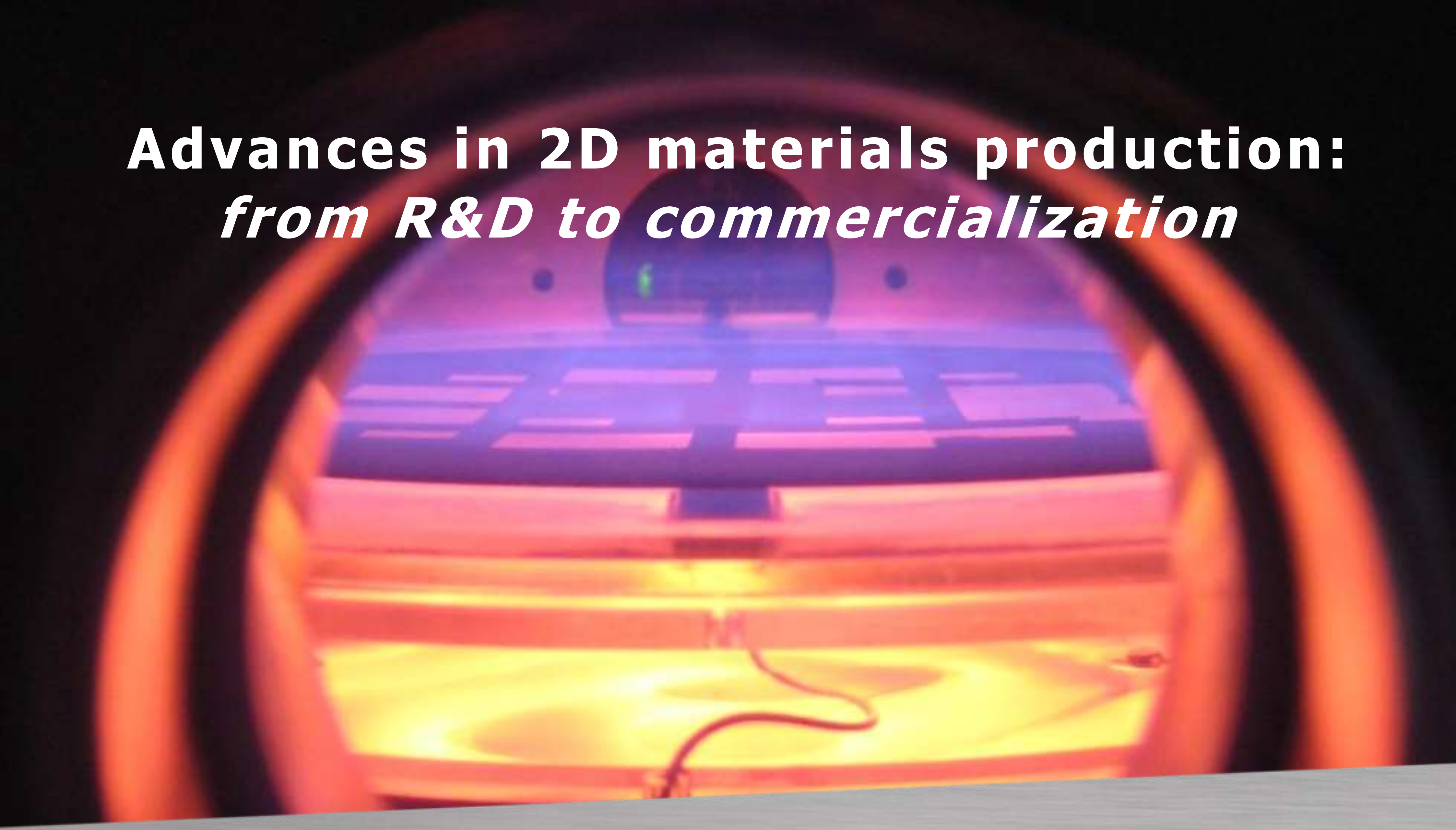
Advances in 2D materials production: *from R&D to commercialization*



Dr. Paul Wiper
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RPGR 2017

AIXTRON

Outline of Presentation

About us

Our Technologies and Products

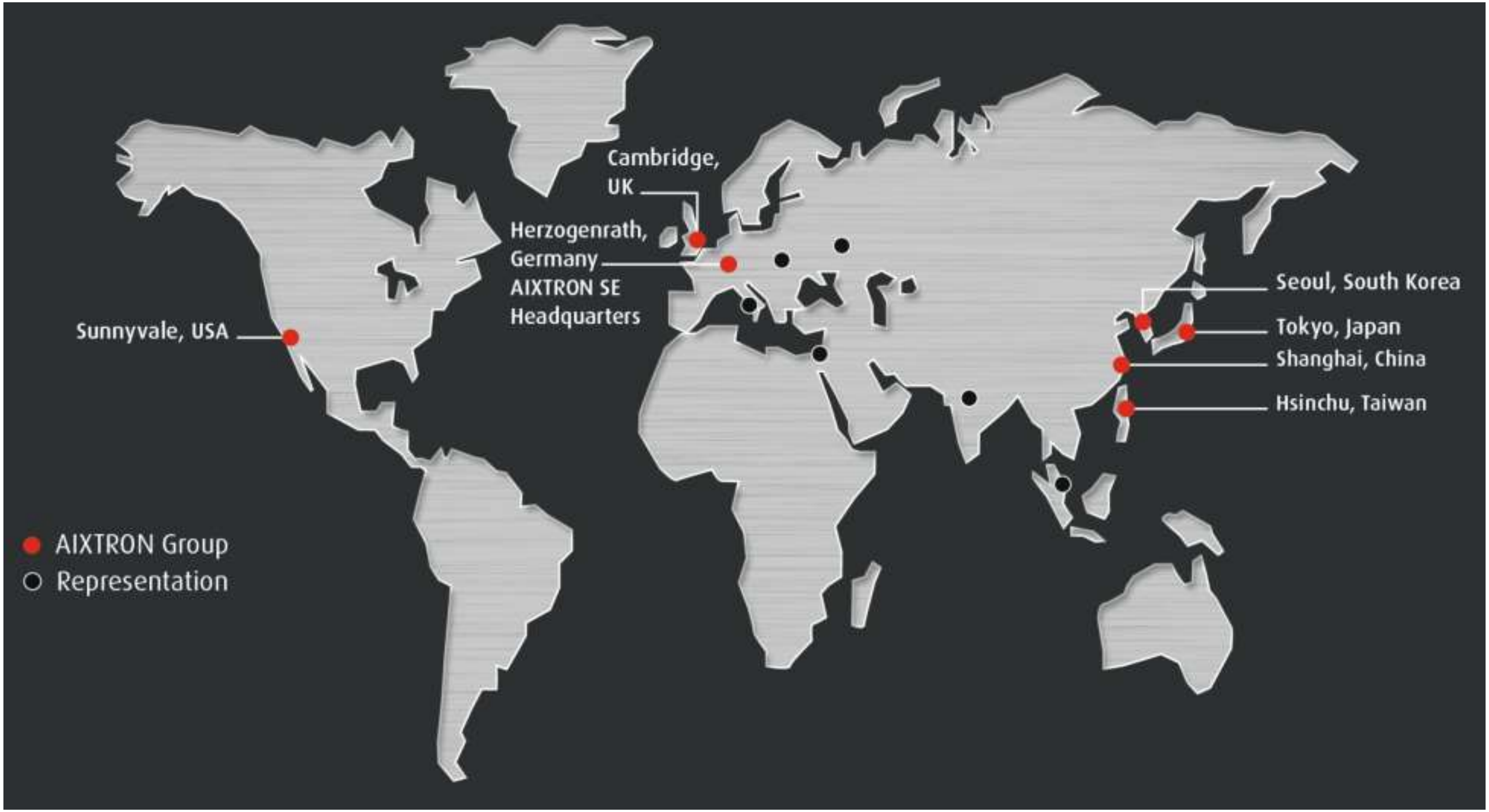
BM Graphene and CNT Product Lines

BM 2D Materials Product Lines

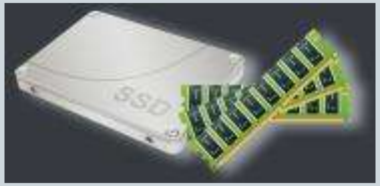
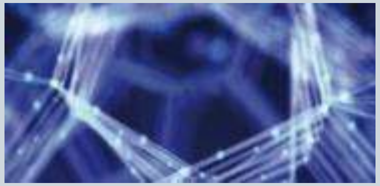
Who we are

