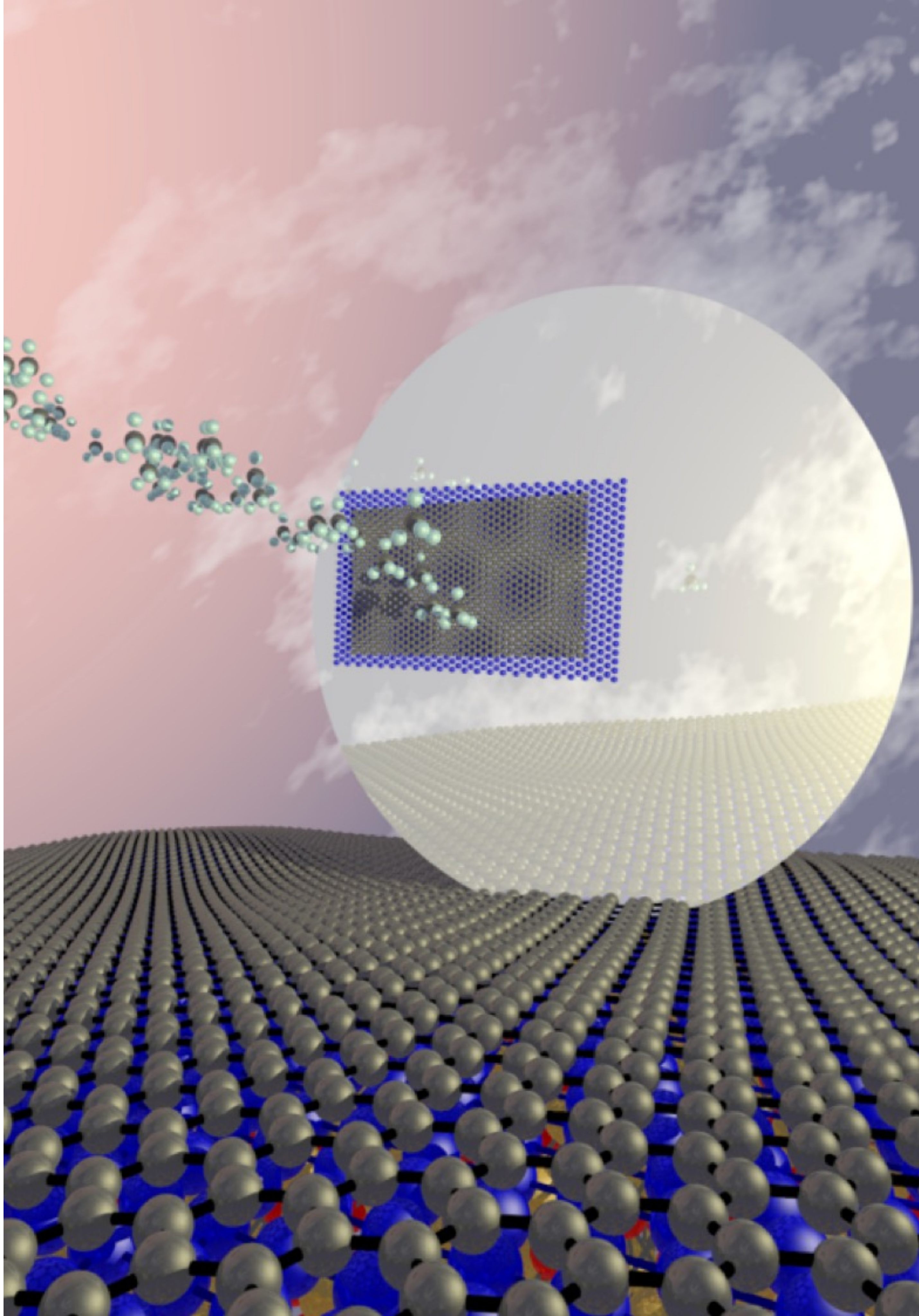
Synthesizing high-quality 2D materials: from surface science to novel applications

Camilla Coletti

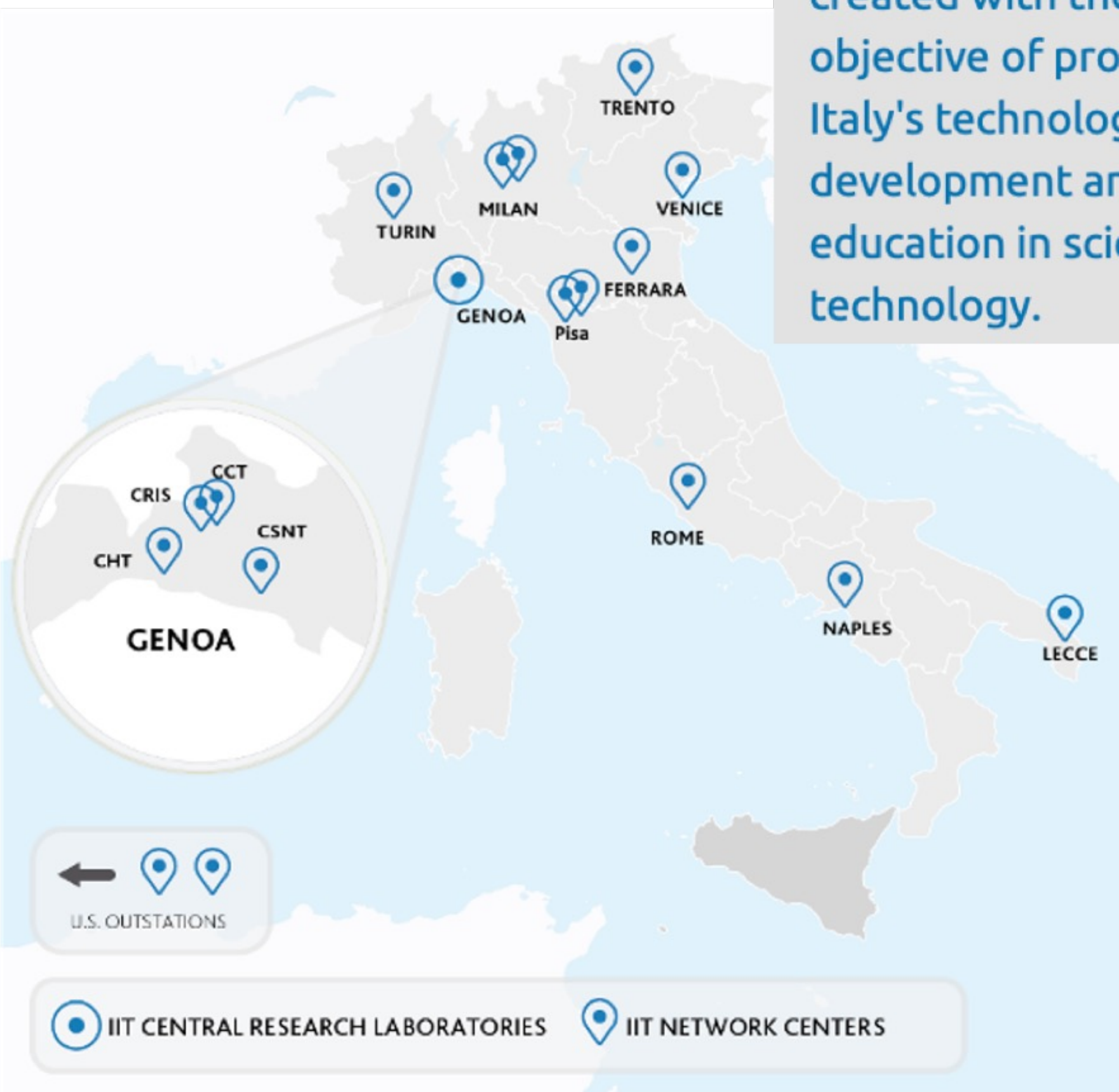
CNI@NEST & Graphene Labs

Istituto Italiano di Tecnologia



Our activities

Istituto Italiano di Tecnologia - IIT - was created with the objective of promoting Italy's technological development and higher education in science and technology.



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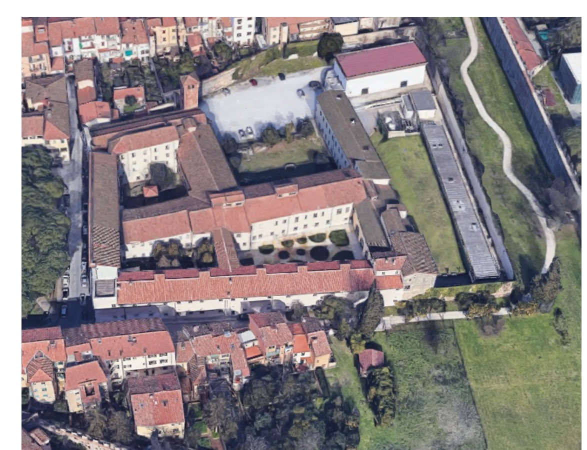
2D Materials Engineering @ IIT-Pisa

The mission of 2D Materials Engineering is to synthesize, investigate and tailor 2D materials and their heterostructures for electronics and photonics.

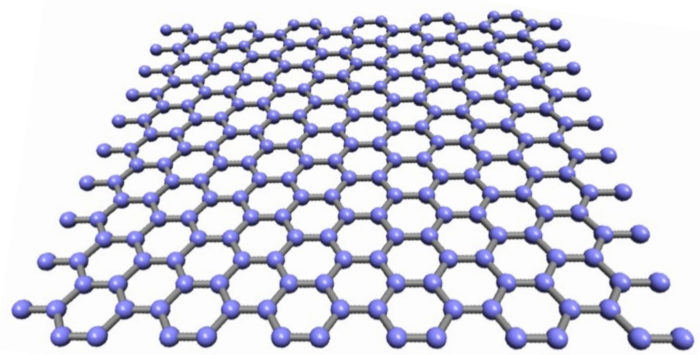
Center for Nanotechnology Innovation @NEST

CNI@NEST is a research center with a focus on nanomaterials for electronics, photonics, nanomedicine, oncology.

