Innovative Technologies

Selected Novel Technologies for Licensing

At 2019 MRS Fall Exhibit Booth 1002

A "Title List" of our new novel technologies

No.	Field	Title
1	Material	**Ultralightweight Al Foam** A New Fabrication Method to Realize Low-cost and Energy Saving
2	Material	**Water-Soluble Polyimides** Polymers Interconverting High Water Soluble and Water Insoluble
3	Material	**Nitrogen-Doped Graphene** Ideal Flat, Semiconductive and Transparent Material for Various Devices
4	Electronic Device	**Oxide/Group-IV Semiconductors** Enhanced Tunneling Drastically Extending Battery Life of IoT Devices
5	Micro/Nano Fabrication	**Graphene Films on SiO_2** Continuous films of multilayer graphene with controllable thickness
6	Micro/Nano Fabrication	**Multilayer Hexagonal Boron Nitride (h-BN)** New CVD Synthesis Methods for Large-area, Multilayer h-BN
7	Micro/Nano Fabrication	**Nano-Spin Motor** Innovative "Nano size Motor" realize GHz rotational speed
8	Software	**Encoding Flow Technology** A common language for flows and Topological Flow Data Analysis
9	Measuring Equipment	**Molecular Structure Identification** X-ray structural analysis without the need for crystallization

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2. We maximize research outcomes by managing research resources under the "Virtual- network based Research Institutes" scheme.
3. We establish Japan's science and technology infrastructure to accelerate science and technology innovation.

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Ultralightweight Al Foam

A New Fabrication Method to Realize Low-cost and Energy Saving

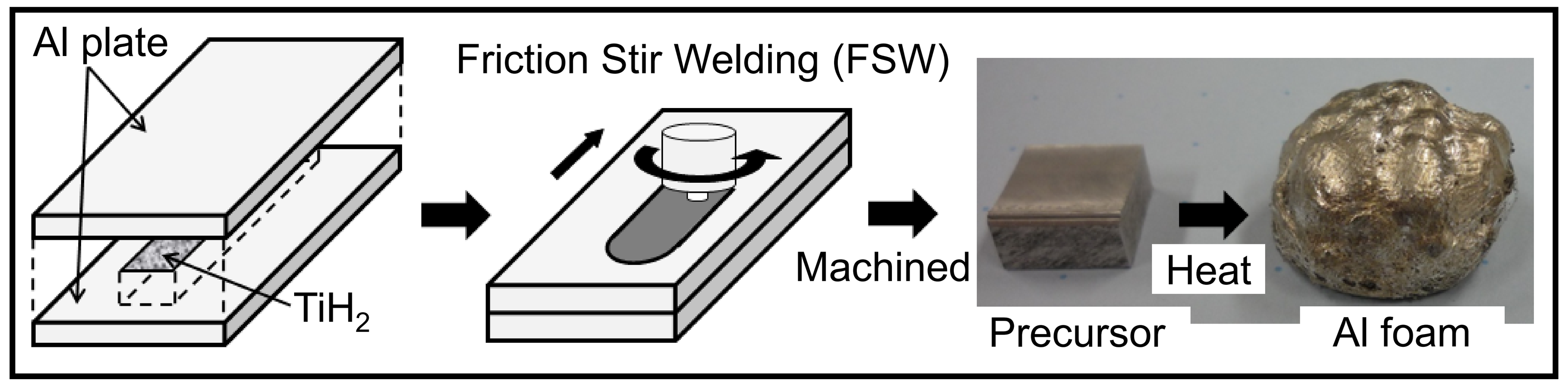
Prof. Yoshihiko HANGAI (Gunma University) et al.

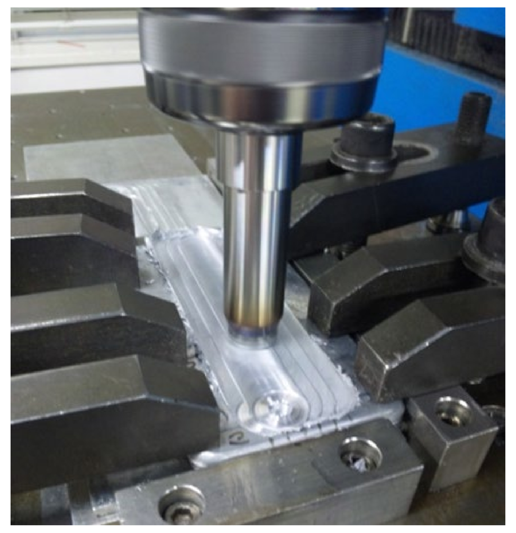
1. Background

Al Foam (Porous Aluminum)

- There is a great demand, but the applications have been narrowed due to its high cost comparing to resin/plastics foam.
- Novel methods to realize low-cost Al Foam has been long-awaited.
- Al Foam has excellent characteristics i.e. ultralightweight, durability against chemicals, endurance against aging

2. (Fabrication Process) Friction Stir Welding

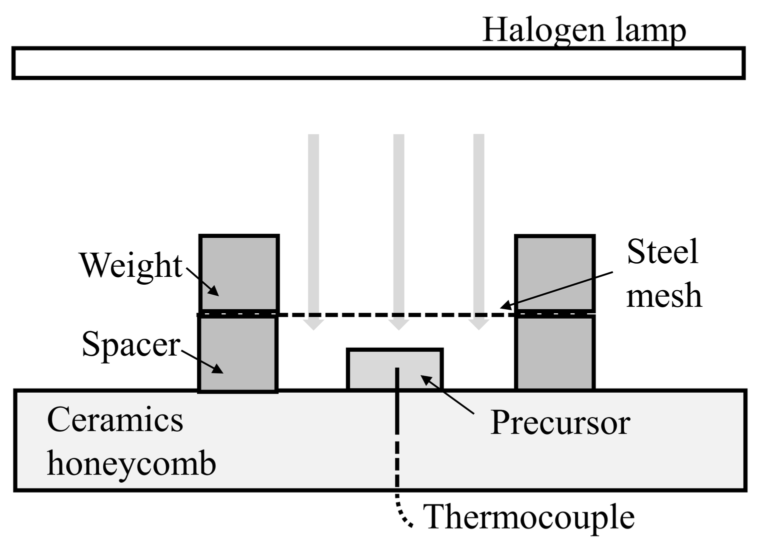


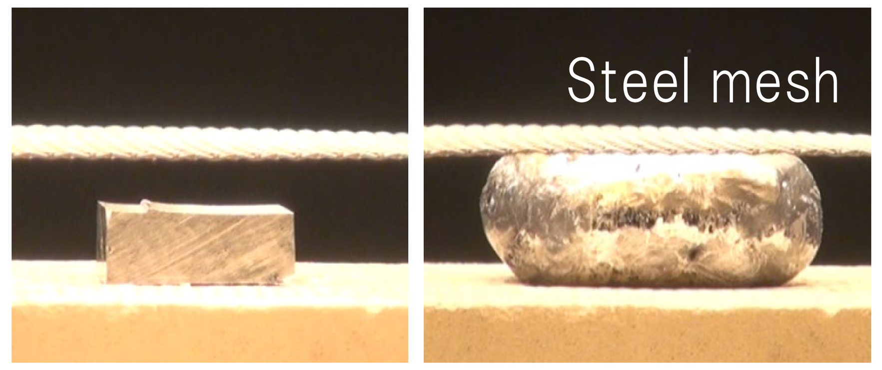


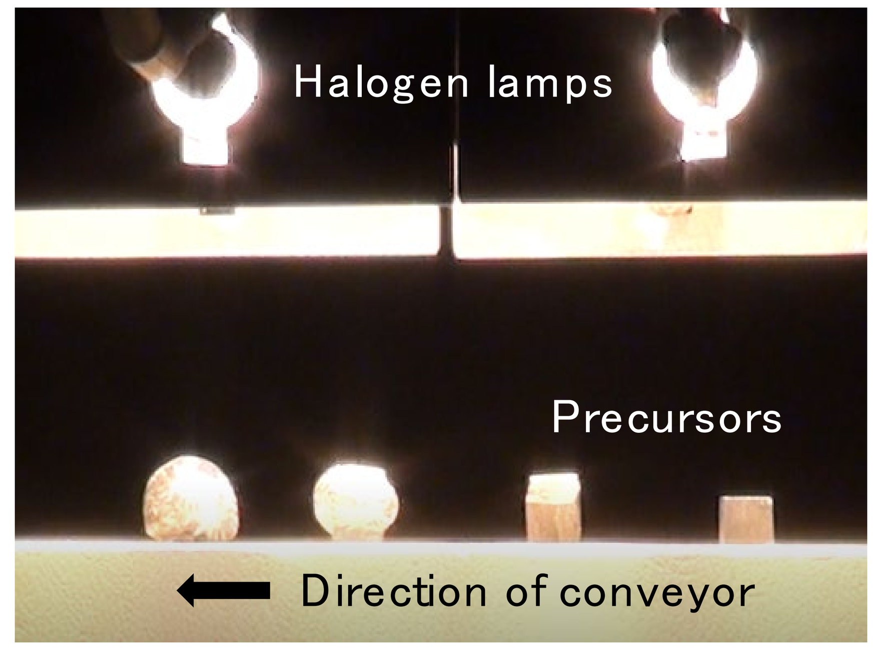
Completely different method of making "Precursor", which is mixture of Al plate and foaming agent, by FSW(Friction Stir Welding) process is adopted instead of the conventional way of sintering mixture of Al powder and foaming agent.

- Not require any special environmental condition for the process i.e. at room temperature, at general factory conditions
- Basic patent of FSW process had expired and the technology of FSW process has been well established. And the facility cost of the process is relatively inexpensive.
- The foaming rate can be controllable by the volume of the foaming agent.

3. (Heating Process of Precursor) Optical Heating







Foaming Process adopt Optical Heating by using halogen lamps. This new method realize "low cost" and "energy saving" at the same time.

- The facility can be easy to scale up and relatively easy to control.
- "Steel mesh" and/or "heat resistant glass", which transmit light and heat, can be used as molds for shaping Al Foam.
- By special placement of small pieces Precursors, large size Al Foam can be produced relatively easy.

4. Prospective Applications

- Train/Vehicle Materials
- Mobile/Wearable Devices
- Construction Materials
- Shock Absorption Materials
- Soundproof/Sound Absorbing Materials

5. Patent Licensing Available

Patent No.: WO2019/013026(JP,US,EP), WO2011/046152 (JP,US,EP), WO2010/029864(JP), WO2010/106883(JP)

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Water-Soluble Polyimides

Polymers interconverting high water soluble and water insoluble.

Prof. Tatsuo KANEKO (Japan Advanced Institute of Science and Technology (JAIST))

1. Background

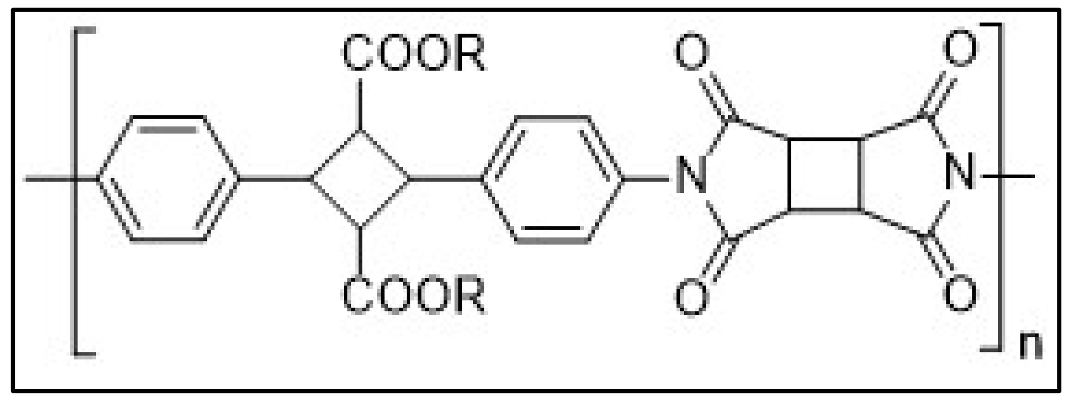
・In order to realize Sustainable Society, the following things for polymers are required.
(1) Reduction of petrochemical resources
(2) “Solvent free” production process

2. Our Technology

(1) Original Invention :

We succeeded in producing “new Polyimides”, which have the advantages listed below;

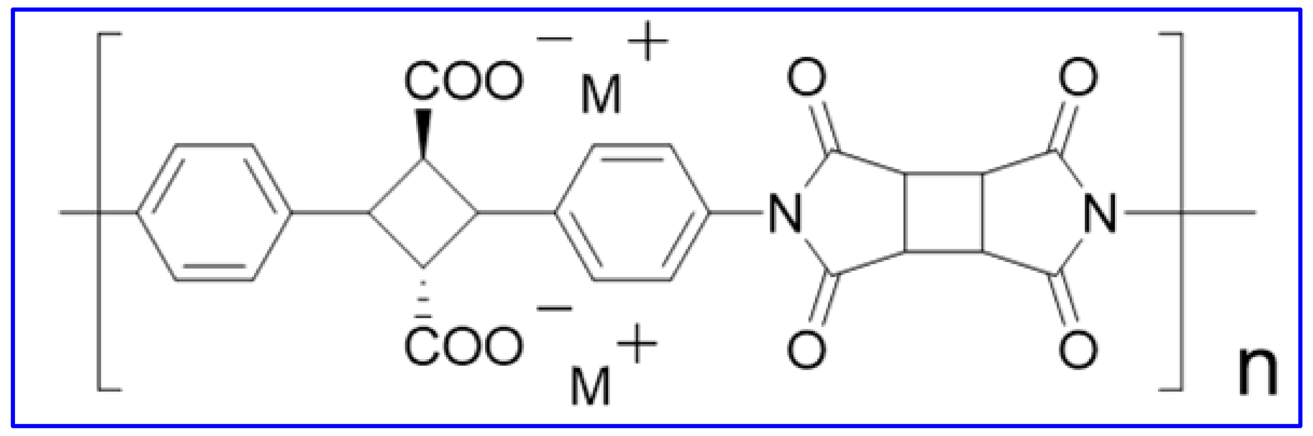
- *High physical strength*
- *High heat resistance*
- *Transparency*
- *Recyclability owing to photodegradability*
- *Production ability from bio-resources*



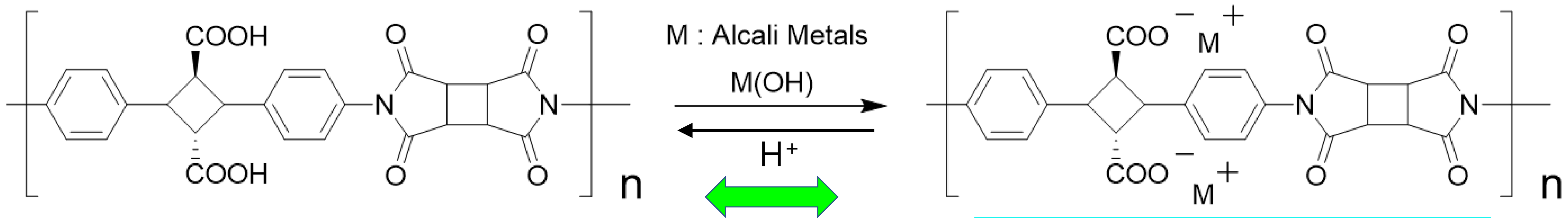
Although the “new polyimides” have several good points, they have disadvantages of “no organic solvent for casting” and “requiring complicated production process”.