- Headquarters based in Herzogenrath, Germany
- Worldwide presence with 13 sales/representatives offices and production facilities
- Company founded in 1983 – over 30 years of experience
- ~ 760 employees
- Technology leader in deposition systems
- More than 3,000 deposition systems delivered all over the world
- State of the art R&D center and demo facilities
- Annual R&D budget of approx. € 60 Million

Global Presence



Our system solutions address multiple key markets

Compound		Silicon	Organics	1D & 2D Materials
MOCVD		**ALD • MOCVD**	**OVPD® • PVPD™ • PECVD**	**CVD • PECVD**
Opto Electronics	**Power Electronics**	**Memory & Logic**	**Organic Electronics**	**Graphene, CNTs & 2D**
• LEDs for display: TVs, mobile phones, tablets, etc. • LEDs for lighting • LEDs for automotive • LEDs for data communication • Telecom lasers • Concentrator photovoltaics	• RF transistors • AC-DC converters • DC-DC converters • Solar inverters • Motor drives in industrial applications automotive and consumer electronics	• DRAM dielectric and metal electrode • Flash inter poly dielectric and metals • Logic gate stack • ReRAM and PCRAM active element and electrode • Logic high mobility channel	• OLEDs for display: TVs, mobile phones, tablets, etc. • OLEDs for lighting • Organic and flexible electronics • Organic photovoltaics	• Transistors • Interconnects • Flexible electronics • Energy storage • Sensors • Composites
				
AIX R6 (GaN) AIX G5 HT • AIX G5+ (GaN) AIX 2800G4-TM (As/P)	**AIX G5 HT • AIX G5+ (GaN) AIX G5 WW (SiC)**	**QXP 8300 ALD LYNX-iXP**	**R&D systems: OVPD®-200 • PRODOS-200 OEC-200 • OPTACAP-200 Production systems up to Gen 8.5**	**BM R&D • BM Pro BM Pro HT • BM GB BM 300T**

Carbon – PECVD

Graphene and Carbon Nanotube Deposition Systems

- Proprietary thermal and plasma enhanced chemical vapour deposition technology
- Excellent uniformity and reproducibility with fast turnaround cycle times
- BM platform: BM R&D (2-inch), BM Pro (4-inch and 6-inch), BM GB (4-inch glovebox), BM HT (high temperature, 1700°C), BM 300T (300 mm)
- Graphene and carbon nanotube films for electronics, energy storage, thermal management, sensors and flexible/transparent applications

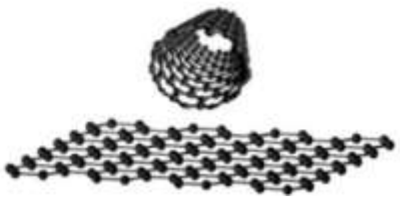
Product Features

- Fast response heater and turnaround
- Thermal CVD
- Substrate and top heating

- Closed loop infrared wafer temperature control
- Plasma enhanced CVD with frequency control
- Flexible processing for different applications

- Low cost of ownership
- Easy maintenance and cleaning
- User management features and growth library

Material Properties + AIXTRON Technology → Enabling Applications



Graphene (2D) and carbon nanotube (1D)

Unique combination of high electrical/ thermal conductivity, mobility, flexibility and transparency

Serving R&D market today

AIXTRON BM Pro

Production ready for tomorrow

AIXTRON BM 300T

BM product line: key technology for carbon deposition

Various patents granted/applied

- Showerhead innovation
- Uniform gas distribution
- Top and bottom heaters
- High temperature ramp rates
- High throughput and reproducibility
- Cold wall reactor design
- Simple user-interface

BM product line for graphene / CNT growth on foil

Towards large scale industrial applications

2011

R&D tool:
4-inch/6-inch

2017

Batch tool:
BM Pro 8”x8” foil

2018

R2R Production tool:
Demo tool on 300mm foil web width

BM product line: Carbon deposition systems portfolio

BM R&D
(2-inch)

BM Pro
(4 & 6-inch)

BM GB
(4-inch)

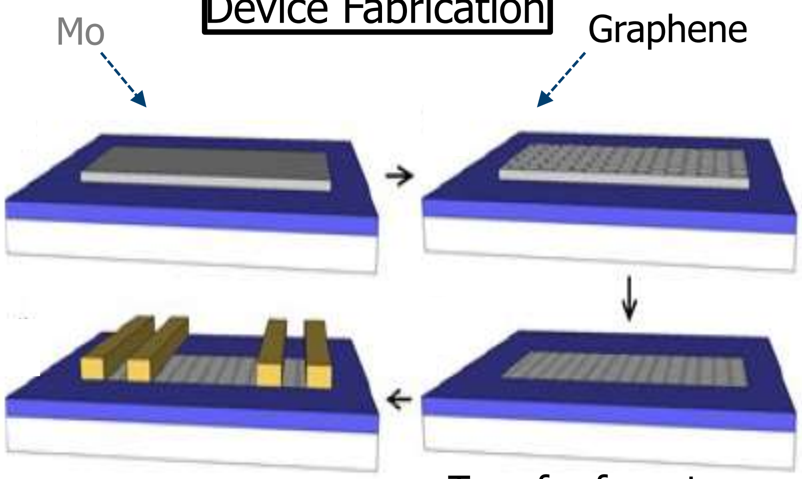
BM Foil
(2x8-inch)

BM 300T
(12-inch)

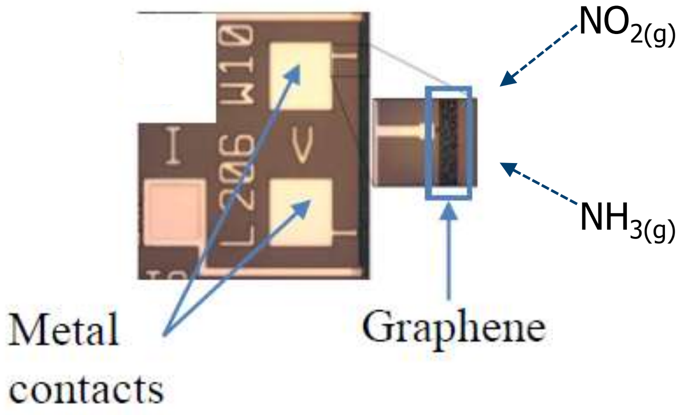
BM Pro 2, 4 & 6-inch Systems: *Academic and R&D Markets*

Gas sensors

Device Fabrication



Device & Gas Detection



Sensitivity of devices

	Sensitivity (ppm^{-1})	
	NO_2	NH_3
Device (10)	$1.9x10^{-2}$	$5.8x10^{-5}$
Device (5)	$2.4x10^{-2}$	$9.3x10^{-5}$

