Selected Novel Technologies for Licensing

2015 MRS Fall

Innovative Technologies for Your Products

Material

- Very Low-temperature Growth of Graphene
- High Heat resistant and Transparent Bioplastics
- Mechanochromic Fluorescent Polymers
- ”floats on water” AFS (Aluminum Foam Sandwich) Material
- New OLED Structure for High-Definition Display Panel

Device

- Highly Efficient and Compact Leaky Wave Antenna Using Non-Reciprocal Metamaterials
- Printable Elastic Conductors with a High Conductivity for Electronic Textile Applications
- InGAIN-Based Nitride TFTs
- Nano-Rheology Printing Technology for Metal-Oxide Patterns and Devices

Equipment

- Precisely Controlled Nanocluster Assembly
- New Measurement Method for Materials : THz Nanometry Microscope
- Low-Energy Inverse Photoemission Spectroscopy (LEIPS)
- Spin-Polarized Electron Source and the Application

Japan Science and Technology Agency

Material

Very Low-temperature Growth of Graphene

Prof. Jun-ichi FUJITA (University of Tsukuba)

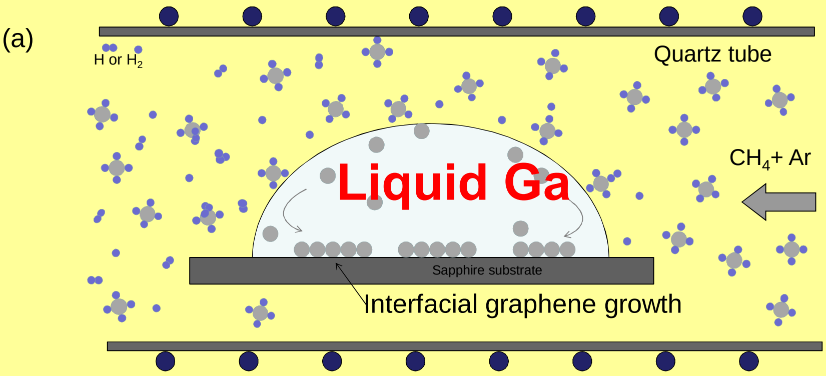
1. Background

- **Graphene** has great potentials for several next-generation devices, such as the electrode pattern on heavy-duty/industrial use printed circuit boards and the transparent elements.
- The prominent **fabrication methods of a large area/low cost graphene,** especially ***under low-temperature environment***, have been long-awaited.

flexible display

2. Newly Invented "Very Low-temperature Fabrication Method"

- The very low-temperature fabrication method is newly invented.
- The technique can sufficiently reduce the synthesis temperature and combine graphene synthesis with conventional ***Si-based processing***.
- This "two-stage graphene growth technique" should prove useful for a wide variety of graphene-based electrical device applications.



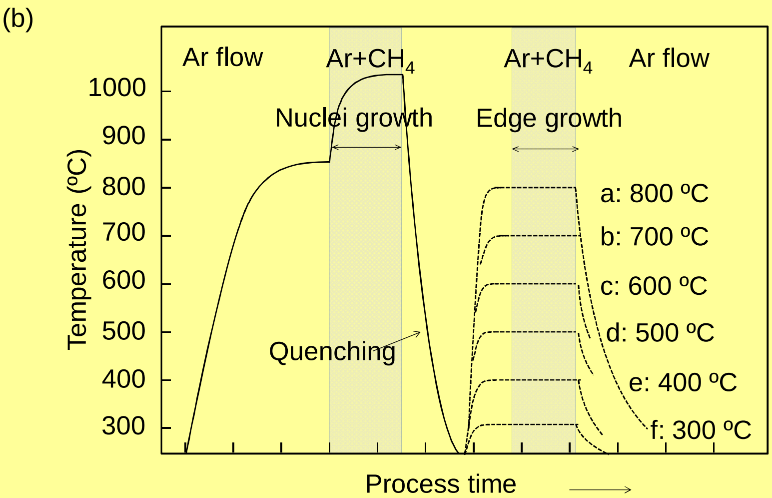


Fig. (a) interfacial CVD growth using molten gallium

∗ The source gas of methane is effectively decomposed on molten gallium at 300℃ , which can be easily confirmed by the dark-colored gallium surface after CVD. Although carbon is insoluble in gallium, the concentration of carbon on the gallium surface was large and thus the molten gallium can transport the carbon to feed edge growth around the nuclei.

Fig. (b) temperature controlling for the two stages of graphene growth

∗ A large area of graphene was grown at 300℃ at the interface of a molten gallium catalyst on a sapphire substrate under diluted methane gas ambient of partial pressure of 1/10000atm (≈10 Pa). Although the nucleation of graphene requires much higher temperatures of >1000℃, the graphene growth around the nuclei continued even when the temperature was reduced to 300℃.

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Material

High heat resistant and transparent bioplastics

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

1. Technical Summary

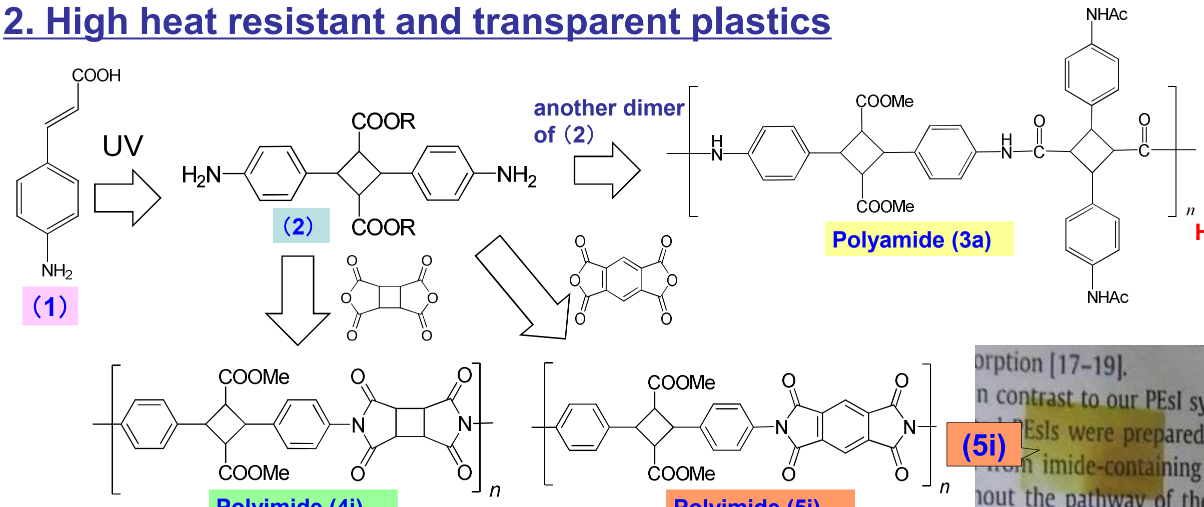
■A combination of microbial production and photochemical reaction created monomer (2).

① We have established a microbial production conditions of 4-aminocinnamic acid (1), which was generally considered difficult due to incompatibility of aniline compound with microorganisms.

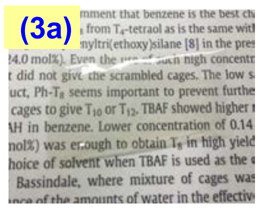
② By photochemical reaction of 4-aminocinnamic acid (1), the monomer (2) was synthesized in almost 100 % yield.

■Polyamides and polyimide films have been synthesized from monomer (2). These films have a very high heat resistance and excellent transparency.

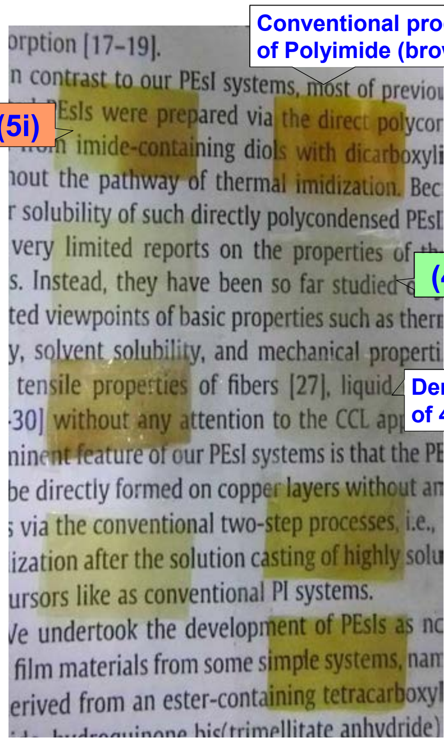
2. High heat resistant and transparent plastics



Heat resistant temperature 390℃ Heat resistant temperature 425℃



Heat resistant temperature 370℃



Various polyimide films processed in our works

Biocompatibility
No interruption to cell proliferation

3. Comparison with the conventional techniques

Conventional Techniques	Temperature
The melting point of lead-free solder	183℃～378℃
The heat-resistant temperature of Kapton® (DuPont)	>500℃
The heat-resistant temperature of fluorinated transparent polyamides	About 335℃

4. Potential applications

The highest heat-resistant in transparent resin	
Thermal decomposition temperature	Maximum 425℃
Linear thermal expansion coefficient (The values are comparable with metals.)	<10 ppm / K
Transmittance (High transparency)	88% @ 450 nm
Young's modulus (Strength of the material)	10 GPa
High refractive index	1.6
Other Features: Ultraviolet degradation, self-extinguishing, high dielectric breakdown resistance	

- LED sealant
- Flexible Display Devices & Components
- Tempered glass-alternative materials
- Automobile, body material of the aircraft

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Patent : WO2013/073519

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Mechanochromic Fluorescent Polymers

Assistant Prof. Shohei SAITO (Nagoya Univ.), Associate Prof. Hiroshi YABU (Tohoku Univ.)