(2) Enhanced Invention :

We succeeded in producing “enhanced new polyimides”, which eliminate the disadvantages



3. Experiments



H type : Water Insoluble

Salt type : Water Soluble

High physical property

☞ **Comparable properties “ester type”**

Very high water-solubility (>66wt%)

☞ **Reduced environmental load**

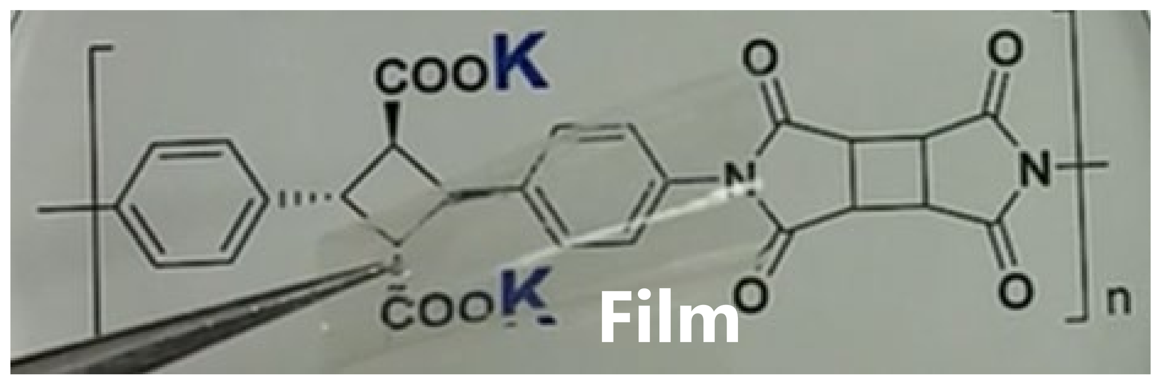
[**Physical properties**]

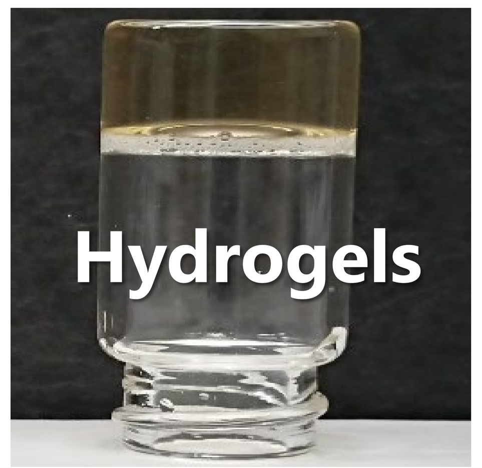
		Counter cations (M)	T_{d10} (°C)	Strength at break (MPa)	Elongation at break (%)	Transparency (%T @450nm)
(2) Enhanced Invention	H type	H	406	109	3.6	90.0
	Salt type	Li	366	42	8.1	90.0
	Salt type	Na	352	37	10.1	89.0
	Salt type	K	362	29	10.9	90.1
(1) Original Invention		Me	390	94	1.8	99.0

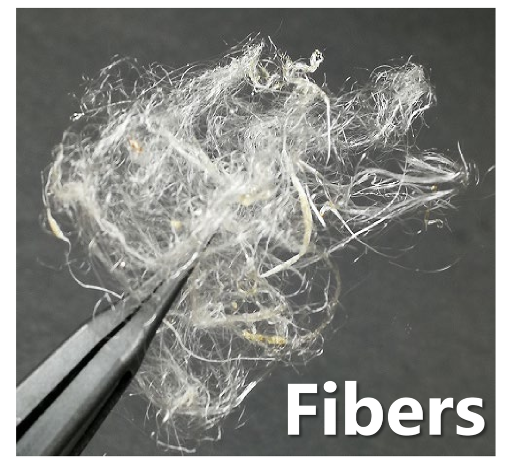
4. Application Example

■Electrode materials (for Lithium ion battery etc.)

■Soft materials (for 3D-Printer etc.)







5. Patent Licensing Available

Patent No.: WO2019 / 026795 (US, EP, CN, JP, KR)
WO2013 / 073519 (US, EP, CN, JP)

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Nitrogen-Doped Graphene

Ideal Flat, Semiconductive and Transparent Material for Various Devices

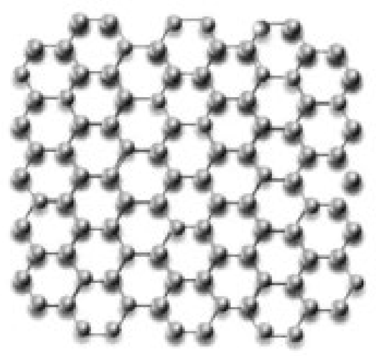
Prof. Nagahiro SAITO (Nagoya University)

1. Back Ground

A certain **Heteroatoms-Doped Graphene** has unique characteristics and especially **Nirtogen-Doped Graphene** has been expected to have excellent characteristics. But there have not been any good methods to produce a "flat" Heteroatoms-Doped Graphene .

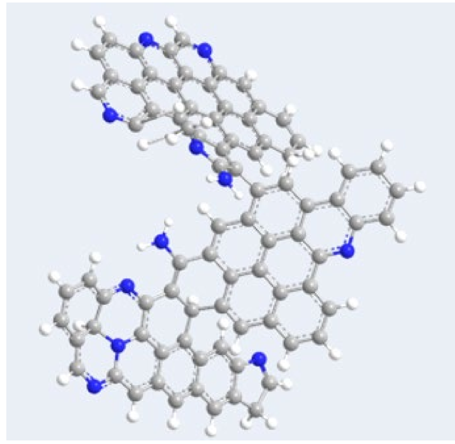
Graphene has ideal flatness structure

Disorder in Graphene



Graphene

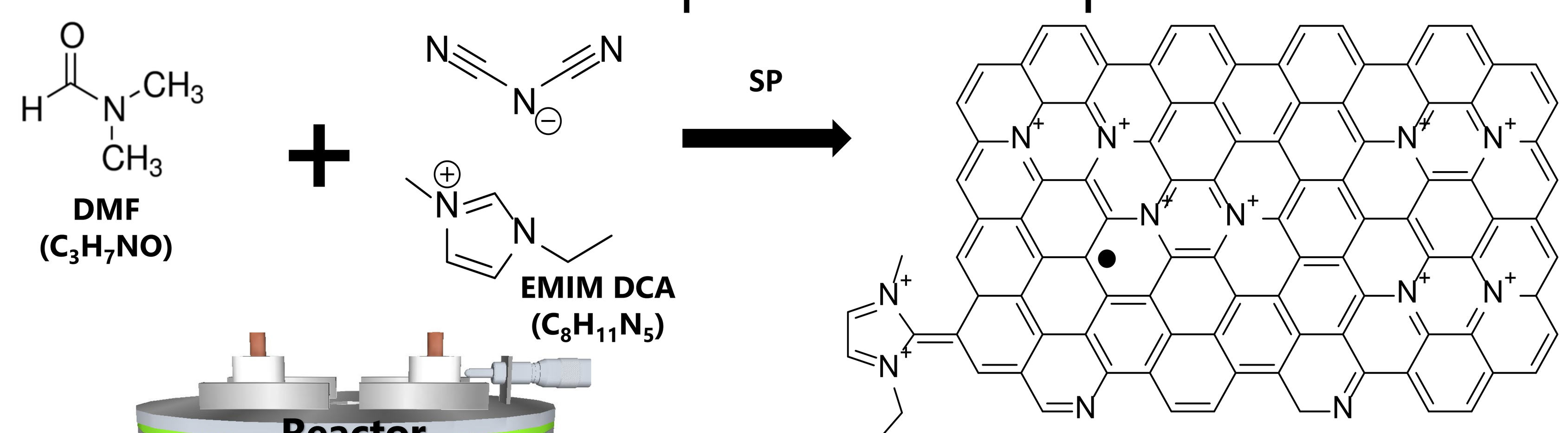
By Doping heteroatoms to Graphene, the flatness of graphene loses.

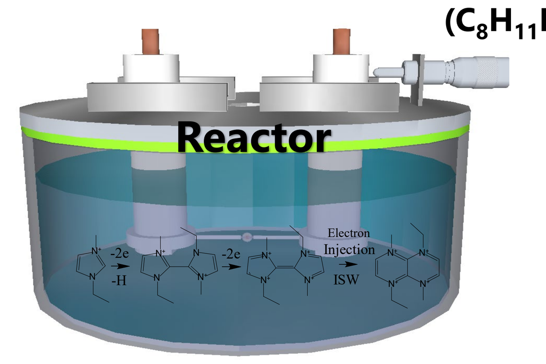


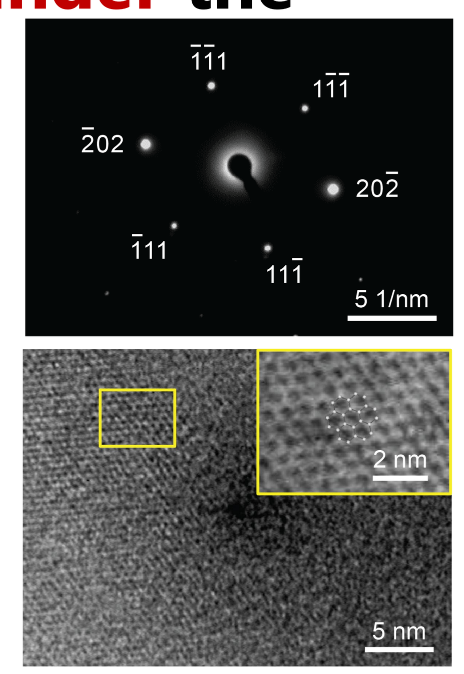
Doped Graphene

2. Our Technology

We invented the **"Solution Plasma Method"** for producing the "flat" Heteroatoms-Doped Graphene. The Method provides **highly doped and ultra "flat" Nitrogen-doped Graphene under the** condition of normal temperature and pressure.





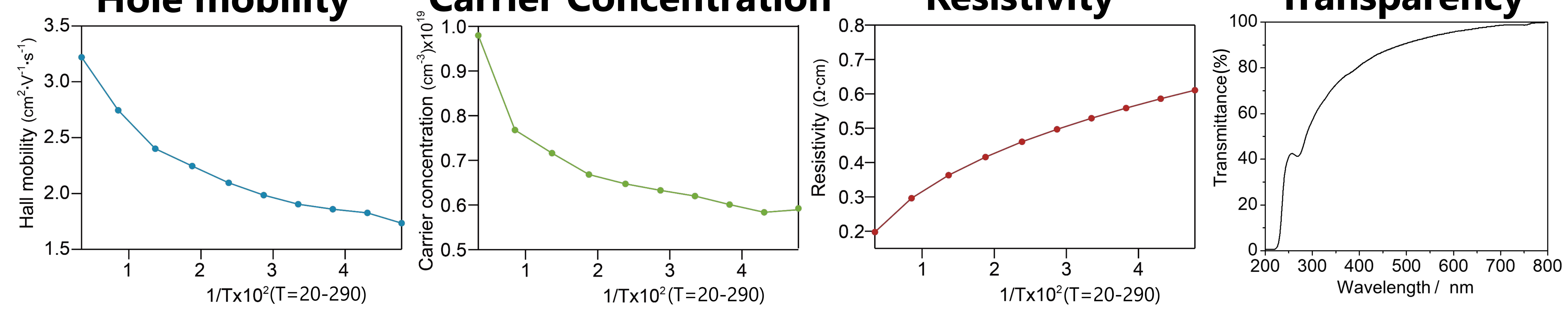


The N-doped flat hetero-graphene has highly crystallinity.

Power Supply	
Electrodes	W – W (Tungsten)
Gap Distance	1 mm
Frequency	200 kHz
Pulse width	1.0 μs
Base solvent	DMF*, 90 wt%
Ionic Liquids	EMIM DCA*, 10 wt%
Time	5 min.

Production method	Dope rate	Flatness	cost	Temp.℃ /pressure
Chemical Vapor Deposition(CVD)	Low	Low	High	800℃ /vacuum
Thermal Annealing Method	Low	Low	Low	1000℃ /normal pressure
(This Work) Solution Plasma Method	High	High	Low	25℃ /normal pressure

3. Characteristics of the Nitrogen-doped Hetero Graphene



The Nitrogen-Doped Graphene shows best values among the other graphene

(1) Hole mobility : **$3.4 cm^2 \cdot V^{-1} \cdot s^{-1}$ at 290K**

(2) P-type Semiconductive, Carrier concentration: **$10^{19} cm^{-3}$ at 290K)**

(3) Sheet resistance : **16 $\Omega \cdot sq^{-1}$**

(4) Transparency : **more than 80 % at 400-800nm(as graphene)**

4. Application Examples

Our Heterographene leads to Transparent, Semiconductive thin film.

Flexible 2D Semiconductive Materials

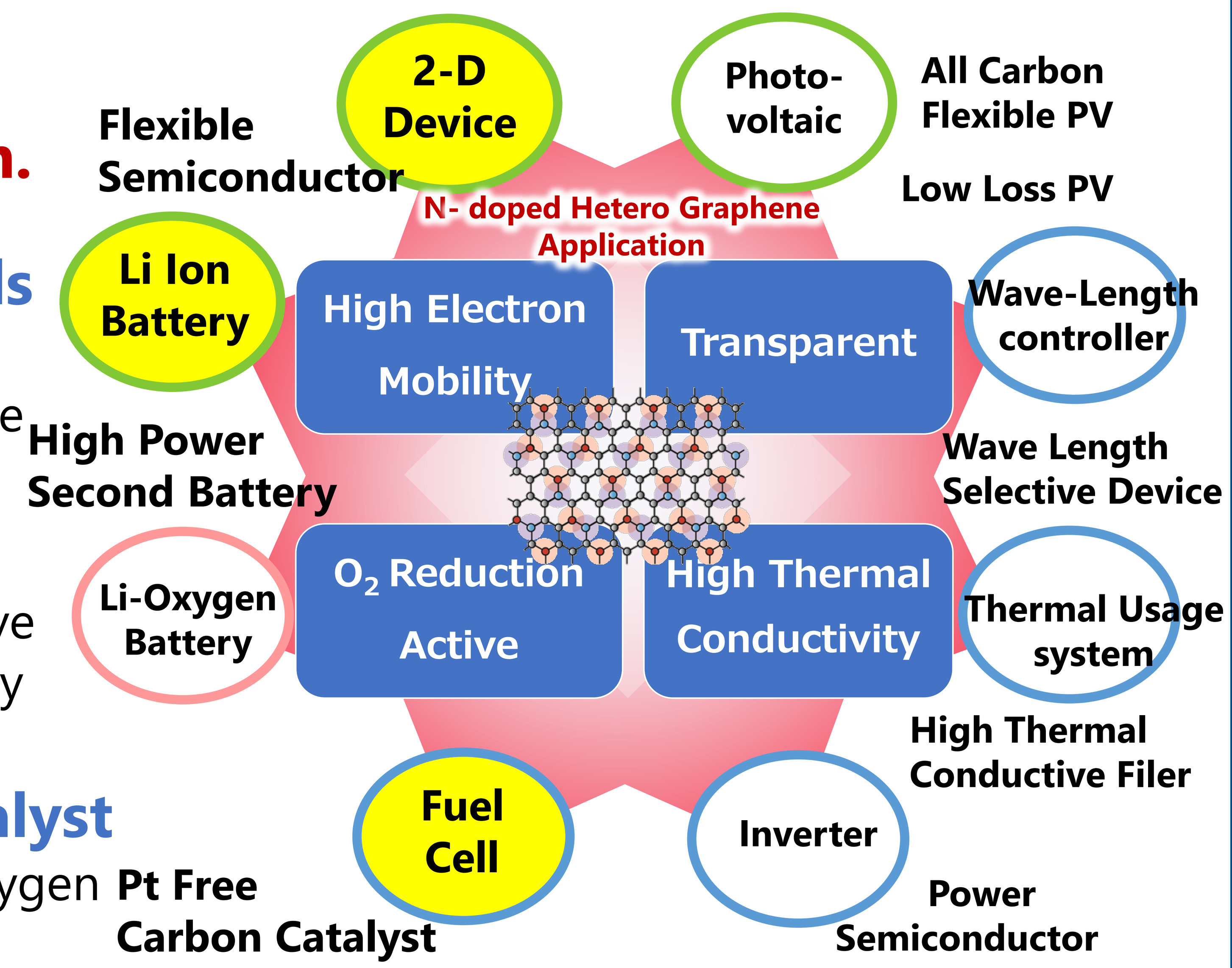
Use of the film in flexible semiconductors and electrode, application for flexible devices can be expanded.

