FNTG Research Society

International workshop on

Advances in Nano-Materials and Nano-Devices

Abstracts

October 24, 2019

Lecture room 232, Engineering Building 2

The University of Tokyo

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University of Vienna, Aalto University,

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The Fullerenes, Nanotubes and Graphene Research Society

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Workshop Venue:

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UTokyo Hongo Campus (7-3-1 Hongo, Bunkyo-ku, Tokyo 113-8656, JAPAN)

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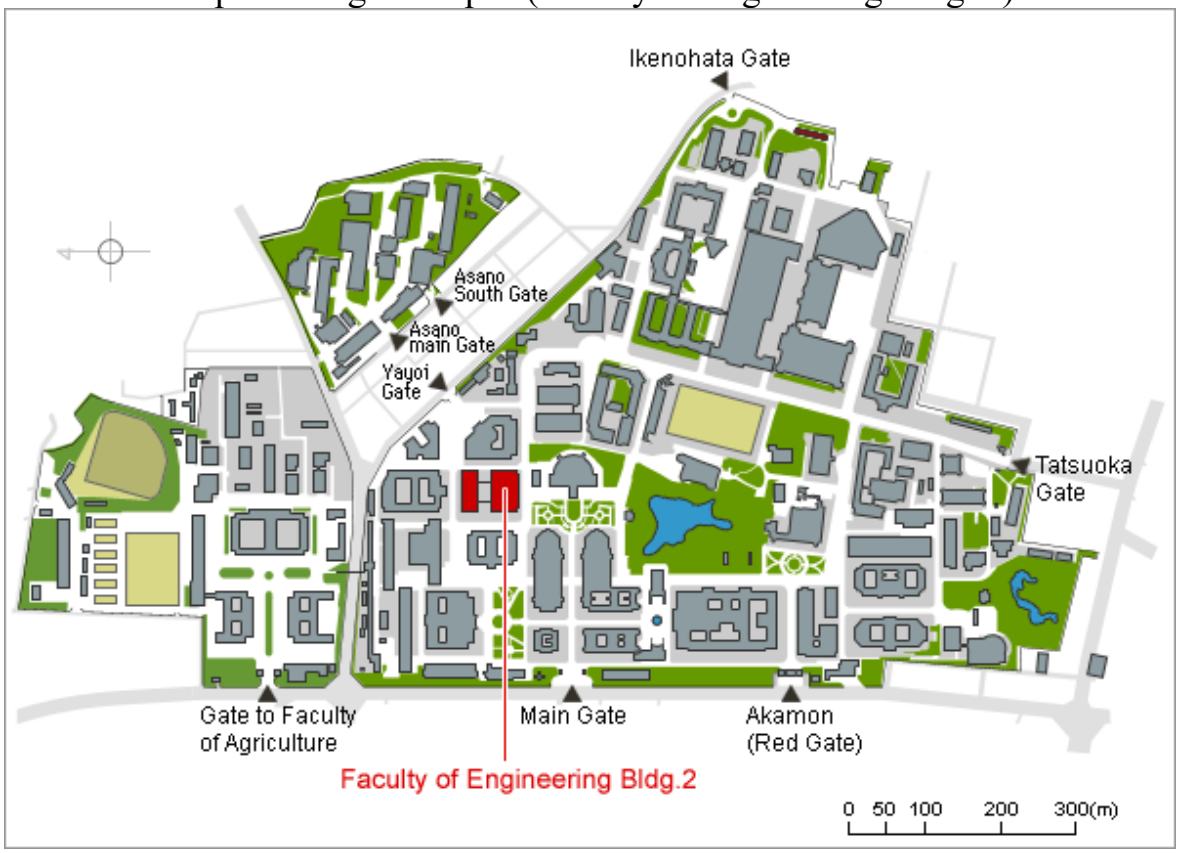

Contact: Dr. Shigeo Maruyama
Department of Mechanical Engineering, The University of Tokyo
TEL: +81-3-5841-6421 TEL&FAX: +81-3-5800-6983
E-MAIL: maruyama@photon.t.u-tokyo.ac.jp, URL: http://www.photon.t.u-tokyo.ac.jp/