- Transfer free CVD graphene chemi-resistor gas sensing devices
- CMOS Mo-catalysts compatible

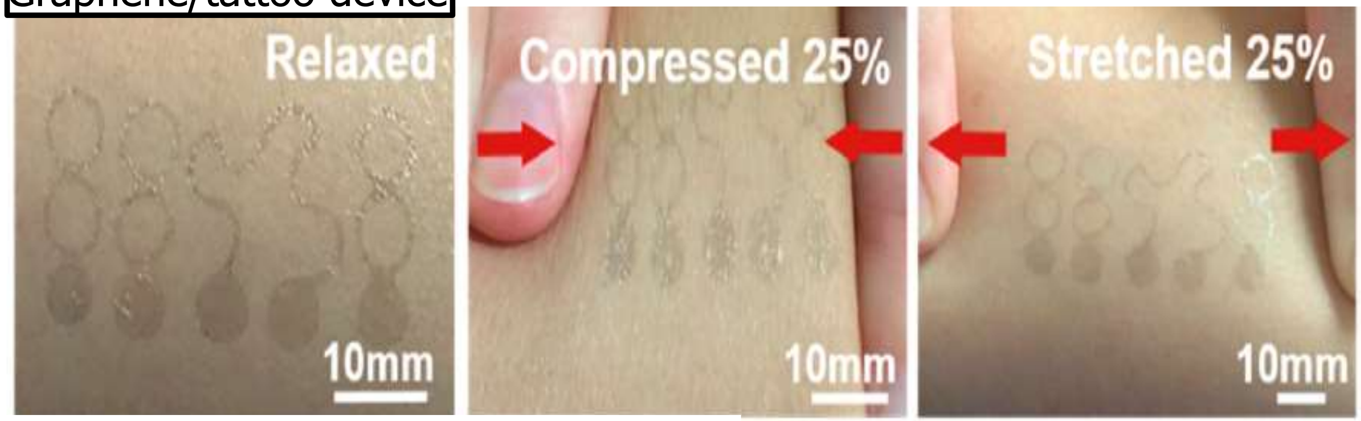
Courtesy of Sten Vollebregt, TU Delft
Conference Paper: IEEE Sensors 2016

BM Pro 2, 4 & 6-inch Systems: *Academic and R&D Markets*

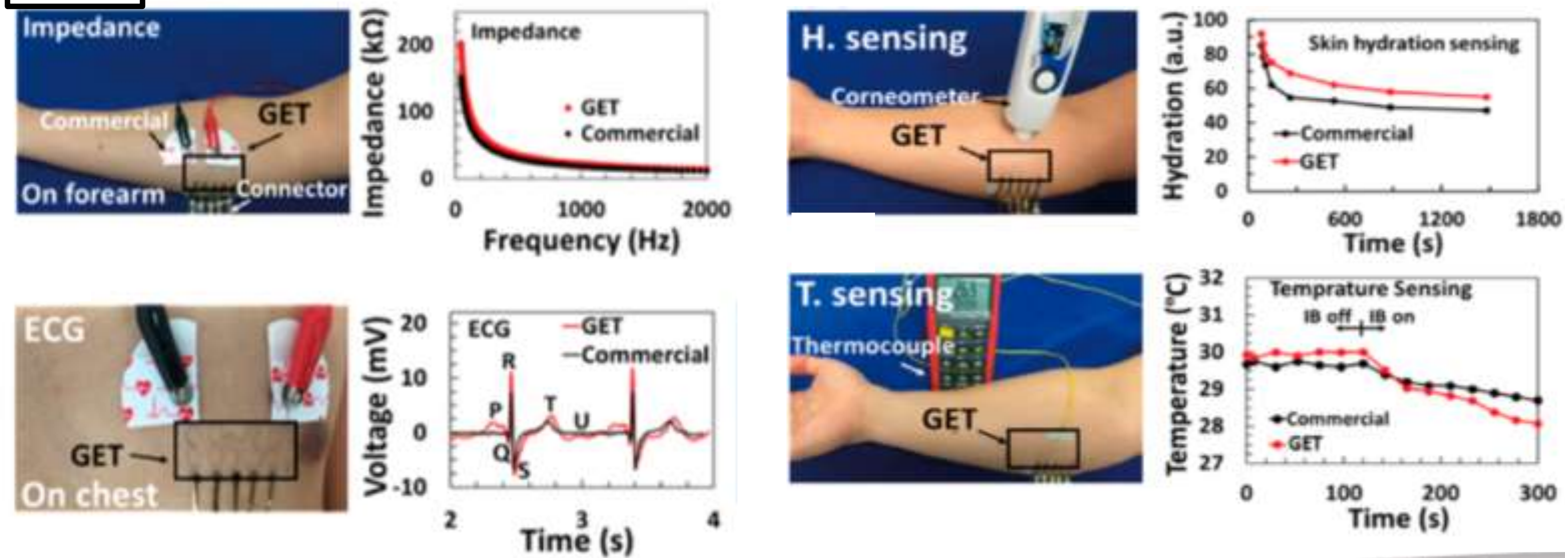
Graphene Tattoos

Courtesy of Deji Akinwande, UT Austin

Graphene/tattoo device



Results



ACS Nano, **2017**, 11 (8)

AIXTRON

BM product line: BM 300T: *Electronic & Optoelectronic Markets*

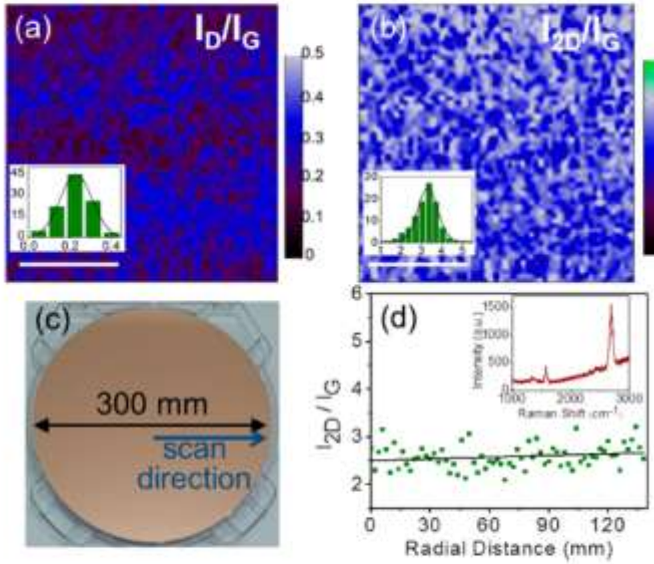
BM 300T – 12”
For your production needs

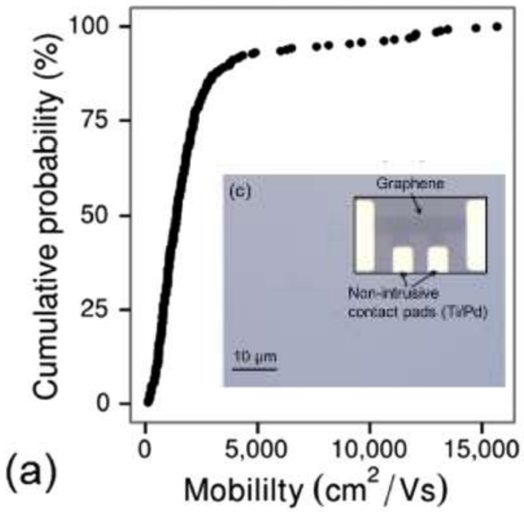
Customer results

Toward 300mm wafer process for graphene transistors

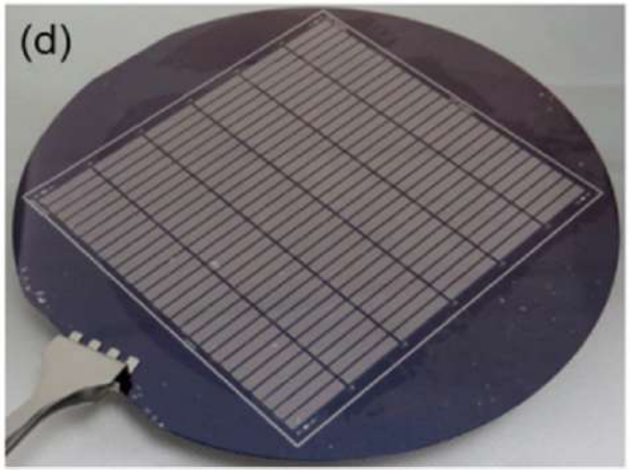
TABLE 1. Comparison of the Material and Electrical Properties of Reported Wafer-Scale Polycrystalline and Single-Crystalline Graphene