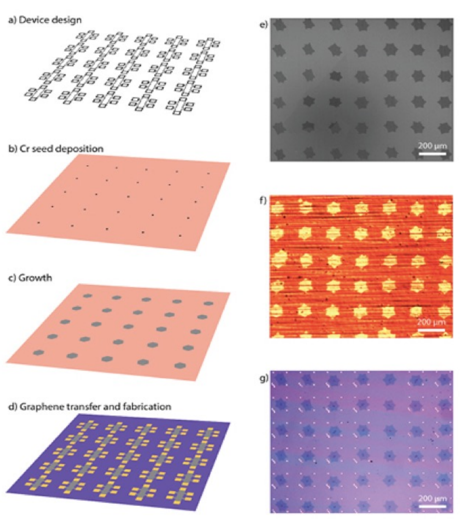


2D Materials Engineering: Our activities

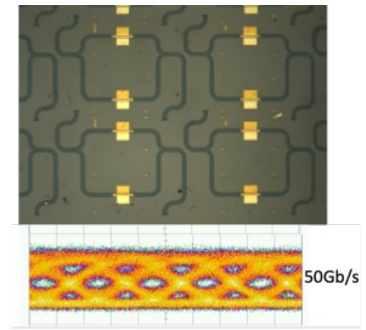
Graphene

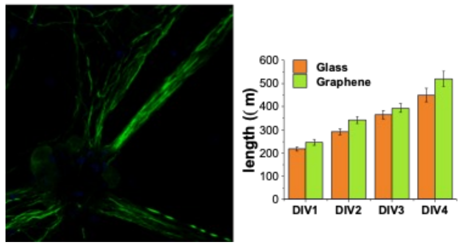


Integration

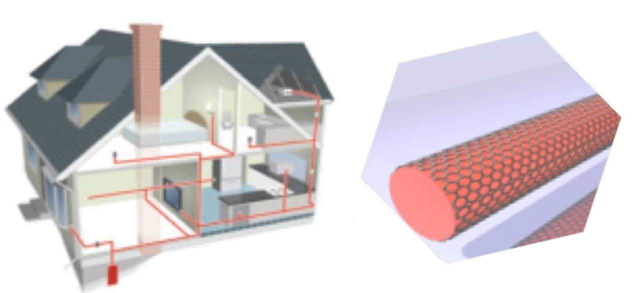


Optoelectronics and Bio

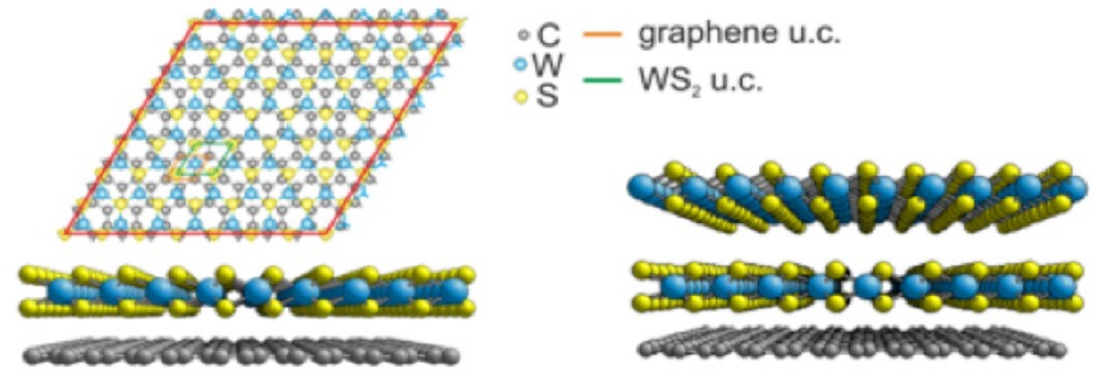




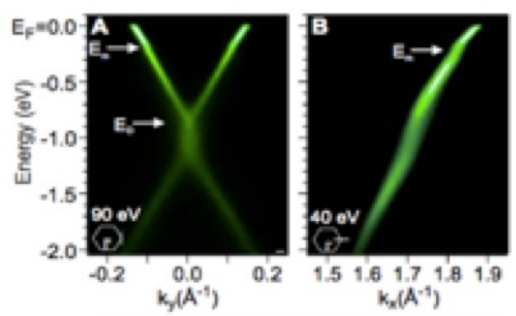
Technology Transfer

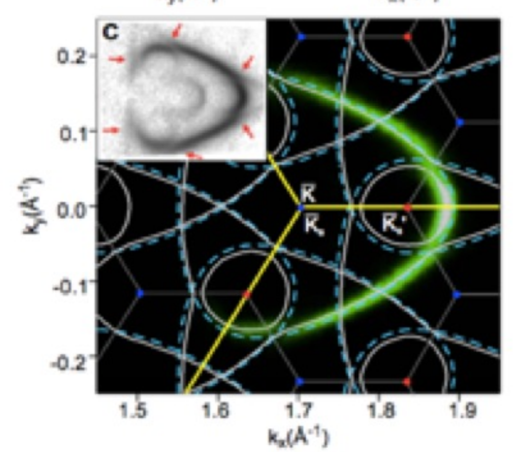


TMDs & van der Waals heterostacks

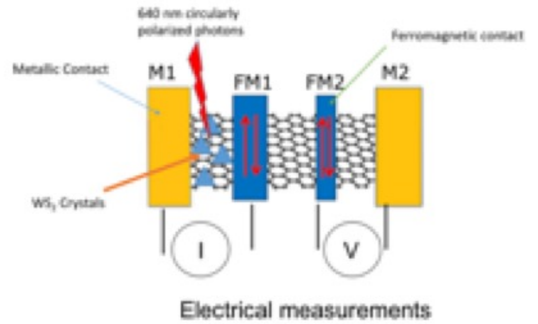


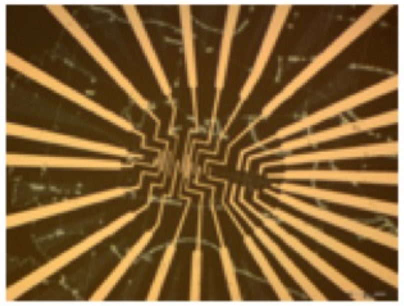
Fundamentals





Novel device concepts

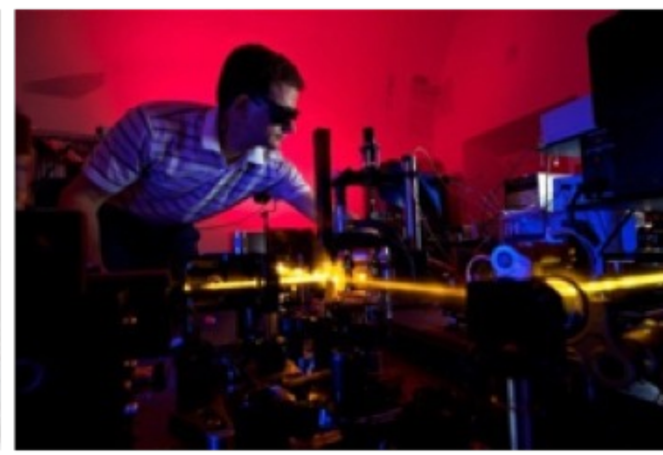




2D-ME @ IIT: labs

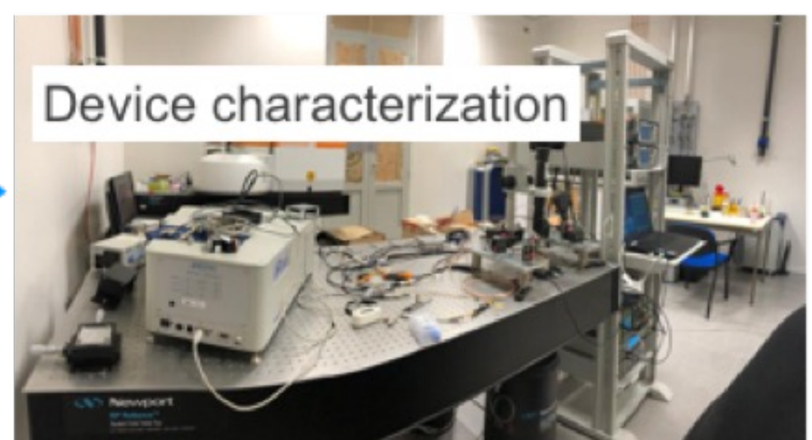
Our Labs

2 Synthesis Labs: 2 commercial PE-CVD reactors; 2 horizontal furnaces; 1 service furnace for oven's parts cleaning; optical microscope; bench for preparation of TMDs; transfer setup for assembling vdW heterostructures; 1 evaporator



Characterisation Labs: LT-STM soon to be equipped with ARPES; LEED equipment; AFM; Raman spectroscopy

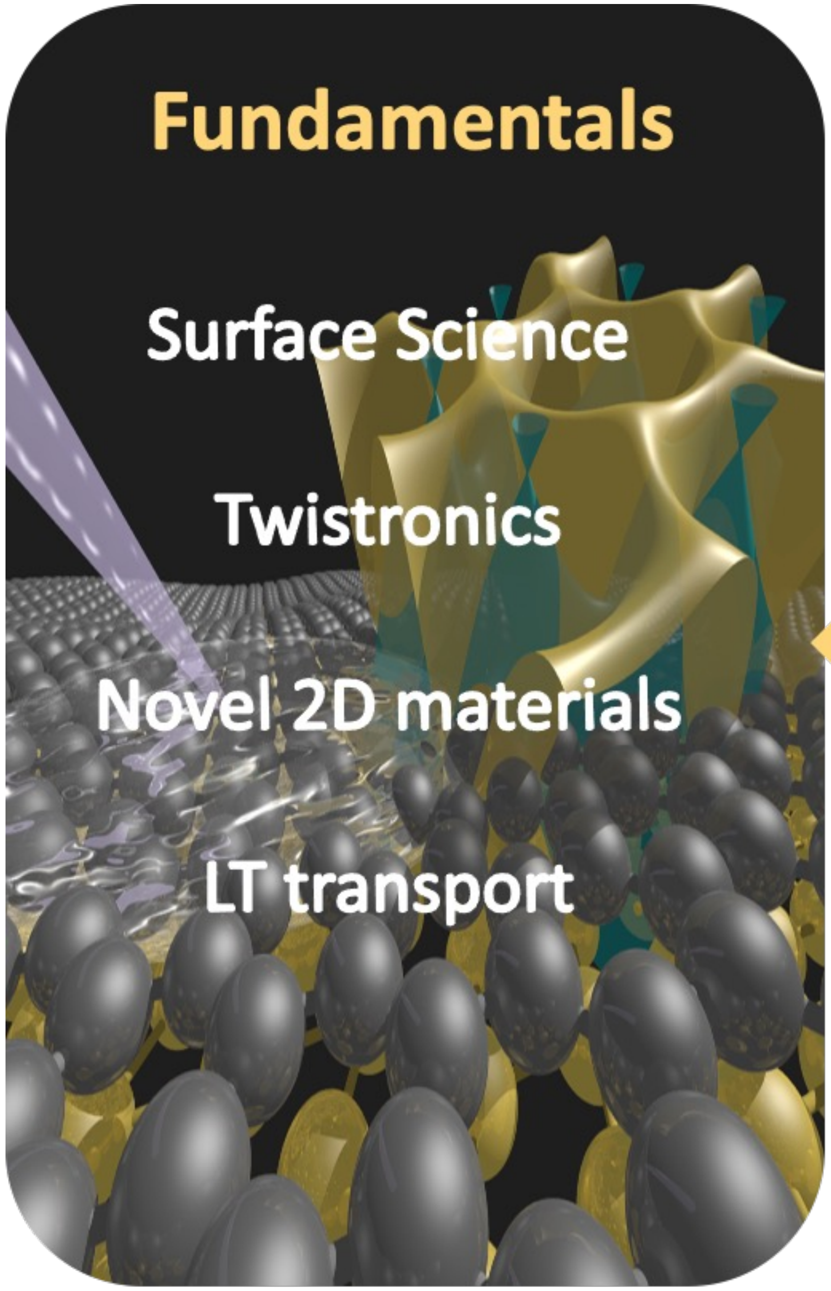
Class ISO7 Cleanroom

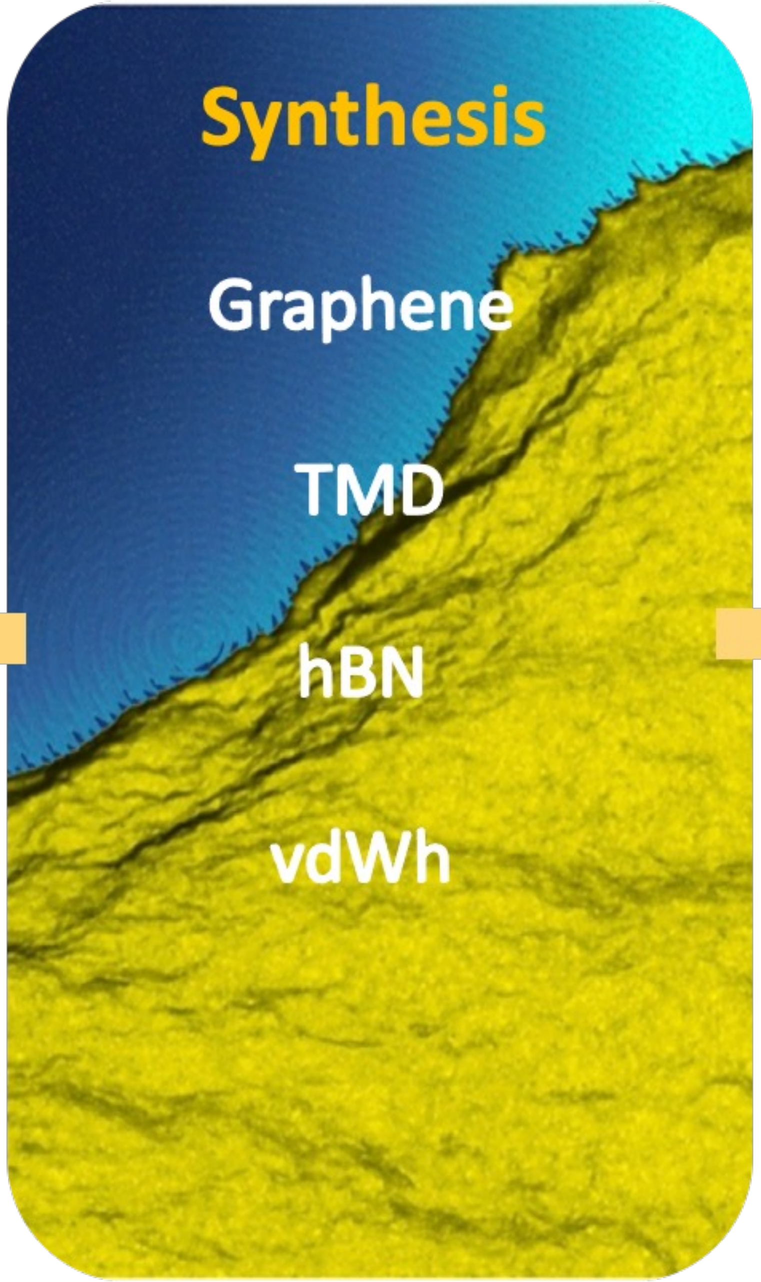


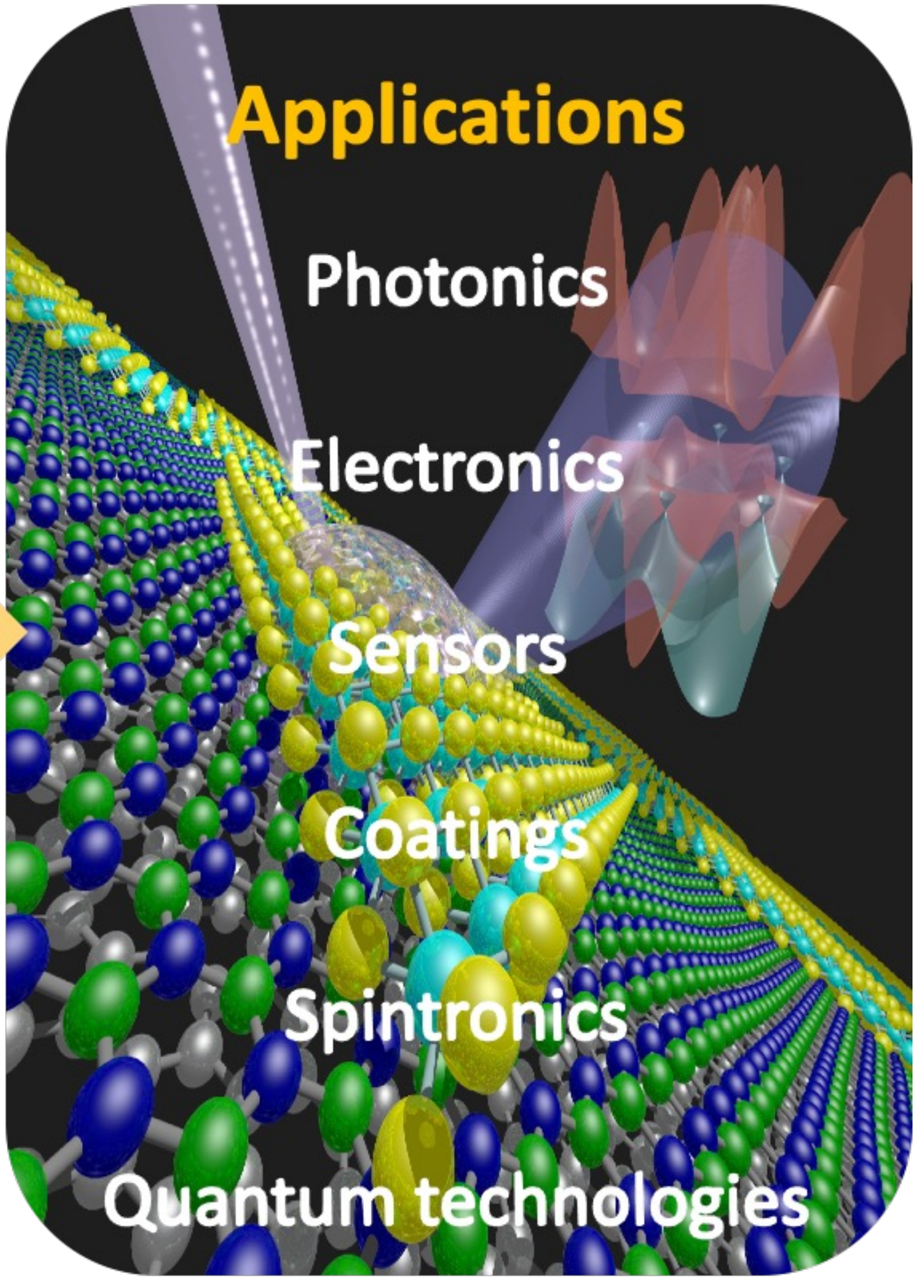
ISO7 Cleanroom: 2 SEM, 1 EBL, 1 EDX, 2 evaporators, 1 RIE, wet benches, spinners.

Device measurement lab: RT 4 point probe measurements with magnet; van der Pauw station; photocurrent measurement system under different ambient conditions.

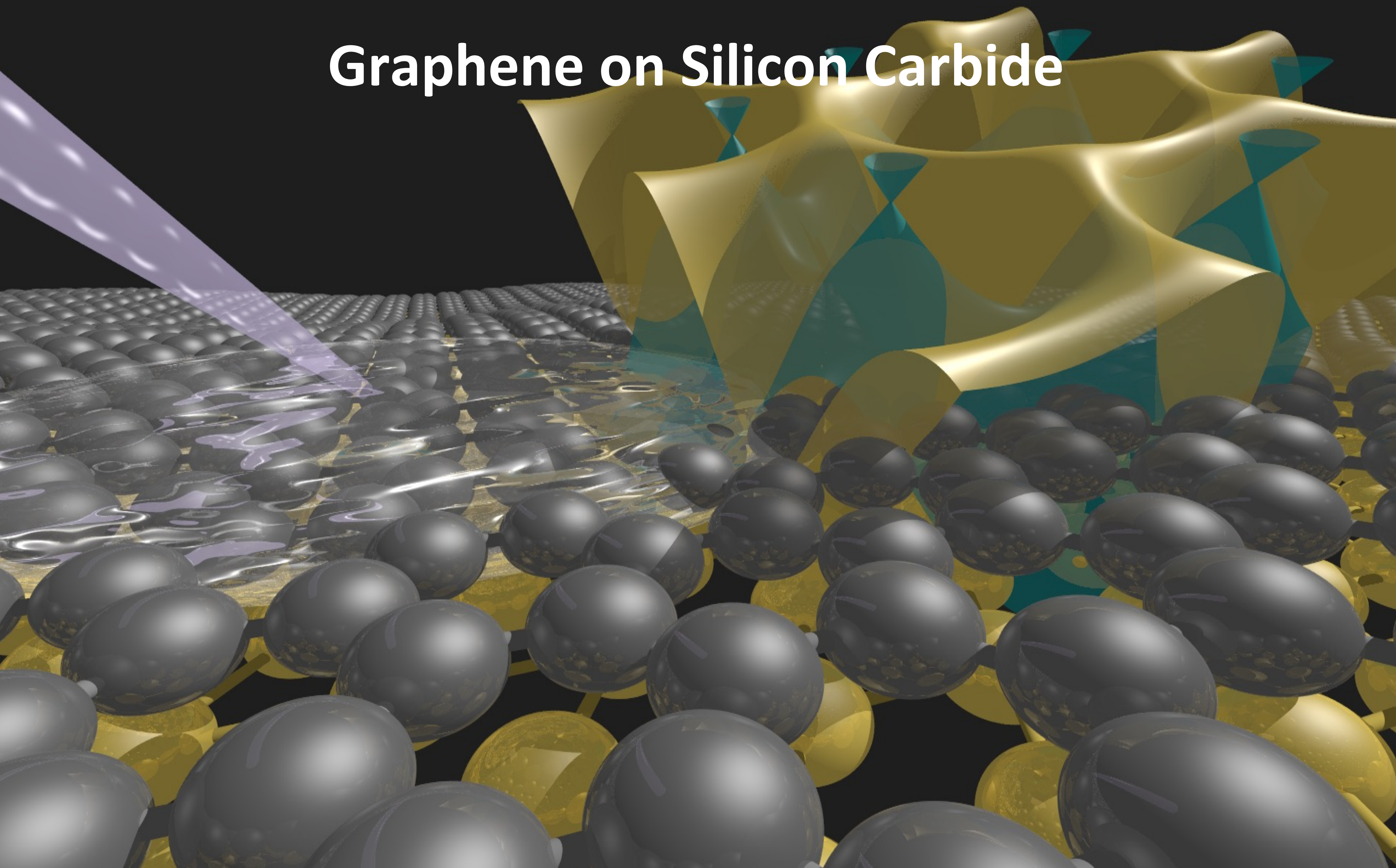
CVD-grown 2D materials: from fundamentals to applications



