challenging due to carrier scattering related to interfacial imperfections, which are unavoidable in traditional semiconductor heterostructures synthesized using advanced epitaxial growth techniques\(^5\). Two-dimensional (2D) materials\(^6\,^7\), with no out-of-plane chemical bonding and pristine interfaces, presents an appealing alternative to traditional semiconductors, and could ultimately eliminate the interfacial imperfections that limit room temperature NDR performance to date. Since 2004 (ref. 6), the overwhelming majority of electronic transport and stacked in 2D materials has been reported using mechanically exfoliated flakes\(^8\). Recently, there has been a concerted effort to directly synthesize layered transition-metal dichalcogenides (TMDs), with powder vapourization\(^9\,^10\), and tungsten disulphide (W\(_x\)S\(_y\)) powders for the powder vapourization route 24, and tungsten hexacarbonyl (W(CO)\(_6\)) and dimethylselenium ((CH\(_3\))\(_2\)Se) for the MOCVD route\(^25\). Molybdenum disulphide is grown via vapourization of molybdenum trioxide (MoO\(_3\)) and sulfur\(^10\). The heterostructure synthesis process is summarized in Fig. 1. The first TMD layer of the heterostructure, WSe\(_2\) or MoS\(_2\), is grown on tri-layer EG (Fig. 1a) at 950 °C and 750 °C for WSe\(_2–\)EG (Fig. 1b) and MoS\(_2–\)EG (Fig. 1c,d), respectively. Following this first TMD growth step, the surface coverage of the WSe\(_2\) or MoS\(_2\) on EG is typically >60%, with a lateral size of 2 μm and 300 nm for WSe\(_2\) and MoS\(_2\), respectively. Subsequently, the MoS\(_2–\)WSe\(_2–\)EG vertical heterostructure is created via a second \textit{ex situ} growth of MoS\(_2\) on WSe\(_2–\)EG at 750 °C (Fig. 1c). Similar to our previous work\(^26\), we find that wrinkles in the graphene as well as defects and edges within the WSe\(_2\) promote vertical growth of the MoS\(_2\), and ML MoS\(_2\)/WSe\(_2\) is primarily achieved in pristine regions of WSe\(_2\) (Fig. 1c and Supplementary

Results

Formation of vertical vdW heterostructures. The heterostructure is achieved by sequentially growing two dissimilar TMD MLs on multilayer (three layers) epitaxial graphene (EG) (Fig. 1a)\(^\text{23}\). The individual TMD layers are grown \textit{ex situ} via powder vaporization or MOCVD. Tungsten diselenide (WSe\(_2\)) is synthesized using both routes: tungsten trioxide (WO\(_3\)) and selenium (Se) powders for the powder vapourization route\(^24\), and tungsten hexacarbonyl (W(CO)\(_6\)) and dimethylselenium ((CH\(_3\))\(_2\)Se) for the MOCVD route\(^25\). Molybdenum disulphide is grown via vapourization of molybdenum trioxide (MoO\(_3\)) and sulfur\(^10\). The heterostructure synthesis process is summarized in Fig. 1. The first TMD layer of the heterostructure, WSe\(_2\) or MoS\(_2\), is grown on tri-layer EG (Fig. 1a) at 950 °C and 750 °C for WSe\(_2–\)EG (Fig. 1b) and MoS\(_2–\)EG (Fig. 1c,d), respectively. Following this first TMD growth step, the surface coverage of the WSe\(_2\) or MoS\(_2\) on EG is typically >60%, with a lateral size of 2 μm and 300 nm for WSe\(_2\) and MoS\(_2\), respectively. Subsequently, the MoS\(_2–\)WSe\(_2–\)EG vertical heterostructure is created via a second \textit{ex situ} growth of MoS\(_2\) on WSe\(_2–\)EG at 750 °C (Fig. 1c). Similar to our previous work\(^26\), we find that wrinkles in the graphene as well as defects and edges within the WSe\(_2\) promote vertical growth of the MoS\(_2\), and ML MoS\(_2\)/WSe\(_2\) is primarily achieved in pristine regions of WSe\(_2\) (Fig. 1c and Supplementary

