Chemical Preparations of Metal Nanoparticle-Modified Electrodes

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Associate Professor

Department of Material Chemistry

Graduate School of Engineering

Kyoto University, Japan

Kyoto in Japan

Capital: 1869-

Top Universities for Chemistry

QS World University Ranking 2018

http://www.topuniversities.com/...../chemistry

1.	**MIT**	**U. S.**
2.	**University of Cambridge**	**U. K.**
3.	**University of California**	**U. S.**
4.	**Stanford University**	**U. S.**
5.	**Harvard University**	**U. S.**
6.	**University of Oxford**	**U. K.**
7.	**NUS**	**Singapore**
8.	**University of Tokyo**	**Japan**
16.	**Kyoto University**	**Japan**

My Research History

1991 **Dr. Sci.** **Kyoto University**

Research Associate **Nagoya University**

Kyoto University

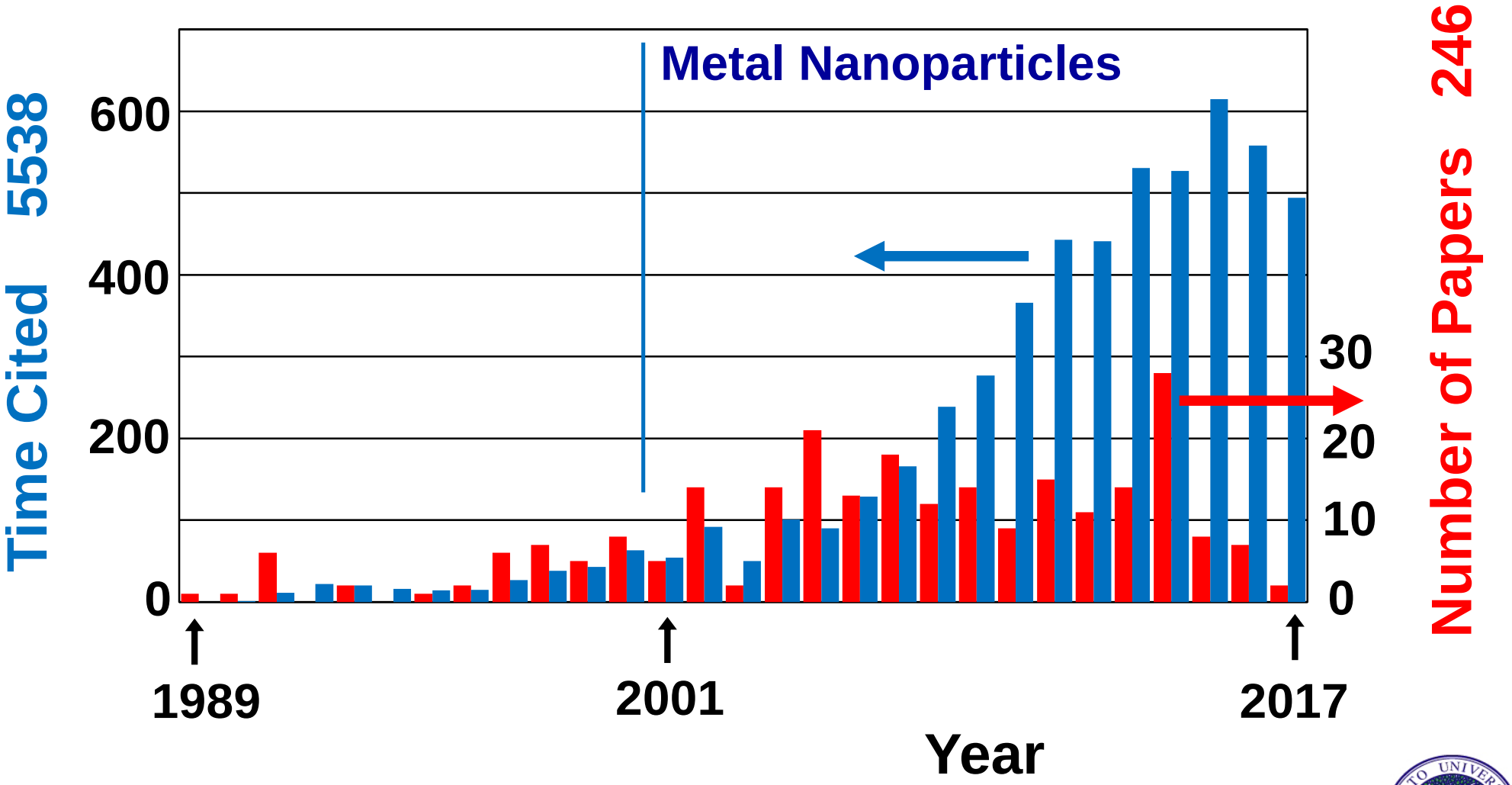
Spectroelectrochemistry in Organic Solvents