International workshop on

Advances in Nano-Materials and Nano-Devices

Date: October 24, 2019, 10:00-17:40

Venue: Lecture room 232, 3rd Floor, Engineering Building II, The University of Tokyo

7-3-1 Hongo, Bunkyo-ku, Tokyo 113-8656, JAPAN

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10:00-	**Opening**
10:00-10:40	**Esko I. Kauppinen (Aalto University)** *On the control of the single walled carbon nanotube (SWNT) atomic structure and thin film color during the floating catalyst chemical vapor deposition (FCCVD) synthesis*
10:40-11:00	**Jani Kotakoski (University of Vienna)** *Atomic-resolution study of nanomaterials in Vienna*
11:00-11:20	*Break*
11:20-12:00	**Shigeo Maruyama (The University of Tokyo)** *One-dimensional hetero-structures based on SWCNTs*
12:00-13:20	*Lunch break*
13:20-14:00	**Clemens Mangler (University of Vienna)** *Experimental manipulation and microscopy setup in Vienna*
14:00-14:20	**Yongjia Zheng (The University of Tokyo)** *CVD growth of one-dimensional hetero-structures*
14:20-14:40	**Ming Liu (The University of Tokyo)** *Synthesis of MoS_2 nanotubes based on BN nanotubes*
14:40-15:20	**Gregor Leuthner (University of Vienna)** *Physical and chemical manipulation of nanomaterials in the electron microscope*
15:20-15:40	*Break*
15:40-16:20	**Harriet Åhlgren (University of Vienna)** *Computational studies of atomic-scale manipulation*
16:20-17:00	**Yutaka Ohno (Nagoya University)** *Doping control for low-voltage operation of CNT-based CMOS ICs*
17:00-17:40	**Kimmo Mustonen (University of Vienna)** *In situ electronic measurement during atomic resolution imaging*
17:40-	**Closing**
18:00-20:00	*Reception (Sanjo Conference Hall)*

On the control of the single walled carbon nanotube (SWNT) atomic structure and thin film color during the floating catalyst chemical vapor deposition (FC-CVD) synthesis

Esko I. Kauppinen

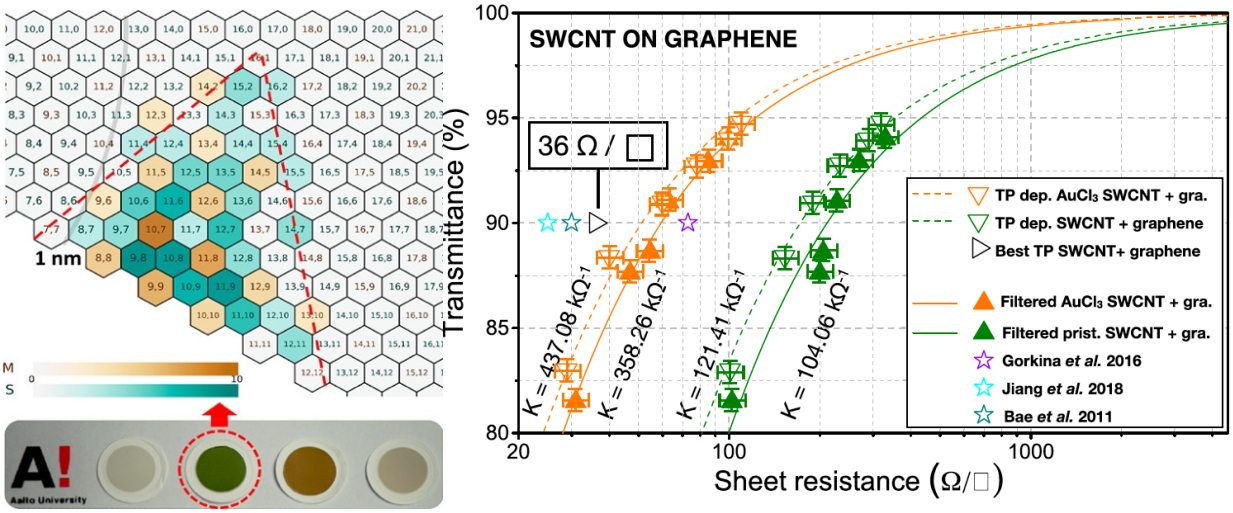
Aalto University School of Science, Department of Applied Physics