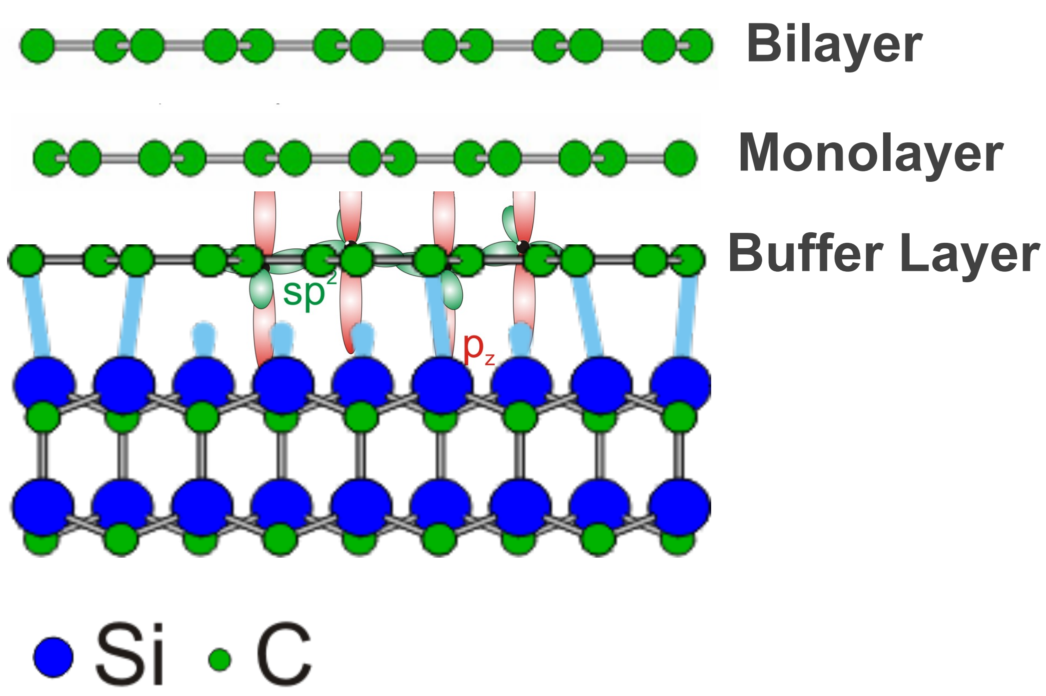


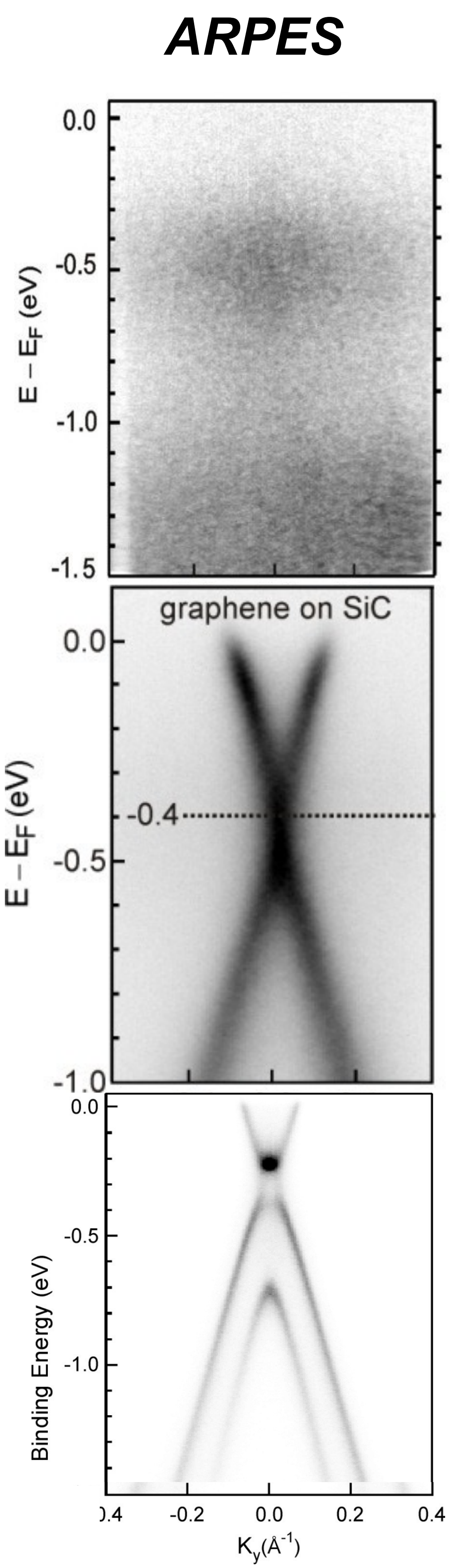


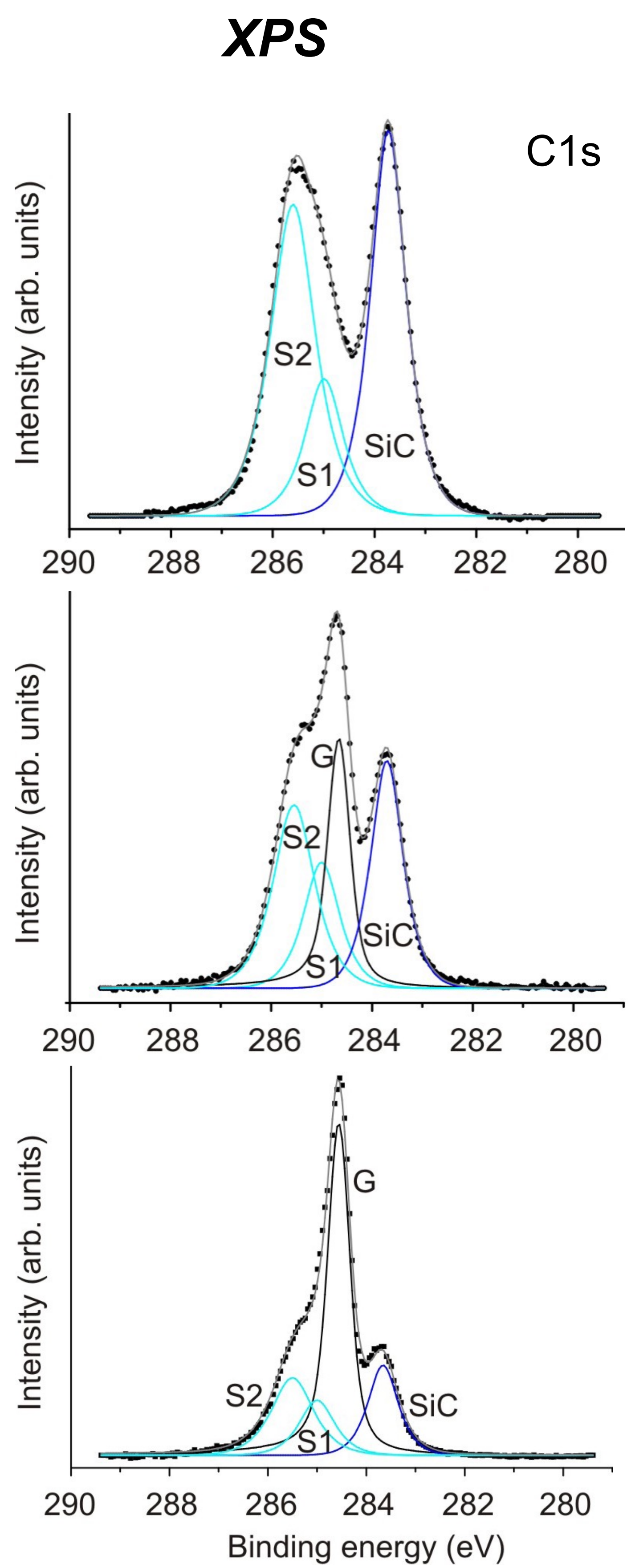
Graphene on Silicon Carbide



Epitaxial graphene on silicon carbide (SiC)

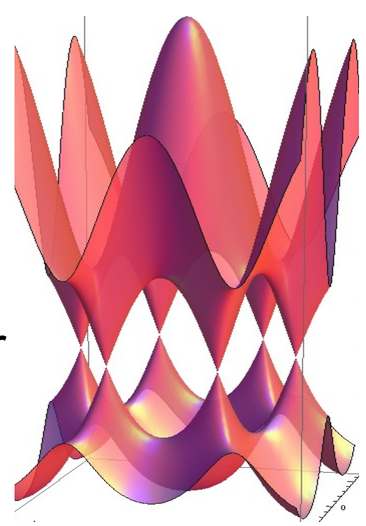






Limitations:

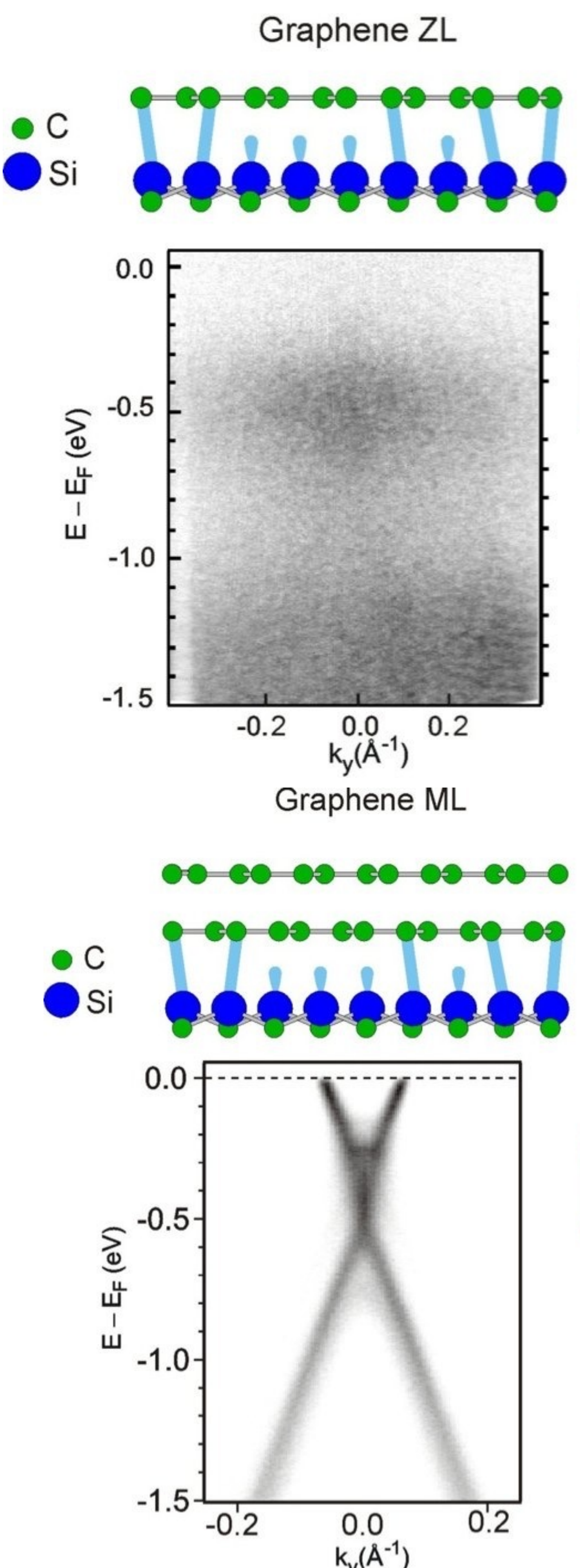
- High doping level
- Small bandgap in BL
- Reduced charge carrier mobility ca. 1000 cm^2/Vs

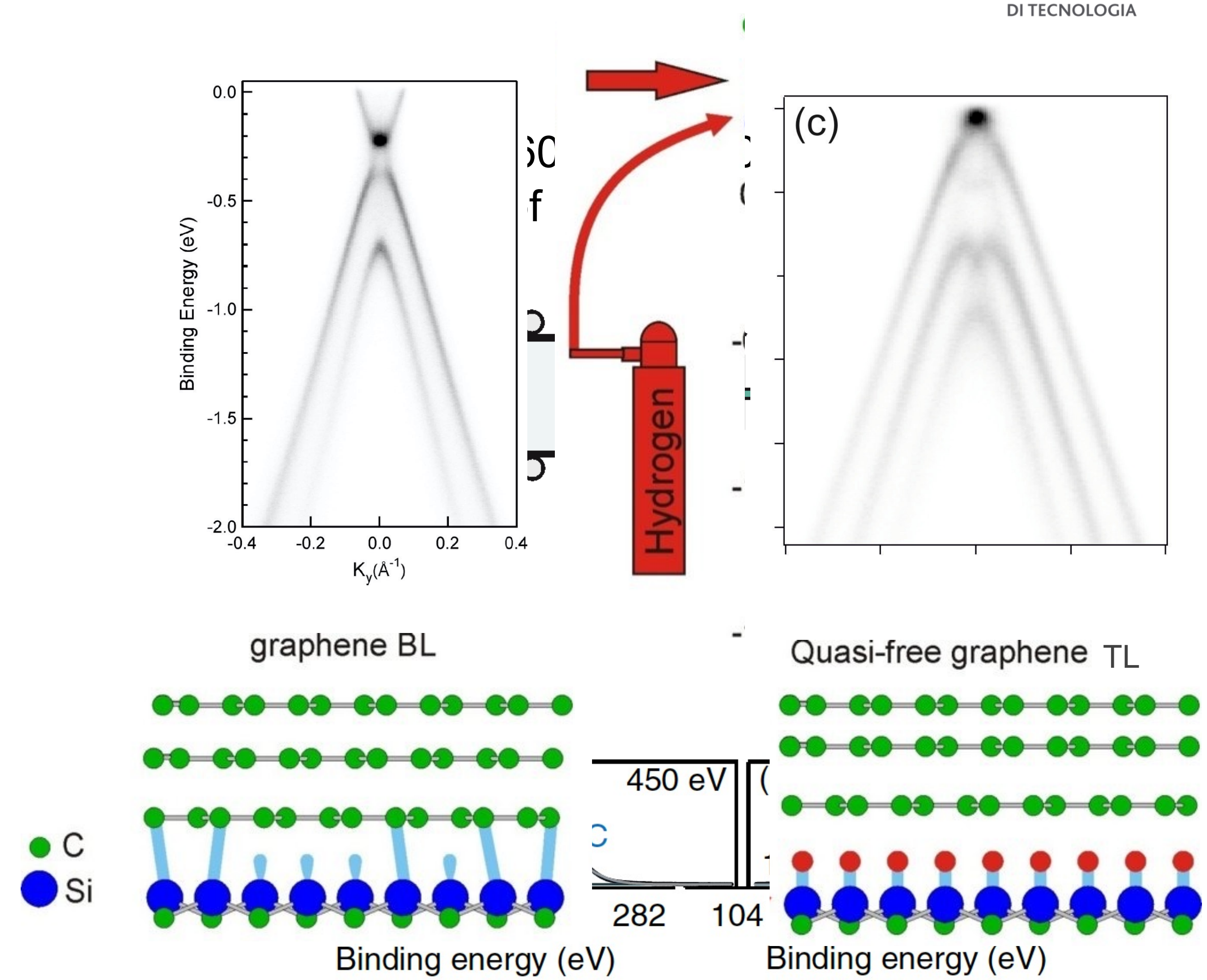


Starke, Forti, Emtsev, Coletti, MRS Bulletin 37 (12), (2012)

Riedl, Coletti et al., PRL 103, 246804 (2009)

Hydrogen Intercalation





✓ Charge neutral large area quasi free-standing ML, BL, TL

✓ BL and TL with electric field unable BG

✓Higher mobilities (5000 cm²/Vs)

✓Limited flexibility: SiC substrate and difficult scalable transfer

✓Playground to synthesis novel 2D materials!