2001 **Associate Professor** **Kyoto University**

Nanomaterials Laboratory

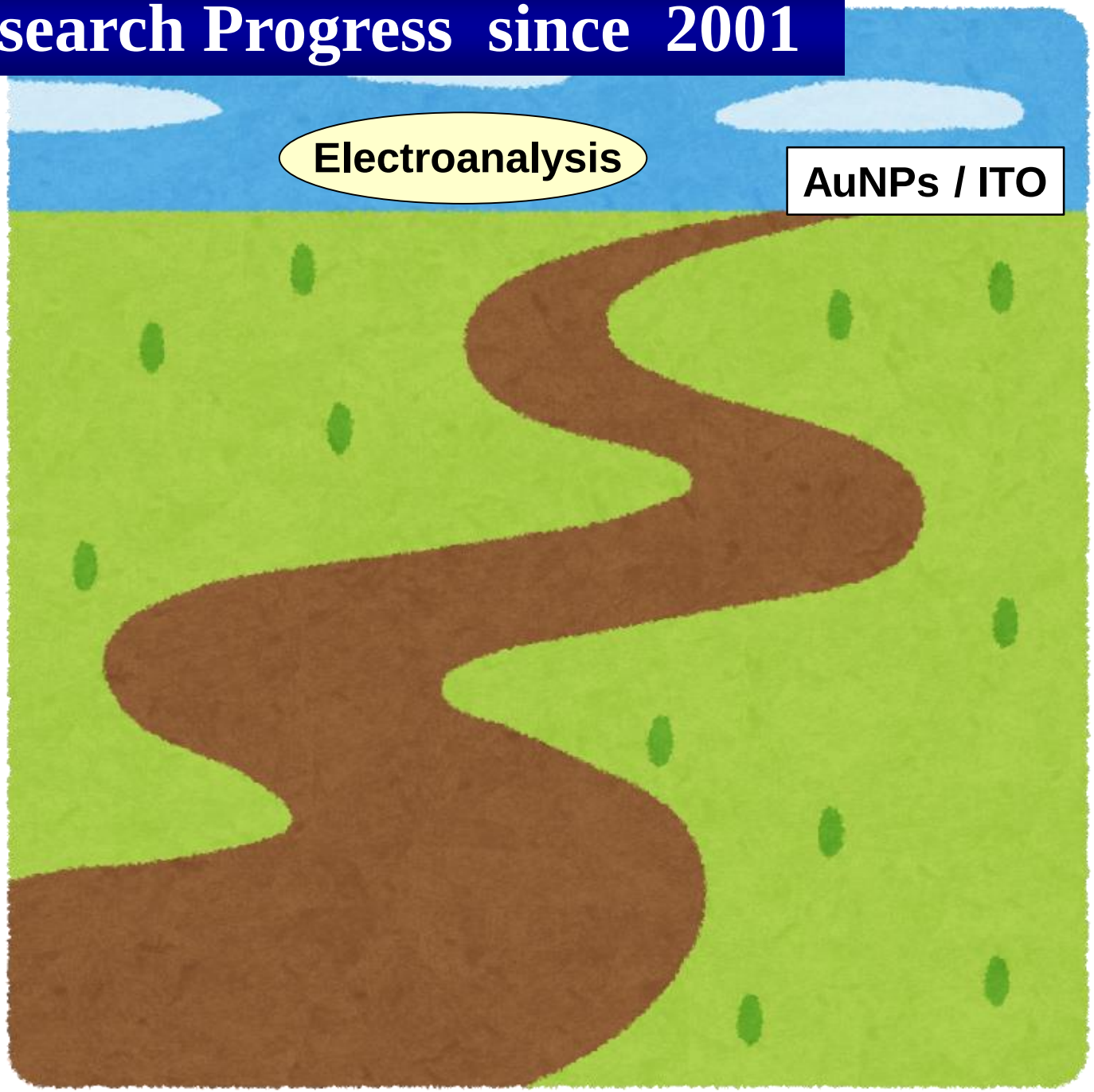
Metal Nanoparticles Modified Electrodes

Time Cited and Number of Papers

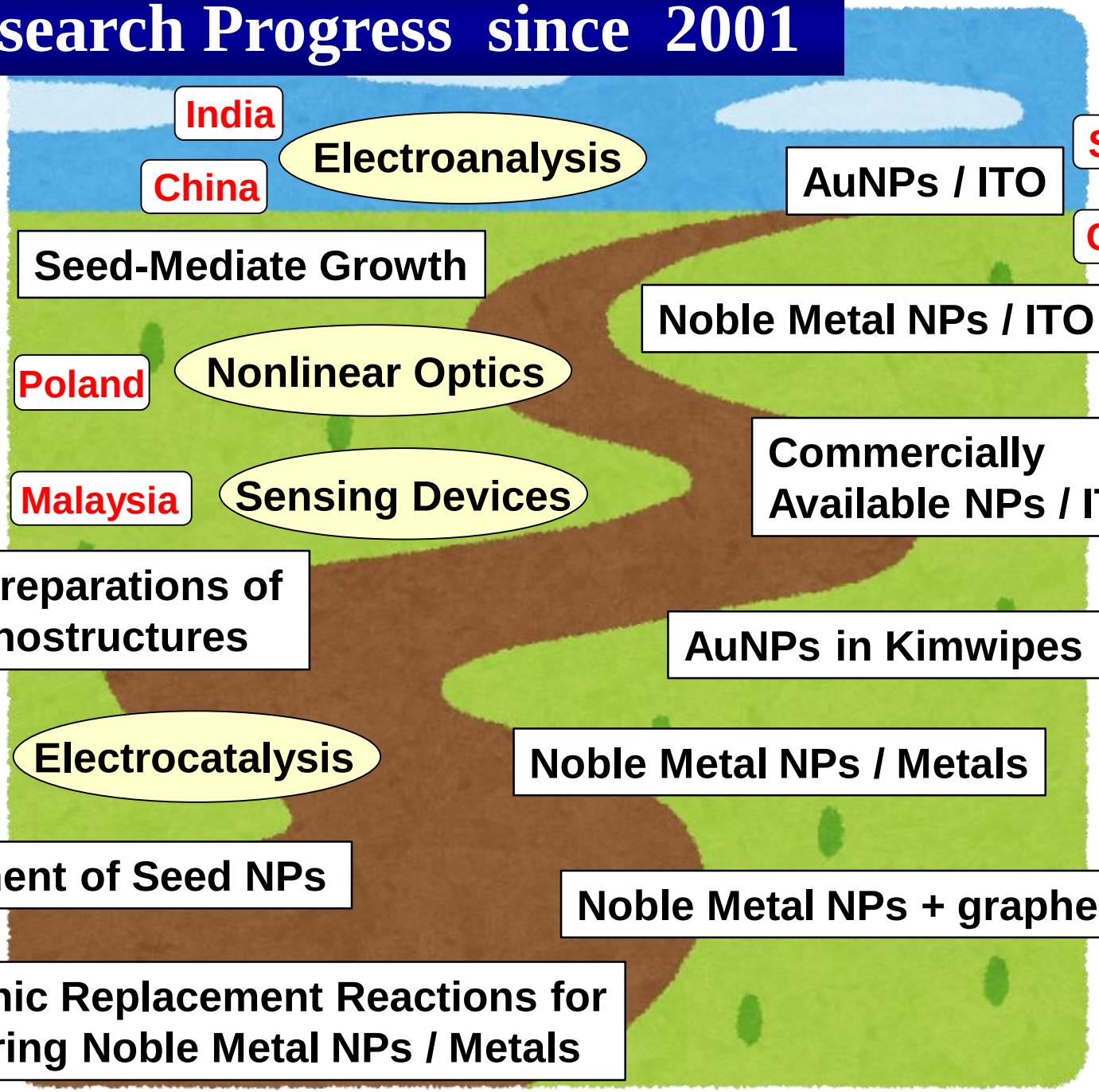


Graduate School of Engineering, Kyoto University

Our Research Progress since 2001



Our Research Progress since 2001