Materials for Secondary battery

Use of the film in electrode catalyst or conductive additives of the secondary batteries, the capacity and life of the batteries can be improved.

Materials for Oxygen Reduction Catalyst

This film is expected for low cost material for oxygen reduction catalyst of fuel cell.



5. Patent Licensing Available

Patent No.: WO2019/066013 (International phase)

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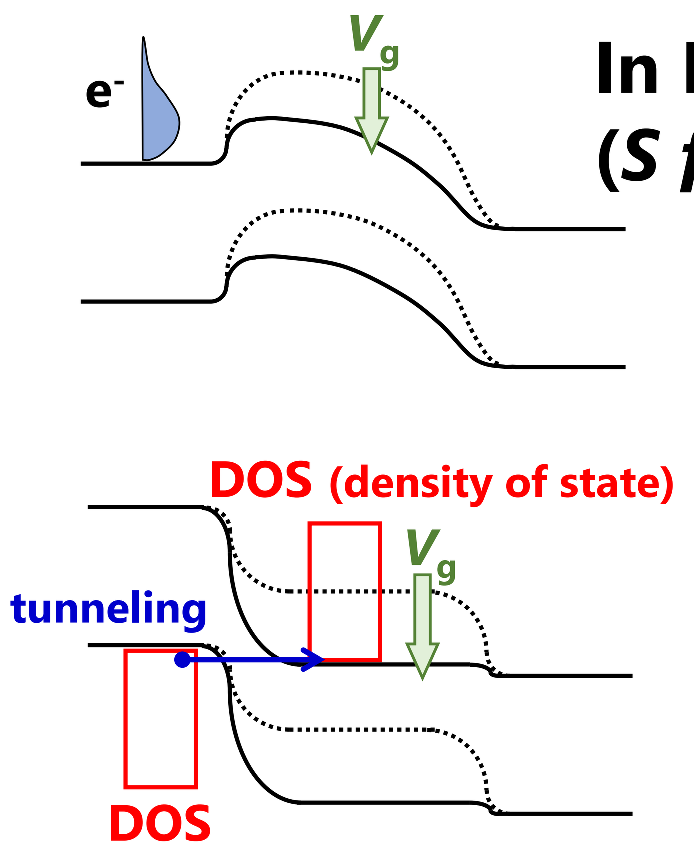
Oxide/Group-IV Semiconductors

Enhanced Tunneling Drastically Extending Battery Life of IoT Devices

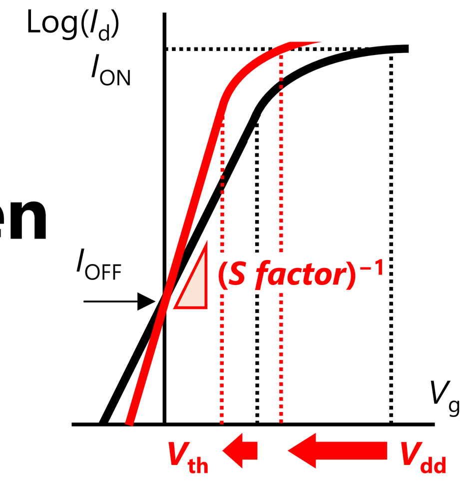
Dr. K. KATO, Prof. S. TAKAGI, Prof. M. TAKENAKA, Prof. H. TABATA, Prof. H. MATSUI (The University of Tokyo)

1. Tunneling Field-Effect Transistors (TFETs)

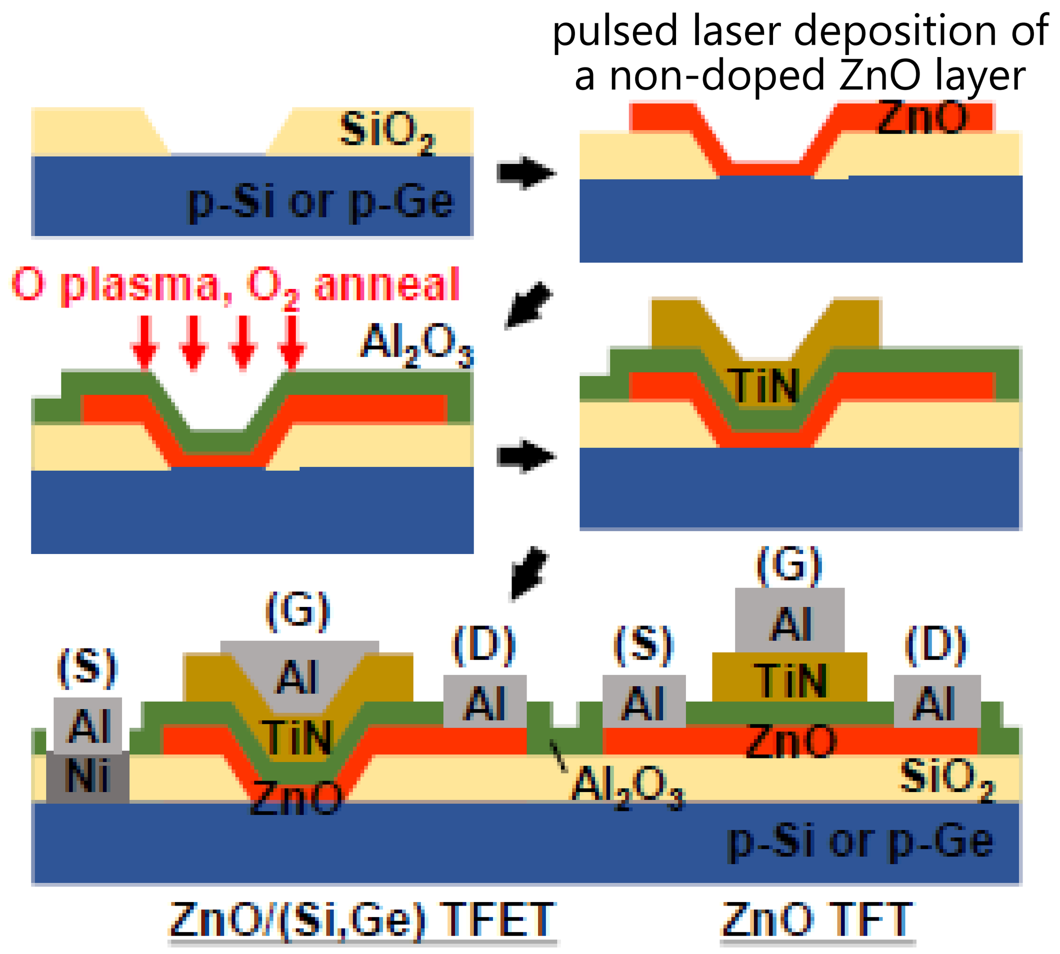


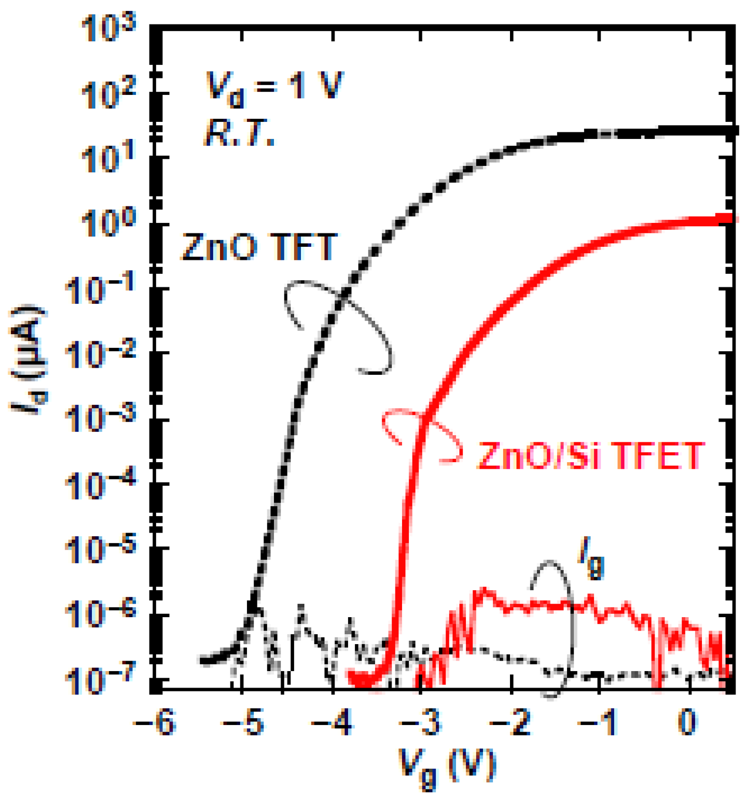
In FETs, drain current of subthreshold swing (*S factor*) is limited by Boltzmann tail of carriers.

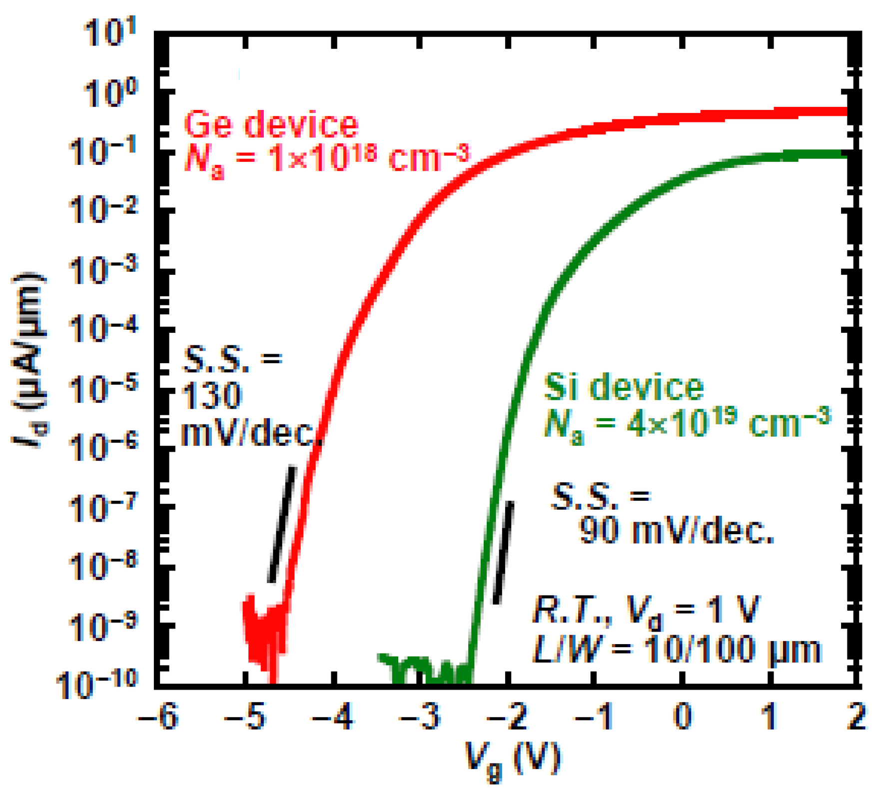
***S factor* of TFETs is small**
because overlap of DOS between source and channel is directly modulated by gate voltage.
However, *ON current* of homo-junction TFETs and *ON/OFF current ratio* of III-V heterojunction TFETs were low.