PO Box 15100, FI-00076 Aalto, FINLAND

esko.kauppinen@aalto.fi

Abstract

We discuss the FC-CVD synthesis of SWNTs, especially the tuning of tube atomic structure i.e. (n,m) distributions and subsequently the thin film color. Ferrocene has been used as the catalyst nanoparticle precursor and CO, CH_4, C_2H_4, ethanol, methanol and toluene as the carbon precursors, with CO_2, H_2O, tiophene and H_2S as the respective additives to tune (n,m) distributions. By introducing various amount of CO_2 in FC-CVD with CO as the carbon source and in-situ ferrocene decomposition generated Fe catalyst nanoparticles, we directly synthesized and dry-deposited SWNT films with tunable (n,m) i.e. helicity distribution as well as tunable colors [1]. When operating the FC-CVD reactor at the ambient pressure and at 850 ^oC temperature with 0.25 and 0.37 volume percent of added CO_2, the SWNT films display green and brown colors, respectively. We ascribed various colors to suitable diameter and narrow (n,m) distributions, which were determined in detail using the electron diffraction. We will present results on using ethylene as the carbon source in nitrogen carrier gas with the addition of H_2O vapor to synthesize SWNTs with extremely narrow (n,m) distribution and directly deposit colorful films. Also, we will discuss studies on SWNT synthesis when using alcohols as carbon sources with tiophene as the growth promoter. When adding methanol with ethanol, we enhance the fraction of semiconducting tubes. Finally, we present interesting recent results on the simultaneous FC-CVD synthesis of fullerenes, graphene and SWNTs, as well as combining SWNTs and graphene for the transparent, conducting film [2].



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[1] Y. Liao et al. *J. Am. Chem. Soc.* **140**, 31, 9797 (2018).

[2] Y. Liao et al., *ACS Nano* **DOI: 10.1021/acsnano.9b05049** (2019).

Atomic-resolution study of nanomaterials in Vienna

Jani Kotakoski[1]

[1] Faculty of Physics, University of Vienna

The group Physics of Nanostructured Materials operates two atomic-resolution electron microscopes: a Nion UltraSTEM 100 since 2015 and a FEI Titan 80-300 since 2018. In this presentation, I will introduce the Vienna research group working with these instruments, and give an overview of the related research activities. The main focus of this presentation will be on the study of the three-dimensional shape of two-dimensional materials and their heterostructures. The topics will include defect-induced out-of-plane buckling of graphene [1–3], intrinsic ripples in graphene and their control in the electron microscope [4], and buckling of graphene in a graphene–h-BN [5] and graphene–carbon nanotube [6] van der Waals heterostructures.

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3. C. Hofer, V. Skakalova, M.R.A. Monazam, C. Mangler, J. Kotakoski, T. Susi & J.C. Meyer, Direct visualization of the 3D structure of silicon impurities in graphene, Appl. Phys. Lett. 114, 053102 (2019).
4. U. Ludacka, M.R.A. Monazam, C. Rentenberger, M. Friedrich, U. Stefanelli, J.C. Meyer & J. Kotakoski, In situ control over graphene ripples and strain in the electron microscope, npj 2D Materials and Applications 2, 25 (2018).
5. G. Argentero, A. Mittelberger, M.R.A. Monazam, Y. Cao, T.J. Pennycook, C. Mangler, C. Kramberger-Kaplan, J. Kotakoski, A.K. Geim & J.C. Meyer, Unraveling the 3D atomic structure of a suspended graphene/hBN van der Waals heterostructure, Nano Lett. 17, 1409-1416 (2017).
6. K. Mustonen, A. Hussain, C. Hofer, M. Monazam, R. Mirzayev, K. Elibol, P. Laiho, C. Mangler, H. Jiang, T. Susi, E. Kauppinen, J. Kotakoski & J.C. Meyer, Atomic-Scale Deformations at the Interface of a Mixed-Dimensional van der Waals Heterostructure, ACS Nano 12, 8512-8519 (2018).