substrate/size (mm)	I_{2D}/I_G	I_D/I_G	μ_{max} (cm^2/(V s))	residual carrier density ($\times 10^{11}$ cm^{-2})	ref
Cu film/100—300	2.6—3.3	0.03—0.22	15 660	3.4—29	this work
Cu film/200	1.8	0.13	3800	1.49[d]	Gao, 2014[11]
Ge(110)[a]/50	3.5	0.03	10 600	3[d]	Lee, 2014[10]
SiC[a]/ 100	1.6—1.9	0[b]	2700	10—100	Kim, 2013[8]
Cu film/100	3	0.2	4900	10[d]	Tao, 2012[12]
Cu film/150	4.5	0.3	23 000[c]	10—40	Heo, 2011[13]
Ni/Cu films/75	3.5	0.25	3000	28[d]	Lee, 2010[9]





Rahimi et al, ACS Nano
DOI:10.1021/nn5038493



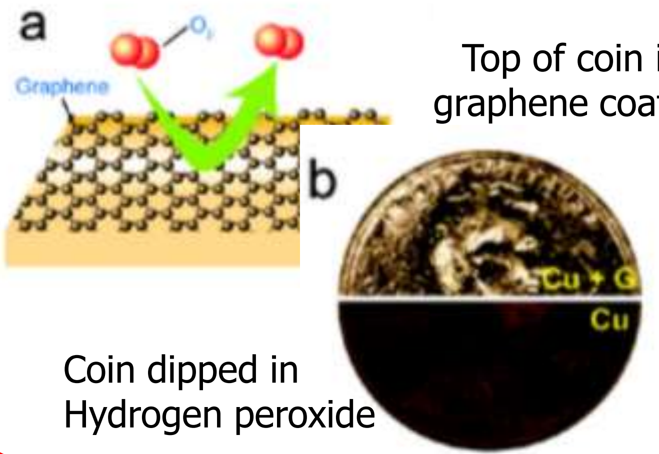
BM spider – Roll-to-Roll CVD system

BM Spider key technical features

- Independent annealing and growth zones
- Showerhead gas distribution
- Adjustable temperature up to 1000 C
- Non vacuum process
- Foils and/or sheets up to 100um thick and 300mm wide
- Auto webbing system
- Speeds up to 8m/hr (for carbon based materials)
- Small footprint (4.5m x1.6m)