1. Issues to be cleared for mechanochromic fluorescent polymers

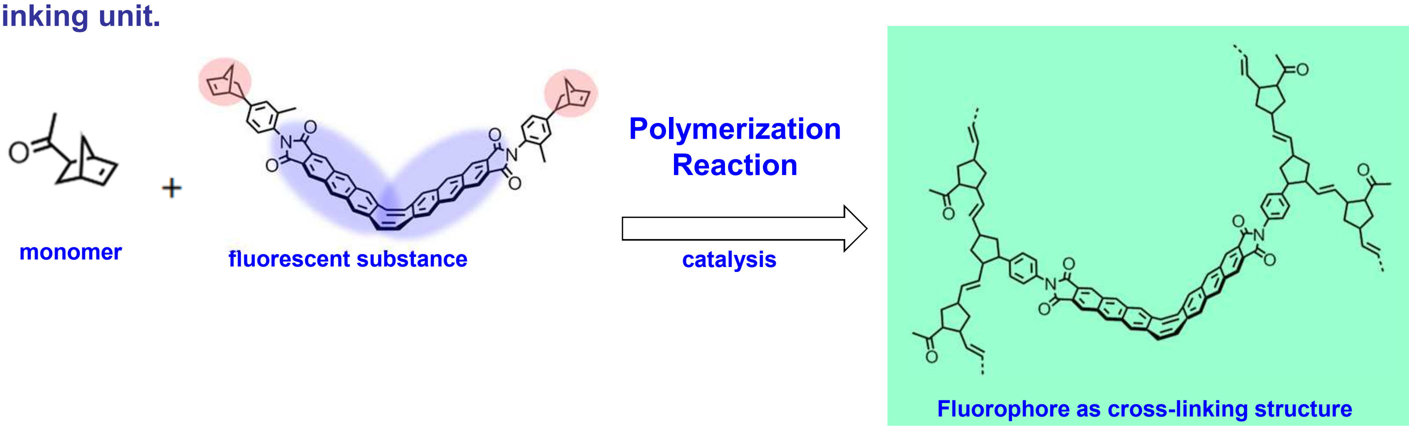
- Fluorescent color often changes when grinding the powder sample.
- By merely mixing the fluorescent materials with the polymer, fluorescent color does not change.
- Some of fluorescent polymers show an irreversible color change.
- Reported fluorescent polymer that shows a reversible color change takes 2 hours to be restored.

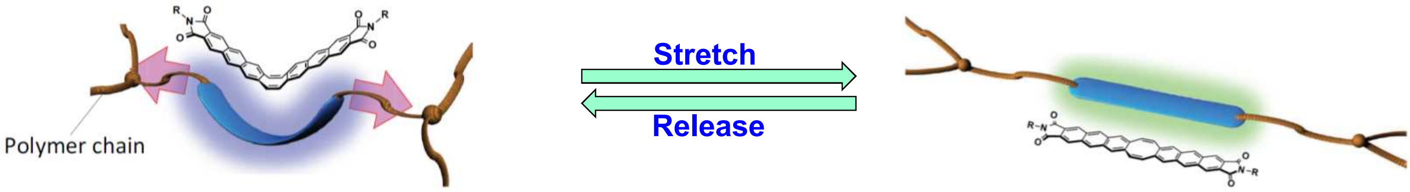
⇩

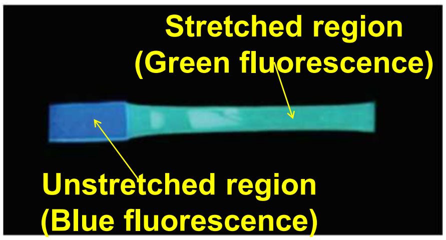
We have created a rapid and reversible fluorescent polymers.

2. Material Properties

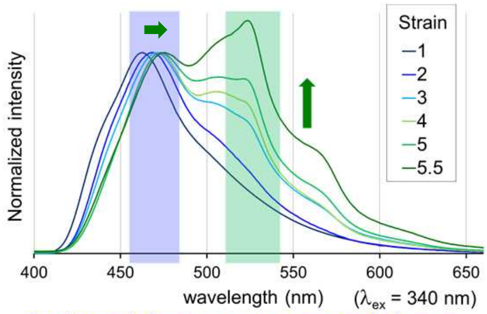
The fluorescent polymers were synthesized with monomers and fluorescent substance as a cross-linking unit.



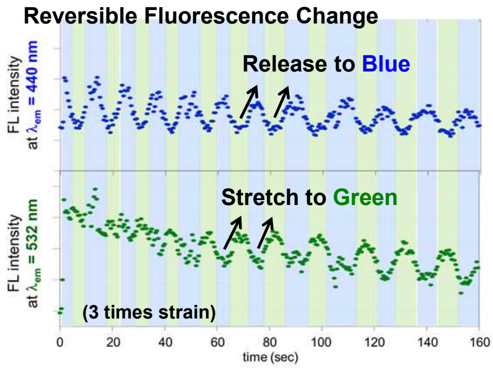




Rapid and reversible response



Strain and fluorescence spectral change



3. Possible applications

- Our polymer can visualize the tension.
- Our polymer can detect the distortion or destruction of objects.

⇨

- Visualization of material distortion on bridges and tunnels
- Visualization of traction force by undifferentiated cells

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Patent : PCT/JP2015/82143

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Material

"floats on water" AFS (Aluminum Foam Sandwich) material

Associate Prof. Yoshihiko HANGAI (Gunma University)

1. AFS structured Metal Foam

- An Aluminum Foam Sandwich (AFS) structure is a composite consisting of an aluminum foam core with two dense metallic face sheets.
- The AFS structured material can be used as the light-weight structural components with high stiffness and high energy/vibration absorption properties.

2. AFS product appearance

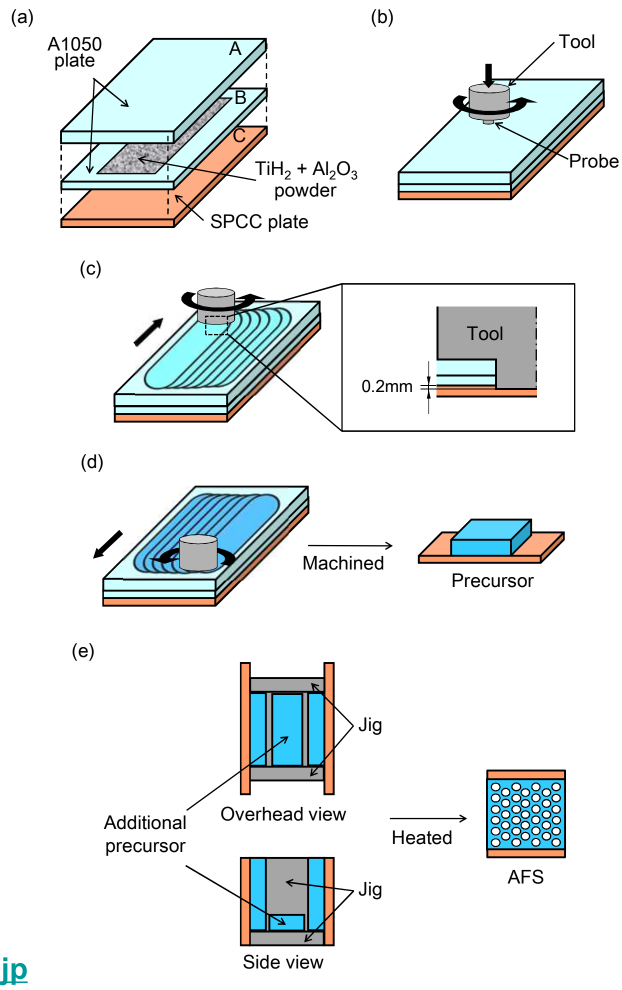
(AFS structured panel)

(AFS structured cylinder)

3. New "Friction Stir Processing (FSP)" for fabricating AFS structure

- The low cost **"AFS structured material"** i.e. panel, cylinder, pipe is developed by using the **"Friction Stir Processing (FSP)"** .
- Newly developed **"metal bonding method"** is highly productive and NO adhesive is necessary for **"AFS structured material"**.