One-dimensional hetero-structures based on SWCNTs

Shigeo Maruyama[1,2]

[1] Department of Mechanical Engineering, The University of Tokyo, Tokyo 113-8656, Japan
[2] Energy NanoEngineering Lab., National Institute of Advanced Industrial Science and Technology (AIST), Tsukuba 305-8564, Japan
maruyama@photon.t.u-tokyo.ac.jp

We have synthesized a new coaxial nanotube structure, in which mono- or few-layer hexagonal boron nitride nanotube (BNNT) seamlessly wrap around a single-walled carbon nanotube (SWCNT), and result in an atomically smooth coaxial tube consisting of two different tubes as in Fig. 1(a) [1]. TEM-EELS clearly demonstrated the BNNT-SWCNT coaxial structure. We can clearly observe the coaxial structure from originally isolated SWCNTs as in Fig. 1(b). Through nano-diffraction TEM measurements, we found no correlation between chiral angle of inner SWCNT and outer BNNT for 'double-walled' SWCNT-BNNT. Based on this non epitaxial nature, we concluded that these are one-dimensional van der Waals heterostructure. We have further developed the 1D coating CVD for transition metal dichalcogenide nanotubes, such as MoS_2 nanotube and WSe_2 nanotubes. We can grow MoS_2 nanotubes around relatively large diameter SWCNT or SWCNT@BNNT. Optical properties of isolated and film samples are discussed, though absorption, Raman and photoluminescence. So far, the coating growth can be efficiently performed for vertically aligned array of SWCNT, suspended SWCNTs between pillars, zeolite based bulk sample and dry-deposited random network films. So far, the full coverage of BNNT is not possible on horizontally alighted SWCNT on crystal quartz. For field effect transistor (FET) devices, we need to start from suspended tubes. Instead, we will discuss applications of film samples such as saturable absorber of mode-lock fiber lasers or as hole transport layer & electrode of perovskite solar cells.

Reference: [1] R. Xiang, T. Inoue, Y. Zheng, A. Kumamoto, Y. Qian, Y. Sato, M. Liu, D. Gokhale, J. Guo, K. Hisama, S. Yotsumoto, T. Ogamoto, H. Arai, Y. Kobayashi, H. Zhang, B. Hou, A. Anissimov, Y. Miyata, S. Okada, S. Chiashi, Y. Li, E. I. Kauppinen, Y. Ikuhara, K. Suenaga, S. Maruyama, arXiv:1807.06154 (2019).

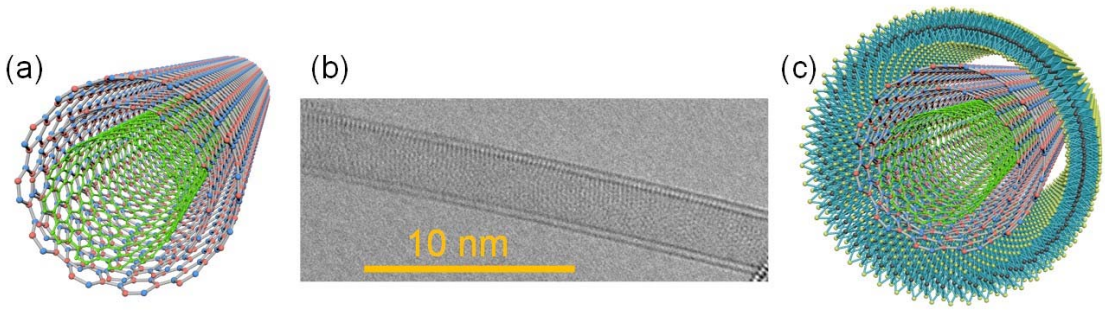

Fig.1 (a) Schematics of SWCNT@BNNT, (b) High resolution TEM image of SWCNT@BNNT, (c) Schematics of SWCNT@BNNT@MoS_2NT

Experimental manipulation and microscopy setup in Vienna

Clemens Mangler[1]

[1] Faculty of Physics, University of Vienna

Carrying out experiments on low dimensional materials can be challenging due to their very high surface-to-volume ratio. As a consequence it is important to focus on carefully managing the environmental conditions during the whole experimental life-cycle of these materials. Typical setups usually include ultra-high vacuum (UHV) technology.

