LAYER TRANSFER OF TWO-DIMENSIONAL MATERIALS AND SEMICONDUCTORS FOR NEXT-GENERATION FLEXIBLE AND HETEROSTRUCTURE-BASED ELECTRONICS

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by

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This thesis is dedicated to

My Parents

For

Their Love and Support

Certificate

This is to certify that the research work presented in this dissertation entitled "**Layer Transfer of Two-Dimensional Materials and Semiconductors for Next-Generation Flexible and Heterostructure-based Electronics**" is being submitted by **Mr. Madan Sharma** for the award of the degree of **Doctor of Philosophy** to Indian Institute of Technology Delhi. The work embodied in this dissertation is a record of bona fide research carried out by him under my guidance and supervision. This thesis to the best of my knowledge does not contain any results which have been submitted in part or full for a degree or diploma at any other Institute or University.

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Abstract

This research has studied the optimization of novel layer transfer methods for two-dimensional (2D) materials, mainly transition metal dichalcogenides (TMDCs). It is now widely accepted that 2D materials are the future hope for replacing silicon CMOS technology due to their exceptional multifunctional properties such as high optical absorption and transparency, superior mobility, high surface to volume ratio, tunable bandgap, mechanical compliance, absence of dangling bonds and so on. 2D materials alleviate the limitations of traditional Si technology, such as high leakage current, short channel effect, etc. Earlier, 2D material-based electronic, optoelectronic, twistronic, flextronic, and biomimetic devices were fabricated on micromechanically exfoliated single crystal flakes, but large-area growth techniques like chemical vapor deposition (CVD), metal-organic CVD (MOCVD), molecular-beam epitaxy (MBE), and physical vapor deposition (PVD) have made steady progress in recent years. However, the direct growth of 2D materials on the substrates that are desirable for commercial applications is frequently constrained by the requirement of high growth temperatures, chemically active growth precursors and promoters, and the requirement for epitaxy. This has led to the development of layer transfer methods for transferring 2D materials with varying degrees of cleanliness and homogeneity. Layer transfer requires optimization to minimize the damage of transferred film and ensure clean interfaces.

We first investigated the limitations of existing transfer methods and then optimized a water-soluble layer-based transfer (WSLT) process that yields clean, damage-and wrinkle-free large-area transferred 2D films. WSLT process outperforms conventional transfer methods in maintaining the structural integrity and uniformity of the as-grown film after transfer. Although the WSLT technique is superior to conventional methods, it still uses PMMA and chemical etchant (NaOH), which could contaminate the transferred film and reduce device performance.

Therefore, we developed a novel transfer process named the quasi-dry layer transfer method, free from PMMA residues and chemical etchants. Furthermore, the compatibility of this transfer method with the nanoscale device fabrication technology is demonstrated by fabricating 2D heterostructures and flexible broadband photodetectors. We fabricated 2D/2D (MoS_2/WS_2 and vice versa) and 2D/3D (MoS_2/β-Ga_2O_3) *van der Waals* (vdW) heterostructures. In MoS_2/β-Ga_2O_3 heterostructure, we observed a straddling (type I) band alignment, with a valence band offset (VBO) of 2.28 eV and a conduction band offset (CBO) of 0.73 eV.

We also fabricated MoS_2/mica and MoS_2/β-Ga_2O_3/mica flexible photodetectors and studied their performance with bending/tensile strain and temperature. The responsivity and detectivity of the MoS_2/mica flexible photodetector were found to be 1.10 mA/W and 3.86×10^{10} Jones, respectively. The PDCR, responsivity, and detectivity of the MoS_2/β-Ga_2O_3/mica flexible photodetector were calculated to be 10^3, 7.21 mA/W, and 2.4×10^{11} Jones, respectively. The photocurrent and responsivity were increased by 155% and 136% under 0.61% tensile strain. Additionally, no noticeable change in the photocurrent was observed even after 500 bending cycles, revealing excellent flexibility and robustness of the device. The device was also thermally stable at high temperatures up to 125℃. We also studied the blistering kinetics in 4H-SiC and optimized a two-step annealing process for large-area exfoliation for potential layer transfer applications. This research will appeal to communities interested in manufacturing devices based on 2D materials for electronics, optoelectronics, flexible electronics, straintronics, spintronics, twistronics, radiation-tolerant electronics, bio-inspired electronics, and edge sensors for internet of things (IoT) devices. This work will also be interesting to scientists who want to use 2D materials with different platforms such as Si augmentation/replacement, the Internet of Things, 2D/2D or 2D/3D heterostructures, etc.