* FSP was developed from the basic principles of friction stir welding (FSW), which is a solid-state bonding process.



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Patent : PCT/JP2010/067979, PCT/JP2009/065097, PCT/JP2010/052821

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New OLED Structure for High-Definition Display Panel

Material

Prof. Hideo HOSONO (Tokyo Institute of Technology)

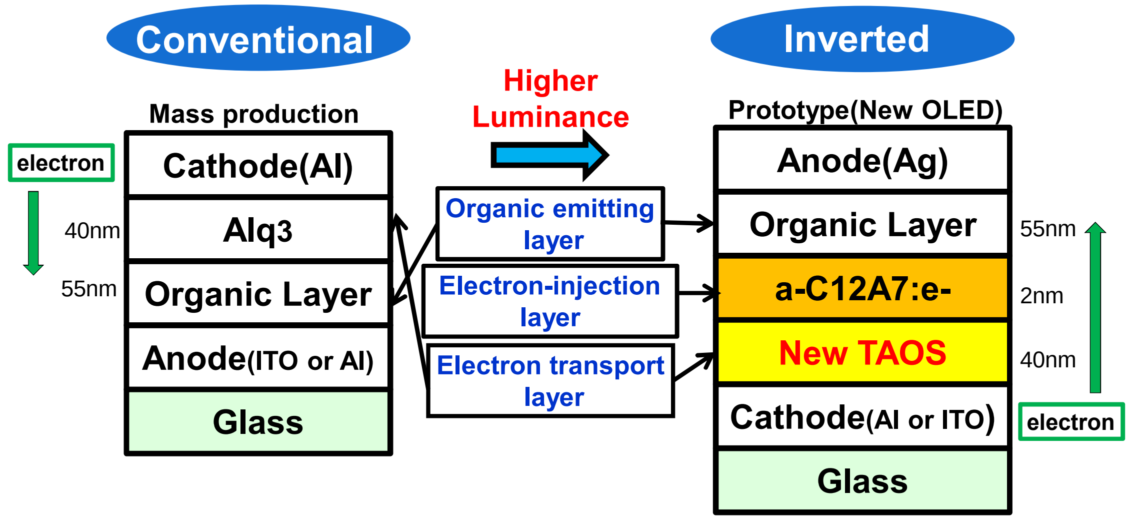
Succeeded in developing the higher luminance OLED than conventional OLED, by combining New TAOS and Electride.

1. OLED Structure

ITO : tin-doped indium oxide

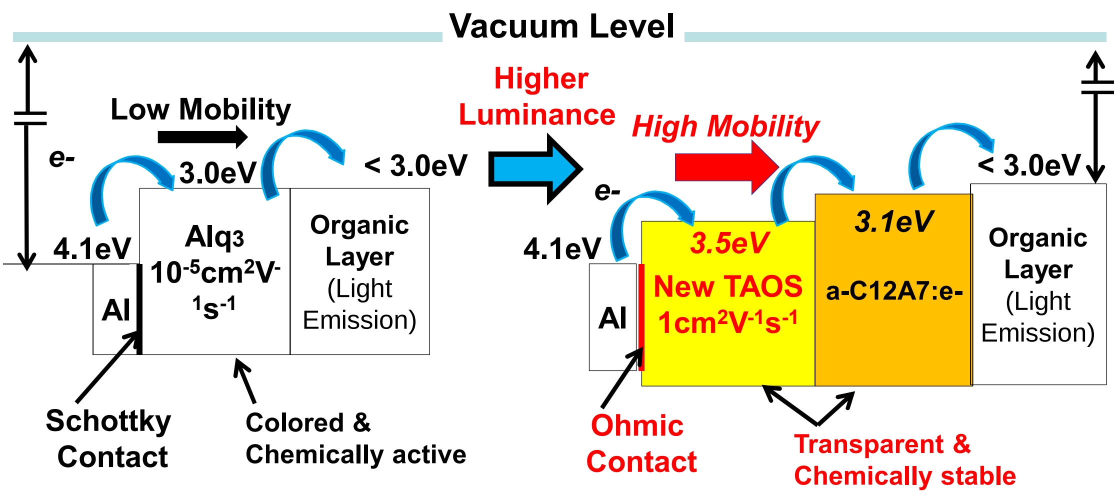
a-C12A7:e- : electride

TAOS : Transparent Amorphous Oxide Semiconductor



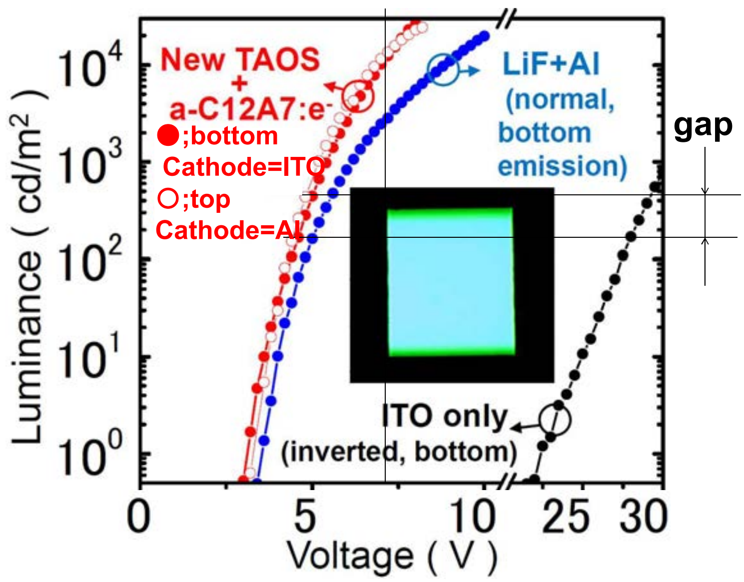
2. Energy Diagram

Smooth Electron transfer for high luminance



3. Luminance

Comparison with a high luminant conventional OLED (LiF+Al)



Material Stability

New OLED : Stable

LiF : Sealing is necessary to prevent deterioration

Application for New OLED

- Display panel
- Lighting equipment (high luminance and thin type)
  - -Embedded light in a mirror and a window
  - -Low profile interior lighting
  - -In the future
    New application using a flexible printed circuit

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Device

Highly Efficient and Compact Leaky Wave Antenna Using Non-Reciprocal Metamaterials

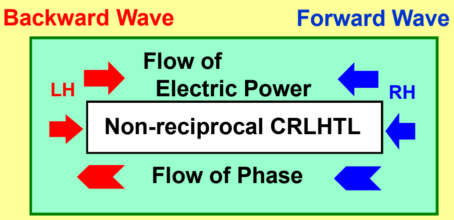
Associate Prof. Tetsuya UEDA (Kyoto Institute of Technology)

Non-reciprocal Metamaterial Technology :

A new concept that combines the non-reciprocal circuit with different transmission characteristics by the propagation direction and the metamaterial technology.

1. Utilization of Non-Reciprocal Metamaterial Technology to Antennas

Non-reciprocal CRLHTL(*)



(*)Composite Right/Left Handed Transmission Lines

Forward: Positive refractive index (RH)

Backward: Negative refractive index (LH) ➔ *metamaterial*

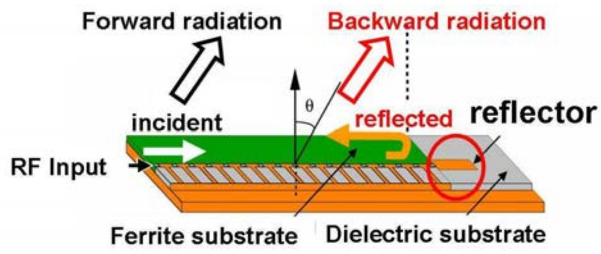
Wave vector is the same direction regardless of the direction of transmitted power

➔ The new technology applied to antenna

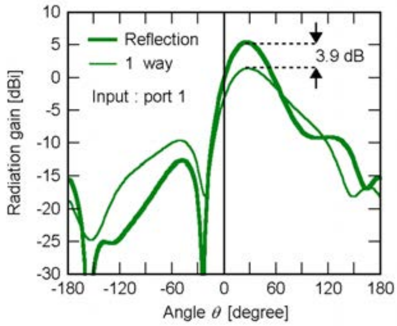
-1 Application to the leaky wave antenna [Non-reciprocal leaky wave antenna]

・It can be reused the radio waves by having reflecting at the end.