BM Spider: R2R Graphene and CNT Coating Applications

Graphene for Corrosion & chemical protection



Top of coin is graphene coated

Coin dipped in Hydrogen peroxide

Energy storage

High performance material for batteries and supercapacitors

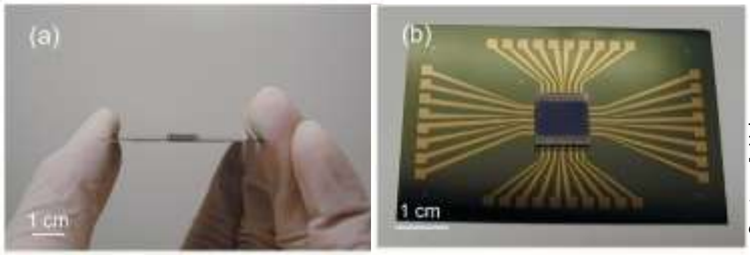
Source: greencarreports.com

CNT on foil

Graphene for Flexible electronics

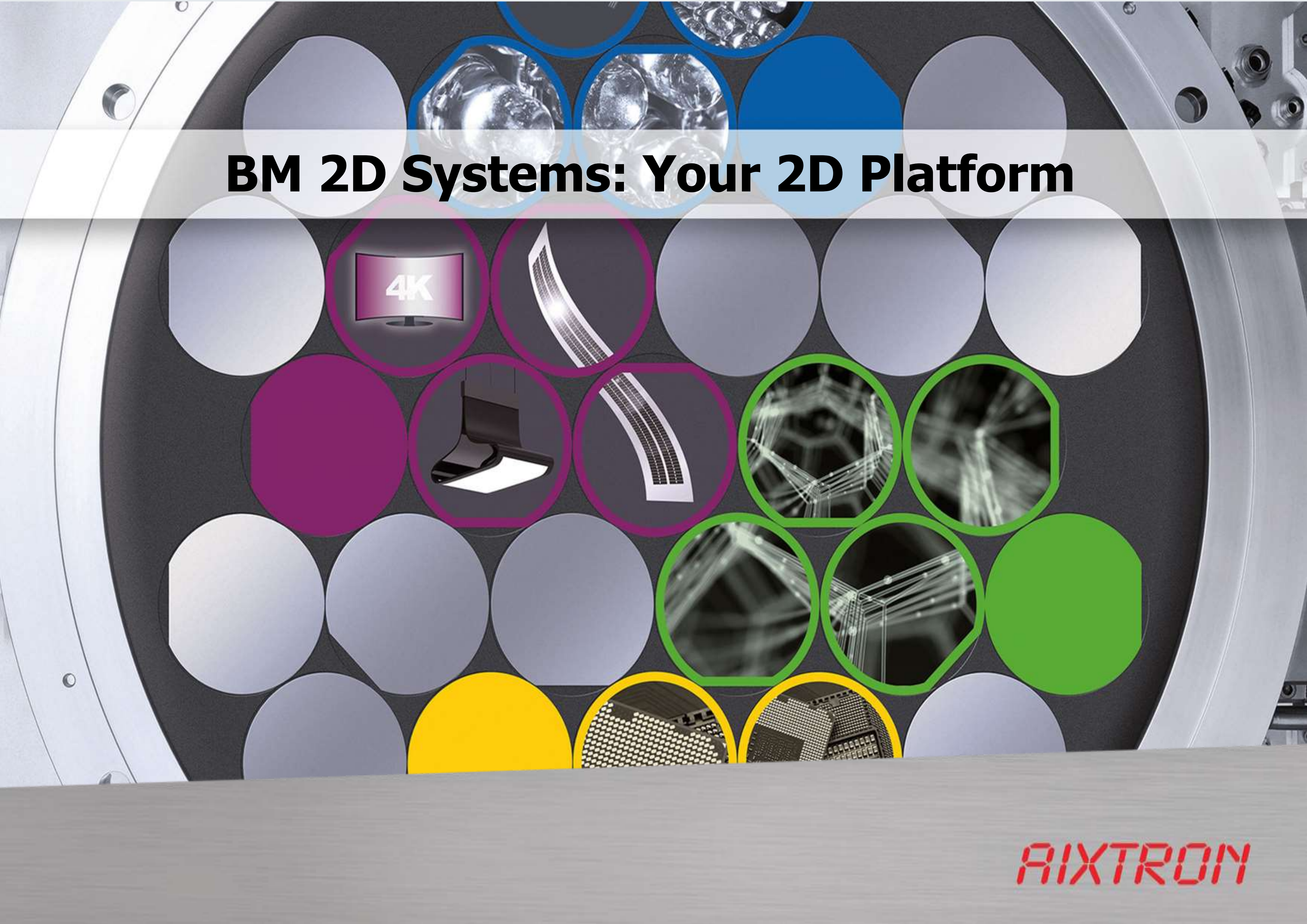
Source: azonano.com

CNT for Thermal management



Courtesy: J. Liu

BM 2D Systems: Your 2D Platform



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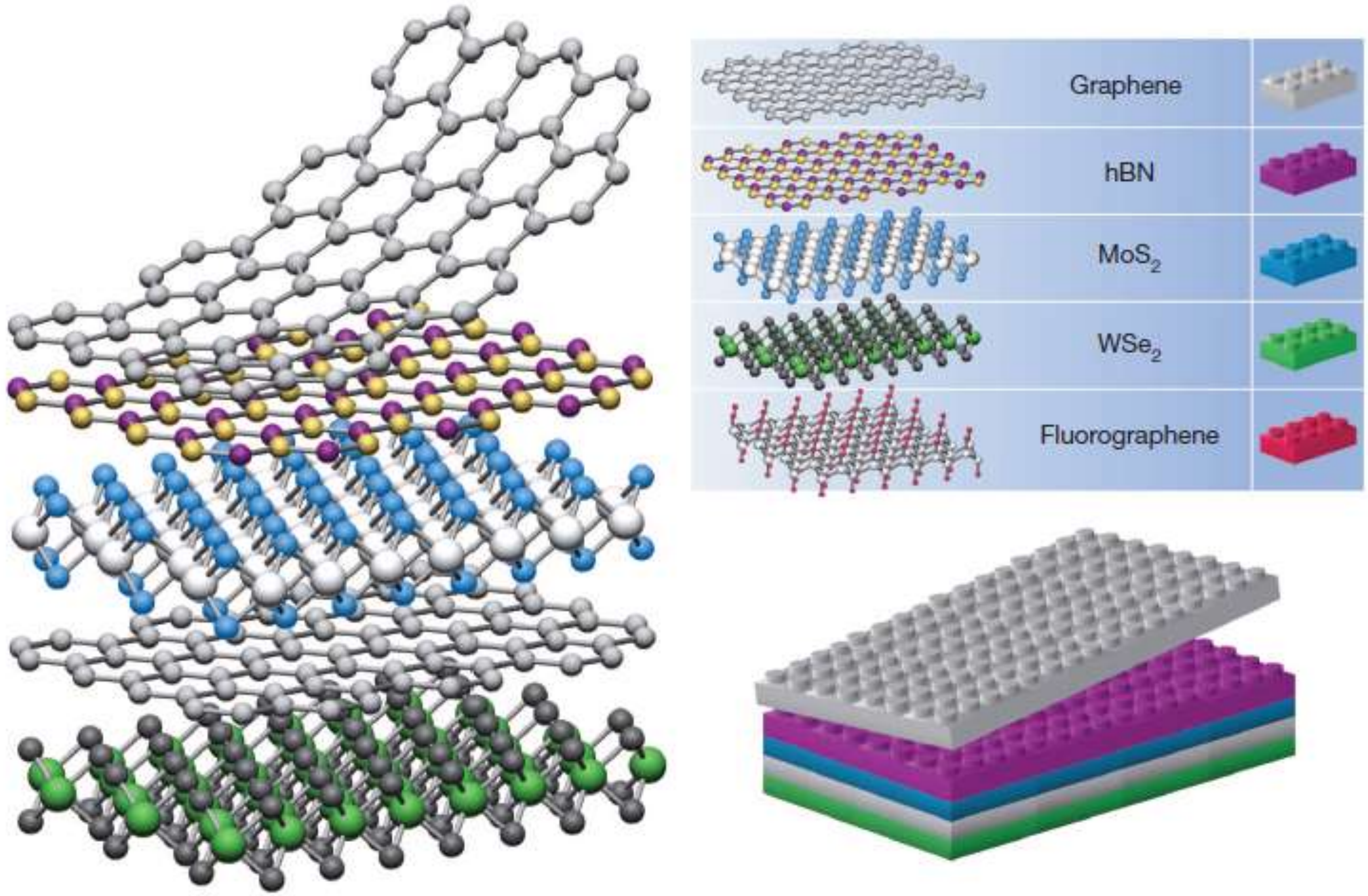
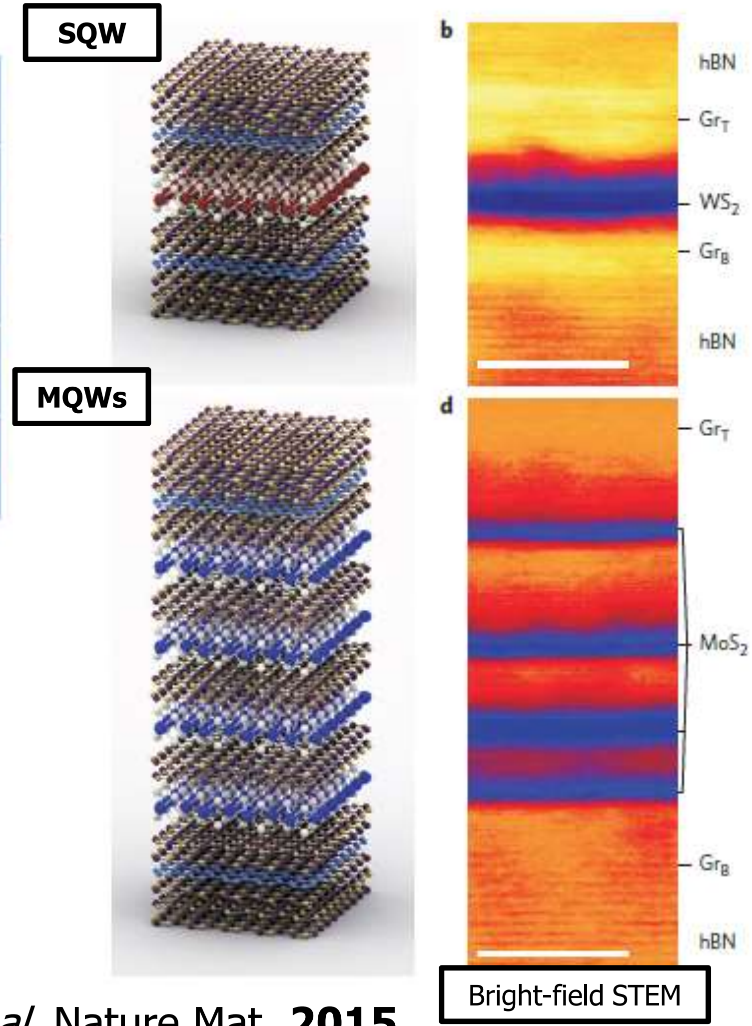
Beyond graphene…

1. Graphene related	2. 2D Chalcogenides	3. 2D Oxides	4. Others
Graphene Graphane Fluorographene Graphene oxide	Transition metal dichalcogenides (TMDs) MX_2; MoS_2, WS_2, $MoSe_2$, WSe_2 etc.	Transition metal oxides (TMOs) Ti/Nb/Mn_xO_y V_2O_5	MAX Phases M = transition metal A = Al or Si X = C or N
hBN Black phosphorous	Transition metal trichalcogenides (TMTCs) MX_3; NbX_3, TiX_3, TaX_3 (X = S, Se or Te)	Trioxides MO_3, WO_3, TaO_3	Metal halides, MX_2 or MX_3 $MoCl_2$
	Transition metal phosphorous trichalcogenides (TMPTCs) MPX_3; $MnPS_3$, $ZnPS_3$, $NiPS_3$	Oxychalcogenides Oxyhalides	Layered silicates Layered double hydroxides (LDHs)
	Group III-VI "Semiconductors" GaX, InX (X = S, Se, Te)	Layered zirconium phosphates and phosphonates	Xene's Silicene etc.