सार

इस शोध ने दो-आयामी (2डी) पदार्थ, मुख्य रूप से संक्रमण धातु डाइक्लोजेनाइड्स (टीएमडीसी), के लिए नई परत हस्तांतरण विधियों के अनुकूलन का अध्ययन किया है। अब यह व्यापक रूप से स्वीकार कर लिया गया है कि 2डी पदार्थ सिलिकॉन सीएमओएस प्रौद्योगिकी को बदलने के लिए भविष्य की उम्मीद है क्योंकि उनमें असाधारण बहुआयामी गुण होते है जैसे कि उच्च ऑप्टिकल अवशोषण और पारदर्शिता, बेहतर गतिशीलता, सतह से वॉल्यूम का उच्च अनुपात, ट्यून करने योग्य बैंडगैप, यांत्रिक अनुपालन, डेंगलिंग बंधन की अनुपस्थिति और भी बहुत सारे। 2 डी पदार्थ पारंपरिक Si प्रौद्योगिकी की सीमाओं को कम करते हैं जैसे कि उच्च रिसाव चालू, लघु चैनल प्रभाव, आदि। इससे पहले, 2डी पदार्थ आधारित इलेक्ट्रॉनिक, ऑप्टोइलेक्ट्रॉनिक, ट्विस्ट्रोनिक, फ्लेक्सट्रॉनिक और बायोमिमेटिक डिवाइस माइक्रोमैकेनिकल एक्सफ़ोलीएटेड सिंगल क्रिस्टल फ्लेक्स पर बनाये गए थे, लेकिन रासायनिक वाष्प जमाव (सीवीडी), धातु-कार्बनिक सीवीडी (एमओसीवीडी), आणविक-बीम एपिटैक्सी (एमबीई), और भौतिक वाष्प जमाव (पीवीडी) जैसी तकनीकों ने हाल के वर्षों में लगातार प्रगति की है जिनसे 2डी पदार्थ की बड़ी परतें बनाई जा सकती हैं। हालांकि, वाणिज्यिक अनुप्रयोगों के लिए वांछनीय सबस्ट्रेट्स पर 2 डी पदार्थ को बनाना अक्सर उच्च ग्रोथ तापमान, रासायनिक रूप से सक्रिय ग्रोथ प्रिकॉजर और प्रमोटरों की आवश्यकता और एपिटैक्सी की आवश्यकता से बाधित होता है। इससे 2डी पदार्थ को साफ-सफाई और एकरूपता की विभिन्न डिग्री के साथ स्थानांतरित करने के लिए परत हस्तांतरण विधियों का विकास हुआ है। स्थानांतरित फिल्म के नुकसान को कम करने और स्वच्छ इंटरफेस सुनिश्चित करने के लिए परत हस्तांतरण के अनुकूलन की आवश्यकता होती है।

हमने पहले मौजूदा स्थानांतरण विधियों की सीमाओं की जांच की और फिर एक पानी में घुलनशील परत-आधारित स्थानांतरण (WSLT) प्रक्रिया को अनुकूलित किया, जो स्वच्छ, क्षति-और शिकन-मुक्त बड़े क्षेत्र में स्थानांतरित 2D फिल्मों का उत्पादन करती है। WSLT प्रक्रिया स्थानांतरण के बाद विकसित फिल्म की संरचनात्मक अखंडता और एकरूपता को बनाए रखने में पारंपरिक हस्तांतरण विधियों से बेहतर प्रदर्शन करती है। हालांकि WSLT तकनीक पारंपरिक तरीकों से बेहतर है, फिर भी यह पीएमएमए और रासायनिक एचेंट (NaOH) का उपयोग करती है, जो स्थानांतरित फिल्म को दूषित कर सकती है और डिवाइस के प्रदर्शन को कम कर सकती है। इसलिए, हमने PMMA अवशेषों और रासायनिक एचेंट से मुक्त, अर्ध-शुष्क परत स्थानांतरण विधि नामक एक नई हस्तांतरण प्रक्रिया विकसित की। इसके अलावा, नैनोस्केल डिवाइस निर्माण तकनीक के साथ इस हस्तांतरण विधि की संगतता 2डी