➔ Radiation efficiency and directivity are improved



Non-reciprocal Leaky Wave Antenna



Improvement of Radiation Efficiency (calculated result)

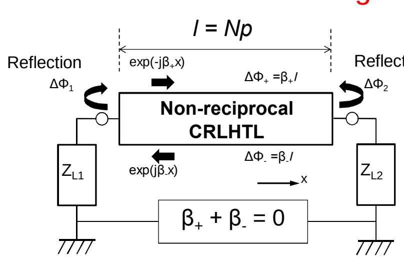
-2 Application to the travelling-wave resonator and the beam-scan antenna

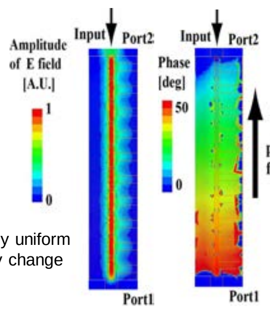
・The resonance condition is automatically satisfied not depending on the resonator size by connecting reflector on both ends.

➔ Possible to change the size of resonator, keeping fixing the resonance frequency. Reduced size of antenna is possible.

・Phase gradient of the electromagnetic field distribution on the resonator is continuously controllable by an externally applied magnetic field.

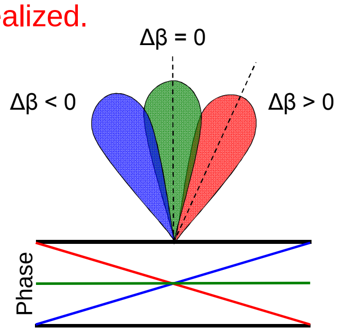
➔ Small and high radiation efficiency beam-scan antenna is realized.





Amplitude: Automatically uniform
Phase gradient: Linearly change

Pseudo-Traveling-Wave Resonator



Beam-scan Antenna

2. Potential Applications

- Beam-scanning antennas for microwave and/or millimeter wave radar
- Beam-scanning antennas for compact wireless communication in which highly-directivity is not required
- Antennas for wireless power transmission

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Patent : WO2008/111460, WO2011/024575, WO2012/014984, WO2012/115245

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Device

Printable Elastic Conductors with a High Conductivity for Electronic Textile Applications

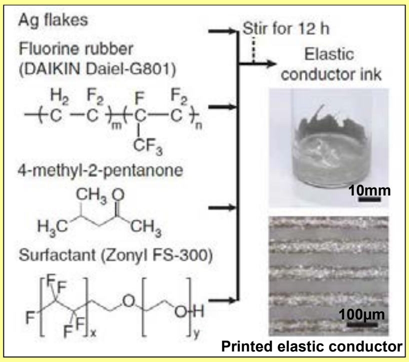
Prof. Takao SOMEYA (The University of Tokyo)

1. Abstract

- New electronic functional ink which exhibits high conductivity and mechanical durability while being printed on textile have been developed.
- The printed conductors by using the new ink realizes a conductivity of 182Scm^{-1} at a strain of 215%, which is currently the highest value reported for stretchable conductors that can be stretched > 150%.

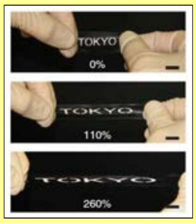
2. Highly Stretchable Elastic Conductors

2-1 Fabrication Process of elastic conductor ink

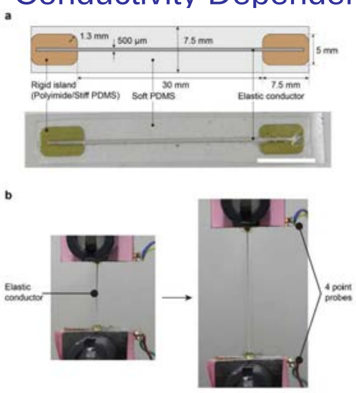


- The elastic conductor ink is prepared by adding Ag flakes as conductive fillers to an elastomeric fluorine copolymer with 4-methyl-2-pentanone as an organic solvent, together with a water-based fluorine surfactant.

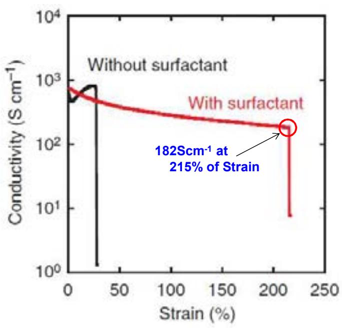
- The conductor ink can be readily printed with conventional printing techniques such as stencil printing or dispensers.



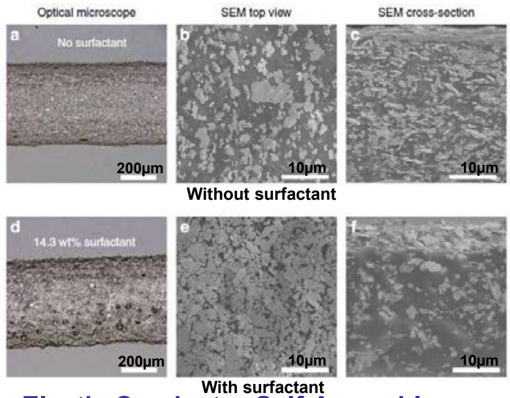
2-2 Conductivity Dependence on Tensile Strain of Printed Elastic Conductor



(a)Evaluation Method

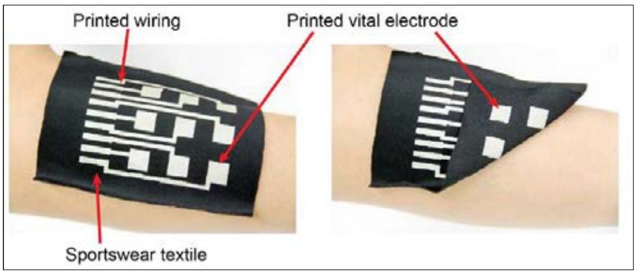


(b)Conductivity vs Strain



Elastic Conductor Self-Assembly by Phase Separation

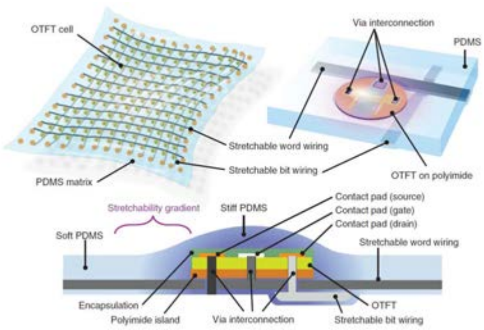
3. As Wearable Sensor



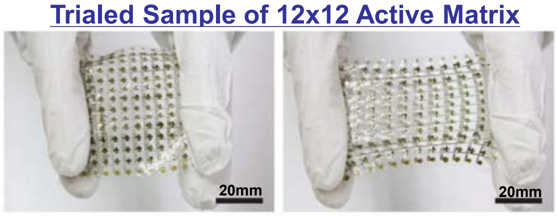
A Muscle Activity Sensor Fabricated on Sportswear Material

- Electrode, wires, and via holes can be printed by a single step printing process. The muscle activity sensor was fabricated combining with an organic transistor amplifier circuit.

4. As Stretchable Organic Transistor Active Matrix



Trialed Sample of 12x12 Active Matrix



Relaxed | Stretched to about 60%

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Patent: WO2015/119217

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InGaAlN-Based Nitride TFTs

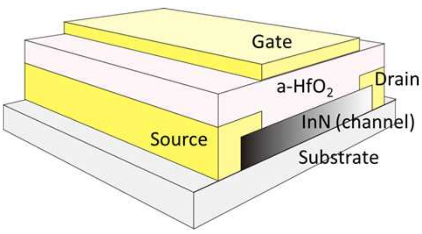
Prof. Hiroshi FUJIOKA (The University of Tokyo)

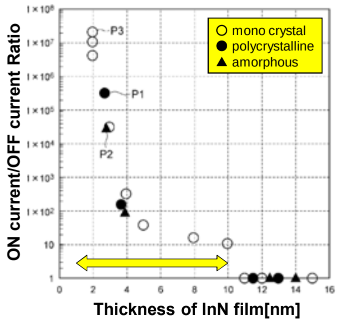
1. Abstract

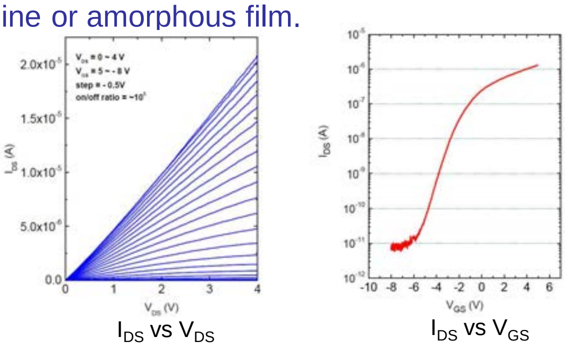
- A transistor having an InGaAlN-based nitride semiconductor layer as a channel is newly invented.
- Even though the film is polycrystalline or amorphous, electrical properties are equivalent to those of a monocrystalline film, in case they are designed in the range of suitable composition ratio of InGaAlN.