3. Experiment



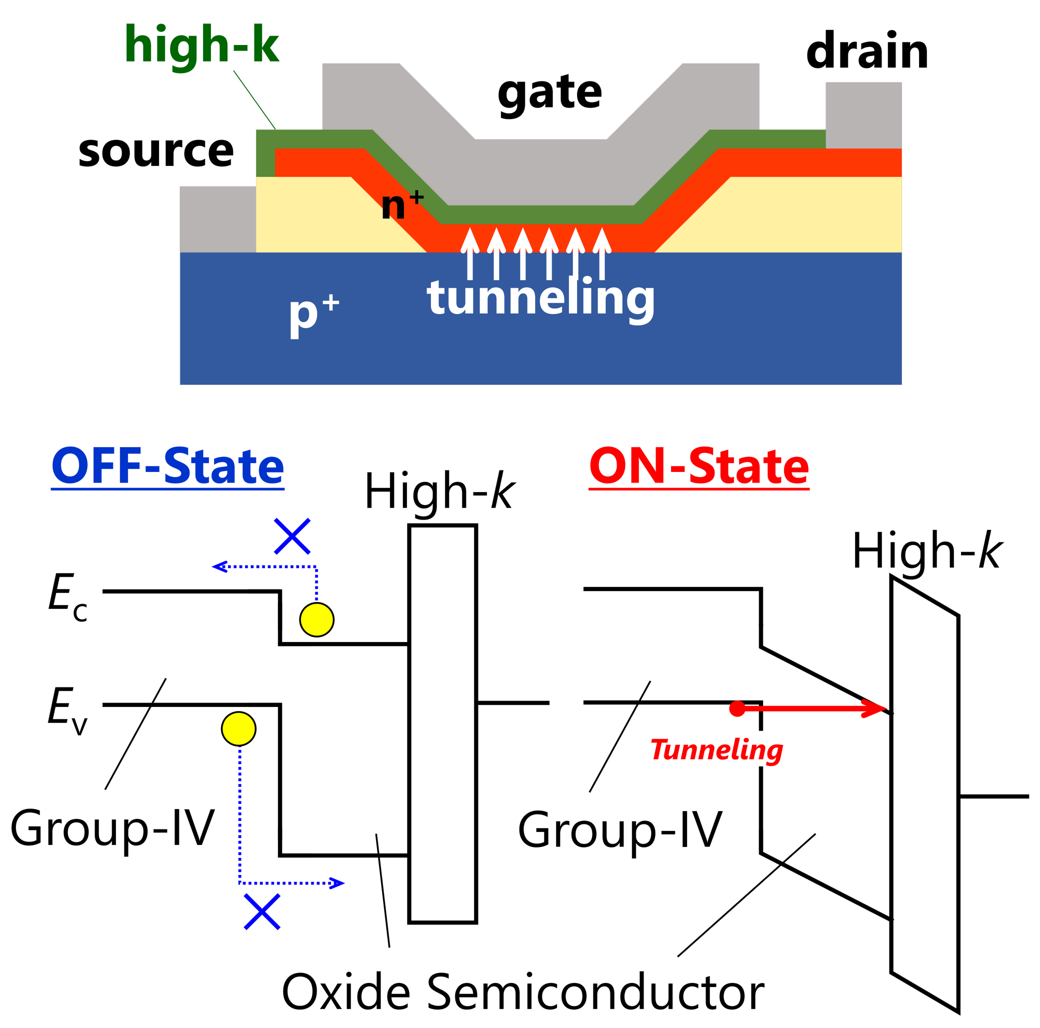




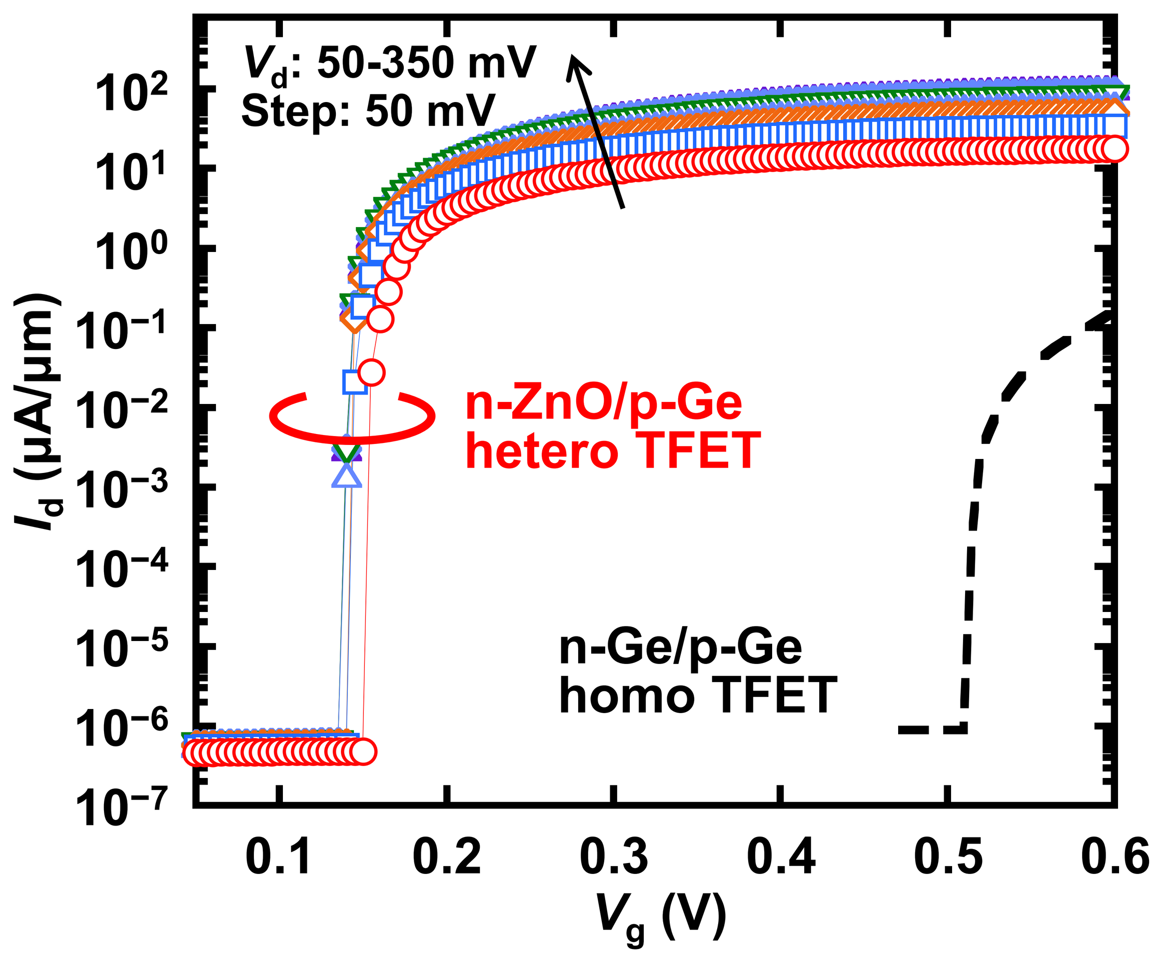
● **Record-high *ON/OFF current ratio* of ~10^8 among TFETs reported so far**

2. Our Technology

Oxide-/group-IV-semiconductor (OS/IV) bilayer TFETs for small *S factor*, large *ON current*, and large *ON/OFF current ratio*

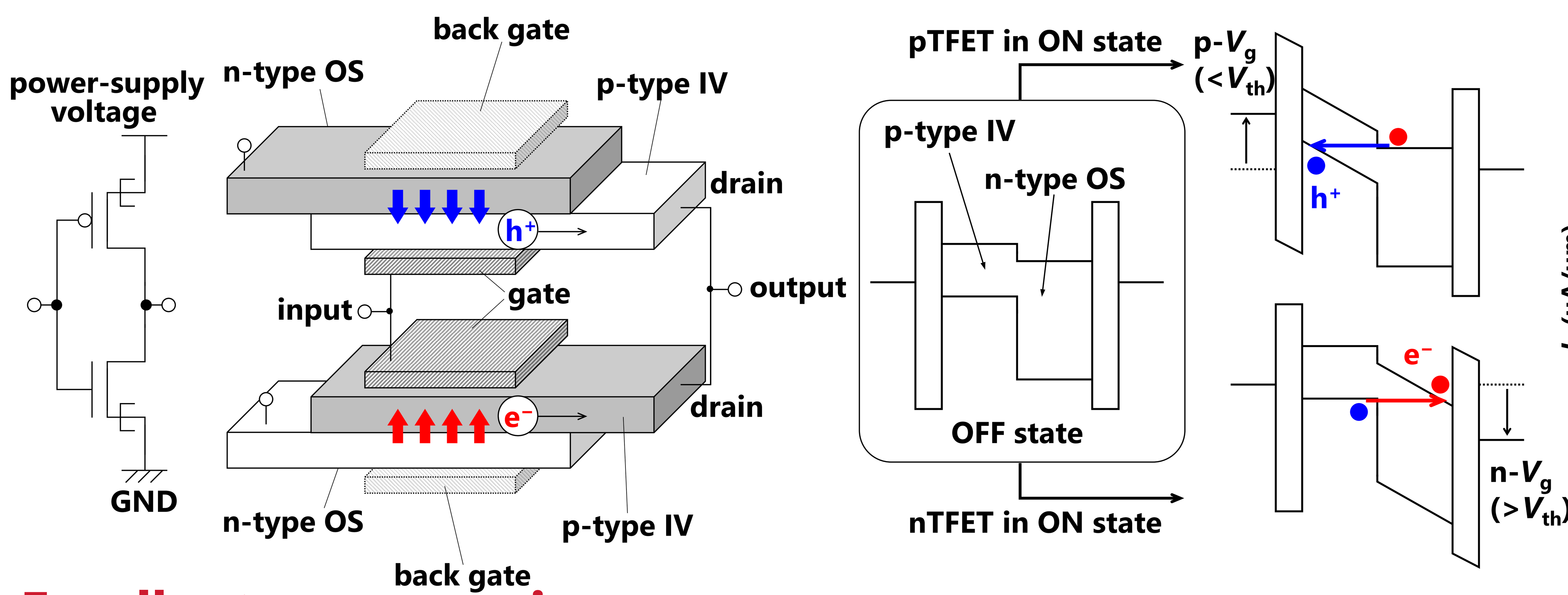


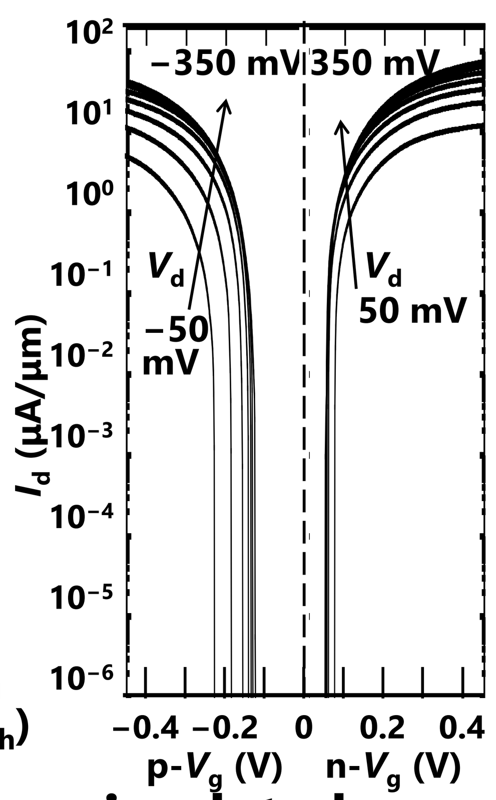
Schematic cross section and energy band diagram of proposed OS/IV bilayer TFET.



Simulated drain current/ gate voltage characteristics of n-OS/p-Ge TFET with various drain voltage.

4. Application Example: Unitary construction of n-/p-TFETs





simulated characteristics

Excellent power saving

⇨ ■ **Energy-efficient IoT (Internet of things) devices**

5. Patent Licensing Available

Patent No.: WO2019/107411

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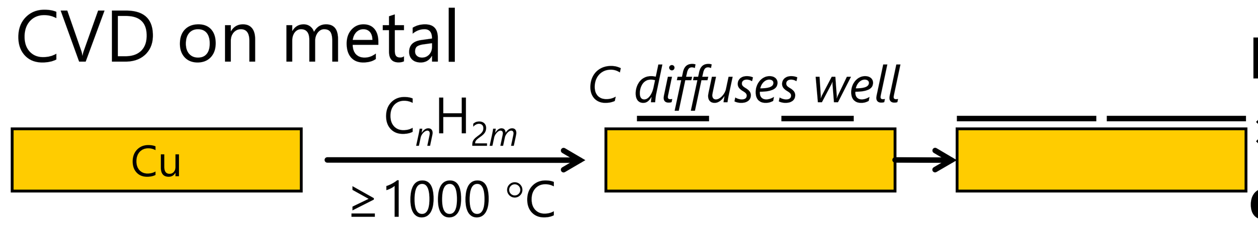
Graphene Films on SiO_2

Continuous films of multilayer graphene with controllable thickness

Prof. Suguru NODA (Waseda University)

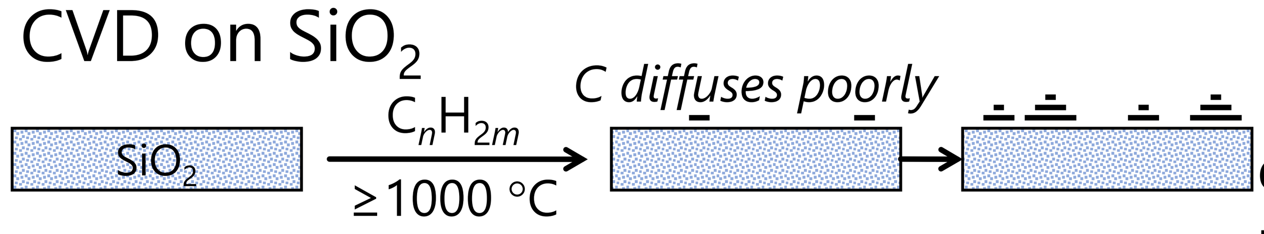
1. Production Methods of Graphene Films

CVD on metal



High quality single-layer on metals
× Graphene films should be damaged during complicated transfer process.

CVD on SiO_2

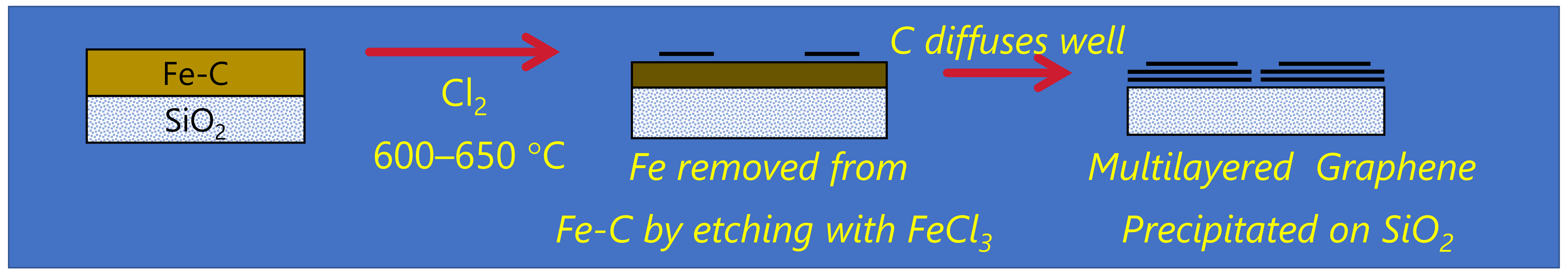


× Only discontinuous film with small domains on SiO_2 can be obtained under the very high temp. process.

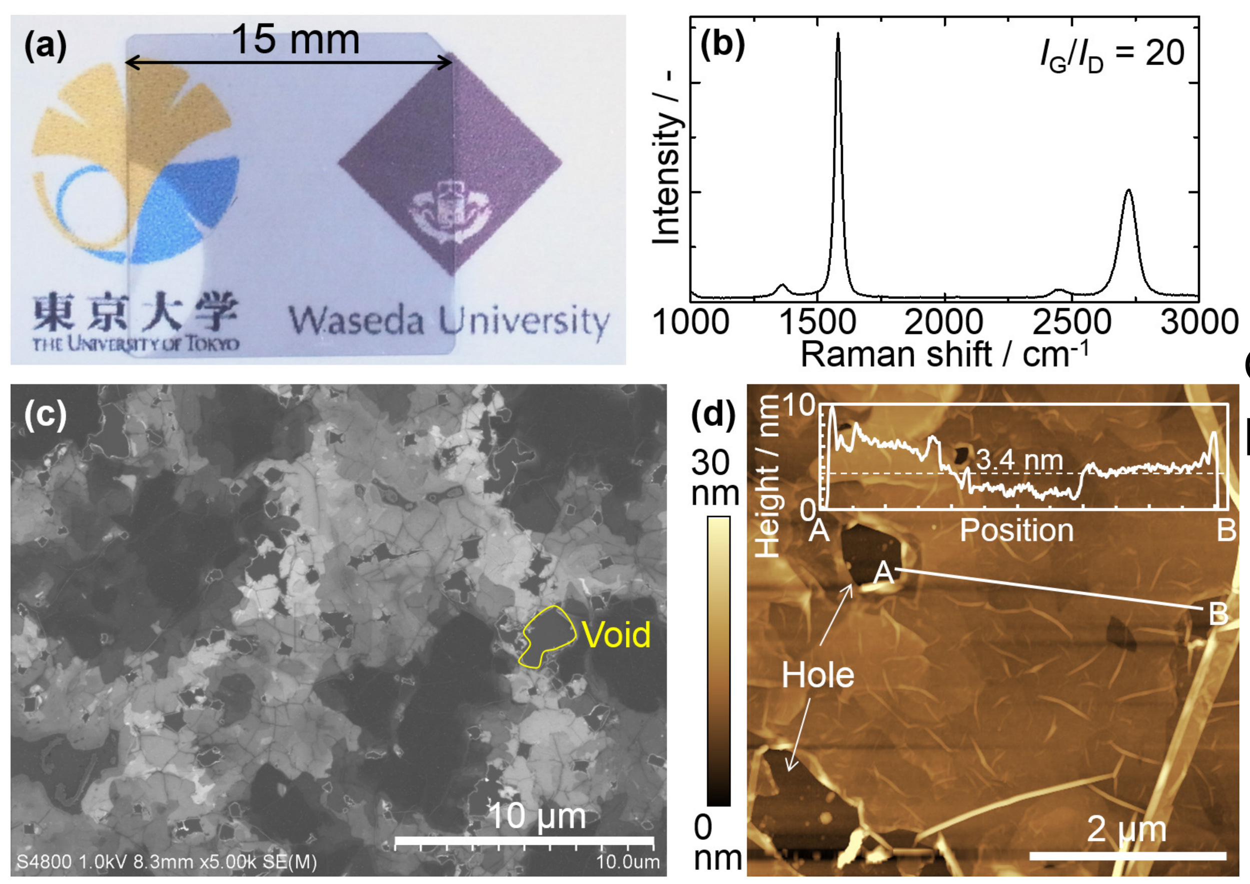
High quality, continuous films with large domains on SiO_2 under moderate temperature process are strongly awaited.

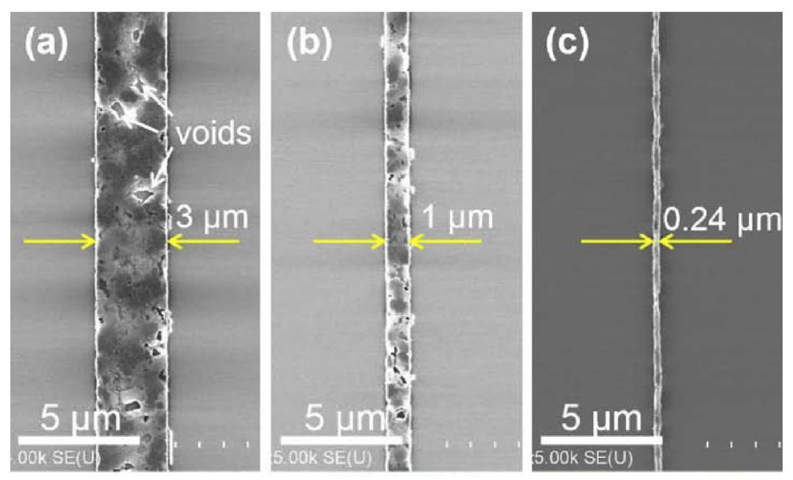
2. Our Technology: Etching-Precipitation Method

Our method adapts the conventional device fabrication processes. At first, the Fe-C films is sputtered on SiO_2 and then the Fe is etched by Cl_2 gas.