India

China

Electroanalysis

Saudi Arabia

AuNPs / ITO

China

Seed-Mediate Growth

Noble Metal NPs / ITO

Morocco

Poland

Nonlinear Optics

Commercially Available NPs / ITO

Poland

Malaysia

Sensing Devices

Chemical Preparations of Various Nanostructures

AuNPs in Kimwipes

UK

China

Electrocatalysis

Noble Metal NPs / Metals

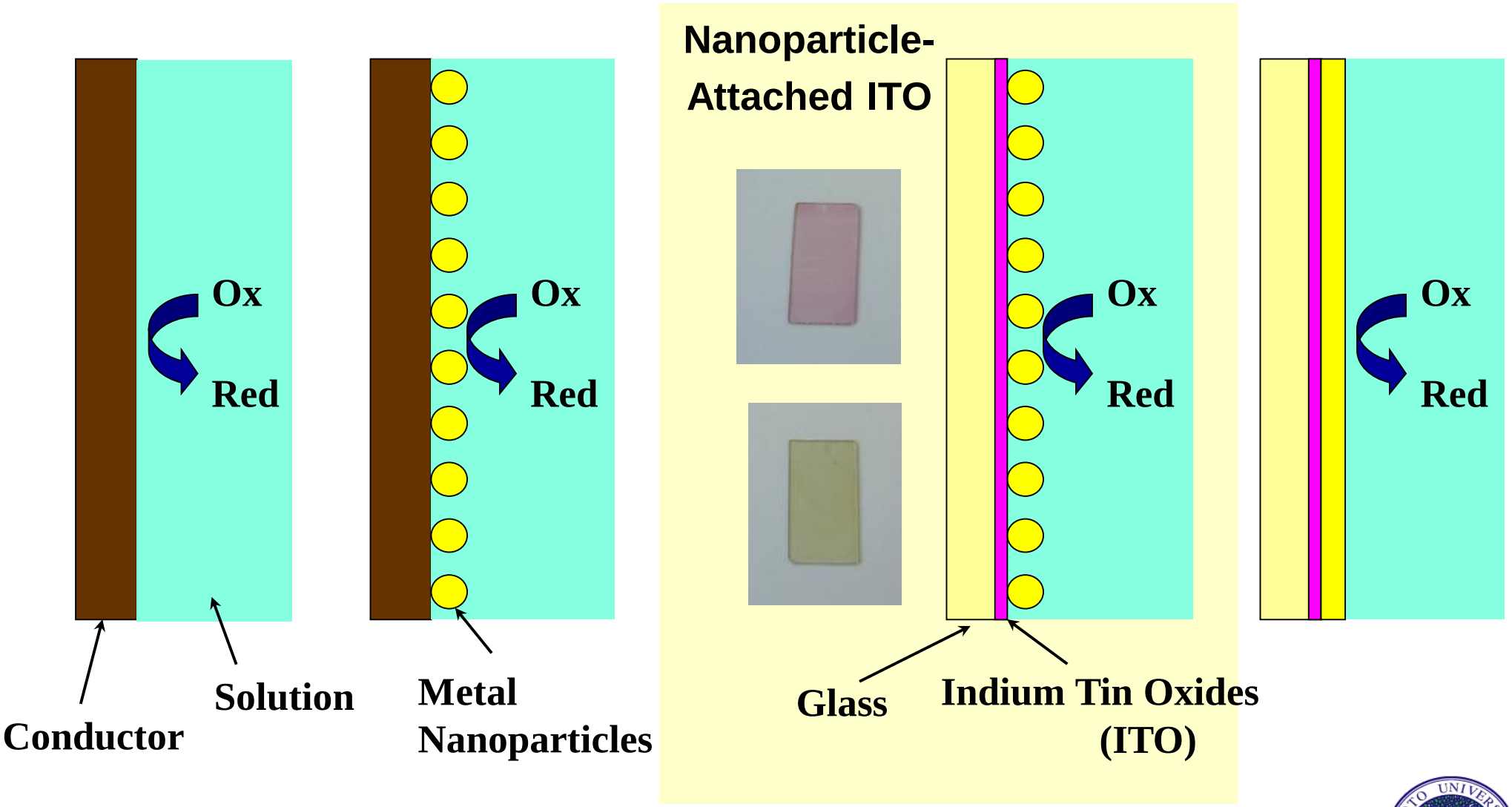
Attachment of Seed NPs

Noble Metal NPs + graphene

China

Galvanic Replacement Reactions for Preparing Noble Metal NPs / Metals

Metal NPs for Electrode Materials



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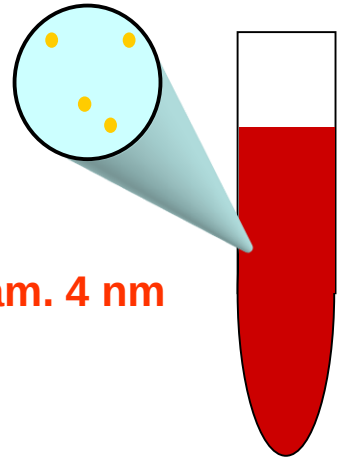
Original of Seed-Mediated Growth Method