Riedl, Coletti et al., PRL 103, 246804 (2009)

Semiconducting 2D gold underneath EG on SiC

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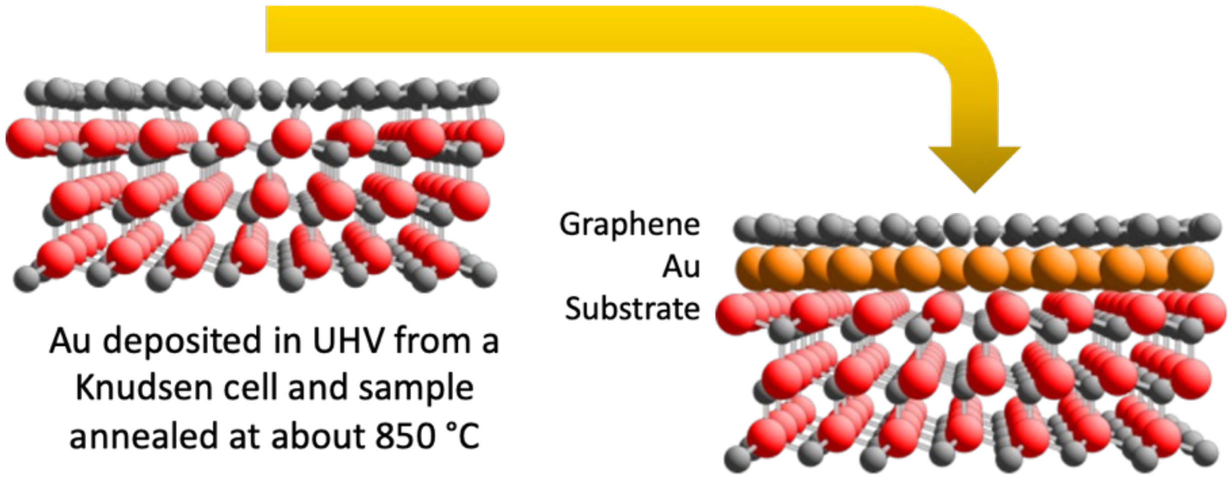


Au deposited in UHV from a Knudsen cell and sample annealed at about 850 °C

- Monolayer of Au intercalated at the heterointerface between SiC(0001) and graphene in UHV
- Every Au atom bound to Si atom at the interface: Au ordered in a (1x1) registry with respect to SiC
- Average 2D Au VBM at -67 meV with respect to EF: **semiconducting material**
- A 225 meV spin-orbit splitting in the sp Au bands, and a -400 meV van Hove singularity
- The electronic properties of 2D Au are layer dependent. A **double layer becomes metallic**

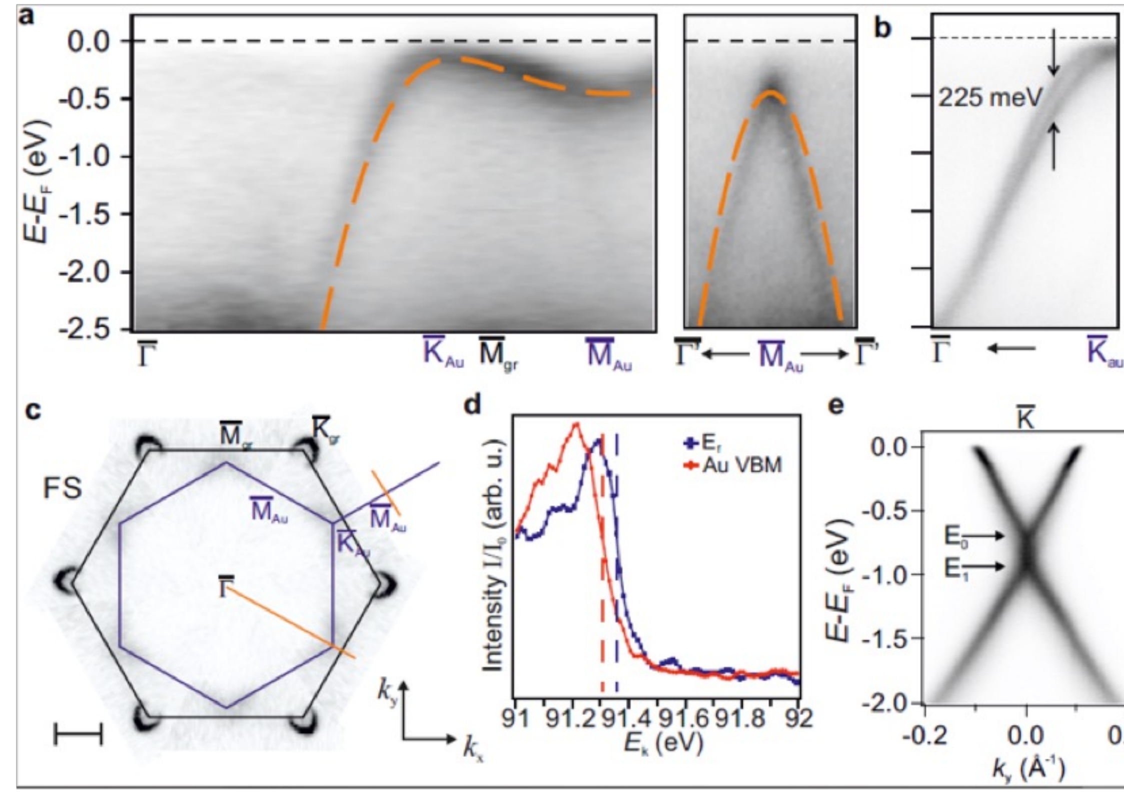
ARTICLE

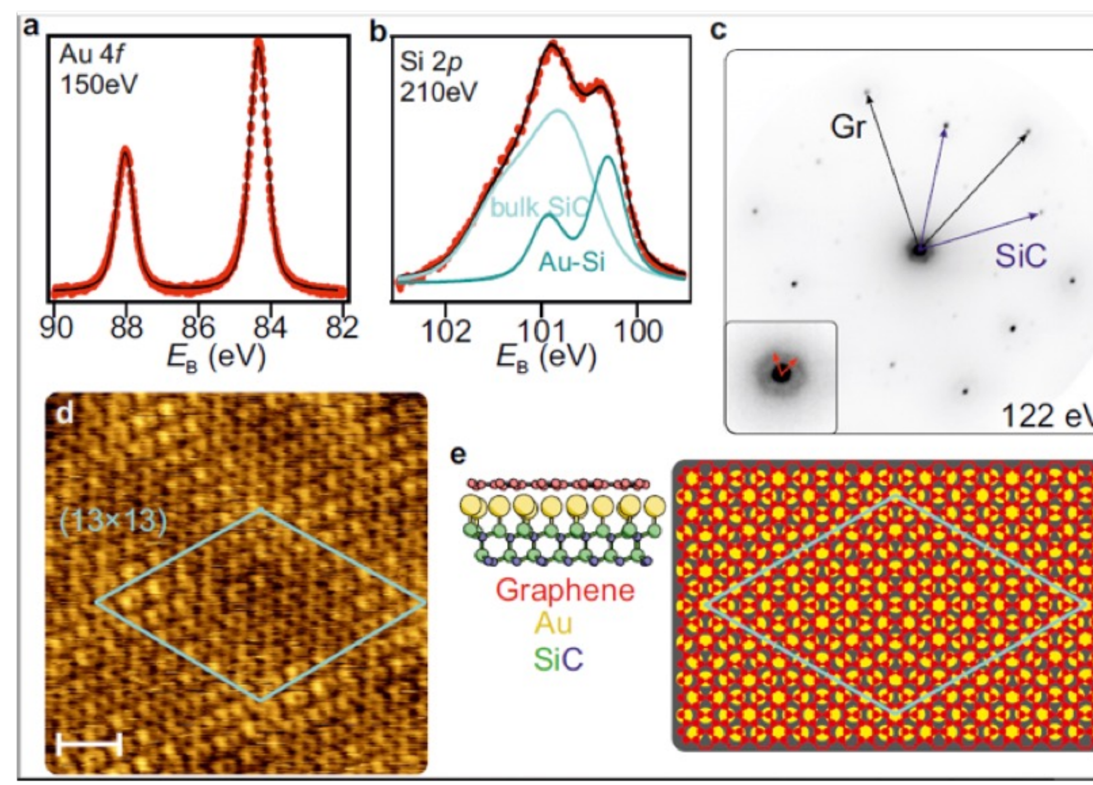
Check for updates

https://doi.org/10.1038/s41467-020-15683-1 OPEN

Semiconductor to metal transition in two-dimensional gold and its van der Waals heterostack with graphene

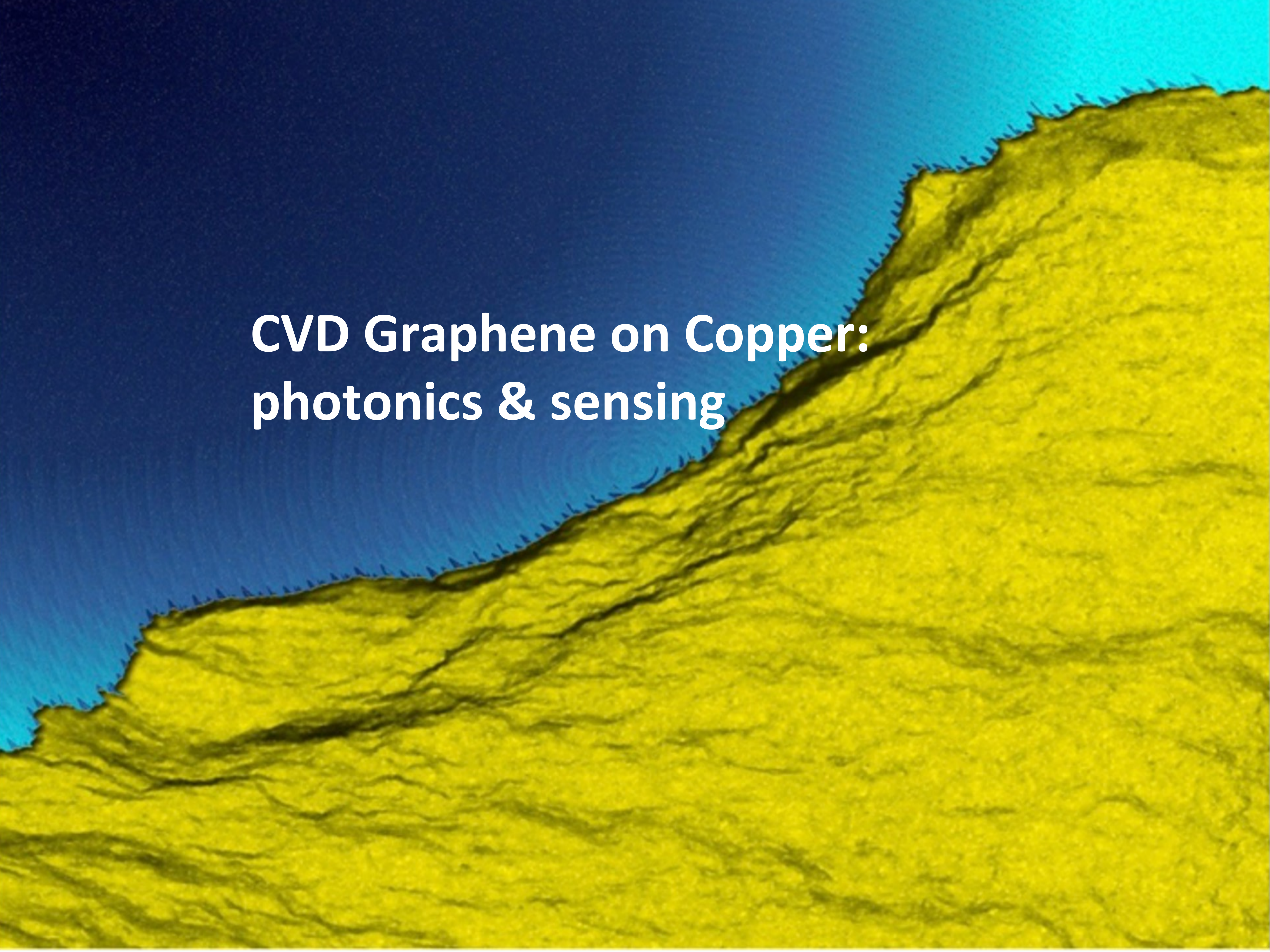
Stiven Forti [1,2], Stefan Link[2], Alexander Stöhr[2], Yuran Niu[3], Alexei A. Zakharov [3], Camilla Coletti [1,4] & Ulrich Starke [2]





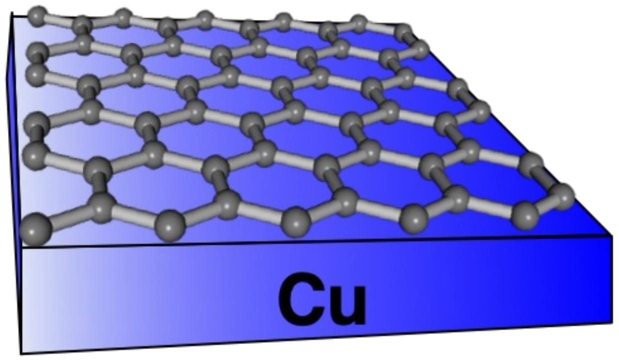
… other 2D materials can be stabilised

CVD Graphene on Copper: photonics & sensing



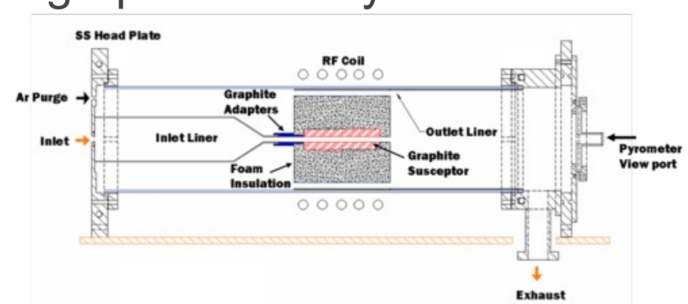
CVD graphene on Cu: growth

$\boxed{CH_4 \rightarrow 2H_{2(g)} + C_{(s)}}$



Cu substrate:

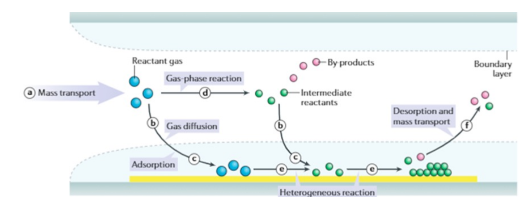
- acts as catalyst
- surface mediated growth
- cost effective
- graphene easily transferred

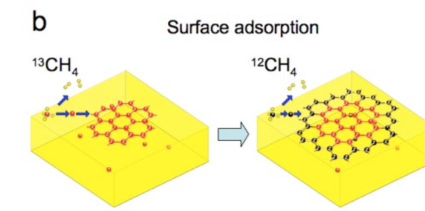


Science

Large-Area Synthesis of High-Quality and Uniform Graphene Films on Copper Foils

Xuesong Li,[1] Weiwei Cai,[1] Jinho An,[1] Seyoung Kim,[2] Junghyo Nah,[2] Dongxing Yang,[1] Richard Piner,[1] Aruna Velamakanni,[1] Inhwa Jung,[1] Emanuel Tutuc,[2] Sanjay K. Banerjee,[2] Luigi Colombo,[3]* Rodney S. Ruoff[1]*