Graphene film formed on SiO_2 at moderate temp. 600 ~ 650 °C.





Graphene line pattern formed by lift-off process and by etching & precipitation Method

Transmittance:~ 86% at 550 nm, corresponding to 6–7 layers.

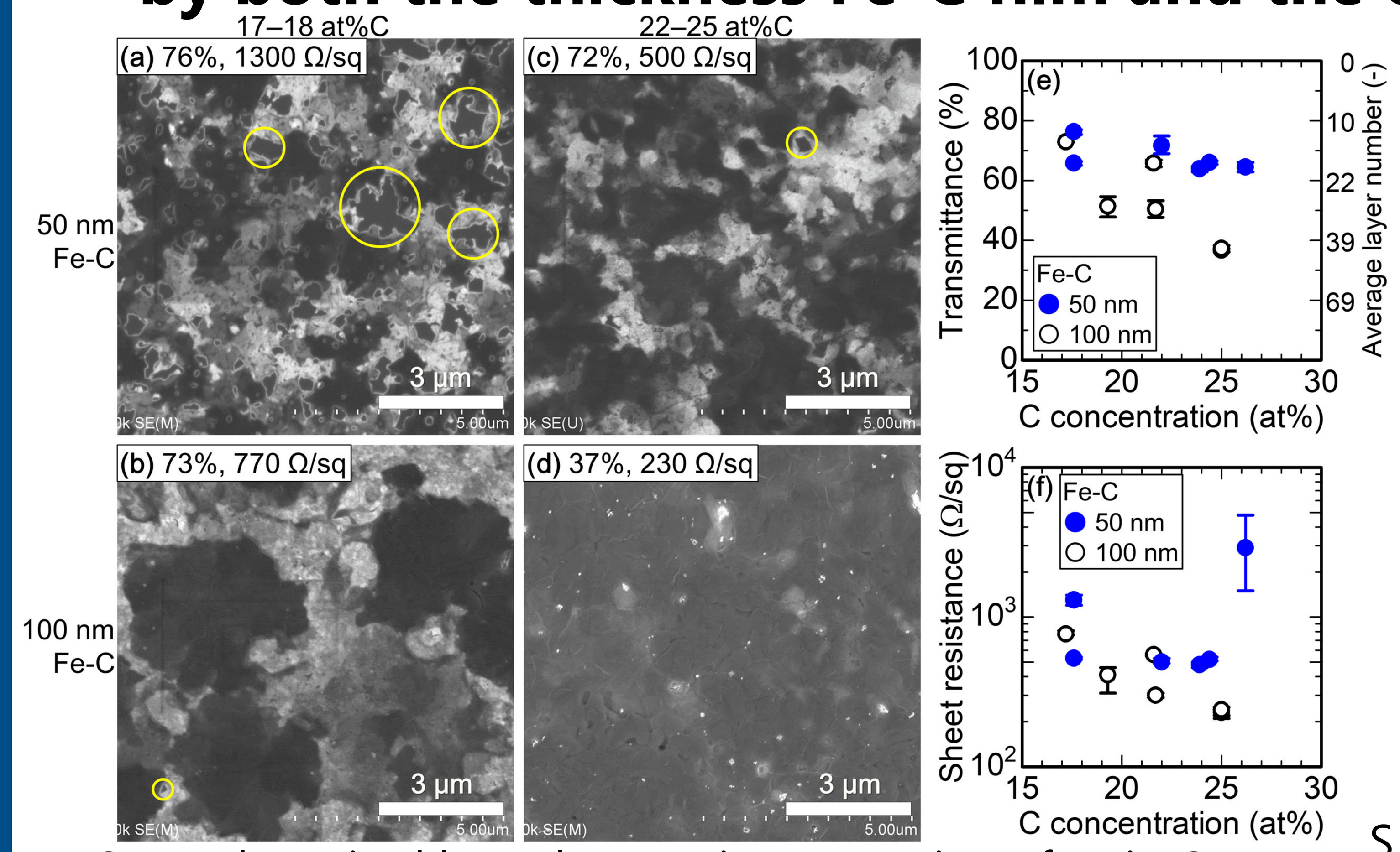
Sheet resistance of 340–460 Ω/sq corresponding to 80–140 μΩ cm.

If the samples had been processed under clean room environment, graphene quality should been improved.

M. Kosaka, et al., Carbon 82 (2015) 254.

3. Controllability of Film Thickness

Thickness of graphene layer can be controlled by both the thickness Fe-C film and the carbon composition of Fe-C film.



Fe-C was deposited here by reactive sputtering of Fe in C_2H_4/Ar.

Average layer number of graphene is varied between 10 and 40.

Sheet resistance is between 300 and 1000 Ω/sq, that will be decreased further to 60 Ω/sq by additional intercalation process with $FeCl_3$.

S. Akiba, et al., Thin Solid Films 675 (2019) 136.

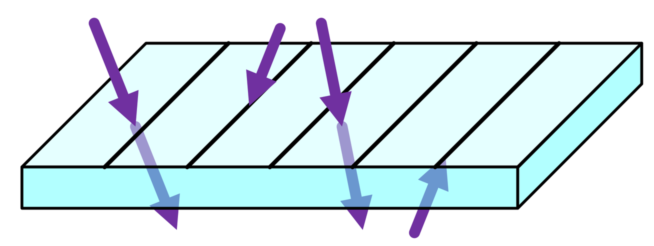
4. Application Examples

Graphene:

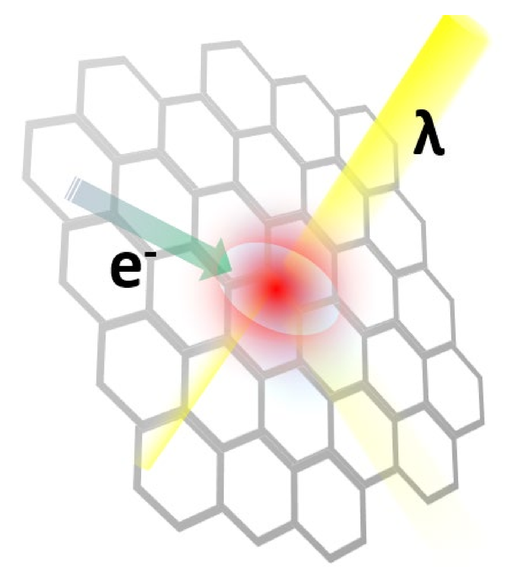
"Transparent, gray or black conductors on glass substrates"

Graphene is optically less shiny and electrochemically more stable than any metals, thus can be used for;

- ☐ Transparent electrodes for smart glass, head-up display
- ☐ Transparent heaters for glass windows
- ☐ Micro-wires of integrate circuits with high current capacity
- ☐ High-speed on-chip blackbody emitter for optical communication



Black graphene grids; conductive, glare-free, stable



Light & heat emitter

Nat. Commun. 9 (2018) 1279.

5. Patent Licensing Available

Patent No.: WO2012/118023 (JP, US, EP, KR, CN, IN)

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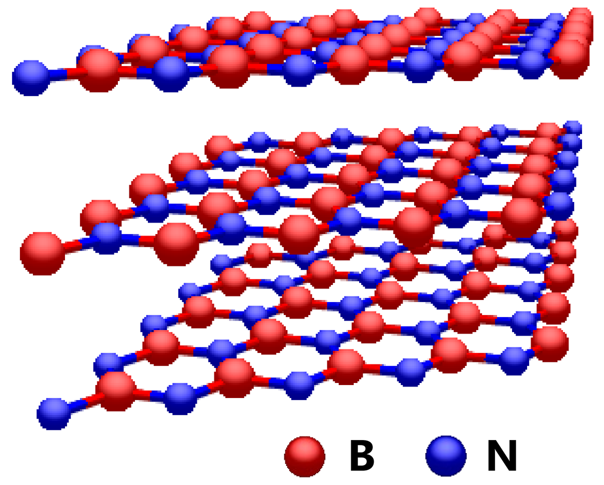
Multilayer Hexagonal Boron Nitride (h-BN)

New CVD Synthesis Methods for Large-area, Multilayer h-BN

Prof. Hiroki AGO (Kyushu University)

1. Importance of Hexagonal Boron Nitride (h-BN)

Multilayer h-BN

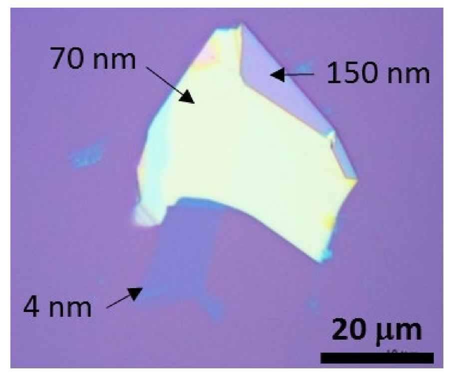


- Two-dimensional insulator with large band gap (5.9eV), atomically flat and dangling-bond-free surface

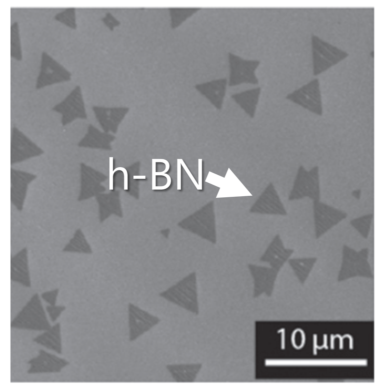
Excellent Insulating material for graphene, TMDC and other 2D materials

TMDC: **T**ransition **M**etal **D**i**C**halcogenide

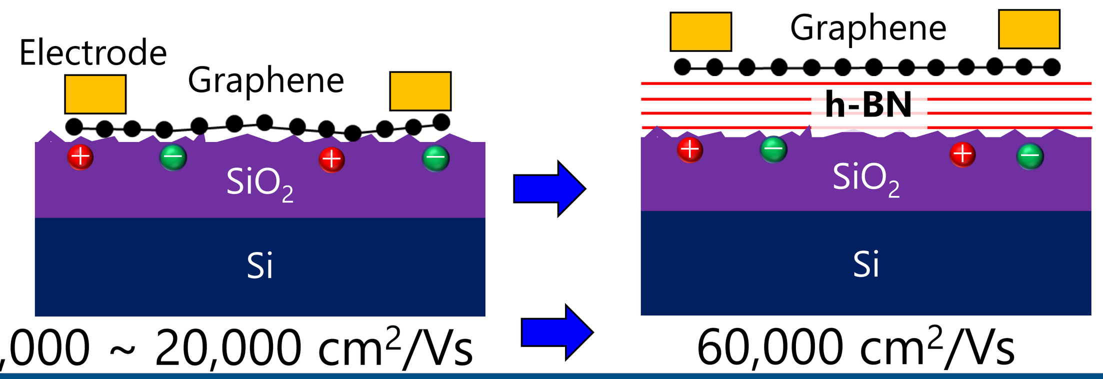
× Mechanical Exfoliation can provide only very small h-BN flakes.



× CVD method can usually give monolayer h-BN, which is not thick enough.

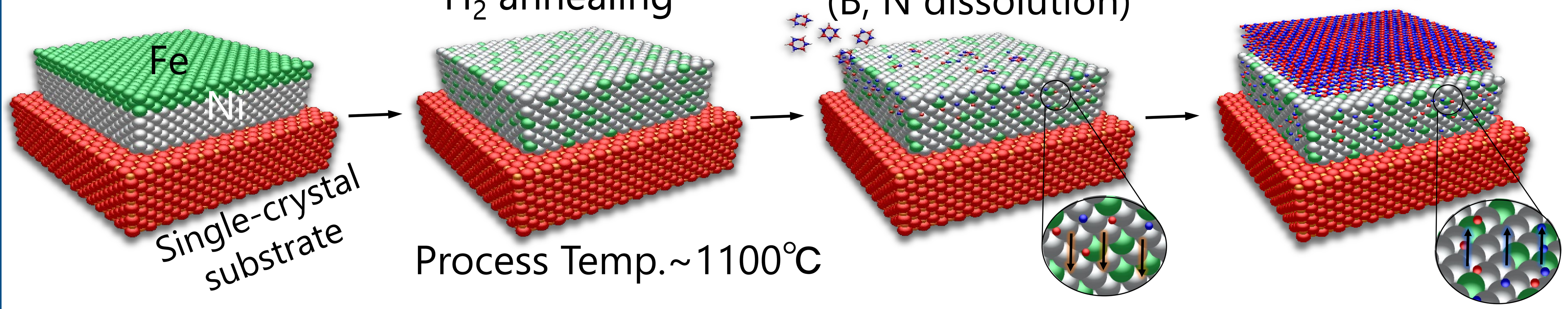


Large-area and multilayer h-BN can reduce surface roughness, optical phonon and charge impurities of SiO_2 surface, and can increase the carrier mobility.

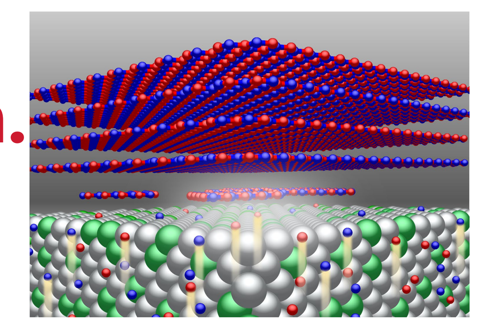


C. R. Dean *et al.*, *Nat. Nanotech.*, **5**, 722 (2010)

2. Our New CVD Synthesis Method



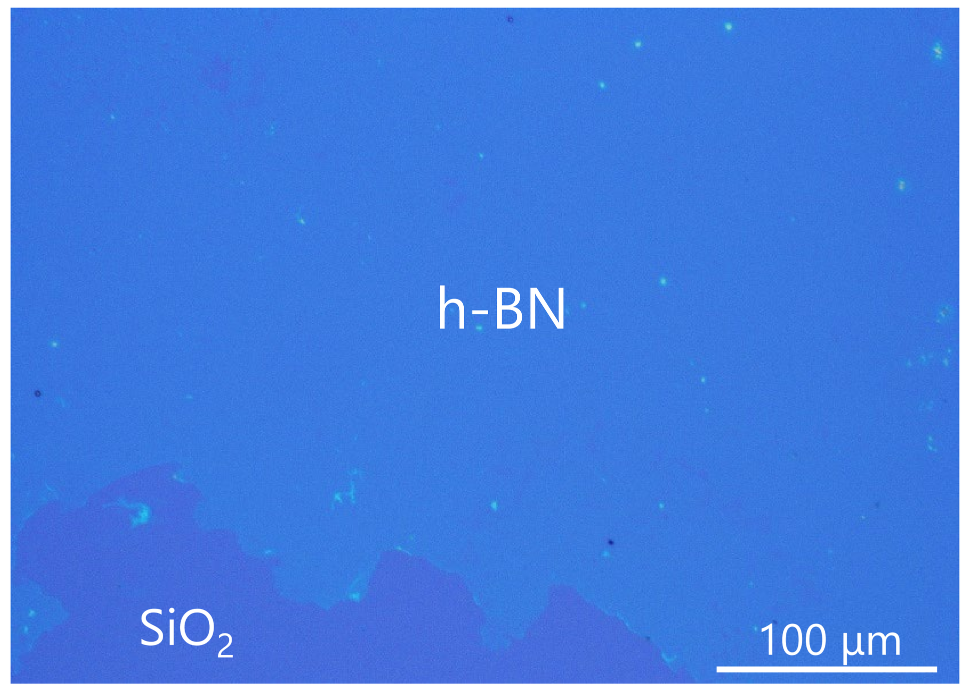
- **Large area & uniform multilayer h-BN can be obtained.**
- **Typical thickness of the h-BN ranges from 3 nm to 10 nm.**
- **The h-BN can be easily transferred onto any substrates by wet process.**