हेटरोस्ट्रक्चर और लचीले ब्रॉडबैंड फोटोडेटेक्टर बनाकर प्रदर्शित किया गया है। हमने 2D/2D (MoS_2/WS2 और इसके विपरीत) और 2D/3D (MoS_2/β-Ga_2O_3) वैन डेर वाल्स (vdW) हेटरोस्ट्रक्चर तैयार किए। MoS_2/β-Ga_2O_3 हेटरोस्ट्रक्चर में हमने 2.28 eV के वैलेंस बैंड ऑफ़सेट (VBO) और 0.73 eV के कंडक्शन बैंड ऑफ़सेट (CBO) के साथ एक स्ट्रैडलिंग (टाइप I) बैंड अलाइनमेंट देखा।

हमने MoS_2/mica और MoS_2/β-Ga_2O_3/mica लचीले फोटोडेटेक्टर भी तैयार किए तथा: तन्यता तनाव और तापमान के साथ उनके प्रदर्शन का अध्ययन किया। MoS_2/mica लचीले फोटोडेटेक्टर की रिस्पोंसिविटी और डिटेक्टिविटी क्रमशः 1.10 mA/W और 3.86×10^{10} जोन्स पाई गई। MoS_2/β-Ga_2O_3/mica लचीले फोटोडेटेक्टर की PDCR, रिस्पोंसिविटी, और डिटेक्टिविटी क्रमशः 10^3, 7.21 mA/W, और 2.4×10^{11} जोन्स पाई गई। 0.61% तन्यता तनाव के तहत फोटो करंट और रिस्पोंसिविटी में 155% और 136% की वृद्धि हुई। इसके अतिरिक्त, 500 बैंडिंगचक्रों के बाद भी फोटो करंट में कोई उल्लेखनीय परिवर्तन नहीं देखा गया, जिससे डिवाइस के उत्कृष्ट लचीलेपन और मजबूती का पता चलता है। डिवाइस 125 डिग्री सेल्सियस तक के उच्च तापमान पर भी स्थिर था। हमने 4H-SiC में ब्लिस्टरिंग काईनेटीक्स का भी अध्ययन किया और बड़े क्षेत्र के एक्सफोलिएशन के लिए दो-चरणीय एनीलिंग प्रक्रिया को अनुकूलित किया। यह शोध इलेक्ट्रॉनिक्स, ऑप्टोइलेक्ट्रॉनिक्स, फ्लेक्सिबल इलेक्ट्रॉनिक्स, स्ट्रेनट्रॉनिक्स, स्पिंट्रोनिक्स, ट्विस्ट्रोनिक्स, रेडिएशन-टॉलरेंट इलेक्ट्रॉनिक्स, बायो-इंस्पायर्ड इलेक्ट्रॉनिक्स, और इंटरनेट ऑफ थिंग्स (IoT) उपकरणों के लिए एज सेंसर के लिए 2D पदार्थ पर आधारित उपकरणों के निर्माण में रुचि रखने वाले समुदायों के लिए रुचिकर होगा। यह काम उन वैज्ञानिकों के लिए भी दिलचस्प होगा जो 2डी पदार्थ का उपयोग विभिन्न प्लेटफार्मों जैसे कि Si संवर्द्धन/प्रतिस्थापन, इंटरनेट ऑफ थिंग्स, 2D/2D या 2D/3D हेटरोस्ट्रक्चर, आदि के साथ करना चाहते हैं।

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List of Symbols

a	Lattice parameter
Å	Angstrom
Ar	Argon
Cu	Copper
Ni	Nickel
GPa	Gigapascal
Au	Gold
Γ	Toughness
H	Hydrogen
Si	Silicon
Ge	Germanium
V	Voltage
cm/mm/μm/nm	Centimeter/millimeter/micrometer/nanometer
λ	Wavelength
θ	Angle
s	Second
ϕ	Work function
W	Watt
˚C	Celsius
K	Kelvin
S	Sulfur
M	Molar
Δω	Frequency difference
eV	Electron volt
d	Interplanar spacing