Figure 1 | The formation of vdW heterostructures. MoS\(_2–\)WSe\(_2–\)EG vertical heterostructures begins with the synthesis of (a) 3L EG from SiC followed by (b) vapour transport or MOCVD of WSe\(_2\) and (c) vapour transport of MoS\(_2\). WSe\(_2–\)MoSe\(_2–\)EG heterostructures are similarly grown, except when (d) MoS\(_2\) is grown first on EG followed by (e) growth of the WSe\(_2\), a Se–S ion exchange occurs, leading to the formation of MoSe\(_2\) from the original MoS\(_2\) layer. The MoSe\(_2\) domains are difficult to topographically identify; however, (f) conductive AFM clearly delineates their location due to enhanced tunnelling at the heterostructures. Raman (g) indicates that preservation of the graphene has occurred during the synthesis process, and Scanning TEM (h,i) confirms that the stacked structures exhibit pristine interfaces, with no intermixing of Mo–W or S–Se after synthesis.
Figure 2 | Coupling in 2D vertical heterostructures. (a) The PL properties of MoS2–WSe2–EG and WSe2–MoSe2–EG reveal significant interlayer coupling, where the (b) MoS2–WSe2–EG and WSe2–MoSe2–EG exhibit the intrinsic PL peaks corresponding to MoS2, MoSe2 and WSe2, and also exhibit interband PL peaks at 1.59 and 1.36 eV, where the excitation wavelength (\( \lambda \)) is 488 nm and 633 nm for MoS2–WSe2–EG and WSe2–MoSe2–EG, respectively. (c) The moiré patterns acquired via AFM in MoS2 on WSe2 indicates an alignment of nearly either 0° or 180° between the top and bottom layer, and (d) STM confirms the moiré pattern with a lattice constant equal to (9.8 ± 0.4) nm. This structure can be reproduced theoretically (e) when the misorientation angle between these layers is ~1.9°. The continuity of the Moiré pattern is interrupted by the formation of a grain boundary and point defects, as indicated in the STM image. (f,g) STS on MoS2–WSe2–EG, WSe2–EG and EG (g, inset) provide evidence that the bandgap of the double junction heterostructure (MoS2–WSe2–EG) is smaller than that of the single-junction (WSe2–EG) heterostructure. The positions of conduction band minimum (CBM), valence band maximum (VBM), and quasi-particle bandgap (Eg) of WSe2 on EG and bilayer on EG are marked.

Fig. 1a[26]. The formation of the WSe2–MoSe2–EG heterojunction occurs during growth of WSe2 on MoS2. During the synthesis, a selenium–sulfur ion exchange occurs when the MoS2 is exposed to the selenium vapour just prior to the growth of WSe2 at 950 °C for 45 min[27]. Standard topographic characterization via atomic force microscopy (AFM) cannot clearly identify the location of the heterostructures (Fig. 1f); however, conductive AFM (CAFM) with platinum (Pt) tip[28] provides a means to map the WSe2–MoSe2–EG junctions and adjacent WSe2–EG regions due to a difference in heterostructure conductivity (Fig. 1f and Supplementary Fig. 1c,d).