In my presentation, I will give an overview of the UHV system built by our group at the University of Vienna featuring various experimental capabilities including aberration-corrected scanning transmission electron microscopy, atomic force microscopy, evaporation and electrical measurements. Also a unique UHV transfer system will be shown.

As an example study with the Vienna setup, the growth of a novel 2D metal structure on suspended 2D materials, graphene and hexagonal boron nitride, will be presented [1]. The structure consists of two atomic layers, one of gold and the other one of copper, that arrange into a hexagonal crystal structure. Until now, islands with sizes up to tens of nanometers have been grown, but there should be no fundamental limit to their size.

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CVD growth of one-dimensional heterostructures

Yongjia Zheng[1], Rong Xiang[1], Taiki Inoue[1], Yang Qian[1], Ming Liu[1], Shohei Chiashi[1], Esko I. Kauppinen[2], Shigeo Maruyama[1,3]

[1] *Department of Mechanical Engineering, The University of Tokyo, Tokyo 113-8656, Japan*

[2] *Department of Applied Physics, Aalto University School of Science, PO Box 15100, FI-00076 Aalto, Finland*

[3] *Energy Nanoengineering Lab., National Institute of Advanced Industrial Science and Technology (AIST), Tsukuba. 305-8564, Japan*

In recent years researchers have focused on two-dimensional van der Waals heterostructures (vdWH), which have generated great interests recently due to the possibility of combining diverse atomic layers to create novel materials and devices [1-2]. In this work, we demonstrate a new one-dimensional van der Waals heterostructures (vdWH) nanotube structure with similar heterostructure interfaces that combines the single-walled carbon nanotubes (SWCNTs), boron nitride nanotubes (BNNTs) and molybdenum disulfide nanotubes (MSNTs) in the radial direction. Techniques involving direct growth of 1D vdWH by chemical vapor deposition (CVD) will be presented in detail. Ammonia borane (BH_3NH_3) as precursor was directly used to synthesize BNNTs with the aid of SWCNTs as a template by a facile CVD technique. MSNTs are synthesized on as-prepared SWCNTs/BNNTs by CVD using MoO_3 and S powders as the reactants. Absorption spectra, Raman spectra, optical images confirmed the existence of boron nitride (BN) and MoS_2. TEM image and electron diffraction pattern clearly shows a ternary nanotube that consists of SWCNT/BNNTs/ MSNTs in a coaxial structure [3]. What's more, different heterostructures, such as SWCNT/BNNTs/CNTs, are also achievable using our CVD method. We believe this vdWH will have a broad interest and impact in many fields, which include but not limited in investigating the intrinsic optical properties of environment-isolated SWCNTs, fabricating BN-protected or gated SWCNT devices and building more sophisticated 1D material systems. It can also be used for photovoltaics and light-emitting devices when combining different kinds of transition metal dichalcogenide monolayers (TMDC) materials.

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[2] K. S. Novoselov *et al., Science,* **353,** 6298 (2016).
[3] R. Xiang *et al.,* arXiv:1807.06154

Corresponding Author: Shigeo Maruyama, Tel: +81-3-5841-6421, Fax: +81-3-5841-6421,
E-mail: maruyama@photon.t.u-tokyo.ac.jp

Synthesis of MoS_2 nanotubes based on BN nanotubes

Ming Liu[1], Yongjia Zheng[1], Yang Qian[1], Rong Xiang[1], Taiki Inoue[1], Shohei Chiashi[1], Esko I.Kauppinen[2], Shigeo Maruyama[1,3]

[1] Department of Mechanical Engineering, The University of Tokyo, Tokyo 113-8656, Japan
[2] Department of Applied Physics, Aalto University School of Science, PO Box 15100, FI-00076 Aalto, Finland
[3] Energy NanoEngineering Lab., National Institute of Advanced Industrial Science and Technology (AIST), Tsukuba 305-8564, Japan