2. FET Having Thin Film of InN as a Channel

- The InN-based nitride semiconductor layer is a polycrystalline or amorphous film having a film thickness of 1-10nm. The InN layer having a film thickness within the range exhibits electrical characteristics equivalent to those of monocrystalline film, even if the layer is polycrystalline or amorphous film.



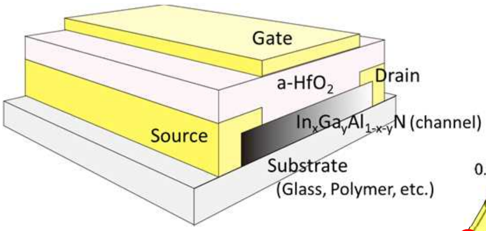


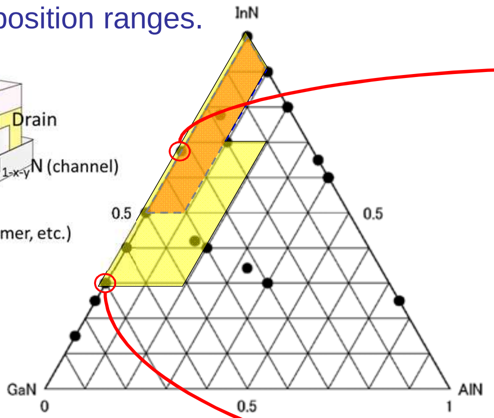


Characteristics of a TFT Consisting of Polycrystalline InN Layer

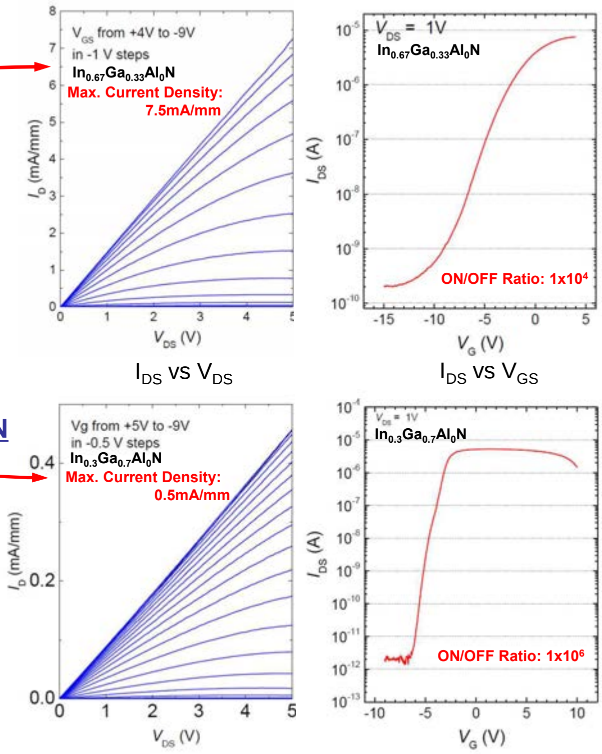
3. InGaAlN-based Nitride Semiconductor

- We have found out that polycrystalline or amorphous InGaAlN-based nitride semiconductors, which consist of non-mono-crystalline films, have the same electrical characteristics as the case of a monocrystalline film under the specific composition ranges.





3-phase composition diagram of $In_xGa_yAl_zN$



Characteristics of TFTs Consisting of Polycrystalline $In_xGa_yAl_{1-x-y}N$ Layer (examples)

Trialed TFTs with InGaAlN-based nitride semiconductor layer:
- The layer is formed by PSD(Pulsed Sputtering Deposition).
- The deposition temperature is lower than 600℃.
- The layer consists of polycrystalline or amorphous film.

➔Good result of ON/OFF current ratio was obtained within the specific composition ranges of $In_xGa_yAl_{1-x-y}N$ $(_{x+y=1.0})$.

These results indicate :
- Constraints in manufacturing conditions are significantly resolved.
- Excellent electrical characteristics are provided at a reduced cost.

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Patent : WO2015/029434, WO2015/029435

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Device

Nano-Rheology Printing Technology for Metal-Oxide Patterns and Devices

Prof. Tatsuya SHIMODA (Japan Advanced Institute of Science and Technology)

1. Abstract

- A new device printing technology is proposed. This device printing utilizes a viscoelastic transformation of the precursor gel when imprinted; it softens at a certain temperature during thermal-imprinting so that the gel can be rheologically imprinted.
- Excellent metal-oxide patterns were formed by this method. Further, thin-film transistors(TFTs) with channel length around 500nm are easily printed under normal atmosphere.

2. The Nano-Rheology Printing(n-RP) Technology

Process of the Nano-Rheology Printing:

(1) A solution is coated and dried to make a semi-solid thin film.

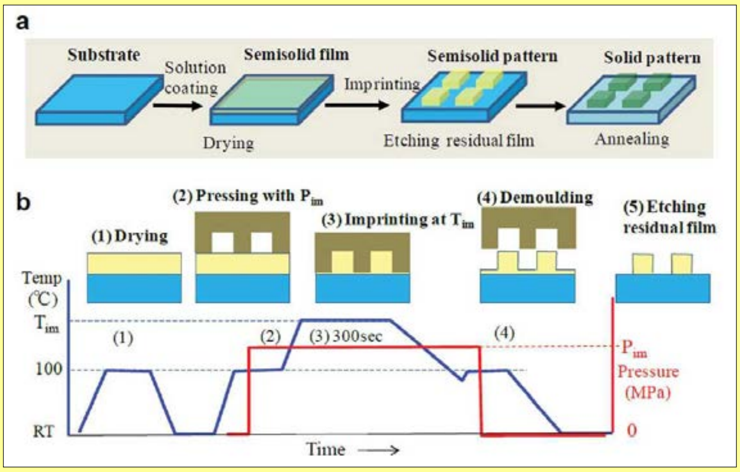
(2) Loaded onto the heating stage of the imprinting machine, after which a mold is set onto the semi-solid film and pressure is applied.

(2)→(3)
When the temperature being increased, the semi-solid film softens at a certain temperature.

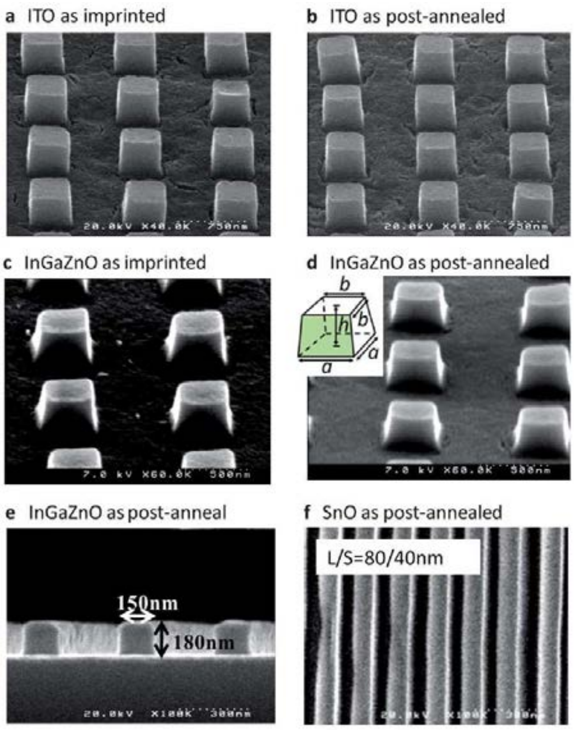
(3) The imprinting temperature(T_{im}) has to be maintained to complete the imprinting.

(4) The temperature being lowered and then the mold is discharged.

(5) The residual film has to be removed by etching in normal atmosphere.