Raman spectroscopy and transmission electron microscopy (TEM) confirm the formation of crystalline, vertical heterostructures (Fig. 1g–i and Supplementary Figs 1–3). A large fraction of the EG remains nearly defect free due to the sequence of TMD growths; however, there are regions of increased defectiveness due to either partial passivation of the graphene/SiC buffer layer[23] or formation of thick TMD layers[29]. Raman spectroscopy (see Supplementary Fig. 3) also confirms presence of significant fractions of ML WSe2 (\( E_{2g}/A_{1g} \) at 250 cm\(^{-1}\) and 2LA at 263 cm\(^{-1}\))[24] and MoS2 (\( E_{2g} \) at 383 cm\(^{-1}\) and \( A_{1g} \) at 404 cm\(^{-1}\))[26], as well as ML MoSe2 (\( A_{1g} \) at 240 cm\(^{-1}\) and \( E_{1g} \) at 284 cm\(^{-1}\))[27]. X-ray photoelectron spectroscopy (see Supplementary Fig. 4 and Supplementary Table 1) also corroborates the absence of any interaction between the two TMDs or graphene, and indicates that the MoS2 exhibits an n-type behaviour, while the WSe2 layer shows a p-type behaviour. Scanning TEM (Fig. 1h,i) also verifies the heterostructure is not a manifestation of the alloying of the constituent TMDs, but indeed are unique layers with pristine interfaces with atomic precision. In the case of MoS2–WSe2–EG, we have focused on a multilayer region of MoS2–WSe2 to ensure pristine layer formation beyond the ML structure (see Supplementary Fig. 2); however, all electrical characterization presented later is on ML heterostructures. The clean interface between MLs can be observed easily using high resolution scanning TEM. The WSe2–MoSe2–EG ordering is confirmed by comparing the intensity with that of bilayer WSe2–EG due to the similar atomic number between W and Mo atom (see Supplementary Fig. 2). Unlike vertical heterostructures based on a single chalcogen (that is, MoS2/WSe2[23]), the ordered layering does not occur when we attempt to grow a vertical structure based on heterogeneous layers where \( M_1 \neq M_2 \) and \( X_1 \neq X_2 \) (\( M = Mo, W; X = S, Se \)) on ‘3D’ substrates such as sapphire or SiO2 (see Supplementary Fig. 5). Instead, all attempts to grow such a structure results in alloying or lateral heterostructures of the layers. Therefore, we hypothesize that EG plays a critical role in the formation of atomically precise vdW heterostructures, where \( M_1 \neq M_2 \) and \( X_1 \neq X_2 \) by providing an atomically smooth surface that is free of dangling bonds, enabling mobility on the surface for TMD layer growth. Sapphire and SiO2 surfaces exhibit high surface roughness, dangling bonds, and are therefore more likely to impede surface diffusion, which catalyzes the alloying process.
Excited holes in MoS$_2$ (MoSe$_2$) valence band transfer to the conduction band of MoS$_2$ (MoSe$_2$) and the band transfer to the WSe$_2$ at 1.65 eV)\(^{30}\); therefore, photoluminescence (PL) spectroscopy (Fig. 2a, b) can provide evidence of electronic coupling between the layers. In addition to the typical PL spectra of the heterostructures exhibit the presence of interlayer excitons at 1.59 eV for MoS$_2$–WSe$_2$–EG and 1.36 eV for WSe$_2$–MoSe$_2$–EG (see Fig. 2d). While the mechanical stacking technique leads to a variety of rotation angles between layers\(^{30}\), the direct growth of vdW layers using our approach appears to have a strict rotational alignment, which may be critical for achieving optimal coupling between the layers\(^{33,34}\).

Scanning tunnelling spectroscopy further provides evidence that the quasi-particle bandgap of MoS$_2$–WSe$_2$–EG is significantly smaller than its WSe$_2$–EG counterpart (Fig. 2f, g, and Supplementary Fig. 6). Based on STS, we infer that, for WSe$_2$–EG, the conduction band minimum is at a sample bias of 0.34 ± 0.03 V and valence band maximum at −1.31 ± 0.03 V, indicating a quasi-particle interlayer $E_g$ of 1.65 eV ± 0.02 V, which is slightly larger than its interlayer $E_g$ at 1.59 eV (Fig. 2d) but smaller than the $E_g$ at 1.90 eV in 1L MoS$_2$–EG\(^{22,31}\). Mapping the tunnel current density of WSe$_2$–EG and WSe$_2$–MoSe$_2$–EG heterostructures via CAFM\(^{28,36}\) (Fig. 1f and Supplementary Fig. 1) provides strong evidence that tunnelling is much more readily achieved in WSe$_2$–MoSe$_2$–EG at a tip bias of 1.5 V, indicating a smaller, resonance tunnelling or both may be occurring. Finally, we note that defects, such as grain boundaries and vacancies disrupt the continuity of the Moiré pattern, further emphasizing that imperfections in layers or the interface will significantly impact the electronic behaviour of vdW heterostructures (Fig. 2d).