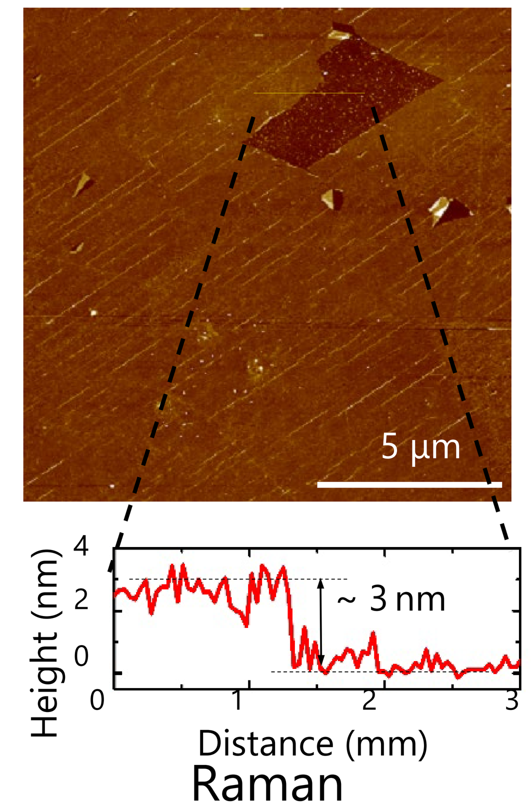
3.h-BN Quality of This Method and Prospective Applications

The below experimental data shows the quality of the h-BN

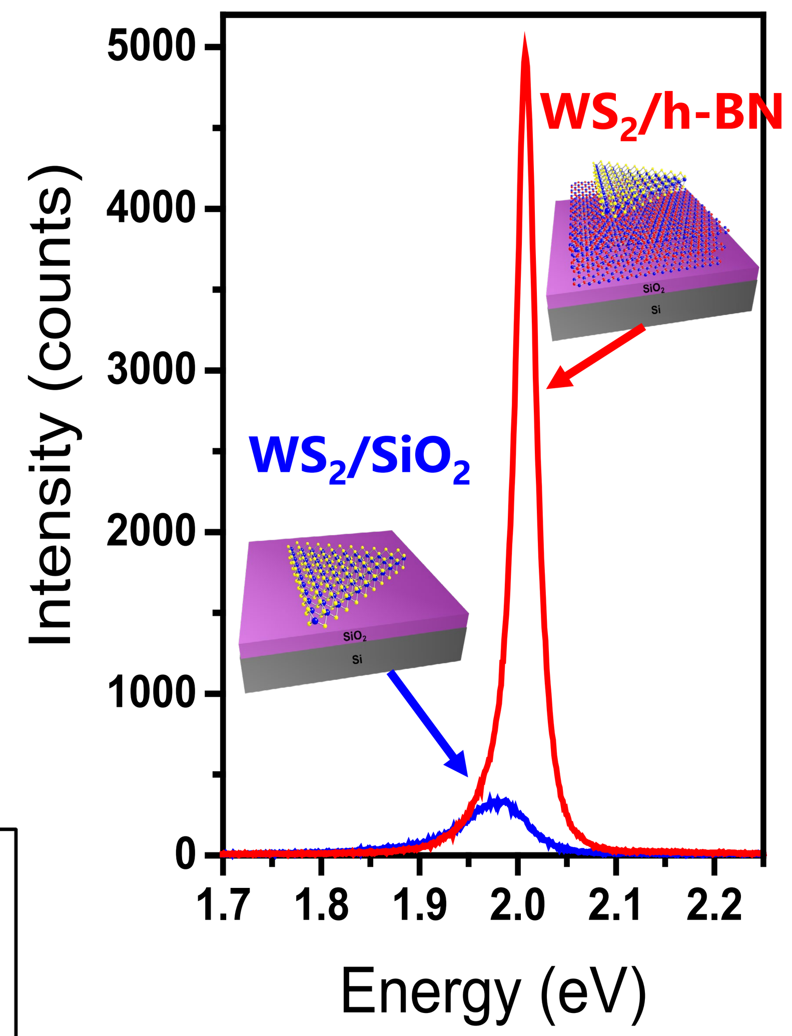
Optical micrograph (Plan-view)



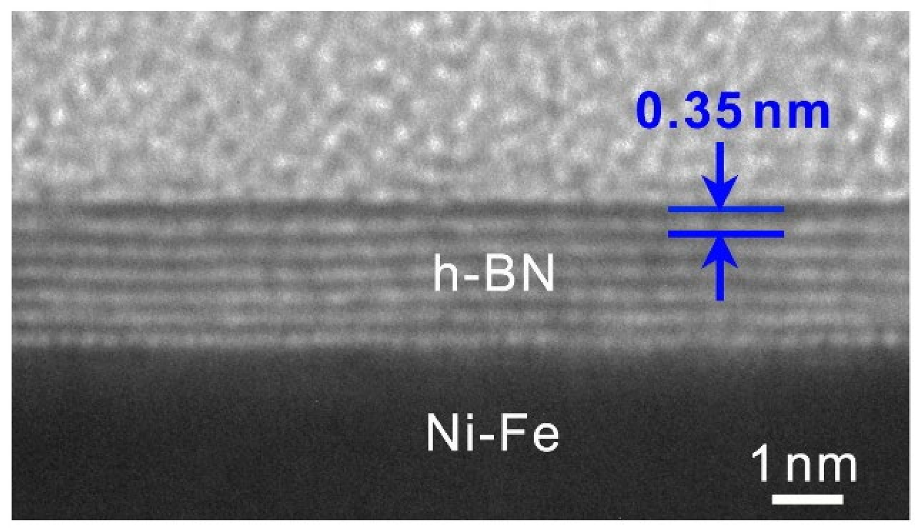
AFM



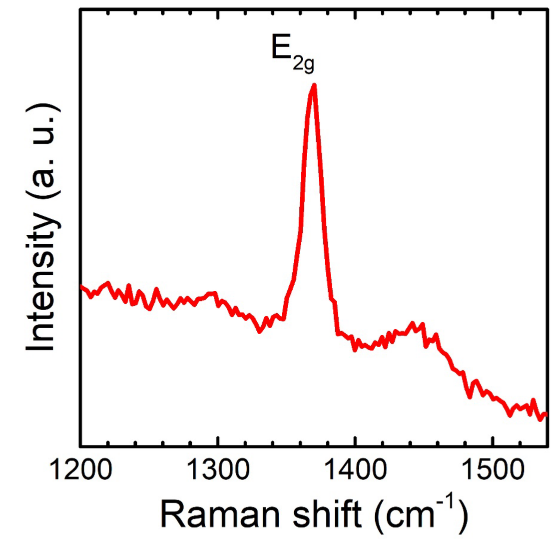
PL of monolayer WS_2



TEM (Cross-section)



Raman



Y. Uchida, H. Ago *et al.*, *ACS Nano*, **12**, 6236 (2018)

Prospective Applications;

- **Ideal insulator layer of hetero-structured 2D device**
- **Tunneling barrier for spin memory device**
- **Light emitting layer of deep ultraviolet device**
- **Oxidation-resistant coating for black phosphorus thin layer device**
- **Highly selective proton permeation for polymer electrolyte membrane**

4. Patent Licensing Available

Patent No.: WO2018/128193 (JP, US, EP, KR, CN,)

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Nano-Spin Motor

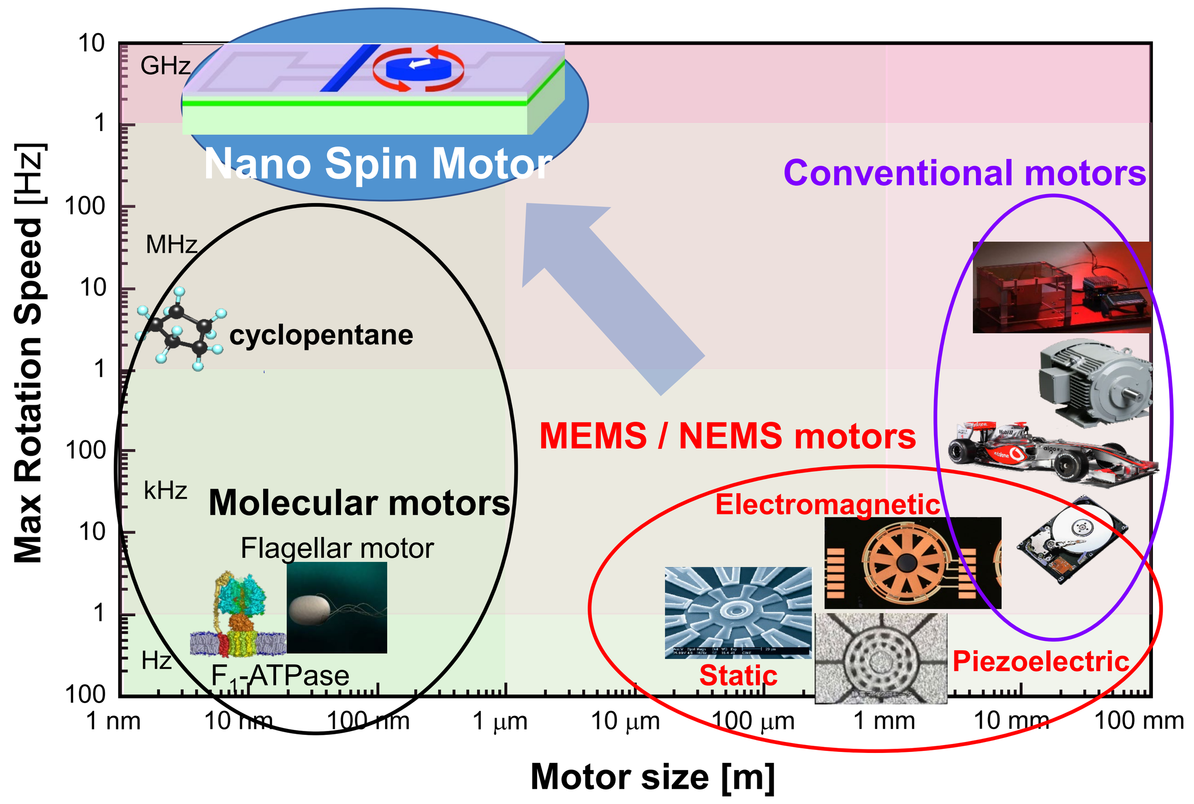
Innovative “Nano size Motor” realize GHz rotational speed

Prof. Atsufumi HIROHATA (University of York, United Kingdom)

1. Motor Innovation Trend

“Smaller”, ”High Rotational Speed” and “Durable” motors have been required.
Several MEMS/NEMS motors have been introduced but they can not realize.

- Size: less than 100 nm
- Rotational Speed: faster than GHz
- Unlimited usage time

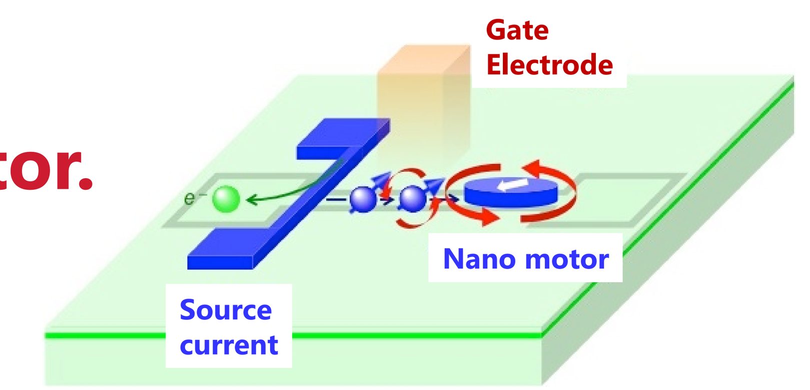


2. Novel “Nano Spin Motor”

Nano-spin motor can be the fastest motor.

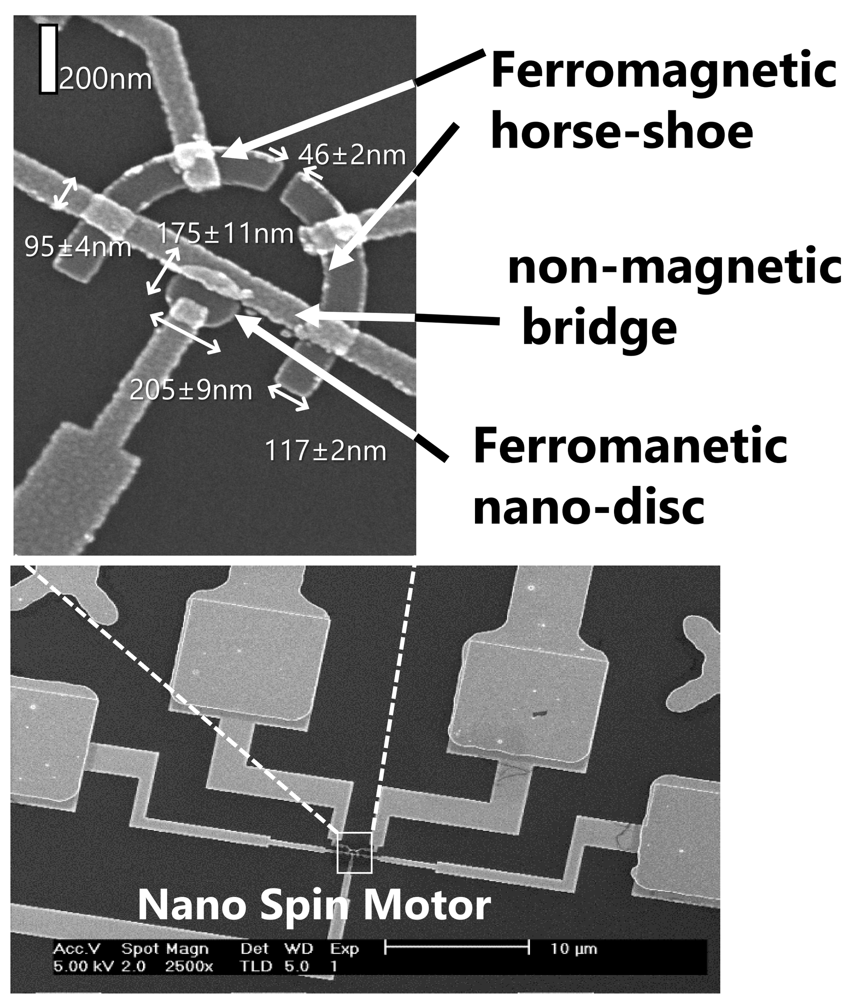
■Mechanism

A spin-polarized current can rotates a magnetic moment in a ferromagnetic disc, which has continuous tunability between 0 and a few GHz reproducibly.