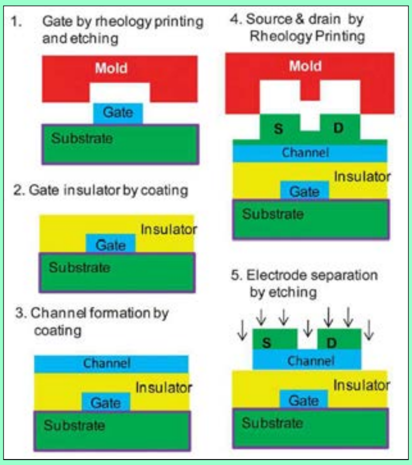


3. Metal-Oxide Patterns Formed by the Nano-Rheology Printing(n-RP) Technology

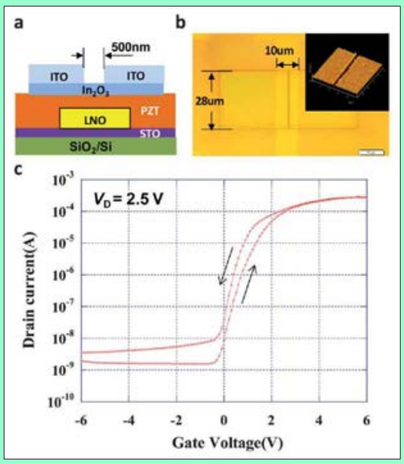


- All samples of formed metal-oxide patterns have a well-defined rectangular shape.
- The deformation after the post-annealing is also slight.

Example of TFT Fabrication by the n-RP Technology



Fabrication Steps of the n-RP



Id-Vg Characteristics of TFT

- The nano-rheology printing technology can fabricate the functional devices, with a short process. These devices can be made by using significantly less raw materials and less energy.
- It is possible to print well-defined patterns of several tens of nanometers necessary for high-performance transistors and circuits.

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Patent : WO2011/138958, WO2013/069686

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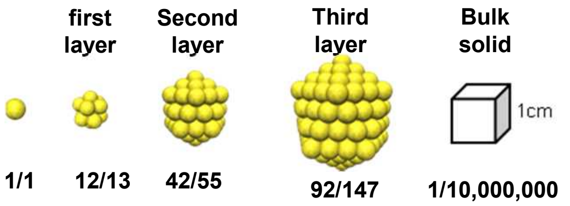
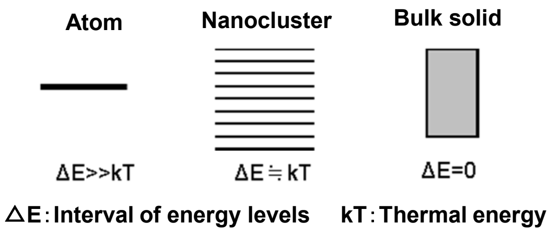
Precisely Controlled Nanocluster Assembly

Prof. Atsushi NAKAJIMA (Keio University)

1. Features of nanoclusters

Nanocluster means an assembly which consists of several to several hundred atoms or molecules.

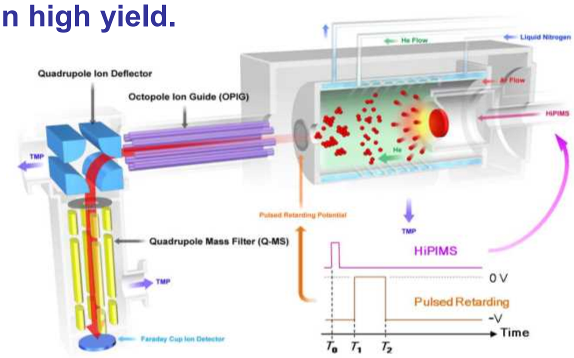


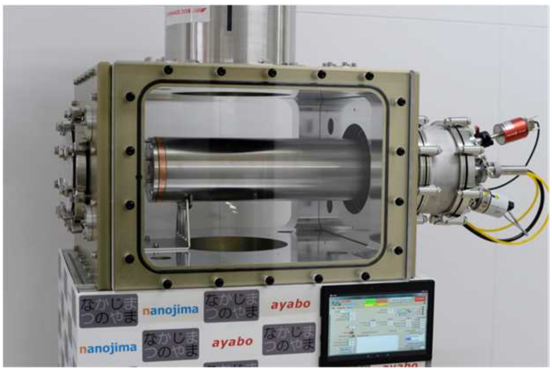


- A high surface atomic ratio.
- As the electronic structure is discrete, a specific catalytic activity point or magnetism appears.

2. Preparation of Nanocluster

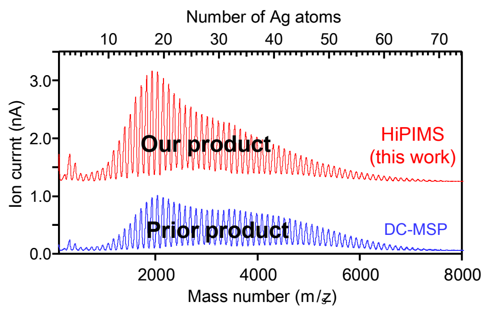
Our Nanocluster can be prepared by the method of high power impulse magnetron sputtering (HiPIMS) in high yield.



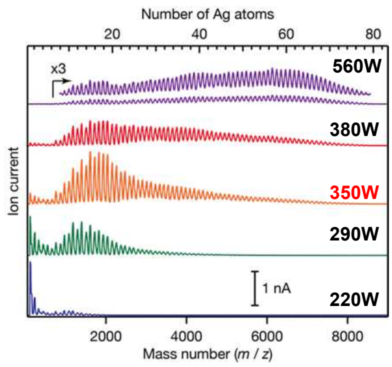


Nanocluster generator
(Trial machine by Ayabo Corporation)

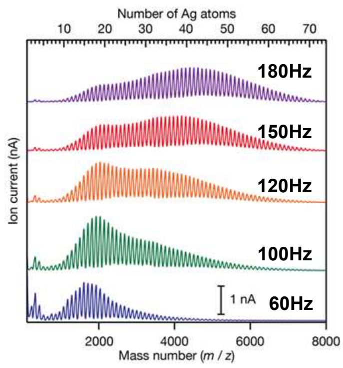
- When the wave modulation is applied to the sputtering voltage, the duty ratio is adjusted at the same time.
- An electric field which has been delayed in synchronization with the sputtering voltage is added at the exit of the electrode of cluster growth chamber.



Mass spectrum of Ag nanocluster



Size control of the discharge power



Size control by the repetition frequency

By a variety of parameter settings, it is possible to synthesize the nano clusters and nano particle size for the purpose.

parameter: Pulse power, Repetition frequency, Pulse waveform, Duty ratio, Voltage blocking electric field, etc.

3. Superiorities to conventional technique and Advantage of our technology

- **1 nm level's nanocluster can be prepared.**
  (2-3 orders of magnitude smaller size than conventional cluster)
- **It is not limited to metal.** Manufacturing of semiconductors and multi-element nanoclusters also possible.

4. Potential Market

- New devices in electronic or magnetic field
- High-performance catalysts

Patent Licensing Available

Patent: PCT/JP2014/063877

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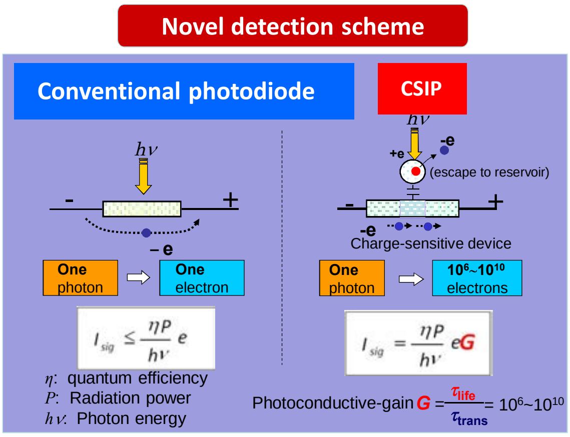
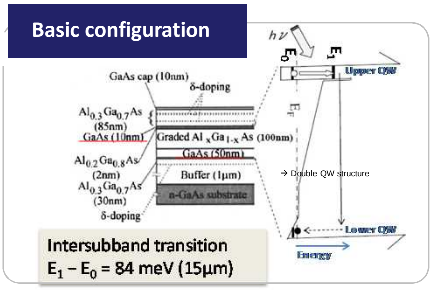
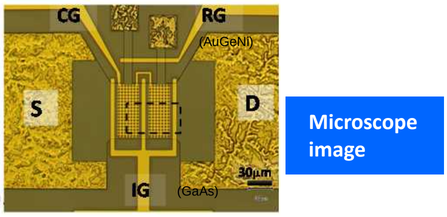
New Measurement Method for Materials THz Nanometry Microscope