Heterostructures and Devices

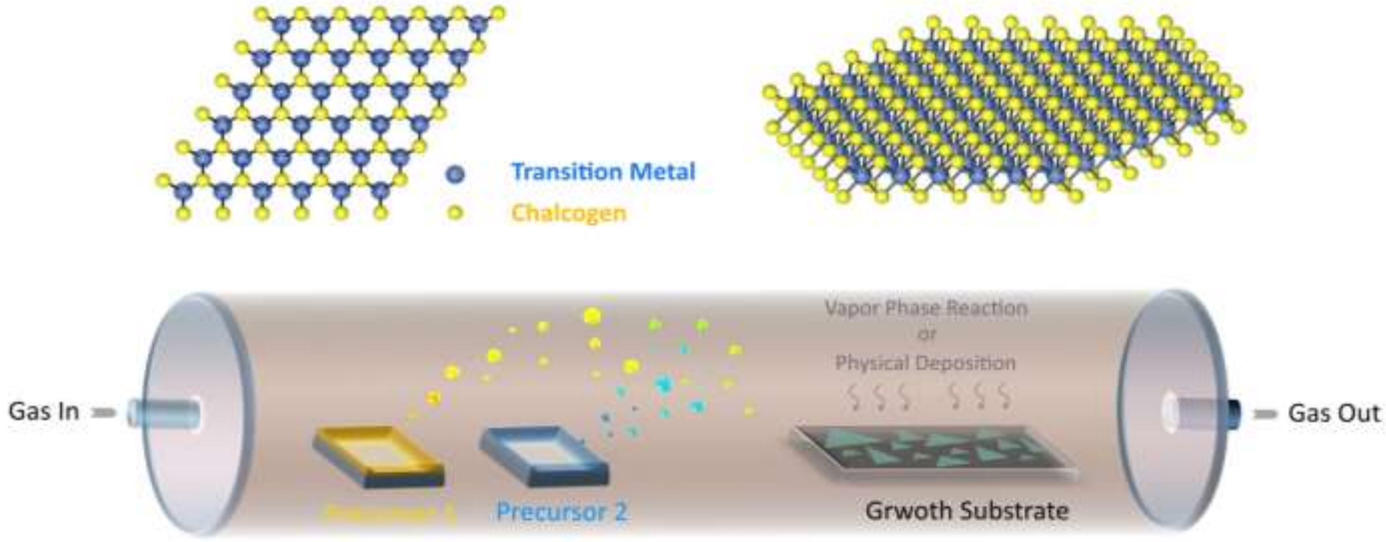


Geim *et al.* Nature 499 (**2013**) 419



Withers *et al.* Nature Mat. **2015**

Tube-based CVD furnace systems



Limitations

- Limited precursors
- Difficult to control uniformity over large areas
- Safety issues

Chem. Soc. Rev., 9, **2015**

BM 2D Systems – Your 2D Platform

BM 2D

Benefits:

- Up to 8-inch substrate size
- Wafer and foil compatible
- Bottom and top heater technology
- Up to 10 gas channels
- Up to 5 MO sources
- Temperatures up to 1050 °C

BM GB 2D – for complex materials deposition

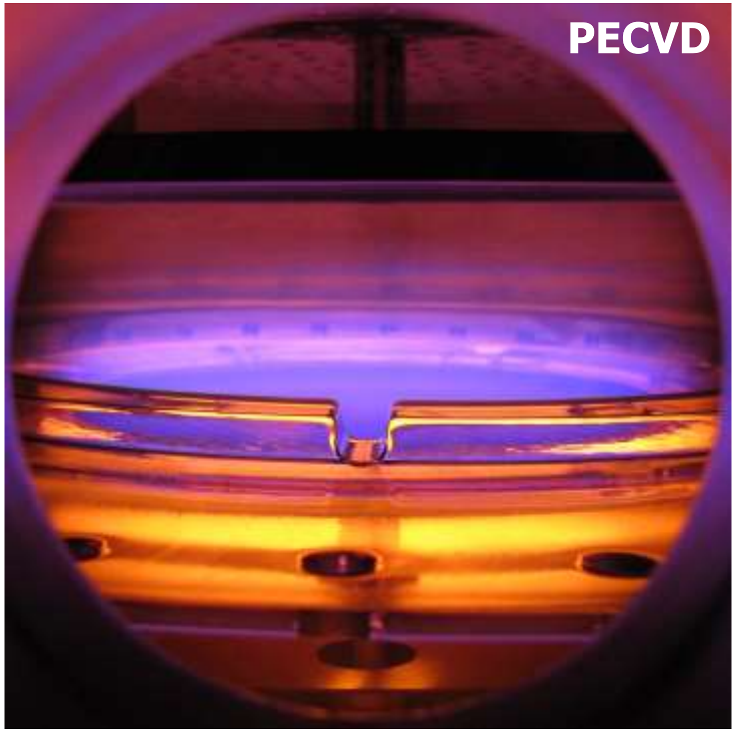
BM GB 2D

Benefits:

- 4-inch substrate size
- Sensitive materials
- Bottom and top heater technology
- Up to 10 gas channels
- Up to 8 MO sources
- Temperatures up to 1050 °C

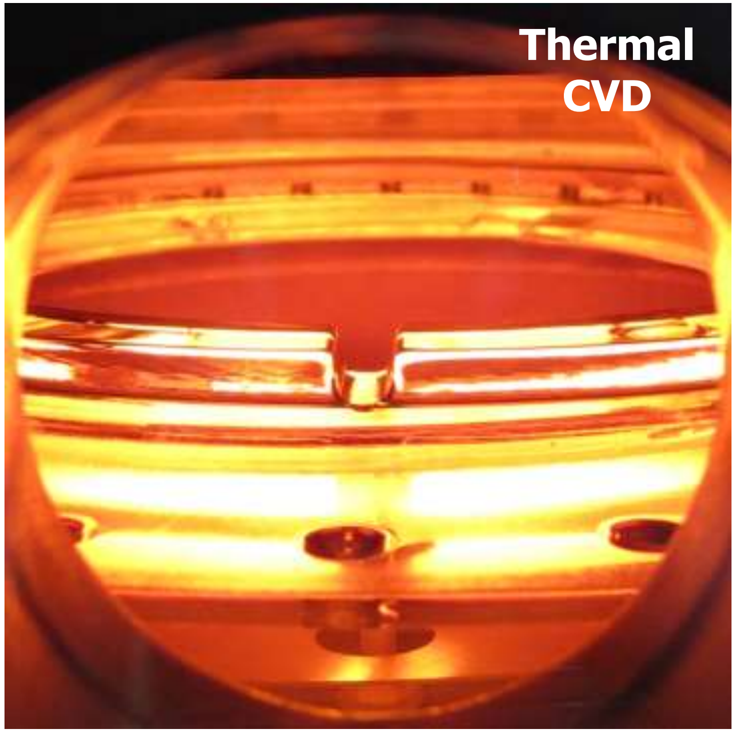
Growth process

Multiple processes available



Benefits

- Adjustable plasma position
- Top plasma for precursor activation
- Substrate plasma for growth alignment
- In-situ reactor cleaning