Seed Solution

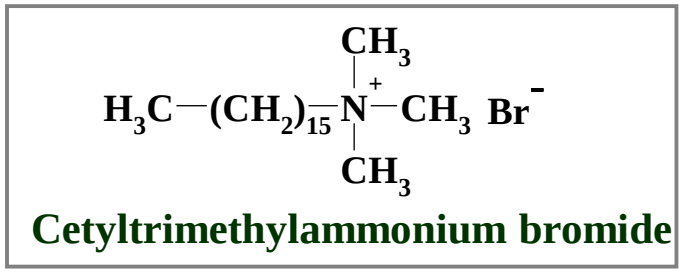
$HAuCl_4$
Sodium Citrate
$NaBH_4$

Au nanospheres, diam. 4 nm

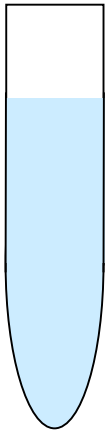


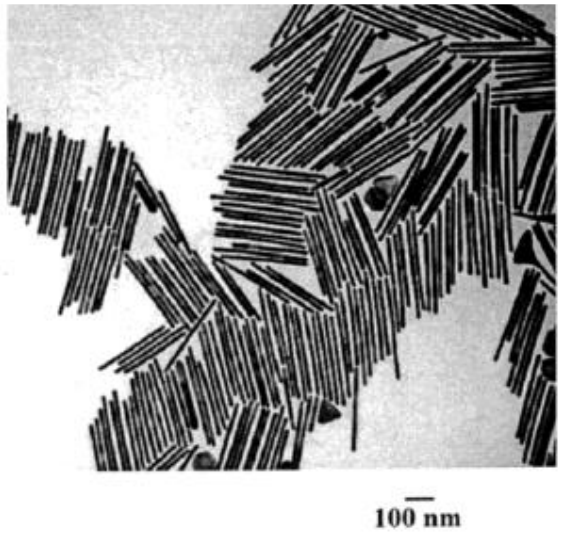
Growth Solution

Cetyltrimethylammonium bromide
$HAuCl_4$
Ascorbis Acid
NaOH



Cetyltrimethylammonium bromide





Murphy and coworkers,

J. Phys. Chem. B, **105**, 4065 (2001). *Adv. Mater.* **15**, 414 (2003).

Graduate School of Engineering, Kyoto University, Japan

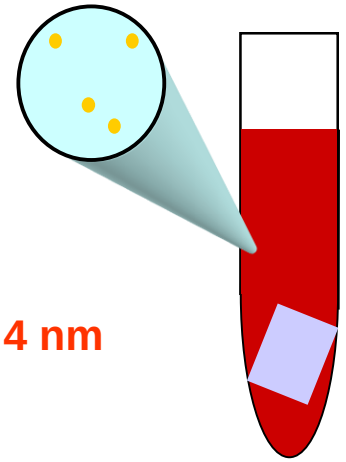
Our Seed-Mediated Growth Method

Substrate **Indium Tin Oxide (ITO)**

Seed Solution

$HAuCl_4$
Sodium Citrate