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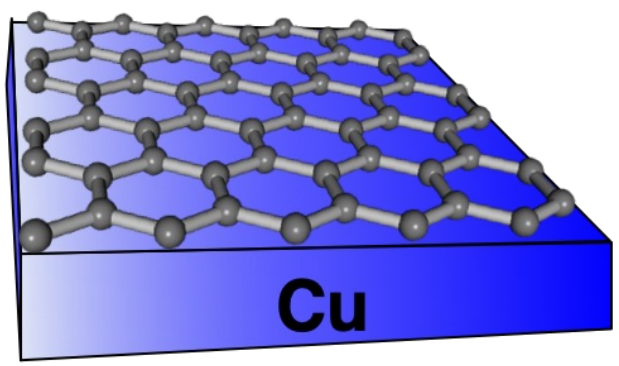

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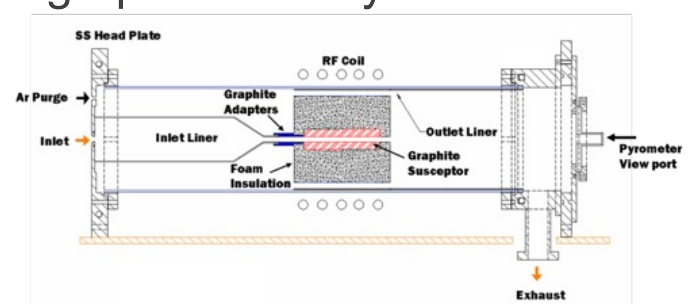

Science

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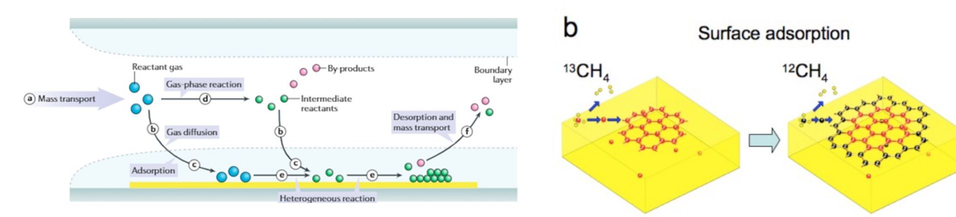
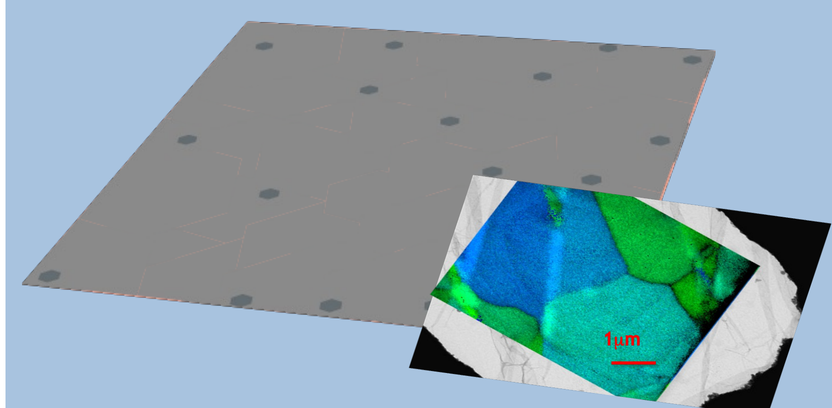


Cu foil

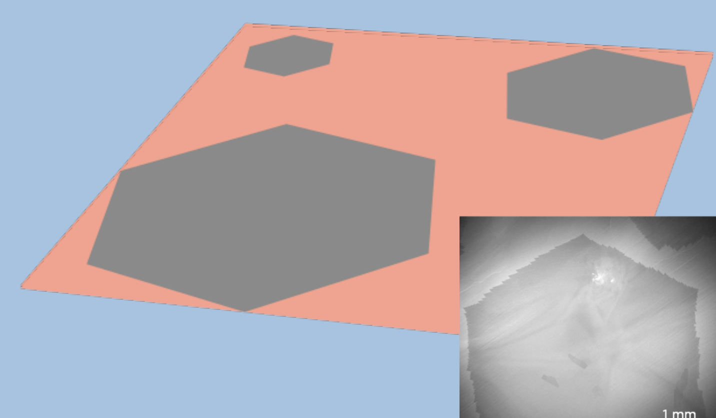
Cu film

Polycrystalline graphene



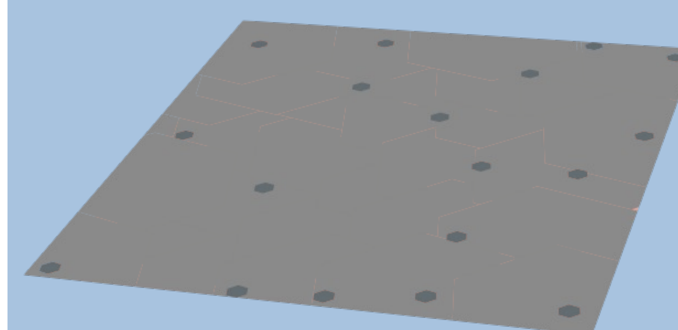
- Typical grain size tens of µm
- RT carrier mobility on SiO_2 ~1000 cm^2/ Vs

Large single-crystal graphene



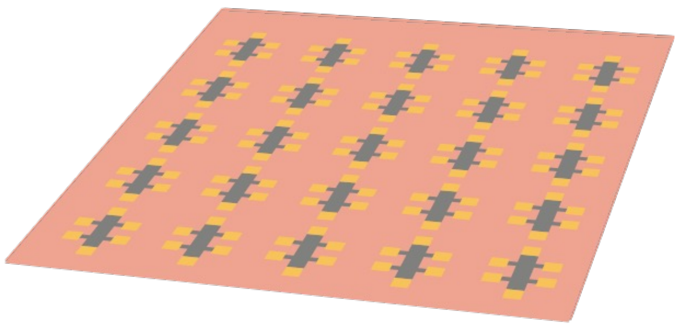
- Grain size tens up to several cm
- Easy transfer
- Higher RT carrier mobility

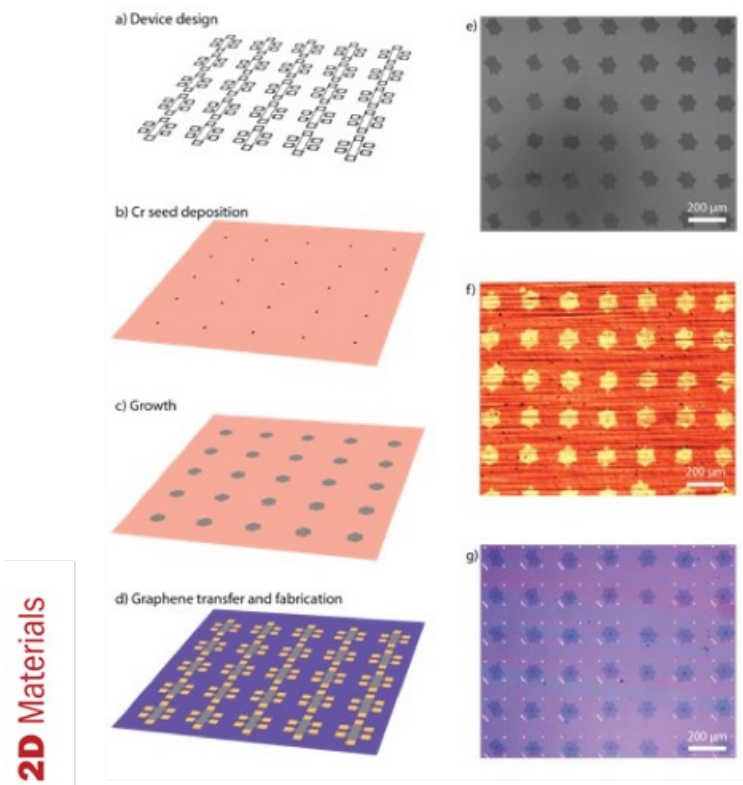
Continuous single-crystal graphene



- High crystallinity
- Transfer not always easy
- Higher RT carrier mobility

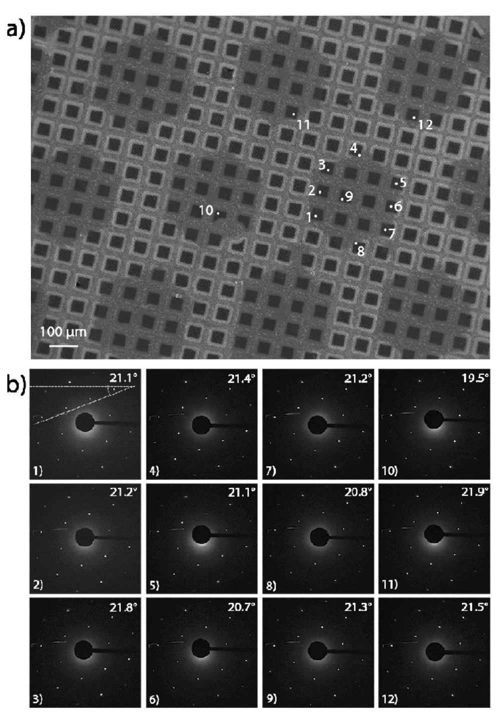
Our approach: seeded growth of graphene single-crystals

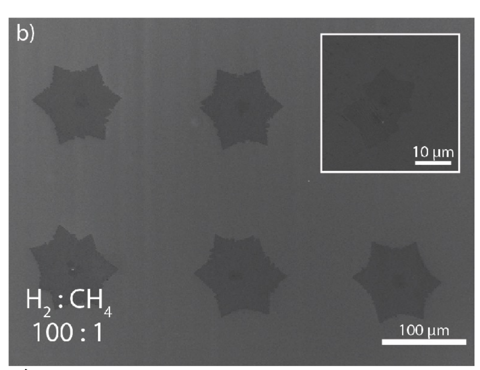


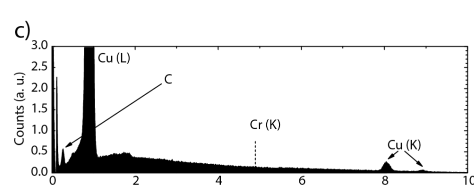


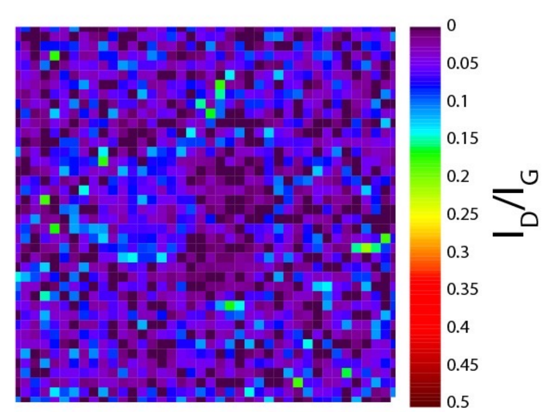
Deterministic patterned growth of high-mobility large-crystal graphene: a path towards wafer scale integration

Vaidotas Miseikis[1,2,3], Federica Bianco[4], Jérémy David[1], Mauro Gemmi[1], Vittorio Pellegrini[2], Marco Romagnoli[3] and Camilla Coletti[1,2]









- Flexible growth as per target wafer layout: graphene is only where is needed
- Implemented in a commercial system: AIXTRON BM Pro
- Single-crystal nature confirmed via SAED
- No Cr residuals as per chemical analysis
- Arrays with up to 1mm periodicity, hundreds of micrometers large crystals
- Rapid process: entire process time for array with 350 µm crystals is 2 h

Miseikis et al., 2D Materials 2017

Deterministic transfer of graphene matrixes

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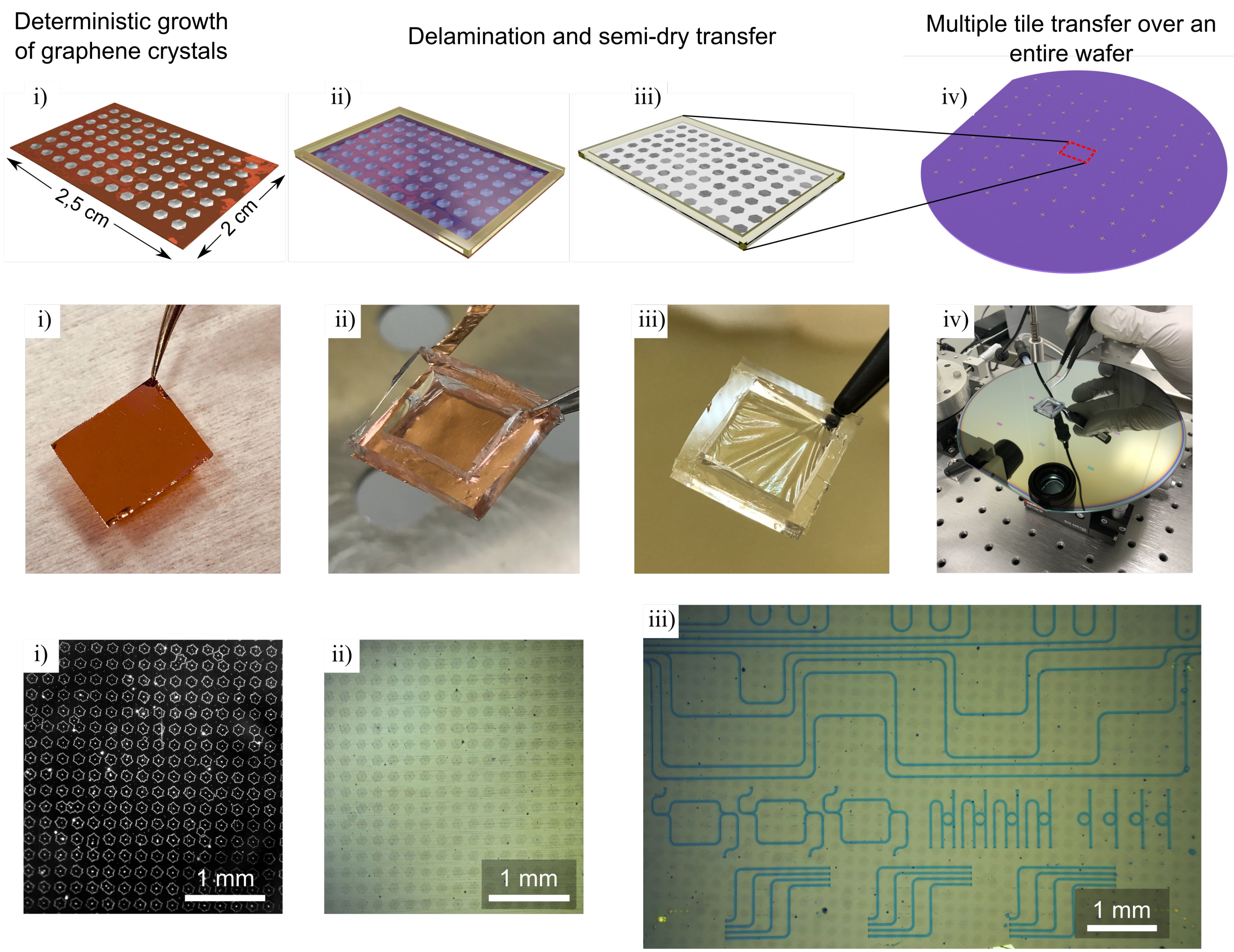
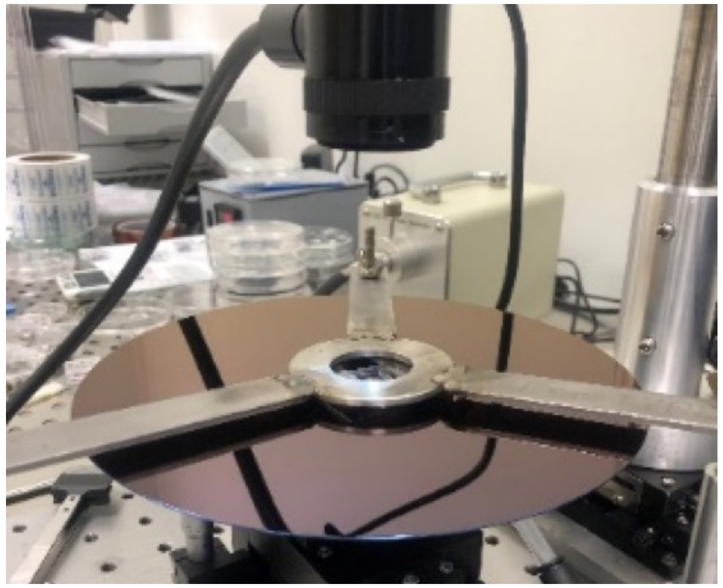
- Deterministic transfer with micro mechanical stage: reduces issues linked to transfer of large-area graphene (strain, wrinkles, adhesion)