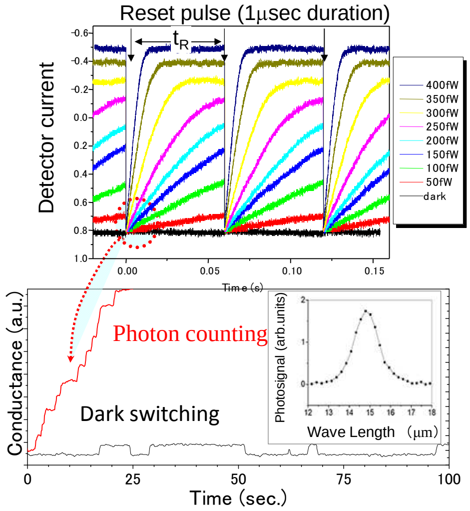
Emeritus Prof. Susumu KOMIYAMA (The University of Tokyo)

1. CSIP: Charge Sensitive Infrared Phototransistor : Single Photon Detector



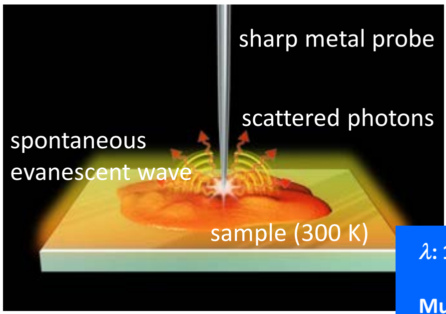






S. Komiyama, *IEEE J. Select. Topics. Quantum Elect.* 17, 54 (2011)

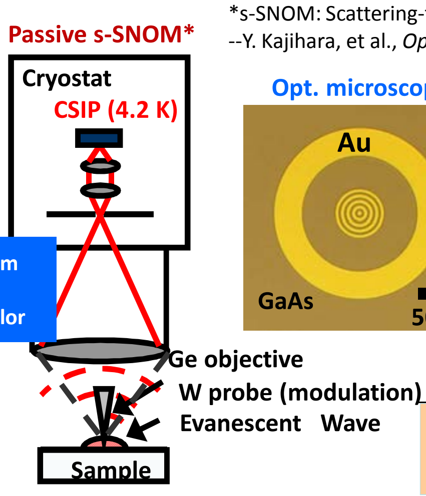
2. Application: THz Nanometry: "passive" near-field microscope



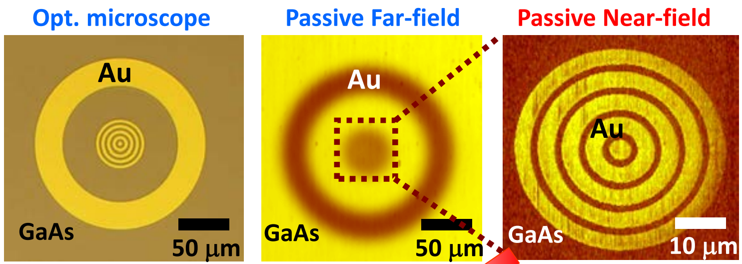
λ: 14.5 μm → Multi-Color

Required Specifications:

1. Detector → CSIP*
2. Resolution → Near-field microscope
3. Remove background noises



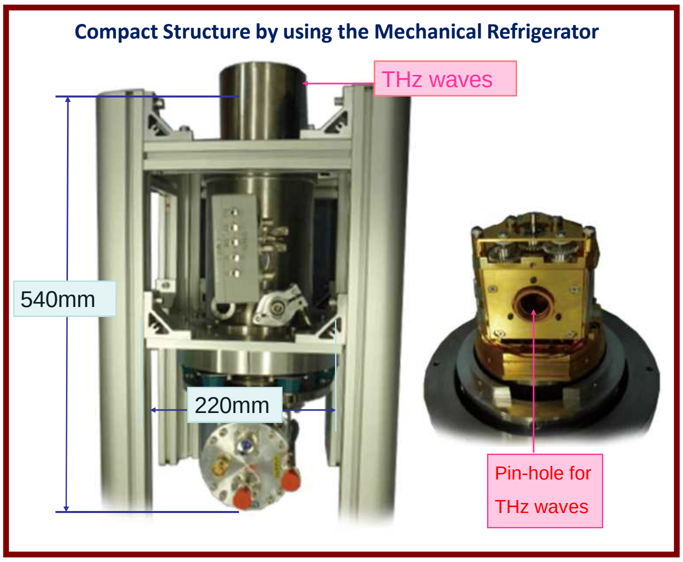
*s-SNOM: Scattering-type Scanning Near-field Optical Microscope
--Y. Kajihara, et al., *Opt. Express* 19, 7695-7704 (2011).

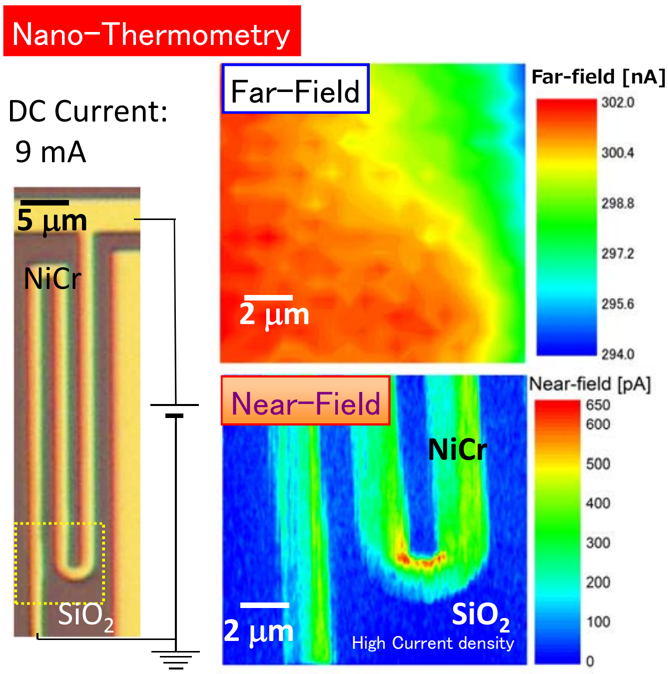


Resolution: 20 nm !

Origin: Thermal charge fluctuation

3. Tabletop ultrasensitive THz detector





Patent Licensing Available

Patent: WO2010/137422, WO2010/137423

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Equipment

Low-Energy Inverse Photoemission Spectroscopy (LEIPS)

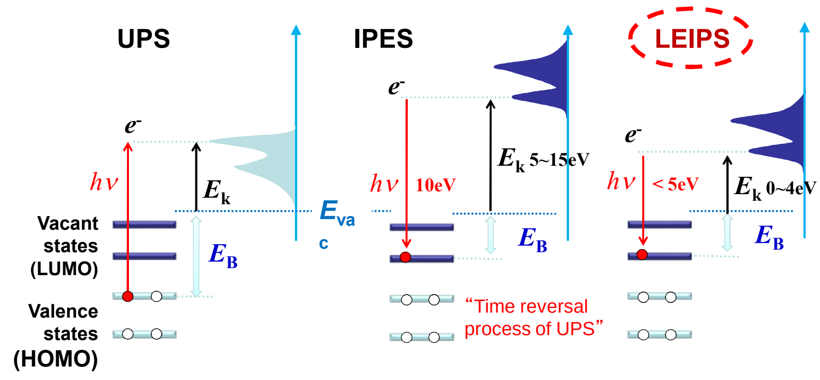
Prof. Hiroyuki YOSHIDA (Chiba University)

1. Abstract

- Electron affinity (LUMO Level) is the key parameter for the R&D of the organic electronics.
- Conventional Inverse Photo-emission Spectroscopy (IPES) has problems:
  Target materials are easy to be damaged and energy resolution is low.
- Low-Energy Inverse Photoemission Spectroscopy (LEIPS) solves the problems.