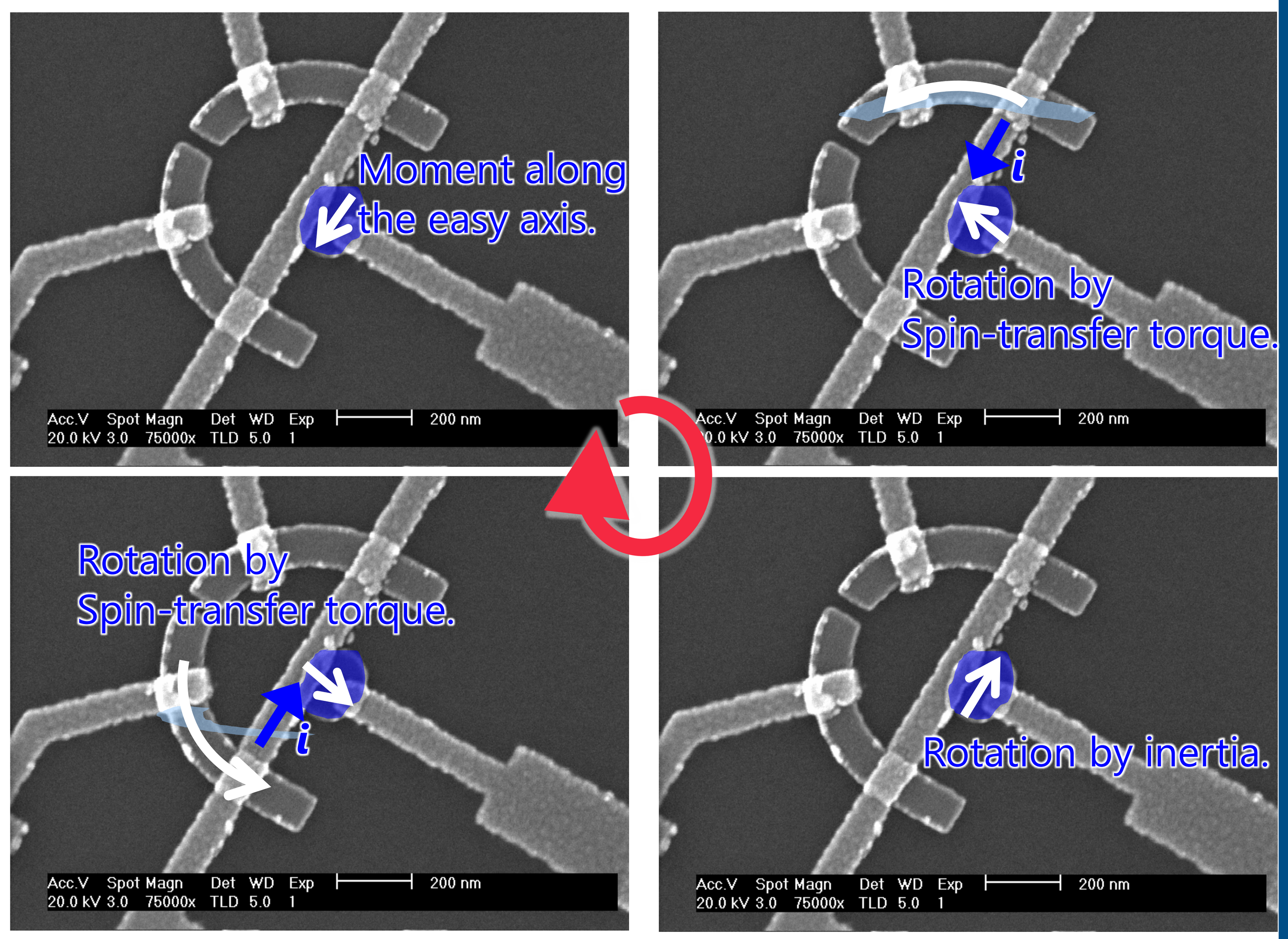


	Conventional	This invention
Rotation Frequency	Hz ~ kHz	Hz ~ GHz
Device size	100 µm ~ 1 mm	100 nm ~ 1 µm

■Electrical operation to rotate magnetic moment

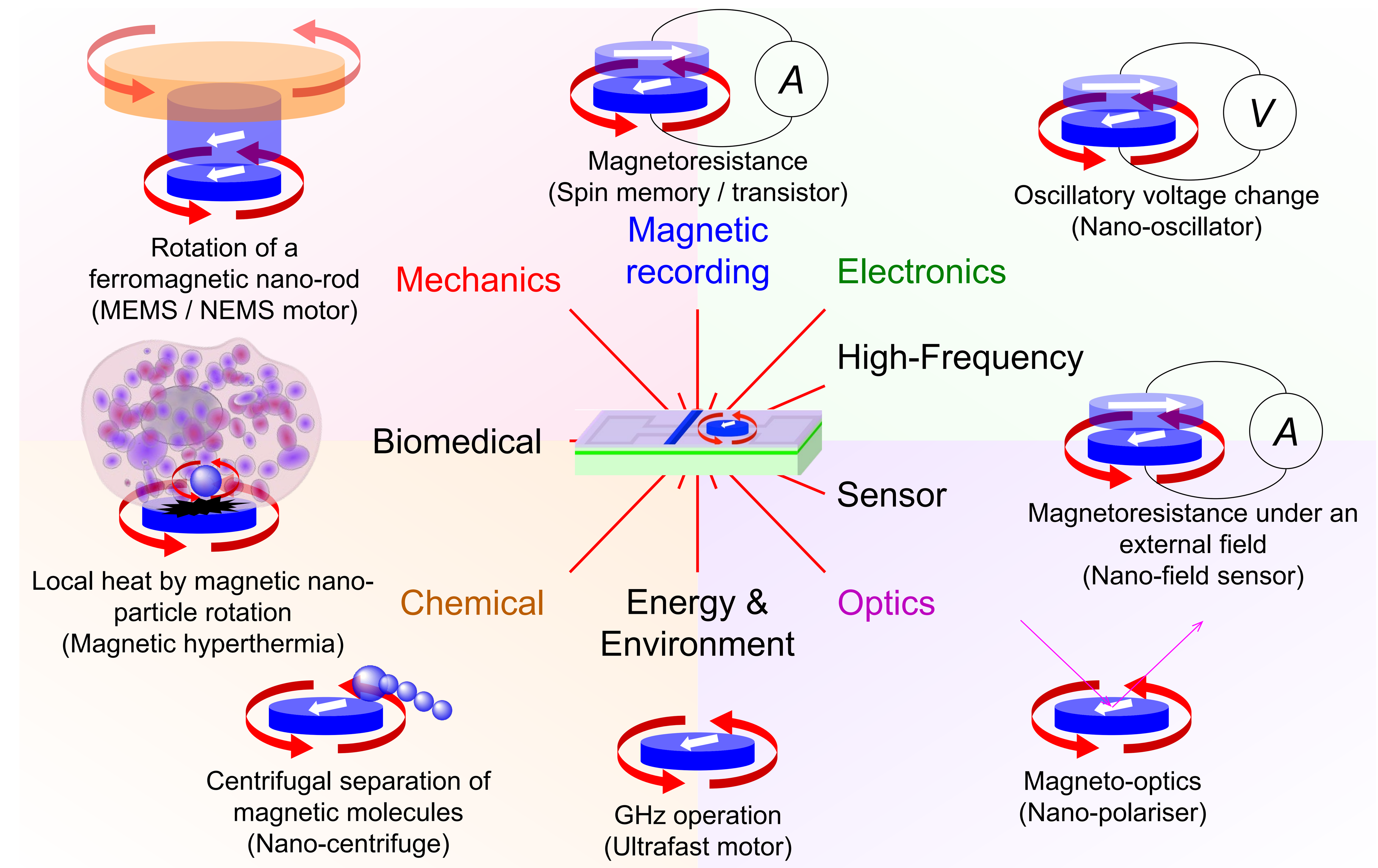


An all-metallic nano-spin motor is fabricated with a split ferromagnetic horse-shoe and a nano-disc as well as a non-magnetic bridge and electrodes.



By alternatively introducing a current from each split horse-shoe, spin-polarised electrons exert a torque onto the nano-disc, achieving the rotation of the magnetic moment in the nano-disc.

3. Potential Applications



■A nano-spin motor, nano-flow controller for a MEMS and NEMS up to GHz rotation.
■Molecules and cells stirrer, using those tagged with magnetic nanoparticles
■Local nanoscale heater via the rotation of a magnetic nanoparticle in a cell for perforation.

4. Patent Licensing Available

Patent No.: WO2014/024697 (JP, US, EP, CN, TW & KR)

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Encoding Flow Topologies

A Common Language for Flows and Topological Flow Data Analysis

Prof. Takashi SAKAJO (Kyoto Univ.) Prof. Tomoo Yokoyama (Kyoto Univ. of Edu.)

1. Conventional Technology

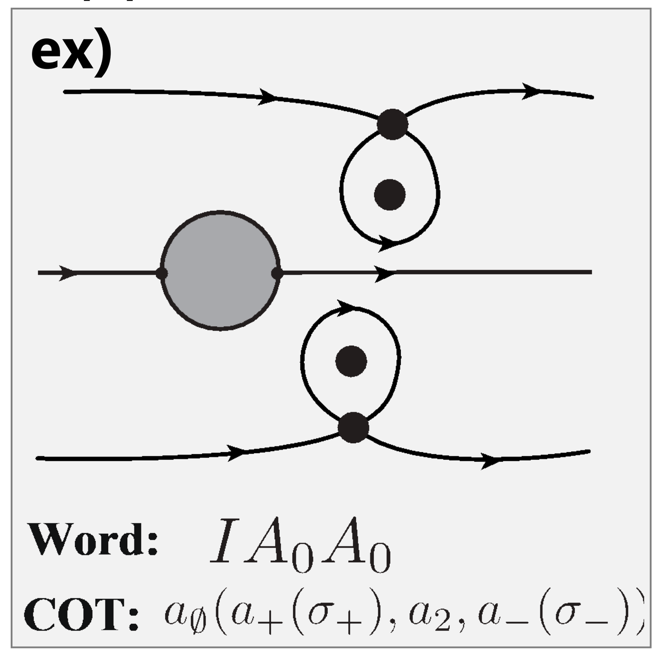
There are not a few Flow Analysis Systems in the market but they have some limitations and disadvantages;

- Limited range of searchable flows
- Lack of accuracy
- Too difficult to realize automatic Image recognition
- Unavailable information extractions

2. Our Technology

What is Topological Flow Data Analysis (TFDA) ?

- **A brand-new methodology for data analysis from topology:** Unique letters, called COT (partially Cyclically Ordered rooted Tree) and word representations, are assigned to a large number of flow patterns. We also obtain some qualitative/quantitative information associated with letters. The letters are able to be used as basis for new data analysis innovation in wide variety of application field.
- **Easy characterization:** Even if you don't know the mathematical theory, you can make the characterization from flow data by understanding the mechanism of conversion rules. And software for automatic assignment of the characterization is now available.



What are the Advantages of TFDA?

- **"DNA information" of Flow can be Extracted:** You can see each assigned letter represents a specific function.
- **Knowledge from the experience about flows can be Extracted:** Characteristic properties can be extracted by statistical processing of the letters.
- **Future predictions of flow patterns can be Available:** You will be able to predict in advance the possible changes of flows in future if you look at the letters without any exception.

3. Experiment: The lift acting on a wing

(a) ,(b)Consider a flow around an inclined flat wing in a uniform flow. It is starting to flow at t=0.0.
(c) In figure, word and COT representations are assigned to stream functions of the flows obtained by numerical simulation.
Numerical computation also yields the time-evolution of the lift-to-drag ratio acting on the flat plate.

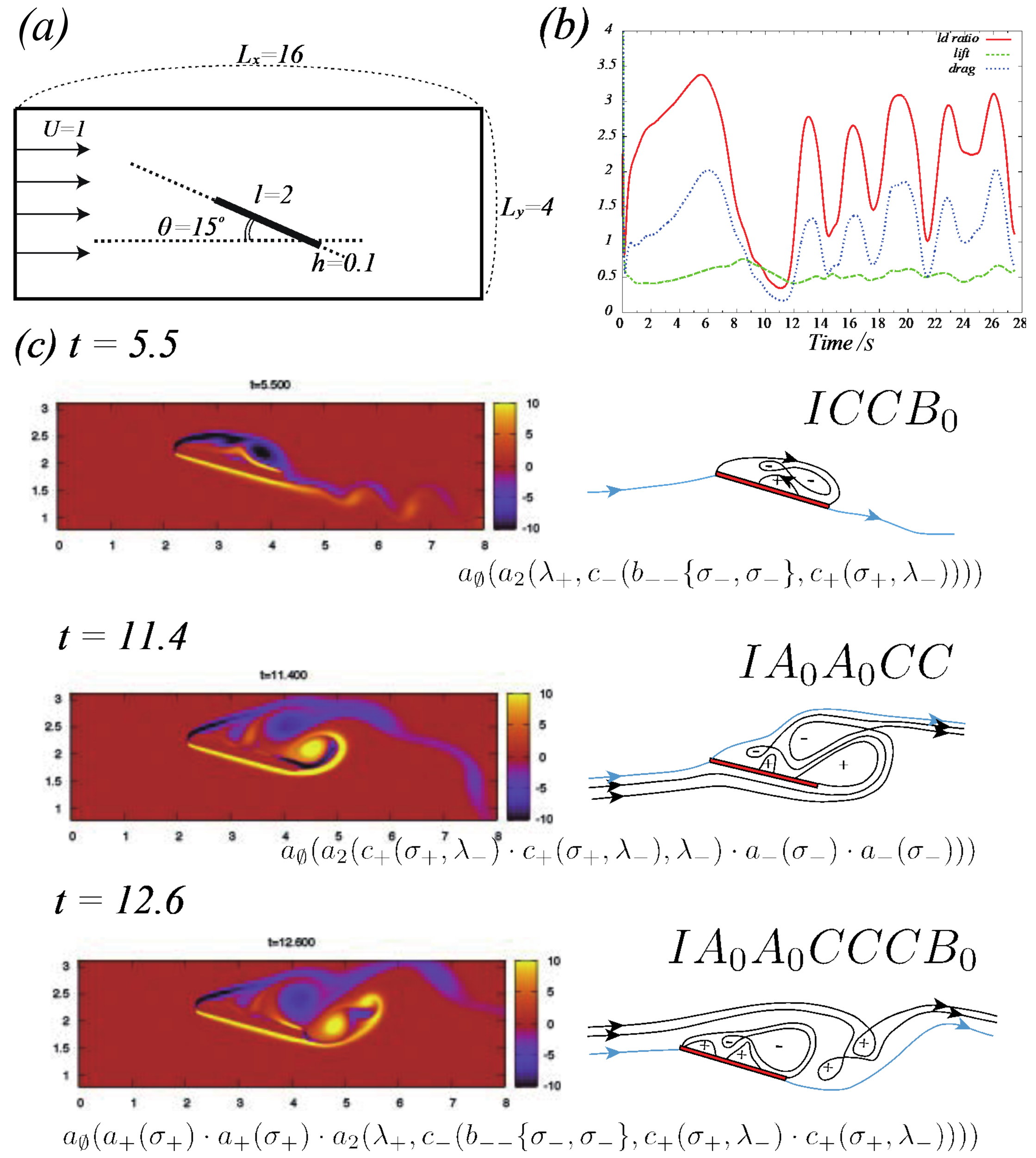
・The lift is maximal at t=5.5 and t=12.6.
・It is minimal at t=11.4.

How should this calculation result relate to this consideration?

Using our technology, the streamline is extracted, and is assigned letters (in (c) right).

・There is the word sequence "CCB_0" at t=5.5 and t=12.6.
・There is no that at t=11.4.
・CCB_0 :The letter is a code for the existence of force enhancing vortex structure entrapped above the plate.

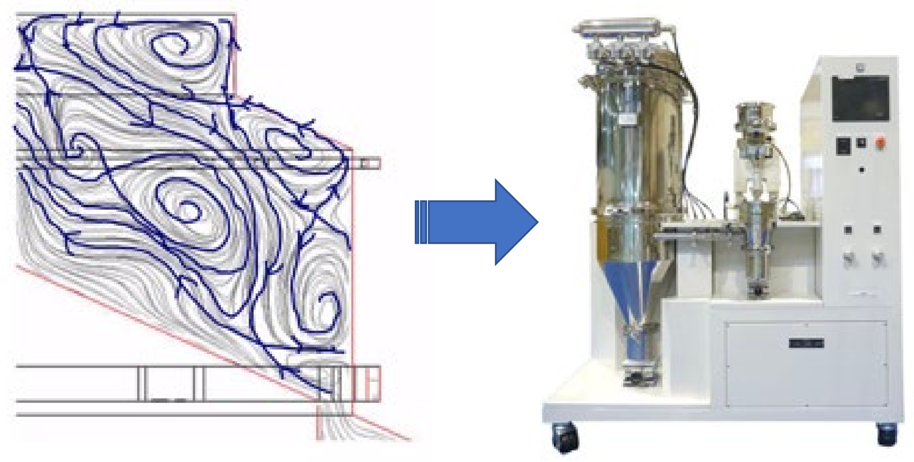
We can efficiently described situation by extracting patterns including "CCB_0" for all data letters.