$NaBH_4$

Au nanospheres, diam. 4 nm

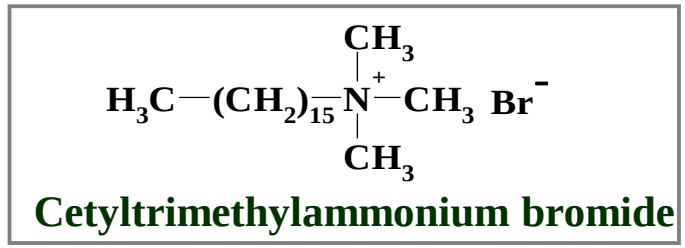


Attachment of Au Nano-seeds

2 hours

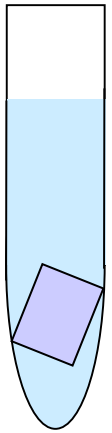
Growth Solution

Cetyltrimethylammonium bromide
$HAuCl_4$
Ascorbis Acid
NaOH



Cetyltrimethylammonium bromide

Two-Step Immersion



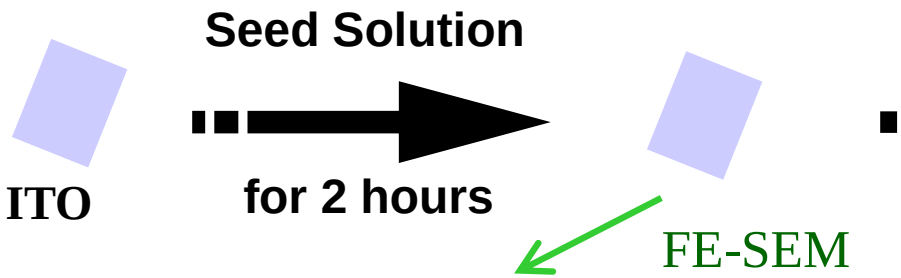
24 hours

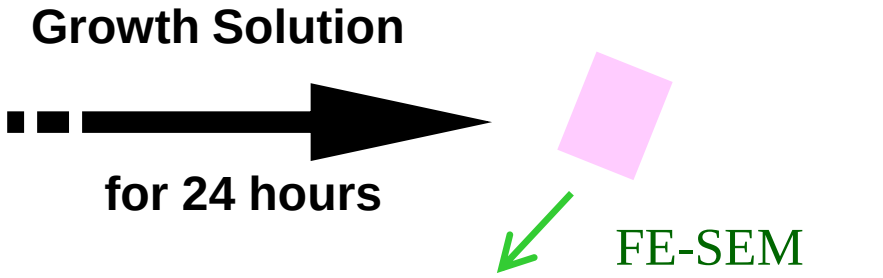
Crystal Growth via Chemical Reduction

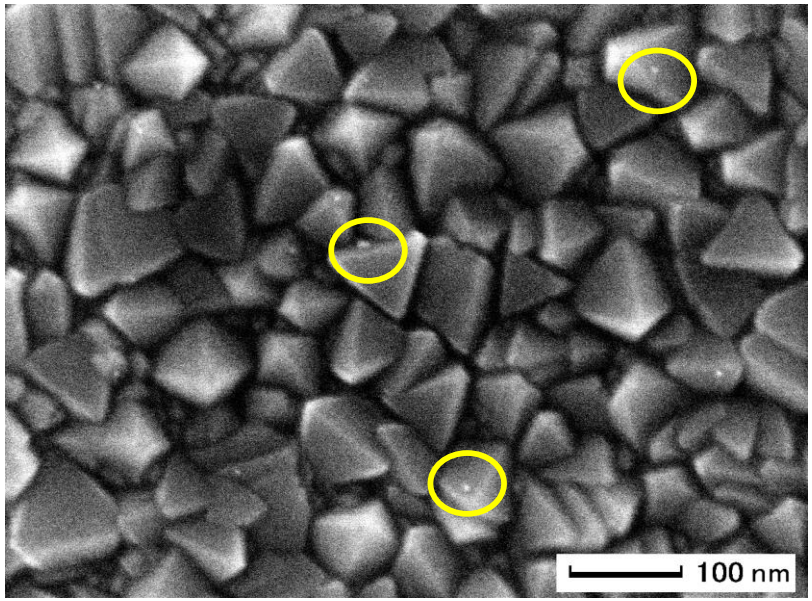
Kambayashi, Zhang, Oyama, *Cryst. Growth Des.*, **5**, 81 (2005).

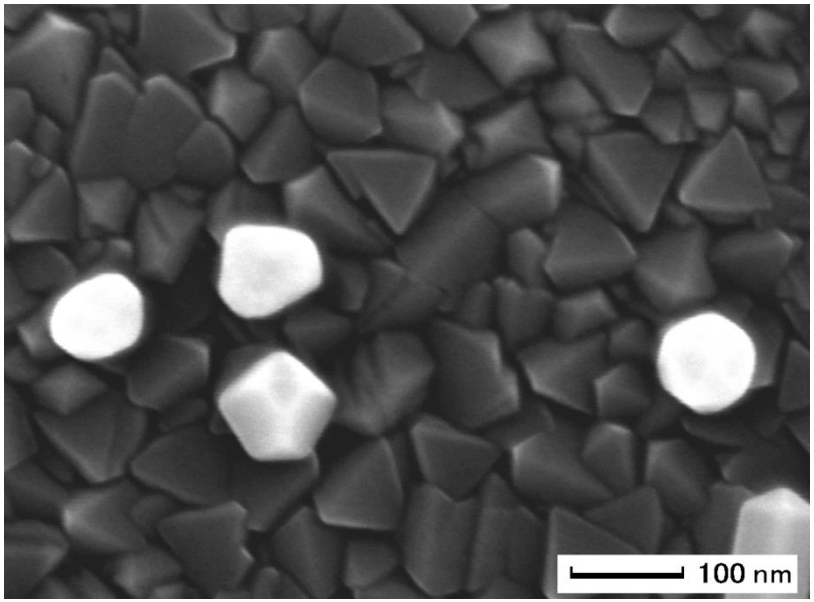
Graduate School of Engineering, Kyoto University, Japan