### Interlayer coupling

ML-semiconducting TMDs exhibit a direct optical bandgap ($E_{\text{opt}}$) at 1.8 ~ 1.9 eV, MoSe$_2$ at 1.55 eV, and WSe$_2$ at 1.6 ~ 1.65 eV)\(^{30}\); therefore, photoluminescence (PL) spectroscopy (Fig. 2a, b) can provide evidence of electronic coupling between the layers. In addition to the typical PL peaks from the direct bandgap transition within the individual layers, the PL spectra of the heterostructures exhibit the presence of interlayer excitons at 1.59 eV for MoS$_2$–WSe$_2$–EG and 1.36 eV for WSe$_2$–MoSe$_2$–EG (see Fig. 2a, b). In this case, the MoS$_2$–WSe$_2$ and WSe$_2$–MoSe$_2$ junctions exhibit type II band alignment\(^{15,20,21,31}\), where electrons in the WSe$_2$ conduction band transfer to the conduction band of MoS$_2$ (MoSe$_2$) and the excited holes in MoS$_2$ (MoSe$_2$) valence band transfer to the valence band of WSe$_2$. Consistent with manually stacked heterojunctions\(^{20,21}\), the position of the PL peak is due to interlayer exciton recombination, which confirms the electronic coupling at the heterojunction between the two ML TMDs.

Additional evidence of coupling comes from the topographical information of the heterostructures. Similar to graphene–hBN heterostructures\(^{32}\), Moiré patterns of MoS$_2$–WSe$_2$ are observed in tapping-mode AFM, which are qualitatively consistent with rotation angles of ~0 or 180° between MoS$_2$ and WSe$_2$. Furthermore, scanning tunnelling microscopy/spectroscopy (Fig. 2d) confirms the presence of a Moiré pattern produced by the misorientation of MoS$_2$ relative to the underlying WSe$_2$ layer. The lattice constant of the Moiré pattern is 9.8 ± 0.4 nm, which corresponds to a misorientation angle of ~1.9°. Modelling the heterostructure with this misorientation produces a consistent Moiré pattern, with a slightly smaller lattice constant of 9.6 nm (Fig. 2e). While the mechanical stacking technique leads to a variety of rotation angles between layers\(^{20}\), the direct growth of vdW layers using our approach appears to have a strict rotational alignment, which may be critical for achieving optimal coupling between the layers\(^{33,34}\).

### Discussion

We have demonstrated the direct synthesis of unique multi-junction heterostructures based on graphene (EG on SiC), MoS$_2$, MoSe$_2$, and WSe$_2$ that yields pristine interlayer gaps and leads to the first demonstration of resonant tunnelling in a atomically thin synthetic stack with the spectrally narrowest room temperature NDR characteristics. Importantly, this work indicates that NDR at room temperature only occurs in TMD heterostructures with truly pristine interfaces, which has been recently corroborated with manually stacked heterostructures where NDR is only evident at liquid nitrogen temperatures\(^{20,38,39}\). This is due to...
resonant tunnelling being highly sensitive to interfacial perturbations such as defects or ‘residue’ from the transfer process, emphasizing the importance of direct synthesis of multi-junction TMD heterostructures for vertical quantum electronics applications. Interestingly, the room temperature full width at half maximum of the NDR in this work is more spectrally narrow than their ‘3D’ semiconductor counterparts (silicon, germanium, III–V) and manually stacked graphene–boron nitride–graphene (Gr–hBN–Gr) heterostructures (Fig. 3b and Supplementary Note 7).49–52 This suggests that the interface of the directly grown vdW heterostructures is superior to that of many previously reported RTD structures.