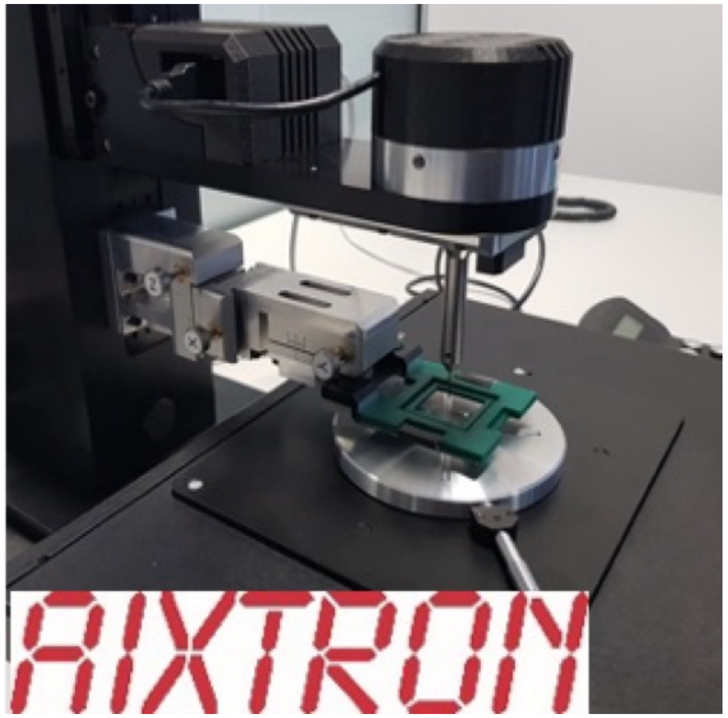
- Precise locations can be targeted, demonstrated over wafer-scale for photonics

Patent PCT/IB2020/052501
Coletti, Giambra, Miseikis, Romagnoli

- Wafer population (now manual) being automatised by Aixtron (WP10)



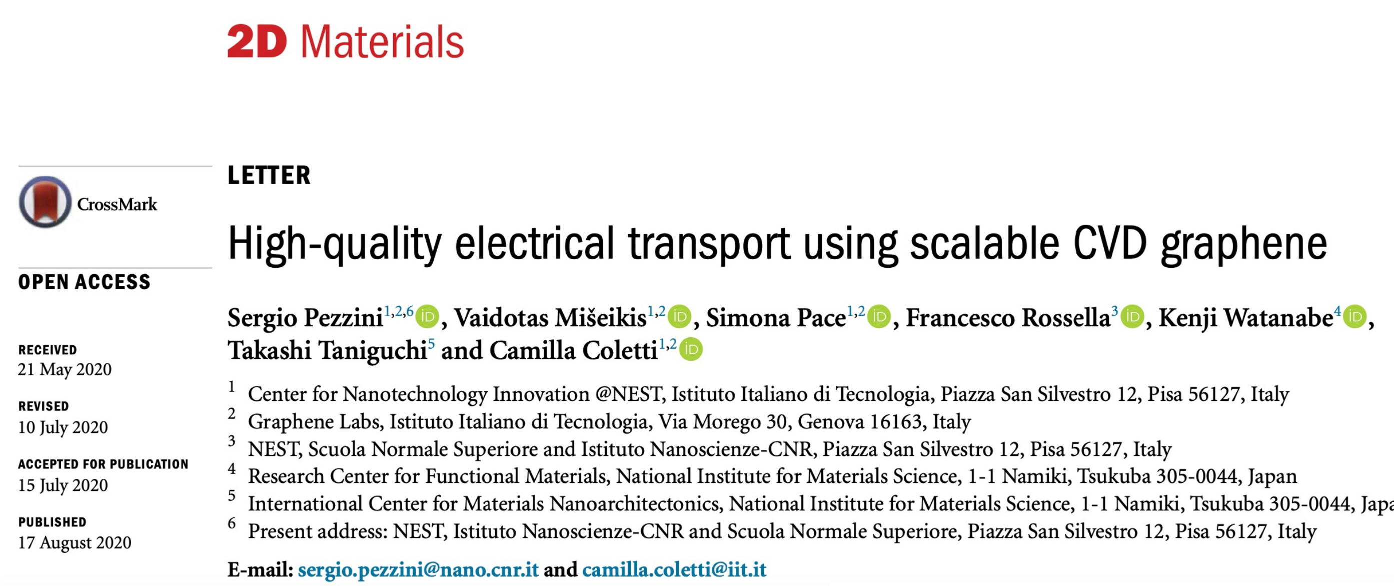




Giambra et al., ACS Nano 2021

Scalable high-quality graphene

- Ideal electric transport in CVD graphene (LT μ_D > 6×10^5 cm^2/Vs at ~10^{11} cm^{-2}) and visualisation of FQHE (Pezzini et al., 2D Materials 2020)



2D Materials

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15 July 2020

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17 August 2020

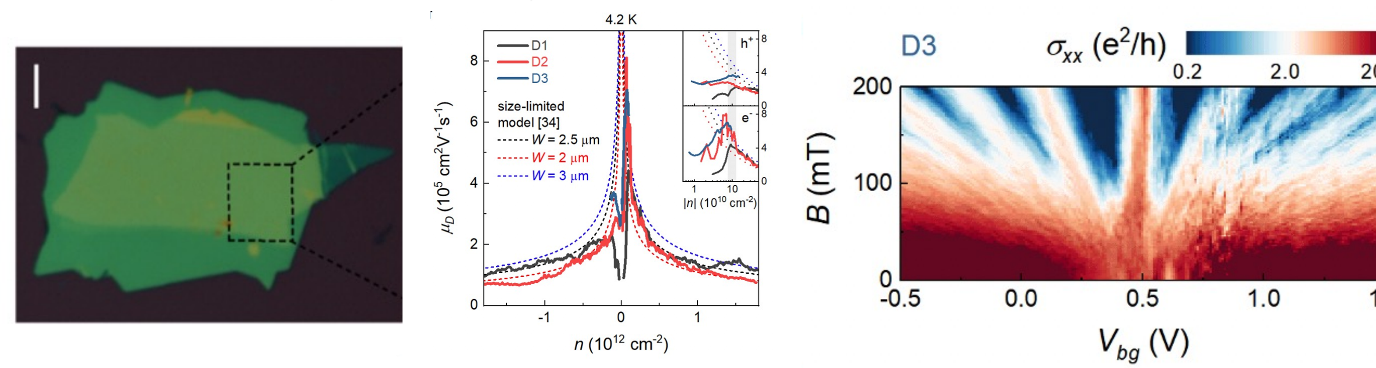
LETTER

High-quality electrical transport using scalable CVD graphene

Sergio Pezzini[1,2,6], Vaidotas Mišeikis[1,2], Simona Pace[1,2], Francesco Rossella[3], Kenji Watanabe[4], Takashi Taniguchi[5] and Camilla Coletti[1,2]

1 Center for Nanotechnology Innovation @NEST, Istituto Italiano di Tecnologia, Piazza San Silvestro 12, Pisa 56127, Italy
2 Graphene Labs, Istituto Italiano di Tecnologia, Via Morego 30, Genova 16163, Italy
3 NEST, Scuola Normale Superiore and Istituto Nanoscienze-CNR, Piazza San Silvestro 12, Pisa 56127, Italy
4 Research Center for Functional Materials, National Institute for Materials Science, 1-1 Namiki, Tsukuba 305-0044, Japan
5 International Center for Materials Nanoarchitectonics, National Institute for Materials Science, 1-1 Namiki, Tsukuba 305-0044, Japa
6 Present address: NEST, Istituto Nanoscienze-CNR and Scuola Normale Superiore, Piazza San Silvestro 12, Pisa 56127, Italy

E-mail: sergio.pezzini@nano.cnr.it and camilla.coletti@iit.it



quality of CVD graphene now comparable to that of exfoliated material!

Scalable high-quality graphene

- Ideal electric transport in CVD graphene (LT μ_D > 6×10^5 cm^2/Vs at ~10^{11} cm^{-2}) and visualisation of FQHE (Pezzini et al., 2D Materials 2020)
- Demonstration of graphene integration over 150 mm wafers: over 12 000 crystals to match final modulator geometry; demonstration of double SLG electro-absorption modulators with data rate 20 Gbps (Giambra et al., ACS Nano 2021) and phootodetectors (Miseikis et al., ACS Nano 2020; Marconi et al. Nature Communications 2021)

ACS NANO
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ARTICLE

Ultrafast, Zero-Bias, Graphene Photodetectors with Polymeric Gate Dielectric on Passive Photonic Waveguides

Vaidotas Mišeikis,◊ Simone Marconi,◊ Marco A. Giambra, Alberto Montanaro, Leonardo Martini, Filippo Fabbri, Sergio Pezzini, Giulia Piccinini, Stiven Forti, Bernat Terrés, Ilya Goykhman, Louiza Hamidouche, Pierre Legagneux, Vito Sorianello, Andrea C. Ferrari, Frank H. L. Koppens, Marco Romagnoli,* and Camilla Coletti*

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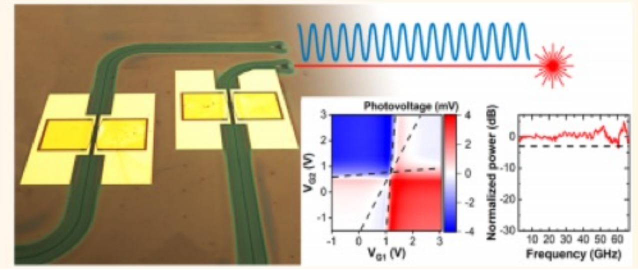


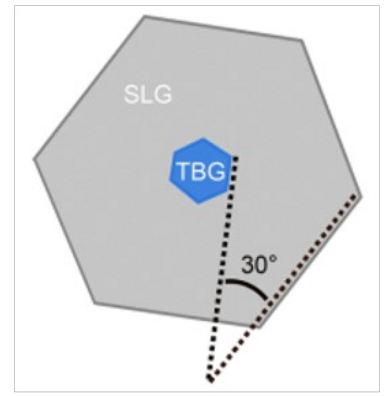
nature COMMUNICATIONS
https://doi.org/10.1038/s41467-021-21137-z OPEN

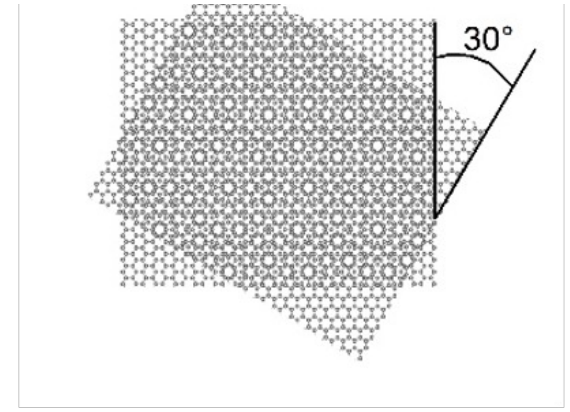
Photo thermal effect graphene detector featuring 105 Gbit s^{-1} NRZ and 120 Gbit s^{-1} PAM4 direct detection

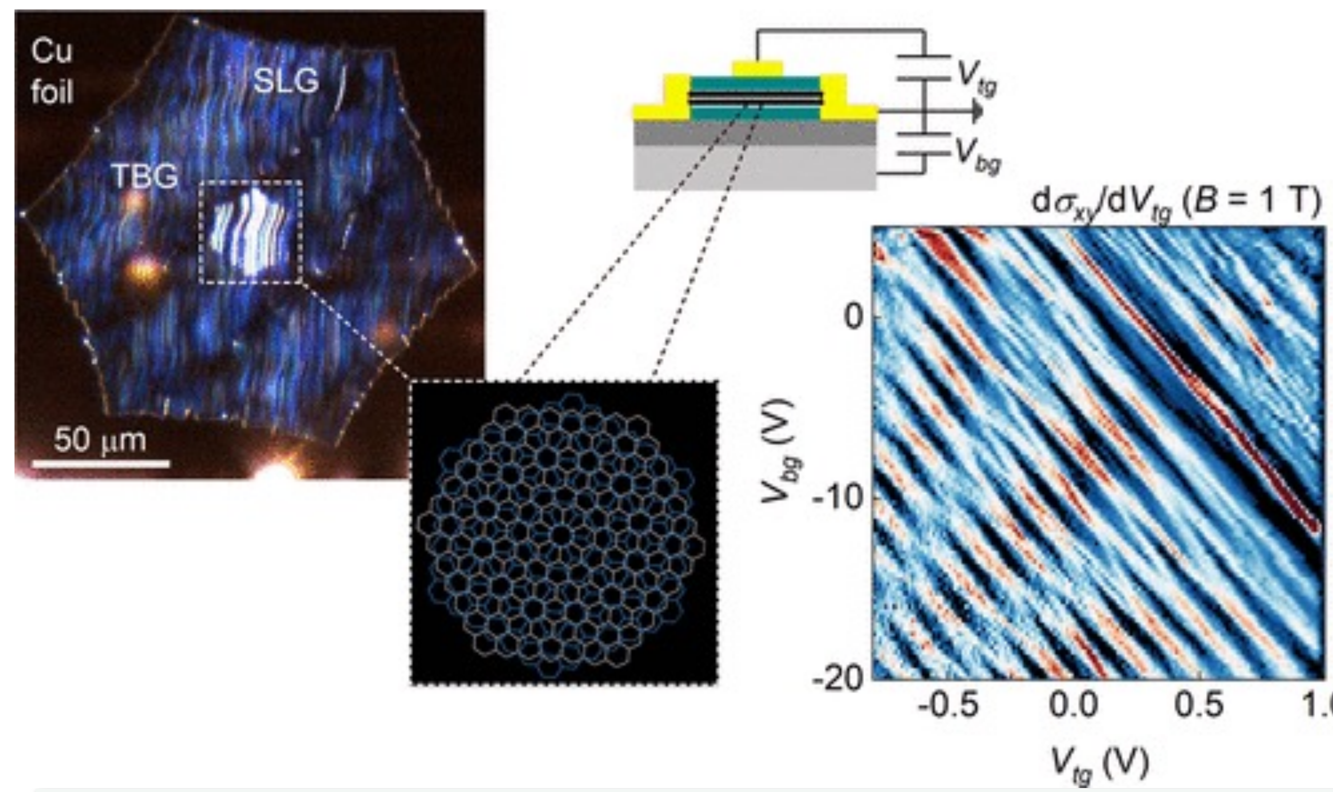
S. Marconi[1], M. A. Giambra[2], A. Montanaro[2], V. Mišeikis[3,4], S. Soresi[2,5], S. Tirelli[2,5], P. Galli[6], F. Buchali[7], W. Templ[7], C. Coletti[3,4], V. Sorianello[2] & M. Romagnoli[2✉]