2. Principle of the Invention

Comparison of the Method of Measurement

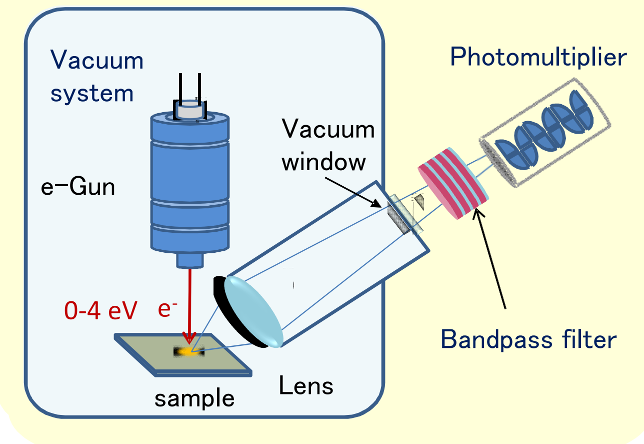


LEIPS features:

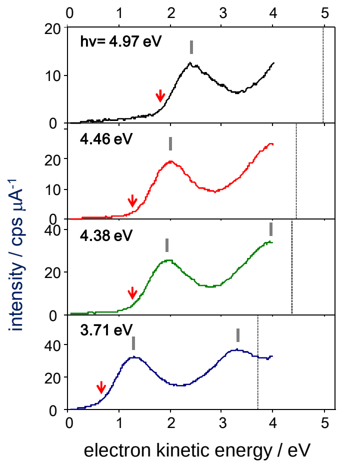
- Use of low-energy electron reduces damage to the target material.
  → Damage: negligible
- Emitted photon energy(*hv*) shifts from VUV to NUV region.
  VUV : Vacuum UltraViolet NUV : Near UltraViolet
  → Easy adaption of the standard spectroscopy technology
- Detection precision/resolution is improved.
  → Resolution improved a factor of 2 (< 0.3 eV)

3. LEIPS system and Measurement Data

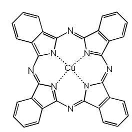
LEIPS system



Precise Determination of the Electron Affinity



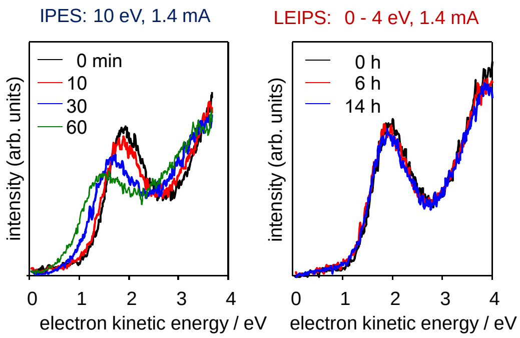
CuPc: Copper Phtharocyanine



Electron Affinity : 3.09±0.05 eV

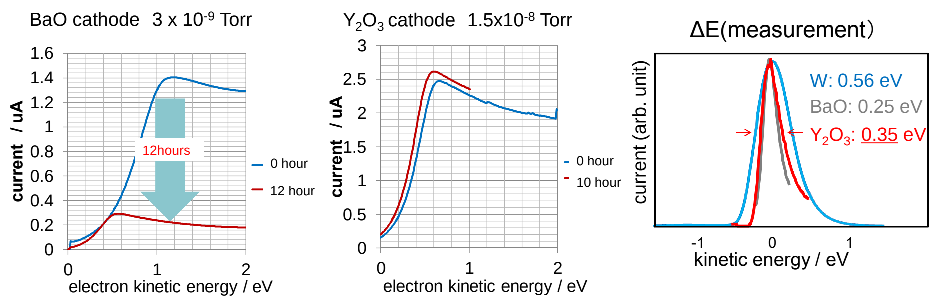
Precise measurement of the Electron Affinity is enabled by changing the energy level of the detection photon.
($hv = E_B + E_K$)

Damages to an organic material



LEIPS available at low vacuum : Y_2O_3 Source & New Optical System

Emission current variation with Time



4. Application

For R&D of organic electronics such as organic light emitting diode and organic photovoltaic cells

Patent Licensing Available

Patent : WO2013/129390 (JP, US, EP) , JP2014-111515

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Equipment

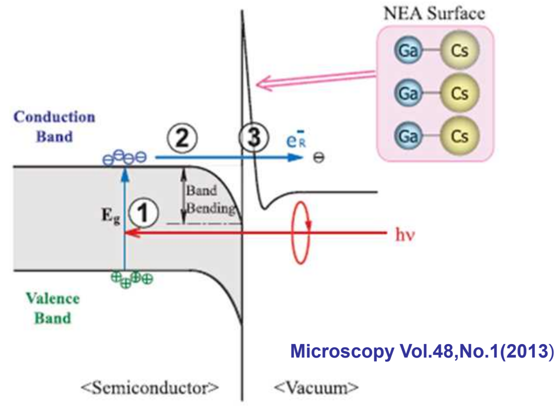
Spin-Polarized Electron Source and the Applications

Prof. Toru UJIHARA (Nagoya University)

1. Abstract

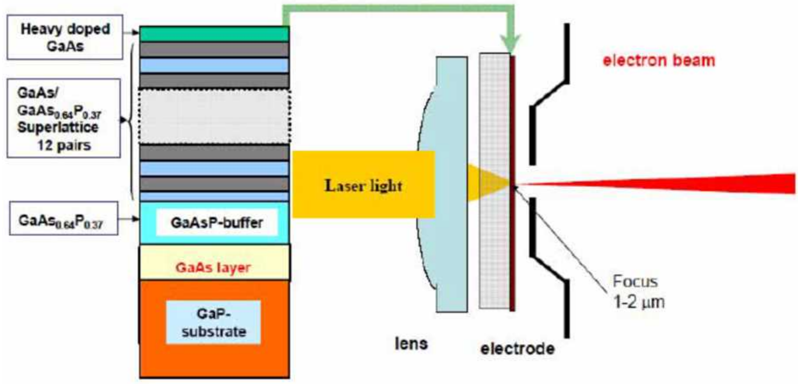
Highly spin-polarized electron source with strained superlattice photocathode has been developed. It can provide high beam brightness and high energy resolution due to the narrow energy spread. Since the electron spin direction and temporal structure are controllable, various kinds of electron microscopes such as transmission electron microscope has been constructed.

2. Principle of the Invention



Microscopy Vol.48,No.1(2013)

Generation process of spin-polarized electron



Spin-polarized electron source with GaAs/GaAsP strained superlattice photocathodes

① In the strained superlattice layer, spin-polarized electrons are excited to the conduction band due to a spin-selective excitation rule by illuminating circularly polarized light.
② The polarized electrons in the conduction band drift to the surface region.
③ The electrons extract into vacuum through an NEA surface with applying a negative electrostatic field. (NEA: Negative Electron Affinity)

3. Comparison of Brightness in various sources

Beam Brightness(A $m^{-2}sr^{-1}V^{-1}$)

Polarized electron beam source

~10^7 Transmission light absorption type electron source (GaAs-GaAsP SL)
ΔE (energy linewidth): <0.3eV
ΔI (current stability /1hour): < several %

~10^4 Conventional SPLEEM (GaAs)
ΔE (energy linewidth): <0.3eV
ΔI (current stability /1hour): < several %

Microscopy Vol.44,No.2(2009)

~10^8 Cold field emission source (W)
ΔE (energy linewidth): 0.3eV
ΔI (current stability /1hour): > 10 %

~10^8 Thermal field emission source(W/ZrO)
ΔE (energy linewidth): 0.7 ~ 1 eV
ΔI (current stability /1hour): < 1 %

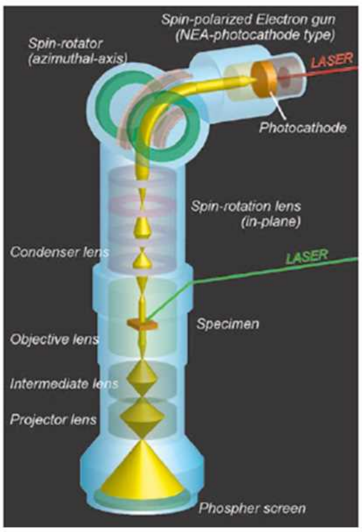
~10^6 Thermal electron source (LaB_6)
ΔE (energy linewidth): 2 ~ 3 eV
ΔI (current stability /1hour): < 2 %

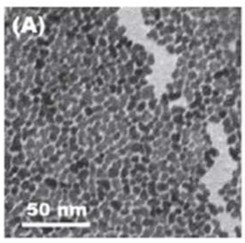
~10^5 Thermal electron source (W)
ΔE (energy linewidth): 3 ~ 4 eV
ΔI (current stability /1hour): < 1 %

Non-Polarized electron beam source

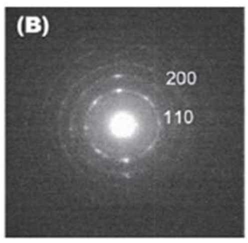
4. Application Examples

Spin-Polarized Transmission Electron Microscope





TEM image of iron



Diffraction pattern

Microscopy Vol.48,No.1(2013)

Other Applications;

Measurement & Analytical Instruments: SEM, AES, EPMA, …..
Electron Beam Lithography

Patent Licensing Available

Patent : WO2011/122171 (JP, US, EP), US 8841615
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We contribute to the creation of innovation in science and technology as the core implementing agency of the fourth phase of the Science and Technology Basic Plan.

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1. To achieve innovation in science and technology through creative research and development.
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