Inorganic nanotubes have initiated a surge of intensive research in the last few years since the discovery of them in 1992 [1]. Lately, MoS_2 nanotubes [2] have started to be produced in decent amounts and with promising potentials for large scale application, mostly as superior solid lubricants and in nanocomposites. The synthesis of single-walled MoS_2 nanotubes is still a challenge in the field, however, and a combined theoretical and experimental work indicated that multiwall MoS_2 nanotubes with an inner core larger than 6 nm are more stable than single-walled nanotubes [3]. In this work, we successfully encapsulated single-walled MoS_2 nanotubes in BN nanotubes and demonstrated the application of BN nanotubes as templates for the formation of core-shell nanotubes structure, see Figure 1(a). The wide cavity of BN nanotubes makes it practically possible for another layered material (MoS_2) to fit into the core and enfold within it, forming a nanotube within a nanotube. Besides, BN nanotubes are electrically insulating (a band gap of ~5-6 eV) with profound chemical and thermal stabilities, which makes the core shell structure promising for investigating the optical properties of single-walled MoS_2 nanotubes. Therefore, the optical properties of single-walled MoS_2 nanotubes are discussed through absorption, Raman and photoluminescence spectra. Meanwhile, the structure of encapsulating MoS_2 nanotubes (Figure 1(b)) and BN nanotubes are examined by TEM characterization.

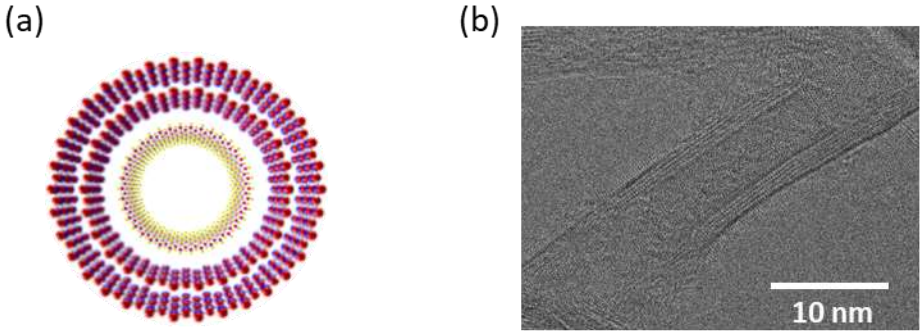

Fig. 1 (a) Schematic structure of MoS_2@BNNT; (b) Representative TEM image of MoS2@BNNT

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[2] Y. Feldman, E. Wasserman, D. J. Srolovitz, R. Tenne, Science 1995, 267, 222.
[3] G. Seifert, T. Kohler, R. Tenne, J. Phys. Chem. B 2002, 106, 2497.

Physical and chemical manipulation of 2D materials in the electron microscope

Gregor T. Leuthner[1]

[1] Faculty of Physics, University of Vienna

Transmission electron microscopy (TEM) is carried out in vacuum to minimize the interaction of the imaging electrons with gas molecules while passing through the microscope column. Nevertheless, in typical devices, the pressure remains at 10^{-7} mbar or above, providing a large number of gas molecules for the electron beam to crack, which can lead to structural changes in the sample. Here, we describe experiments carried out in a modified scanning TEM (STEM) instrument, based on the Nion UltraSTEM 100. In this instrument, the base pressure at the sample is around $2x10^{-10}$ mbar, and can be varied up to 10^{-6} mbar through introduction of gases directly into the objective area while maintaining atomic resolution imaging conditions. This can be used for physical and chemical manipulation of nanomaterials. Examples presented in this talk include oxygen-cleaning of graphene [1], using different chemical reactivity of zigzag and armchair edges in graphene to control edge types in oxygen atmosphere, Stone-Wales defect creation in a STEM at 60 kV with assistance of hydrogen atoms and defect creation in hexagonal boron nitride in hydrogen atmosphere.