Attachment and Growth of AuNPs









Crystal growth of 60 - 80 nm of Au Nanoparticles

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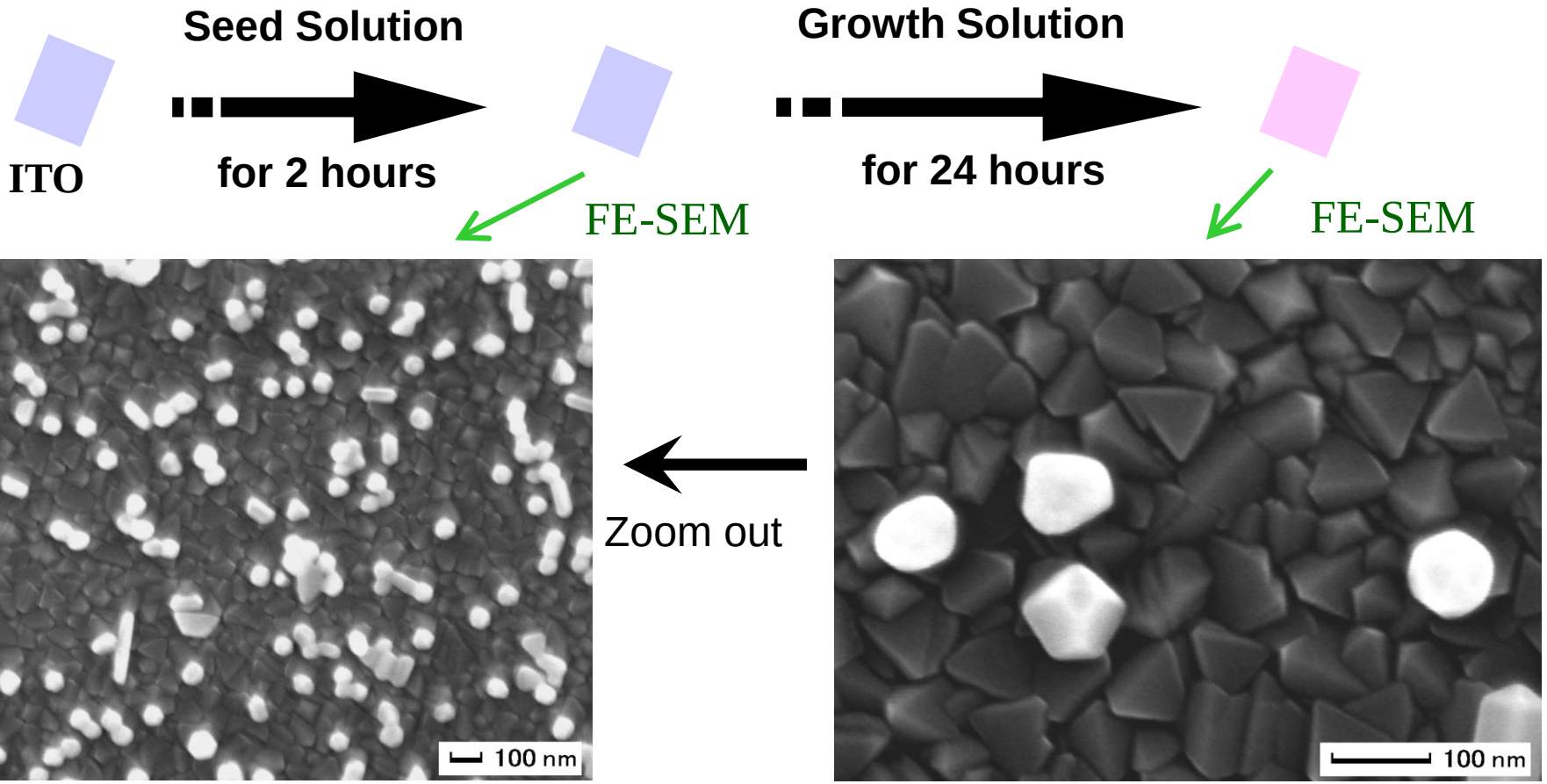
Attachment and Growth of AuNPs