Na	Sodium
A	Ampere
Ag	Silver
R_λ	Responsivity
D^*	Detectivity
I_p	Photocurrent
I_d	Dark current
P_λ	Power density
A_{eff}	Effective area
α	Proportionality constant
β	Empirical constant
I_0	Steady state-current
τ	Relaxation time constant
ε	Tensile strain
t_s	Thickness of substrate
t_f	Thickness of film
R_c	Radius of curvature
η	t_f/t_s
Y	Young's modulus
R_0	Responsivity at zero strain
q	Charge
V_{CPD}	Contact potential difference
h	Plank's constant
ν	Frequency
E_g	Bandgap
ΔE_C	Conduction band offset
ΔE_V	Valence band offset
ϕ_{SBH}	Surface barrier

E_{VBM}	Valence band Maximum
E_C	Conduction band
E_F	Fermi level
Ti	Titanium
I_s	Dark saturation current
n	Ideality factor
k_B	Boltzmann constant
ϕ_B	Schottky barrier height
A^*	Effective Richardson constant
$\Delta R/R_0$	Relative responsivity
E_a	Activation energy
σ_0	Stress in implanted layer
σ_{xx}	Stress in the buckled layer
p_{int}	Internal pressure
p_{ext}	External pressure
Δp_c	Pressure variation
δ_c	Vertical deflection
E	Young's modulus

Abbreviations

CMOS	Complementary metal-oxide-semiconductor
SCE	Short-channel effect
TG-MOSFETs	Trapezoidal-formed gate MOS field effect transistor
UTB-FETs	Ultra-thin-body field-effect transistors
BiSFETs	Bilayer pseudospin field effect transistors
Gr-FET	Graphene field-effect transistor
TMDCs	Transition metal dichalcogenides
SEDNE	Silicene encapsulated delamination with native electrodes
DFT	Density functional theory
CBM	Conduction band minima
VBM	Valence band maxima
CVD	Chemical vapor deposition
PLD	Pulse laser deposition
rGO	Reduced graphene oxide
PMMA	Poly (methyl methacrylate)
DI	Deionised
HF	Hydrofluoric acid
CA	Cellulose acetate
BOE	Buffered oxide etch
PS	Polystyrene
TRT	Thermal release tape
PTAS	Perylene-3, 4, 9, 10-tetracarboxylic acid tetrapotassium salt
LRS	Layer-resolved splitting
RT	Room temperature
CMP	Chemical mechanical polishing
MEMS	Micro-electromechanical systems
SiC	Silicon carbide
SOI	Silicon-on-Insulator

vdW	Van der Waals
OM	Optical microscope
SEM	Scanning electron microscope
FESEM	Field Emission Scanning Electron Microscope
AFM	Atomic force microscopy
TEM	Transmission electron microscopy
CCD	Charged coupled device
SAED	Selected area electron diffraction
HRTEM	High resolution transmission electron microscopy
HRXRD	High resolution X- Ray Diffraction
PL	Photoluminescence
UV	Ultraviolet
Vis	Visible
XPS	X-ray photoelectron spectroscopy
WSLT	Water-soluble layer transfer
2D	Two-dimensional
3D	Three-dimensional
MoS_2	Molybdenum disulfide
WS_2	Tungsten disulfide
Ga_2O_3	Gallium oxide
1L	Monolayer
3L	Trilayer
SiO_2	Silicon dioxide
NaCl	Sodium chloride
GS	Growth-substrate
TS	Target-substrate
FWHM	Full-width half maxima
NaOH	Sodium hydroxide

PEN	Polyethylene naphthalate
PET	Polyethylene terephthalate
PI	Polyimide
PDMS	Polydimethylsiloxane
N_2	Nitrogen
PVDF	Polyvinylidene fluoride
a-MoS_2	As-grown MoS_2
t-MoS_2	Transferred MoS_2
PDCR	Photo-to-dark current ratio
IDEs	Interdigitated electrodes
MBE	Molecular beam epitaxy
EDX	Energy-dispersive X-ray spectroscopy
as-TH	As-transferred heterostructure
KPFM	Kelvin probe force microscopy
HOPG	Highly oriented pyrolytic graphite
BE	Binding energy
SBB	Surface band bending
DUV	Deep ultraviolet
TCO	Transparent conducting oxides
SBH	Schottky barrier height
FvK	Föppl-von Karman
VH	Hydrogen-defect complexes
SICOI	SiC on insulator
FEO	Future electronics and optoelectronics
AI	Artificial intelligence
IoT	Internet of Things