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Computational studies of atomic scale manipulation

Harriet Åhlgren[1]

[1] Faculty of Physics, University of Vienna

The predictive and interpretive power of computational methods provides valuable insight into materials and their properties at the atomic scale. Here we discuss the manipulation of nanomaterials, such as graphene, via ion irradiation using computational modelling tools to understand the processes and give predictions towards experimental work. At low irradiation energies foreign atomic species can be implanted into graphene in substitutional sites [1], or trapped within the interface of two layers or surfaces forming new nanostructures [2,3]. Patterning of nanomaterials is possible with higher energy irradiation resulting in vacancy type modifications that can be controlled by the chosen projectile, its energy and incident angle [4,5].

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Doping control for low-voltage operation of CNT-based CMOS ICs

Fu-Wen Tan[1], Jun Hirotani[1], Shigeru Kishimoto[1], and Yutaka Ohno[1,2]

[1]*Department of Electronics, Nagoya University, Nagoya 464-8601, Japan*
[2]*Institute of Material and Systems for Sustainability, Nagoya University, Nagoya 464-8601, Japan*

Carbon nanotube thin-film transistors (CNT TFTs) exhibit excellent electrical and mechanical properties, and coupled with complementary metal-oxide semiconductor (CMOS)-based circuits, which would enable high-performance flexible electronic devices with low power consumption. Although complementary CNT TFTs have been achieved by featuring n-type TFTs by doping, its counterpart p-type devices are usually left as-fabricated without addressing issues such as switching threshold variability, which may lead to unreliable CMOS circuit operation [1-2]. In this work, we demonstrate more than 100 complementary CNT CMOS inverters at a low operating voltage of 0.5 V by intentional doping of both p- and n-type TFTs to tune the switching threshold.

Bottom-gated complementary TFTs connected in inverter configuration were fabricated on a flexible poly(ethylene naphthalate) (PEN) substrate as illustrated in Fig. 1. Semiconductor-enriched CNTs were utilized as the channel material. We confirmed that all 129 devices showed p-type behavior with on/off ratio ~10^4 and uniform characteristic prior to doping. Then, potassium hydroxide/ benzo-18-crown-6-ether (KOH/CE) was spin-coated only on selective devices to achieve n-type doping. An Al_2O_3 passivation layer was formed by atomic layer deposition on the n-type devices. Next, a similar process was repeated by spin coating silver-bis(trifluoromethane)imide (AgTFSI) on the p-type devices intended for threshold tuning. Then, a polymethyl methacrylate (PMMA) and Al_2O_3 passivation layer was formed on top of the p-type devices.

Figure 2 shows the transfer characteristics of 126 devices after (a) KOH/CE and (b) AgTFSI doping with their respective passivation layers with a yield 95.4 %. The p- and n-doped devices were successfully characterized without significant degradation of on-current and mobility. We confirmed the operation of CMOS inverters, with rail-to-rail voltages of 0.5 V, as shown in Fig. 2(c) and a small hysteresis width of 0.02 V on average.

[1] Y. Zhao *et al.*, ACS Nano **10**, 2193 (2016)
[2] J. Tang *et al.*, Nat. Nanotechnol. **1**, 191 (2018).
Corresponding Author: Y. Ohno
Phone & Fax: +81-52-789-5387,
E-mail: yohno@nagoya-u.jp

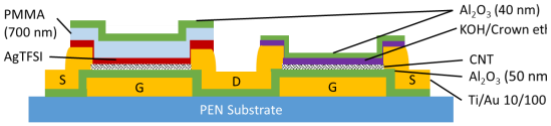

Fig.1 Schematic structure of a complementary CNT TFT device in inverter configuration.