Benefits

- Temperatures up to 1000C
- Heat ramps > 300 C/min
- Excellent gas uniformity

Source types

Flexible source choice

MO type source

Benefits
- Wide process window
- Temperature control for precursors
- Carrier gas controlled

Vapor draw type source

Benefits
- Less expensive than MO
- Narrow process window (WP below precursor VP)
- Temperature control for precursors

Gas type source

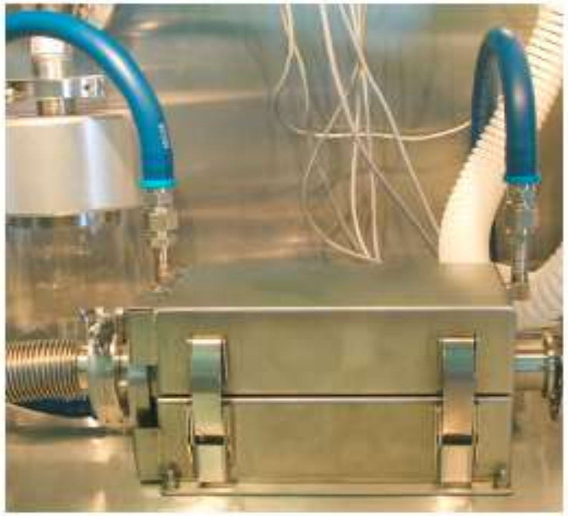
Benefits
- MFC controlled gases
- Metal and chalcogenides precursors possible

- Different source combinations available for process flexibility

Typical TMDC precursors

Type	Name	Chemical formula	CAS	Phase	Vapour pressure
metal	Molybdenum hexacarbonyl	Mo(CO)6	13939-06-5	solid	1.4mbar @45C
metal	Tungsten hexacarbonyl	W(CO)6	14040-11-0	solid	1.6mbar @67C
metal	Niobium (V) ethoxide	Nb(OCH2CH3)5	3236-82-6	liquid	no data available
metal	Tris(diethylamido)(tert-butylimido)niobium(V)	C16H39N4Nb	210363-27-2	liquid	no data available
chalcogenide	Di-tert-butyl disulfide (DTBS)	C8H18S2	110-06-5	liquid	68 mbar @37.7C
chalcogenide	Diethyl sulfide	C4H10S	352-93-2	liquid	53mbar @16C and 140mbar @37.7C
chalcogenide	Dimethyl selenide	C2H6Se	593-79-3	liquid	no data available
chalcogenide	Dimethyl telluride	C2H6Te	593-80-6	liquid	no data available

MO by-products safety



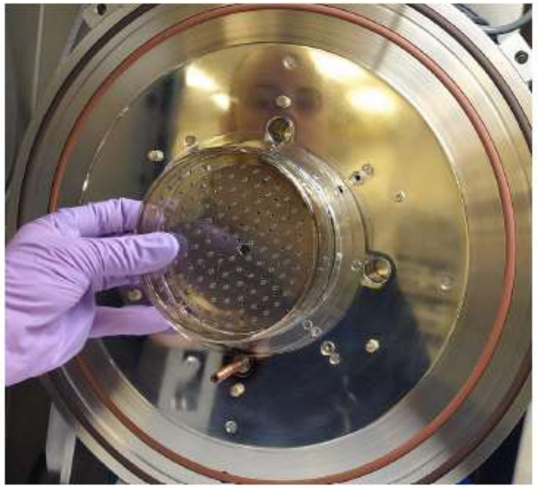
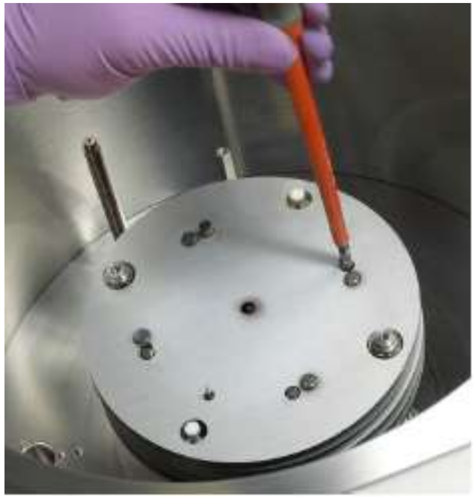
Benefits

- Cooled two stage filtration for unreacted MO precursors condensation
- Improved pump lifetime
- Easy filter regeneration
- Oxidation furnace available

Maintenance

Easy to maintain

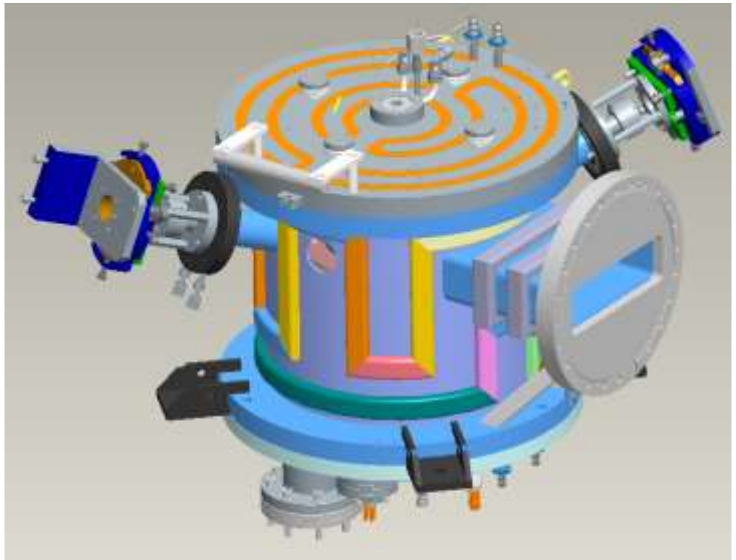


Benefits
- "Plug and play" parts concept
- Changeable quartzware for different processes
- Easy access to reactor inside

In-situ and low pressure options

Ports for in-situ measurements



- Sample temperature measurement
- In-situ ellipsometry for thickness and growth quality