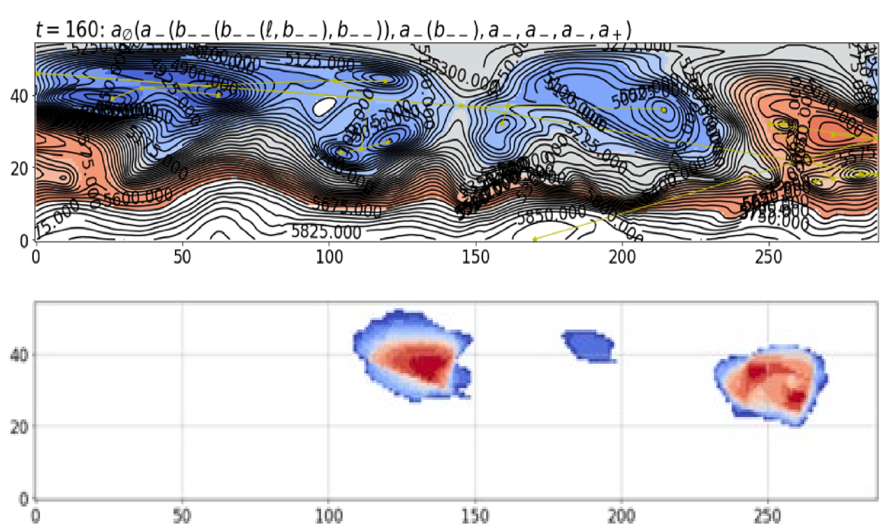


4. Applications

Any types of flow patters can be visualized in the flow field of medical engineering, environments and materials.

- **Design Optimization of Powder Jet Mill:** The design guidelines can be proposed from optimal device counting by streamline analysis for output of numerical calculations.
- **Judgment of extreme weather by weather maps:** By assigning a string of letters to a pattern of weather data to the weather map, the automatic judgement of extreme weather such as blocking phenomenon, can be available.





5. Patent Licensing Available

Patent No.: JP2019-139657, WO2014/041917(JP,US,EP,CN,IN,KR)
WO2016/072515(JP,US,EP,IN), etc

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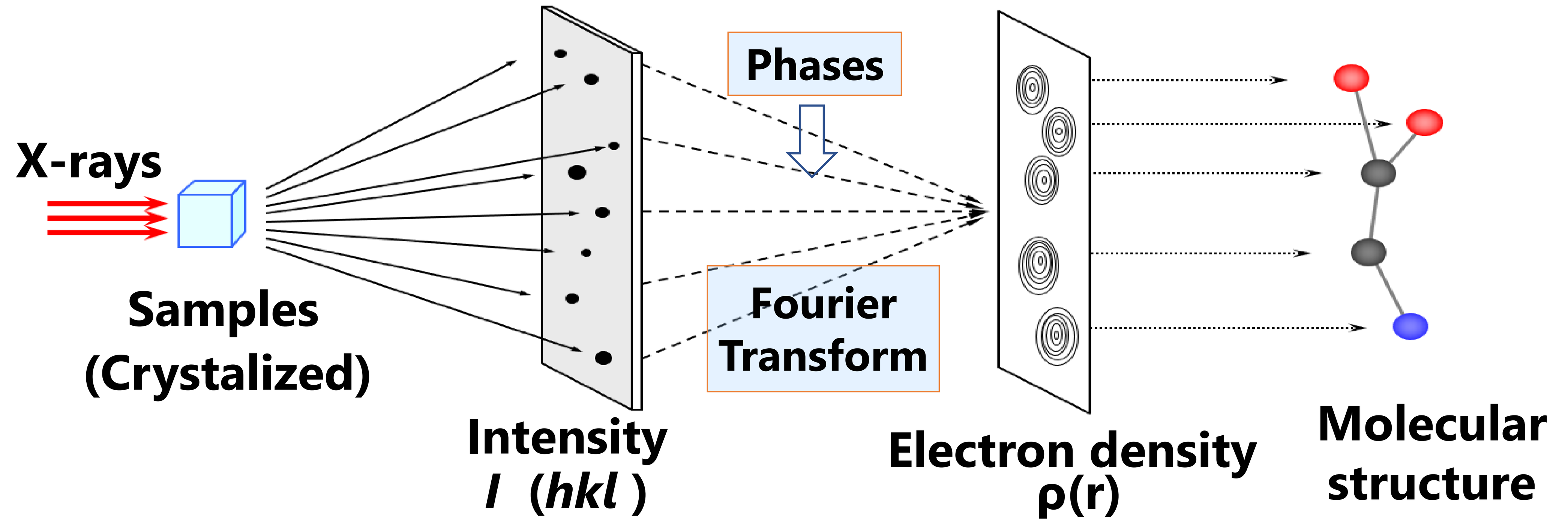
Molecular Structure Identification

X-ray Structural Analysis without Crystallization

University Distinguished Professor, Makoto FUJITA (The University of Tokyo)

1. Conventional X-ray Structure Analysis



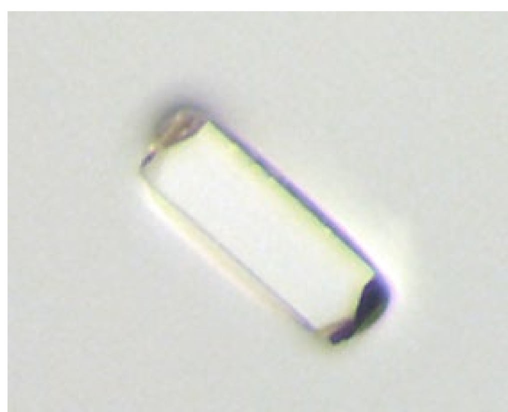
Problem: A high quality <u>single crystal</u> is <u>required</u>.

2. Core Technology : The Crystalline Sponge Analysis

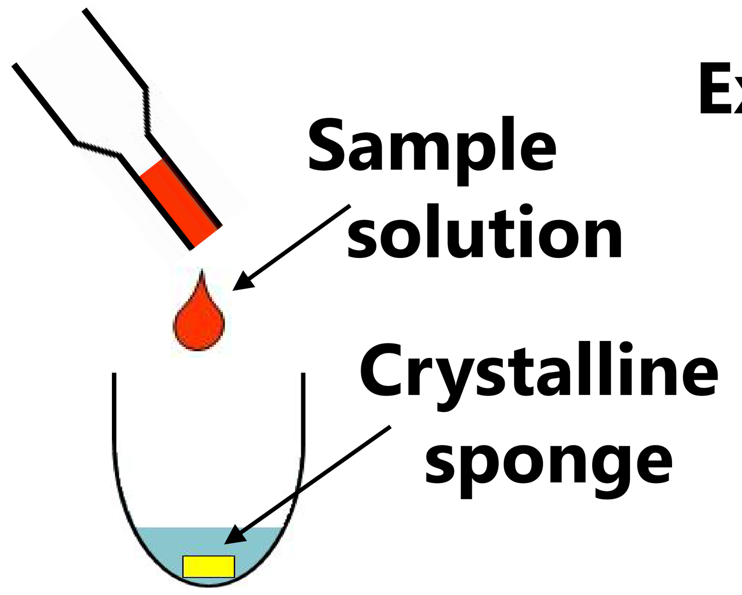
Nature 2013, 495, 461-466

<u>No growth of single crystals</u> required.

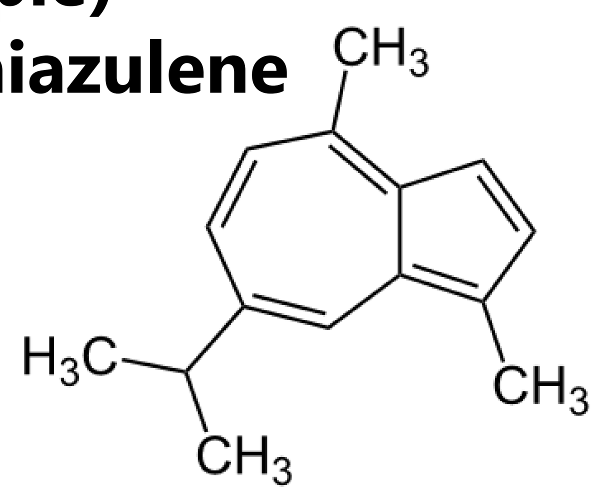
Let sample soak into the perfect single crystalline sponge matrix.

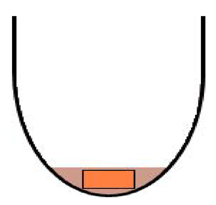


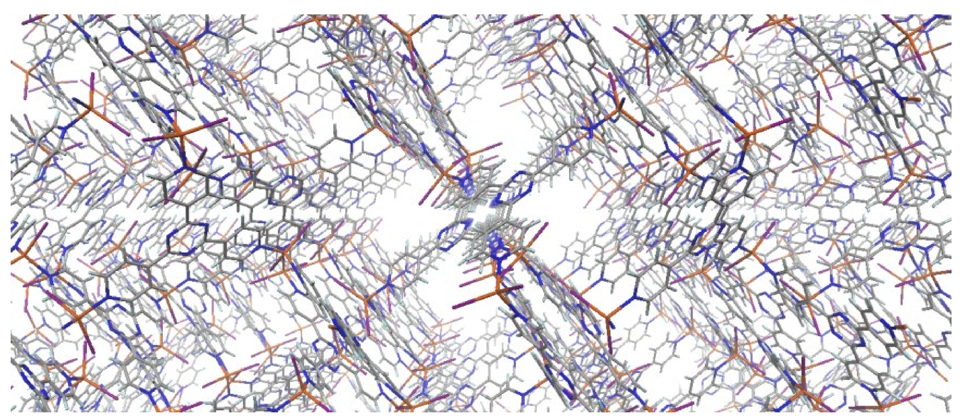
Crystalline sponge
About 0.2 mm in size



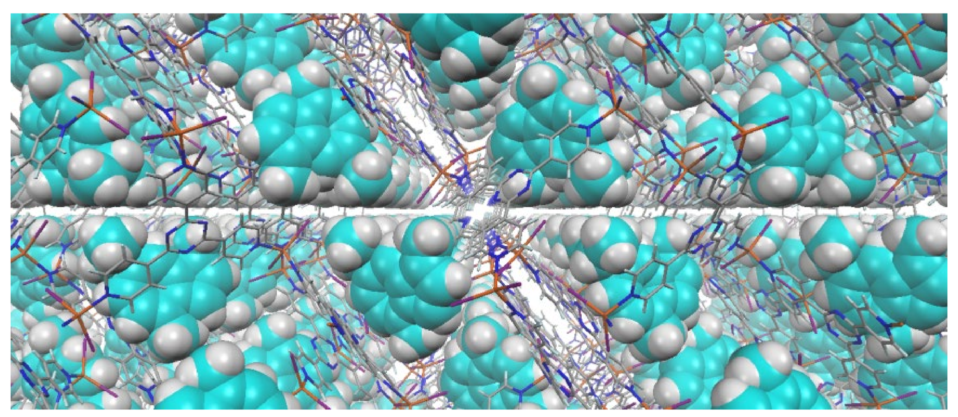
Example)
Guaiazulene







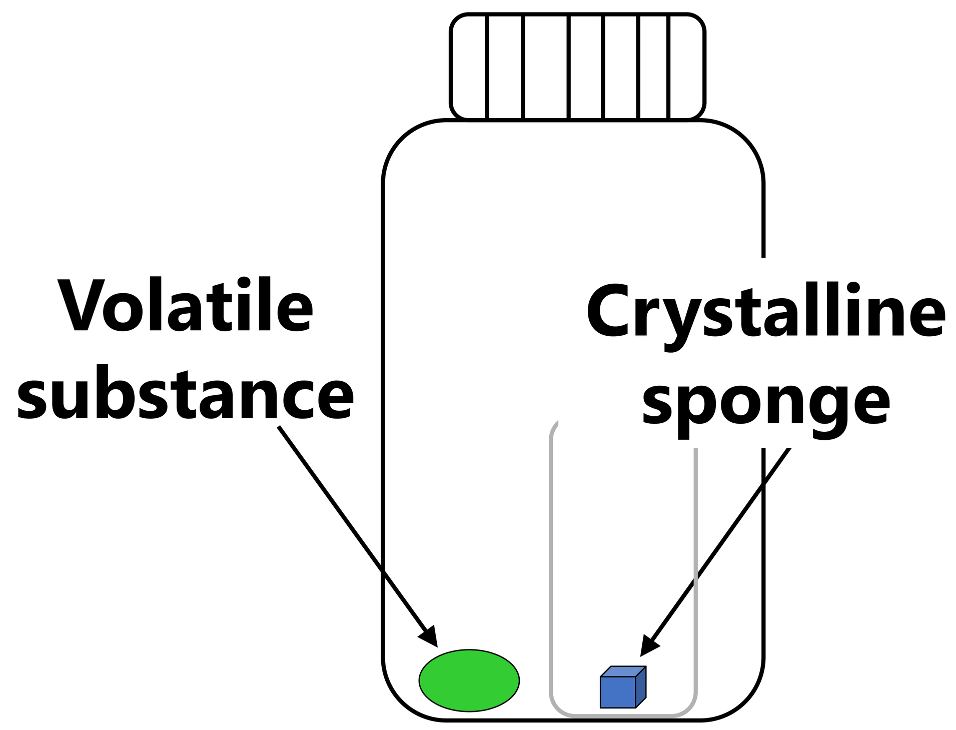
Crystal sponge:
Crystal with Porous framework



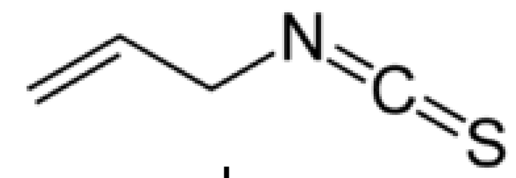
Samples molecules arranged in the crystal sponge

3. Structural analysis of volatile substances possible as well

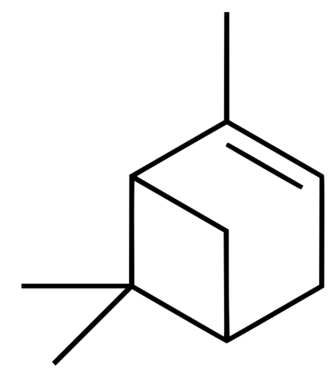
Cited from Rigaku Journal 35(1) 2019



Example



from wasabi



from Cypress essential oil

Structural analysis of <u>nanogram amounts</u> of molecule is possible.

4. Companies licensed to provide service

As of December 2019

- **Merck KGaA, Darmstadt, Germany**
  E-mail: bob.young@milliporesigma.com
  Phone: +1 (240) 447-4579

＊The life science business of Merck KGaA, Darmstadt, Germany operates as MilliporeSigma in the U.S. and Canada.

- **Mitsui Chemical Analysis & Consulting Service, Inc.**
  E-mail: Kazunari.Shiozawa@mitsuichemicals.com
  Phone: +81-3-5524-3853

- **Rigaku Corporation** - Contract Service & Instruments
  E-mail: sponge@rigaku.com
  Phone: +81-42-545-8103

Thanks to Rigaku Corporation and Merck KGaA, Darmstadt, Germany for providing photos and materials

Patent:WO2014/038220,WO2016/017770

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