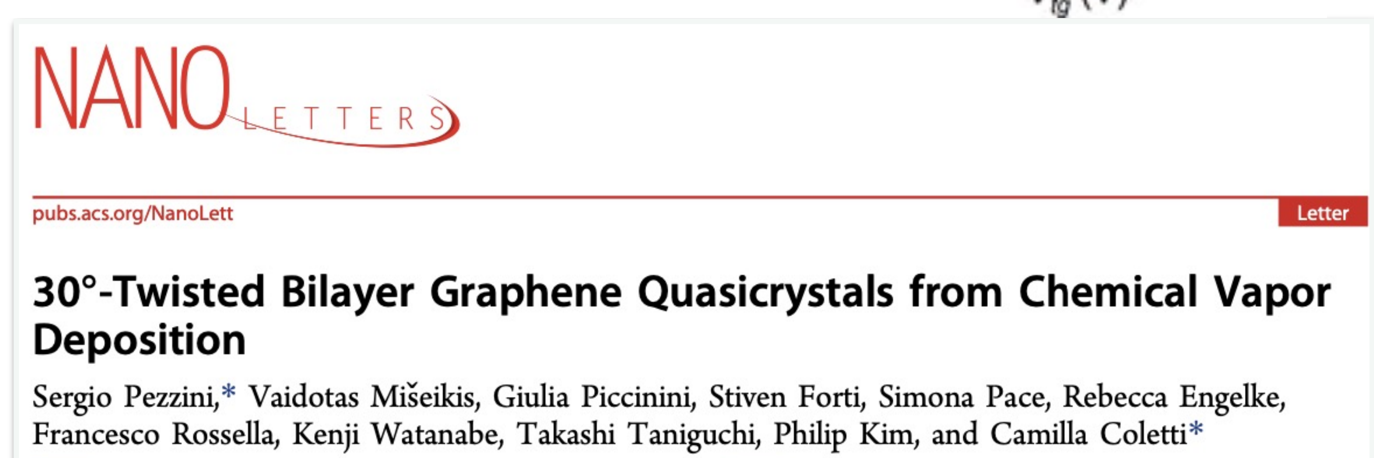
Scalable high-quality graphene

- Ideal electric transport in CVD graphene (LT μ_D > 6×10^5 cm^2/Vs at ~10^{11} cm^{-2}) and visualisation of FQHE (Pezzini et al., 2D Materials 2020)
- Demonstration of graphene integration over 150 mm wafers: over 12 000 crystals to match final modulator geometry; demonstration of double SLG electro-absorption modulators with data rate 20 Gbps (Giambra et al., ACS Nano 2021) and phootodetectors (Miseikis et al., ACS Nano 2020; Marconi et al. Nature Communications 2021)
- CVD 2D graphene quasi-crystals - 30 TBLG allowing investigation of low temperature magneto transport (Pezzini et al., Nano Letters 2020; Piccinini et al. PRB 104 2021)








NANO LETTERS

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30°-Twisted Bilayer Graphene Quasicrystals from Chemical Vapor Deposition

Sergio Pezzini,* Vaidotas Mišeikis, Giulia Piccinini, Stiven Forti, Simona Pace, Rebecca Engelke, Francesco Rossella, Kenji Watanabe, Takashi Taniguchi, Philip Kim, and Camilla Coletti*

Scalable high-quality graphene

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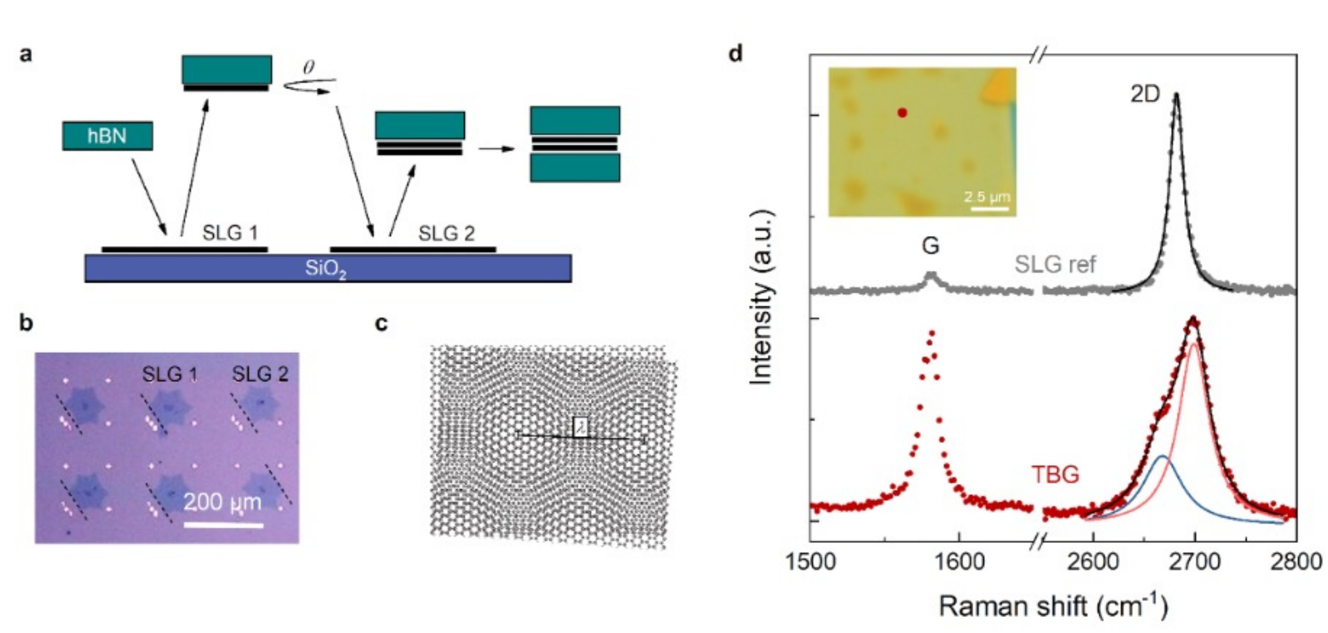
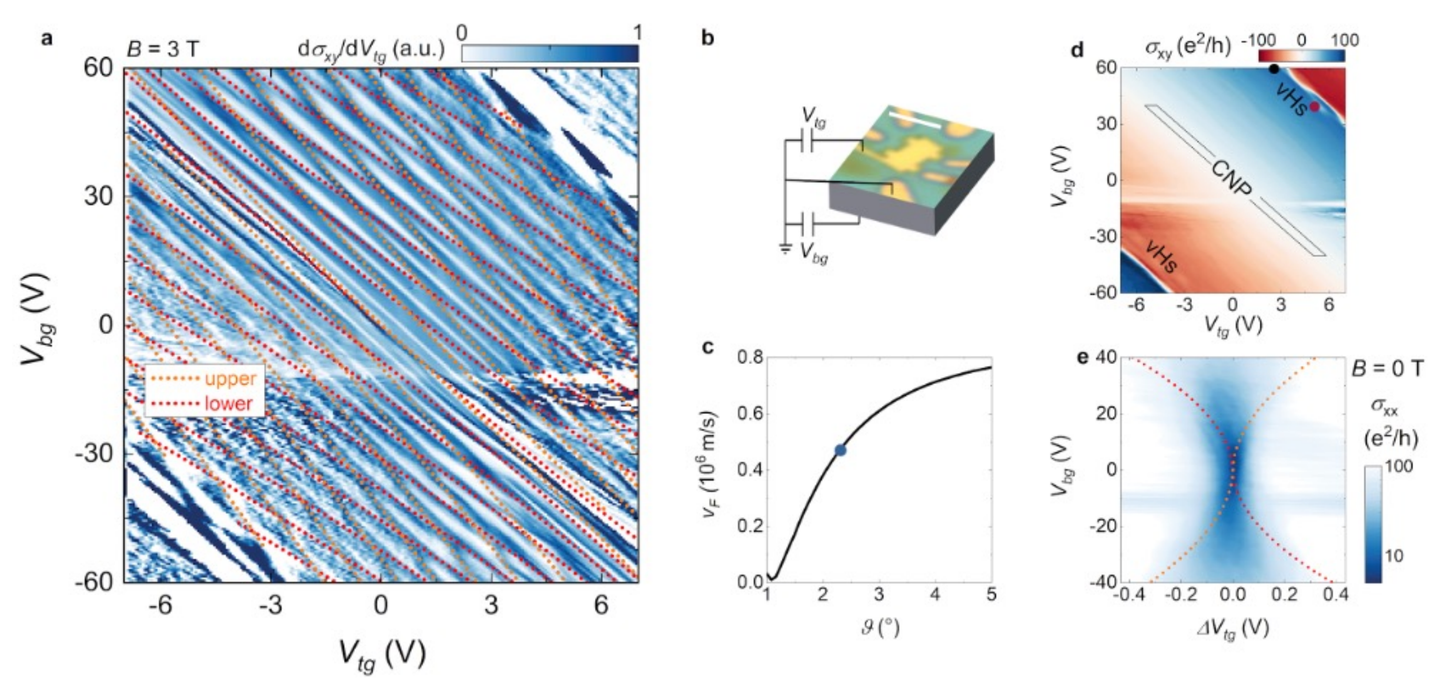
- Twistronics: vdW assembly of CVD bilayer graphene with controlled angle and study of magnetotransport (Piccinini et al., Nano Letters 2022)





Scalable high-quality graphene

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- Twistronics*:* vdW assembly of CVD bilayer graphene with controlled angle and study of magnetotransport (Piccinini et al., Nano Letters 2022)
- Demonstration of up to 9 layers CVD-grown ABC stacked graphene with device compatible size (Bouhafs et al., Carbon 2021)

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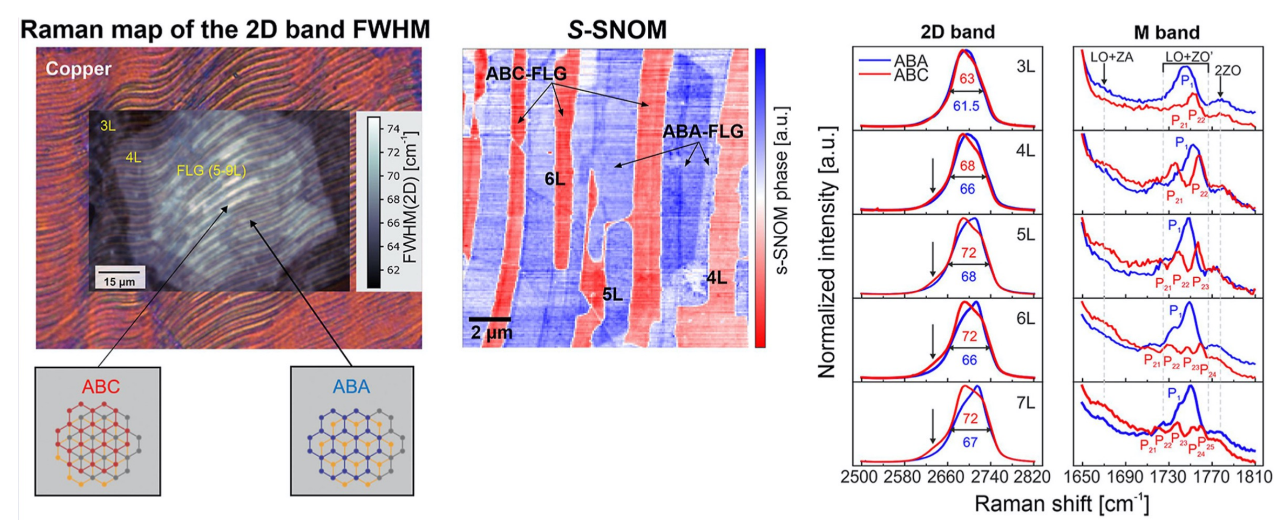
Synthesis of large-area rhombohedral few-layer graphene by chemical vapor deposition on copper

Chamseddine Bouhafs [a, b, **], Sergio Pezzini [a, b, c], Fabian R. Geisenhof [d], Neeraj Mishra [a, b], Vaidotas Mišeikis [a, b], Yuran Niu [e], Claudia Struzzi [e], R. Thomas Weitz [d, f, g], Alexei A. Zakharov [c], Stiven Forti [a], Camilla Coletti [a, b, *]