Methods

EG synthesized from 6H-SiC. Graphene is synthesized on 1 cm² squares of 6H-SiC (0001) in a graphite crucible.6 The 6H-SiC substrate was annealed in H₂ at 1,500°C for 10 min to clean substrate surface prior to graphene growth. At this stage the chamber pressure is 700 torr under a H₂ (50 s.c.c.m)/Ar (450 s.c.c.m) flow. After H₂ annealing, the system temperature cooled to 850°C and pumped/purged with ultra-high pure N₂ at least six times to remove H₂ gas. Subsequently the chamber is filled in Ar gas (500 s.c.c.m.) to 200 torr. The chamber temperature is then heated up to 1,030°C at 100°C/min.38 and dwelled at this temperature for 20 min to grow three layers of graphene within the terraces of substrates via sublimation of silicon on the silicon side of 6H-SiC (0001). The system cooled down naturally to room temperature after the growth.

MoS₂–WSe₂–EG synthesis. WSe₂ can be grown on EG either via vapour phase reaction of WO₃ and Se powders or via MOCVD.44–47 The vapour phase reaction utilizes the vapourization of WO₃ powders in a ceramic boat placed at the centre of a horizontal hot wall reactor with a flow of H₂ (10 s.c.c.m.)/Ar (90 s.c.c.m.). The EG substrates for WSe₂ growth were placed at the downstream side of the tube and heated to 925°C at 25°C/min. Samples were held at 925°C for 15 min and then cooled naturally to room temperature. The total pressure throughout the reaction is held at 700 torr. Utilizing MOCVD, WSe₂ was synthesized in a vertical cold wall reactor using W(CO)₆ and (CH₄)₂Se precursors. The metallic organic precursors were transported into the reactor by carrier gas of 100°C H₂ via a bubbler manifold that allows for controlling each precursor concentration independently. The (CH₃)₂Se and W(CO)₆ were held at temperatures of 22°C and 25°C, respectively, and a pressure of 700 torr. The Se to W ratio was fixed at 20:00. The MOCVD growth of WSe₂ took place at 890°C with a total pressure of 700 torr. The growth time varied between 15 and 30 min. After the completion of WSe₂–EG synthesis, the ex situ MoS₂ growth via the vapour phase reaction using MoO₃ and S powders was carried out in a horizontal hot wall tube reactor at 700°C. During the MoS₂ growth, MoO₃ powder in a ceramic boat placed at the centre of heating zone were heated at 750°C for 10 min. After the MoS₂ growth, the reactor cooled down to room temperature naturally.

MoS₂–MoSe₂–EG synthesis. In this case, the processes are similar, but steps reversed. The MoS₂ is grown first, followed by an ex situ WSe₂ growth via the vapour phase reaction of WO₃ and Se. A Se–S ion exchange occurs in the MoS₂ converting the MoS₂ into MoSe₂.27 Subsequently, the WSe₂ layers grow on MoSe₂–EG as the hot zone is held at 950°C for 45 min, resulting in MoS₂–WSe₂–EG heterostructures.

Sample characterization. The details of characterization performed on the heterostructures can be found in the Supplementary Note 1.

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**Author contributions**

J.A.R. and Y.-C.L. conceived the idea, and J.A.R., S.D., R.M.W., M.J.K. and L.-J.L. directed the research. Y.-C.L., M.-Y.L. and S.M.E. synthesized the heterostructures. Y.-C.L. carried out AFM, Raman, photoluminescence and CAFM measurements. R.A. carried out STM/STS; H.Z. and X.P. carried out XPS; N.L. carried out TEM; and R.K.G. carried out the modelling. Y.-C.L. and J.A.R. wrote the paper with significant inputs from all authors. All authors have read and approved the manuscript. All authors participated in the analysis of the data and discussed the results.

**Additional information**

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