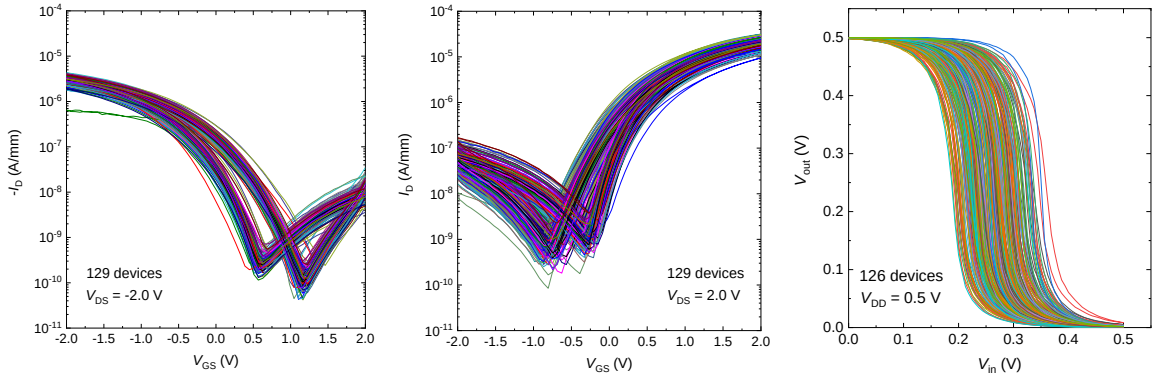

Fig. 2 I_D-V_{GS} characteristics at V_{DS} = | 2.0 V | of 129 CNT TFTs with channel length L_{ch} = 50 μm after (a) AgTFSI and (b) KOH/CE doping. (c) Output characteristics of 126 CNT CMOS inverters at V_{DD} = 0.5 V.

In situ electronic measurements during atomic resolution imaging

Kimmo Mustonen[1], Nan Wei[2], Heena Inani[1], Jun Hee Choi[3], Sang Wook Lee[2], Esko I. Kauppinen[2] and Jani Kotakoski[1]

[1]Faculty of Physics, University of Vienna, Boltzmanngasse 5, A-1090 Vienna, Austria
[3]Department of Applied Physics, Aalto University, Puumiehenkuja 2, 02150, Espoo, Finland
[3]Department of Physics, Ewha Womans University, 03760, Seoul, Korea

Since the introduction of aberration correctors for electron optical systems in the late 1990s [1], scanning transmission electron microscopy (STEM) has emerged one of the most powerful analytical techniques on the field of nanoscience. Currently for example elemental identification at a single-atom level [2], vibrational spectroscopy [3], isotope analysis [4] and controlled manipulation of covalently bound lattice impurities [5] are conducted routinely in the latest generation of STEM instruments. So far this potential has not been fully untapped in the context of *in situ* electronic measurements. In my talk, I will cover our latest transport measurements in a Nion UltraSTEM 100 microscope, concentrating on single-walled carbon nanotubes (SWCNTs) and graphene contacted in 2- and 3-terminal device configurations. I will show that, much like in graphene, current annealing conducted with thin SWCNT networks result in cleaning of the nanotube walls, and eventually in a measurable increase in the network conductivity. Finally, I will present our recent work utilizing 2D materials as support layer stabilizing SWCNTs for STEM experiments [6], and show that this approach can potentially be used to fabricate 3-terminal single-SWCNT devices in which each atom can be directly resolved.

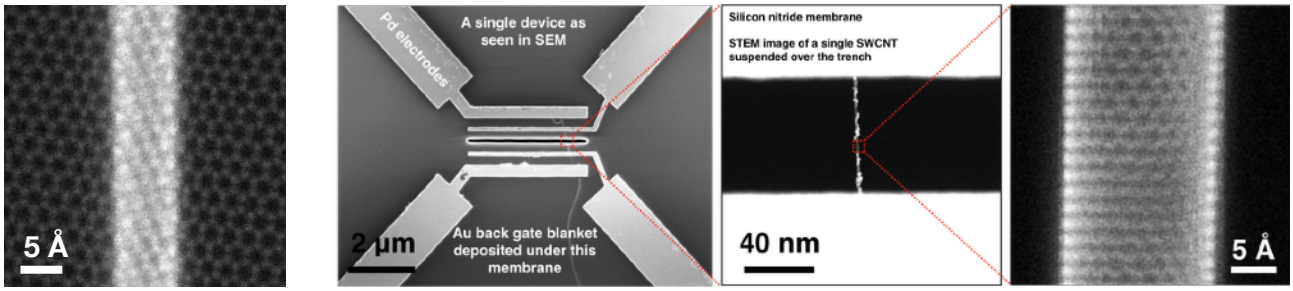

Fig 1. LEFT: Small diameter single-walled carbon nanotube (SWCNT) stabilized on graphene. RIGHT: SWCNT suspended on 3-terminal configuration for electron microscopy experiments.

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