Stable, highly-sensitive graphene Hall sensors

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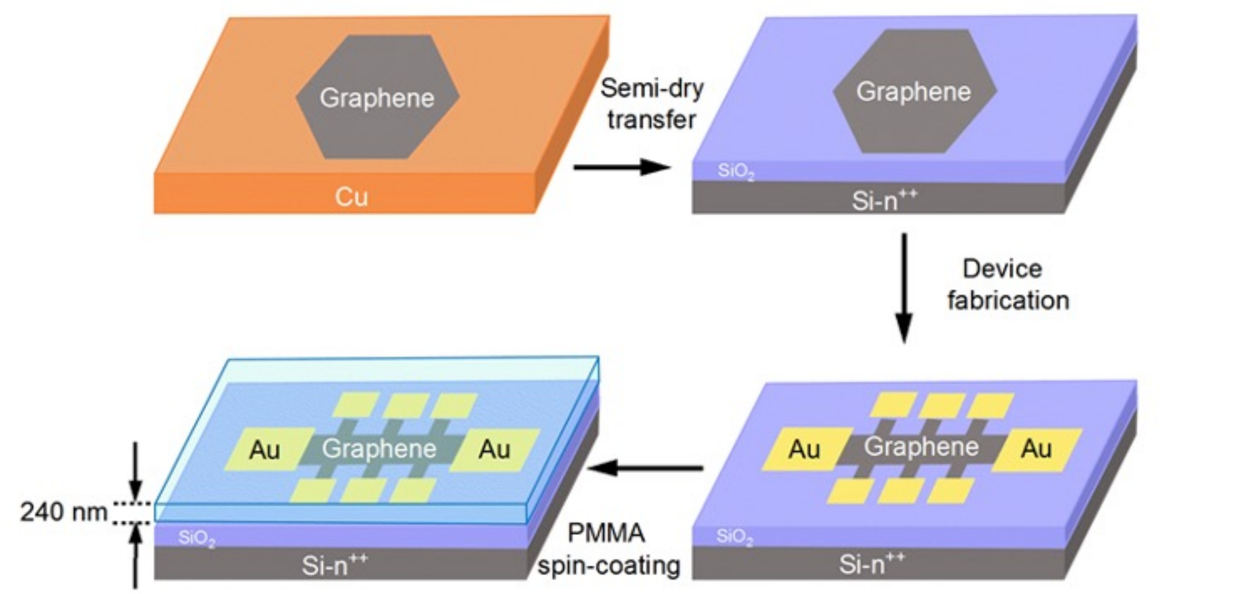
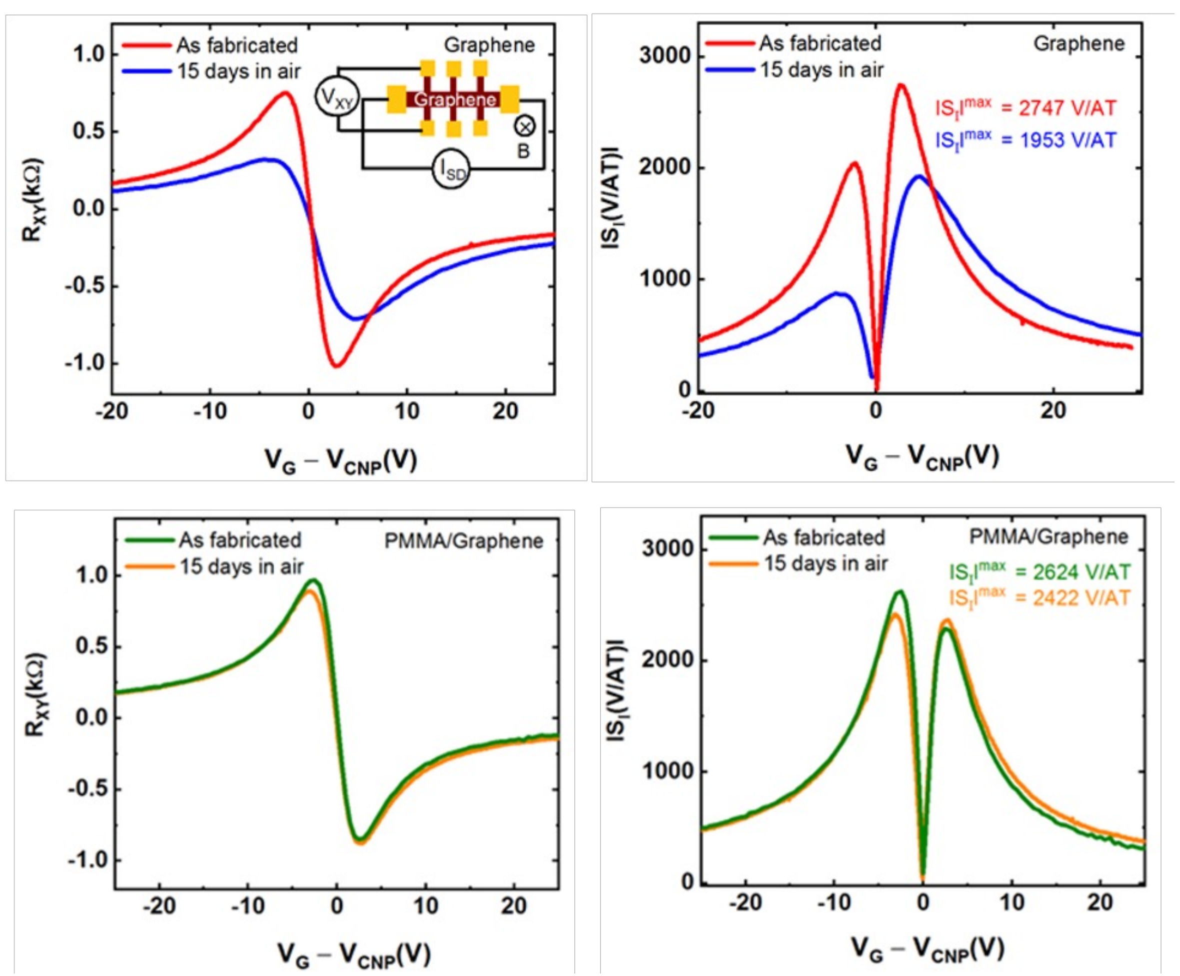
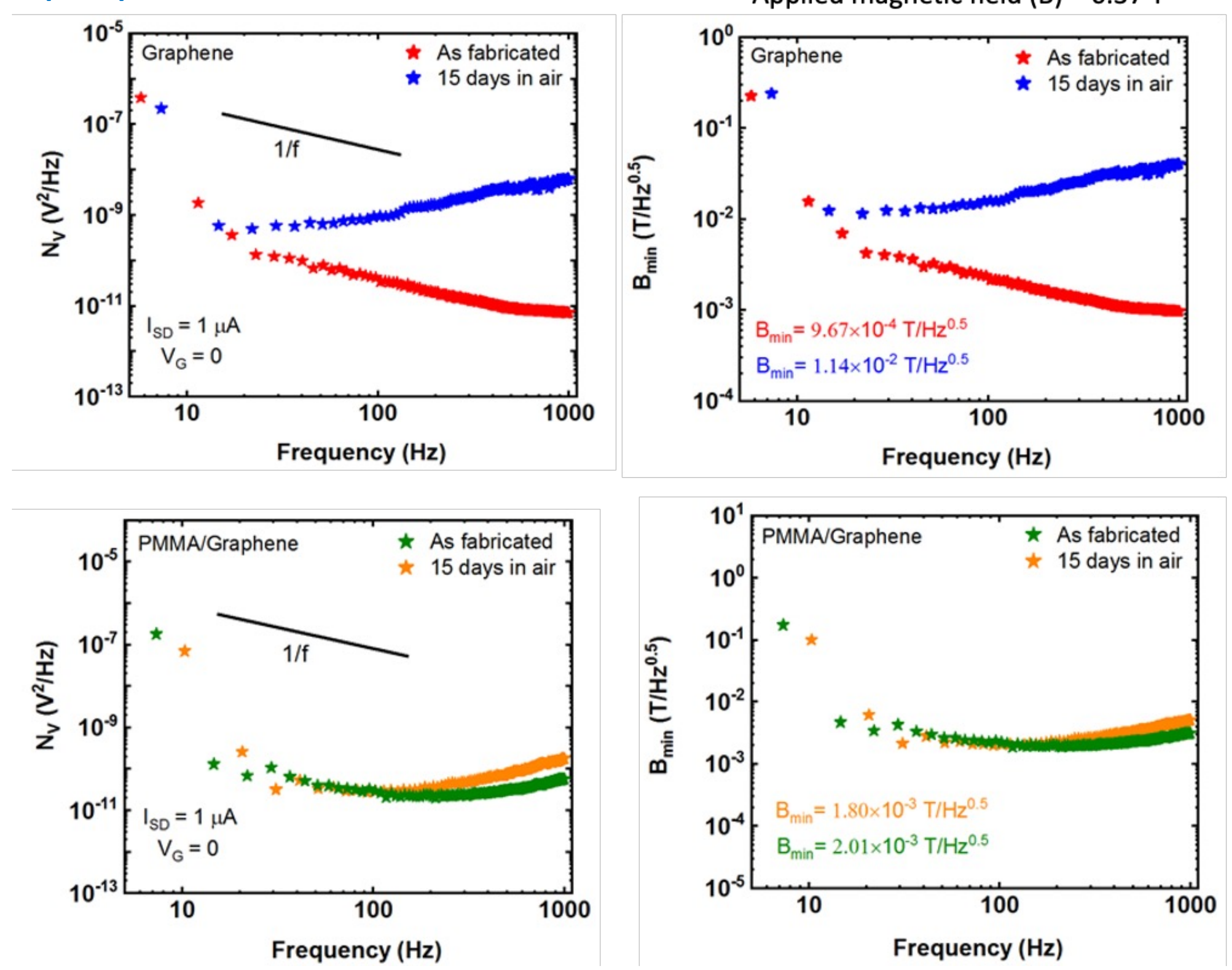
- Graphene-based all sensors: enticing prospects for various applications: from automotive to IoT
- **Goal: Increase sensitivity with highly crystalline graphene and improve stability over time**
- **How: graphene arrays and polymer (PMMA) capping**
- Hall resistance (R_{XY}), current related sensitivity (S_I) significantly decrease (>50% in 15 days) for bare graphene Hall-sensors in ai while noise increases
- Stable values for PMMA/graphene stable **after 15 days** with current sensitivity **S_I =2422 V/AT**, low noise and high B_{min} values observed for array of devices



Tyagi et al., in preparation



Constant current bias (I) = 1 μA,
Applied magnetic field (B) = 0.37 T



Gaseous detectors

Collaboration with INFN, Roma Tre, Sapienza, CERN on the development of gas detector to study radation-matter interaction

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Transmission through graphene of electrons in the 30 - 900 eV range

Alice Apponi*[1,4], Domenica Convertino[2], Neeraj Mishra[2,3], Camilla Coletti[2,3], Mauro Iodice[4], Franco Frasconi[5], Federico Pilo[5], Gianluca Cavoto[6], and Alessandro Ruocco[1,4]

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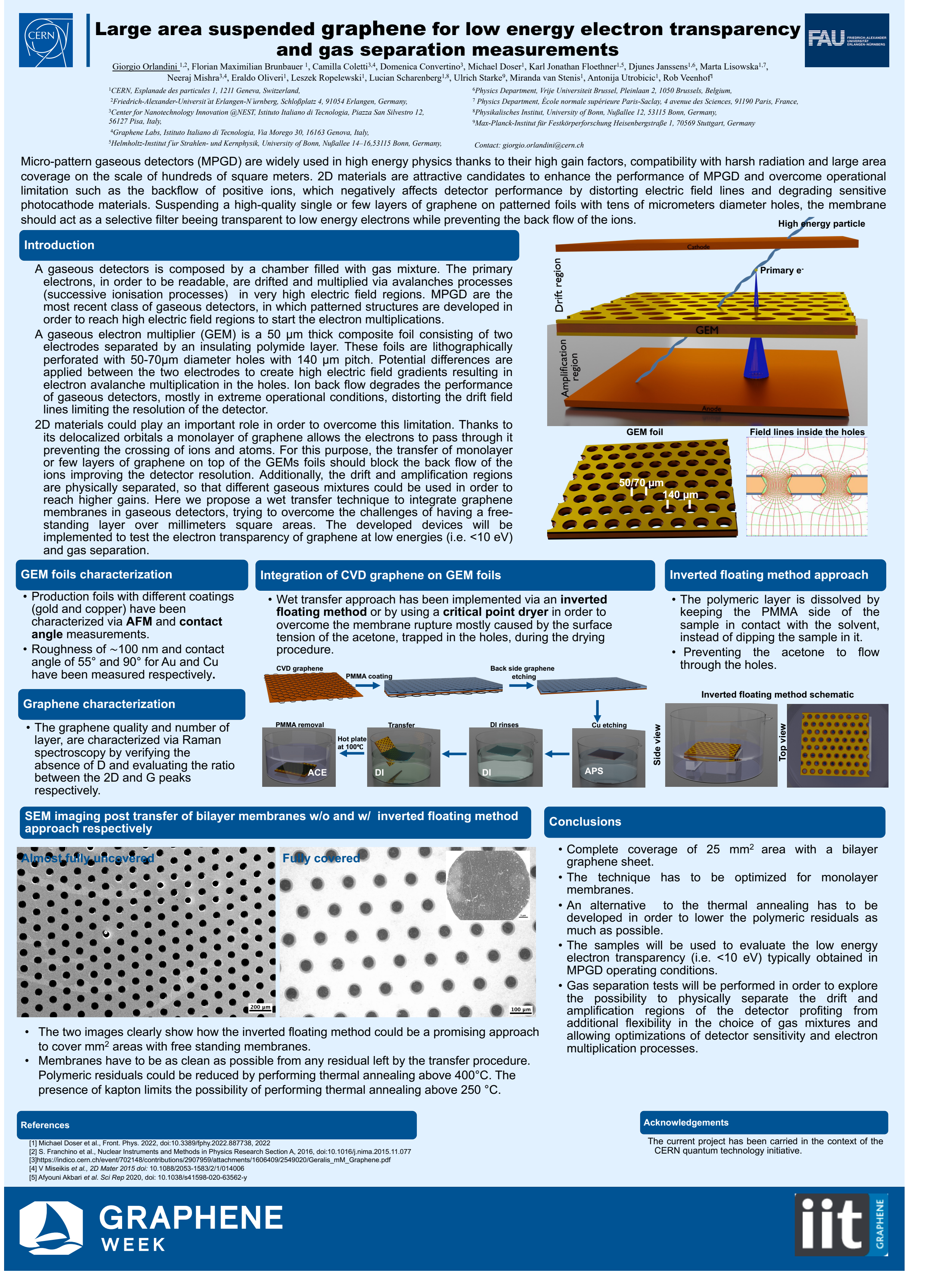
Abstract

We report on the transmission of low-energy electrons through monolayer and trilayer graphene. Polycrystalline graphene was grown via chemical vapor deposition and transferred onto TEM grids. A custom-made monochromatic electron gun has been employed to perform the transmission measurements in the 30 - 900 eV range in vacuum. The transmission of graphene layer(s) is obtained from the absolute measurement of the direct beam current and the transmitted one, by means of a Faraday cup. In order to fully characterize the graphene, we performed spectroscopy with micro-Raman, X-ray photoemission and electron energy loss. We observed evidences of good quality suspended graphene.



Large area suspended graphene for low energy electron transparency and gas separation measurements

Giorgio Orlandini[1,2], Florian Maximilian Brunbauer[1], Camilla Coletti[3,4], Domenica Convertino[3], Michael Doser[1], Karl Jonathan Floethner[1,5], Djunes Janssens[1,6], Marta Lisowska[1,7], Neeraj Mishra[3,4], Eraldo Oliveri[1], Leszek Ropelewski[1], Lucian Scharenberg[1,8], Ulrich Starke[9], Miranda van Stenis[1], Antonija Utrobicic[1], Rob Veenhof[1]

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Micro-pattern gaseous detectors (MPGD) are widely used in high energy physics thanks to their high gain factors, compatibility with harsh radiation and large area coverage on the scale of hundreds of square meters. 2D materials are attractive candidates to enhance the performance of MPGD and overcome operational limitation such as the backflow of positive ions, which negatively affects detector performance by distorting electric field lines and degrading sensitive photocathode materials. Suspending a high-quality single or few layers of graphene on patterned foils with tens of micrometers diameter holes, the membrane should act as a selective filter beeing transparent to low energy electrons while preventing the back flow of the ions.

Introduction

A gaseous detectors is composed by a chamber filled with gas mixture. The primary electrons, in order to be readable, are drifted and multiplied via avalanches processes (successive ionisation processes) in very high electric field regions. MPGD are the most recent class of gaseous detectors, in which patterned structures are developed in order to reach high electric field regions to start the electron multiplications.

A gaseous electron multiplier (GEM) is a 50 µm thick composite foil consisting of two electrodes separated by an insulating polymide layer. These foils are lithographically perforated with 50-70µm diameter holes with 140 µm pitch. Potential differences are applied between the two electrodes to create high electric field gradients resulting in electron avalanche multiplication in the holes. Ion back flow degrades the performance of gaseous detectors, mostly in extreme operational conditions, distorting the drift field lines limiting the resolution of the detector.

2D materials could play an important role in order to overcome this limitation. Thanks to its delocalized orbitals a monolayer of graphene allows the electrons to pass through it preventing the crossing of ions and atoms. For this purpose, the transfer of monolayer or few layers of graphene on top of the GEMs foils should block the back flow of the ions improving the detector resolution. Additionally, the drift and amplification regions are physically separated, so that different gaseous mixtures could be used in order to reach higher gains. Here we propose a wet transfer technique to integrate graphene membranes in gaseous detectors, trying to overcome the challenges of having a free-standing layer over millimeters square areas. The developed devices will be implemented to test the electron transparency of graphene at low energies (i.e. <10 eV) and gas separation.

GEM foils characterization

- Production foils with different coatings (gold and copper) have been characterized via **AFM** and **contact angle** measurements.
- Roughness of ~100 nm and contact angle of 55° and 90° for Au and Cu have been measured respectively.

Graphene characterization

- The graphene quality and number of layer, are characterized via Raman spectroscopy by verifying the absence of D and evaluating the ratio between the 2D and G peaks respectively.

Integration of CVD graphene on GEM foils

- Wet transfer approach has been implemented via an **inverted floating method** or by using a **critical point dryer** in order to overcome the membrane rupture mostly caused by the surface tension of the acetone, trapped in the holes, during the drying procedure.

Inverted floating method approach

- The polymeric layer is dissolved by keeping the PMMA side of the sample in contact with the solvent, instead of dipping the sample in it.
- Preventing the acetone to flow through the holes.

SEM imaging post transfer of bilayer membranes w/o and w/ inverted floating method approach respectively

- The two images clearly show how the inverted floating method could be a promising approach to cover mm^2 areas with free standing membranes.
- Membranes have to be as clean as possible from any residual left by the transfer procedure. Polymeric residuals could be reduced by performing thermal annealing above 400°C. The presence of kapton limits the possibility of performing thermal annealing above 250 °C.

Conclusions

- Complete coverage of 25 mm^2 area with a bilayer graphene sheet.
- The technique has to be optimized for monolayer membranes.
- An alternative to the thermal annealing has to be developed in order to lower the polymeric residuals as much as possible.
- The samples will be used to evaluate the low energy electron transparency (i.e. <10 eV) typically obtained in MPGD operating conditions.
- Gas separation tests will be performed in order to explore the possibility to physically separate the drift and amplification regions of the detector profiting from additional flexibility in the choice of gas mixtures and allowing optimizations of detector sensitivity and electron multiplication processes.

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[2] S. Franchino et al., Nuclear Instruments and Methods in Physics Research Section A, 2016, doi:10.1016/j.nima.2015.11.077
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[4] V Miseikis *et al., 2D Mater 2015 doi:* 10.1088/2053-1583/2/1/014006
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GRAPHENE WEEK

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