Low pressures ($<10^{-5}$ mbar) compatible

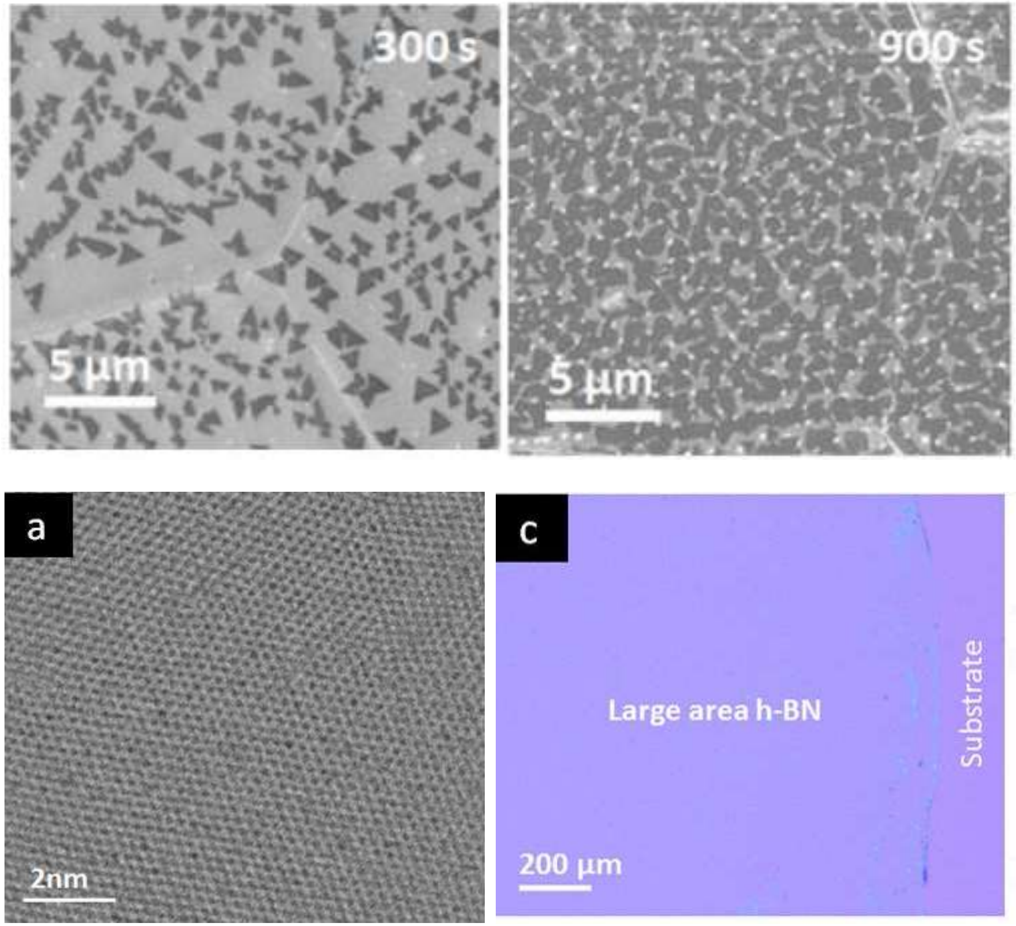
- Turbo pump integration for low pressure processes

Customer results

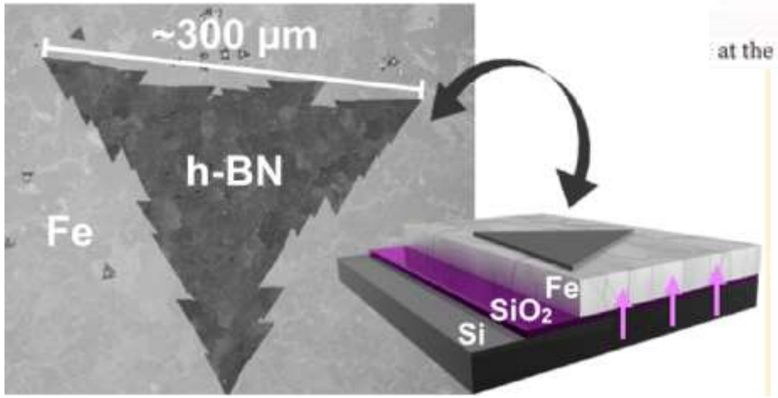
Courtesy of S. Hofmann

Exploring h-BN Growth using BM Systems

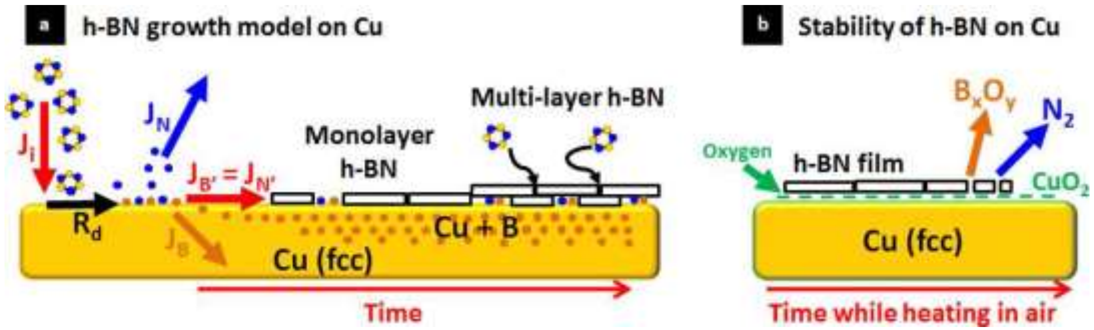
the HOFMANN group at the University of Cambridge



Kidambi et al. Chem. Mater., 2014, 26 (22), pp 6380–6392

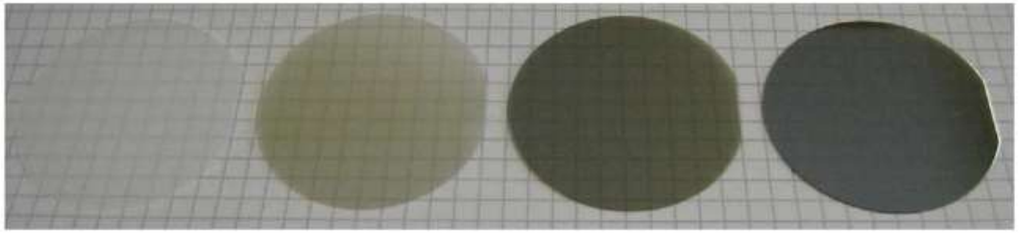


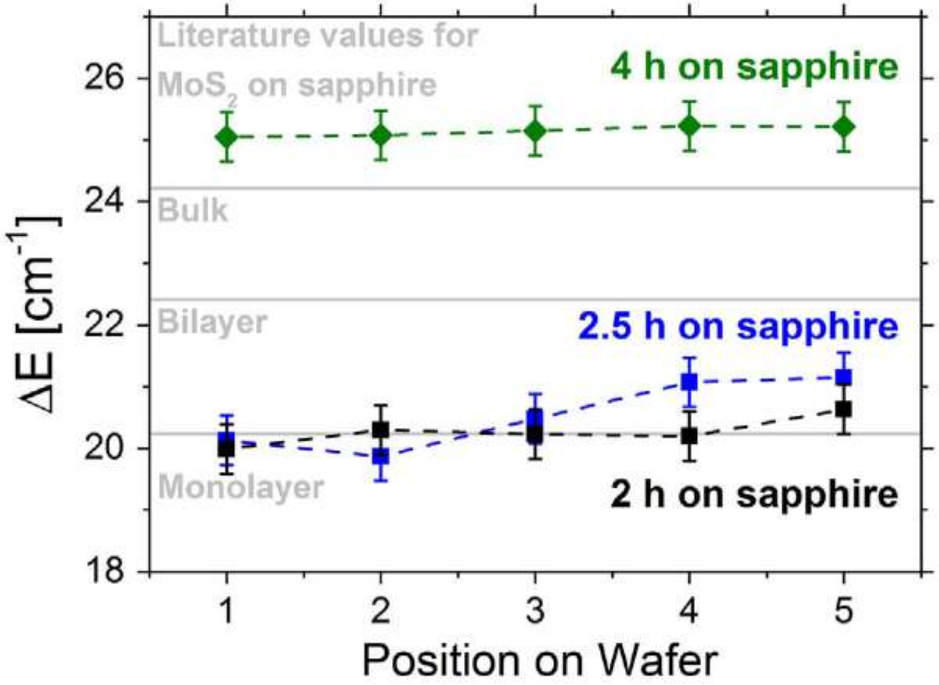
Caneva et al. Nano Lett.2015, 1867-1875



Internal Process and Optimisation for MoS_2 Growth using MOCVD

Precursors: $Mo(CO)_6$ + DBTS
Conditions: substrate, temperature, reaction time, atmosphere…





- Direct deposition of MoS_2 on 2-inch sapphire wafers
- Homogenous monolayer achieved

Marx *et al.* Journal of Crystal Growth *464, 100-104,* ***2017***

Thank you very much for your attention.

If you have any further questions or require more information, please contact us at:

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