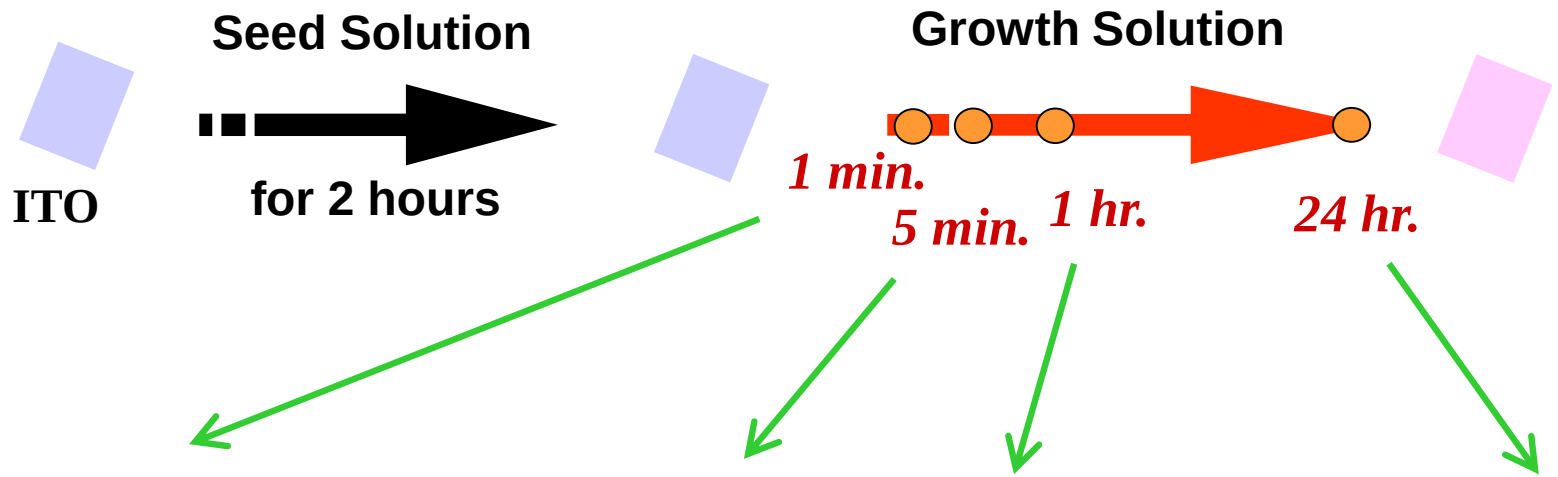


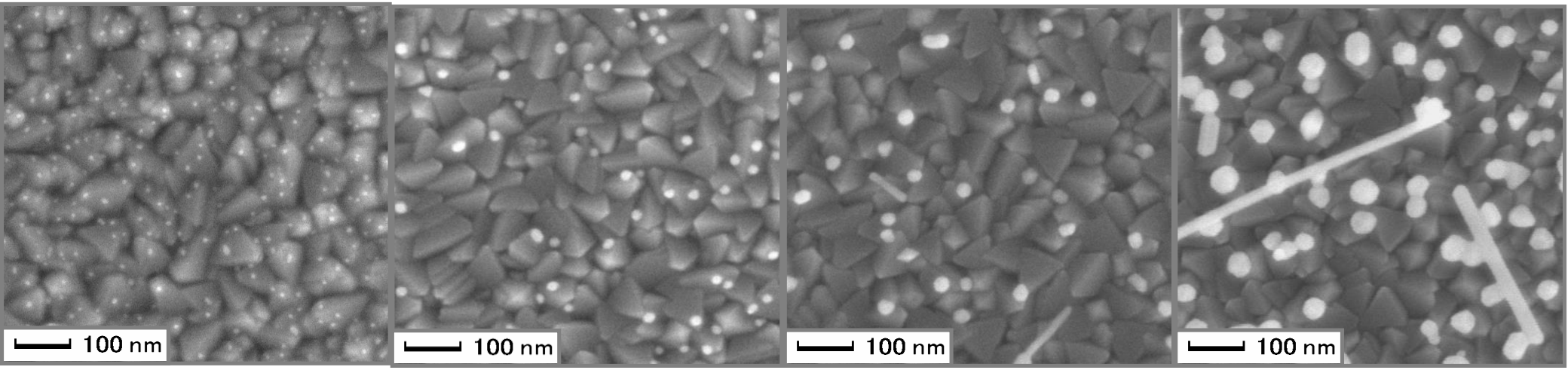
Attachment of Dispersed Au Nanoparticles

Crystal growth of 60 - 80 nm of Au Nanoparticles

Graduate School of Engineering, Kyoto University, Japan

Time Course of Growth of AuNPs





ca. 9 nm

ca. 15 nm

ca. 21 nm

ca. 50 nm

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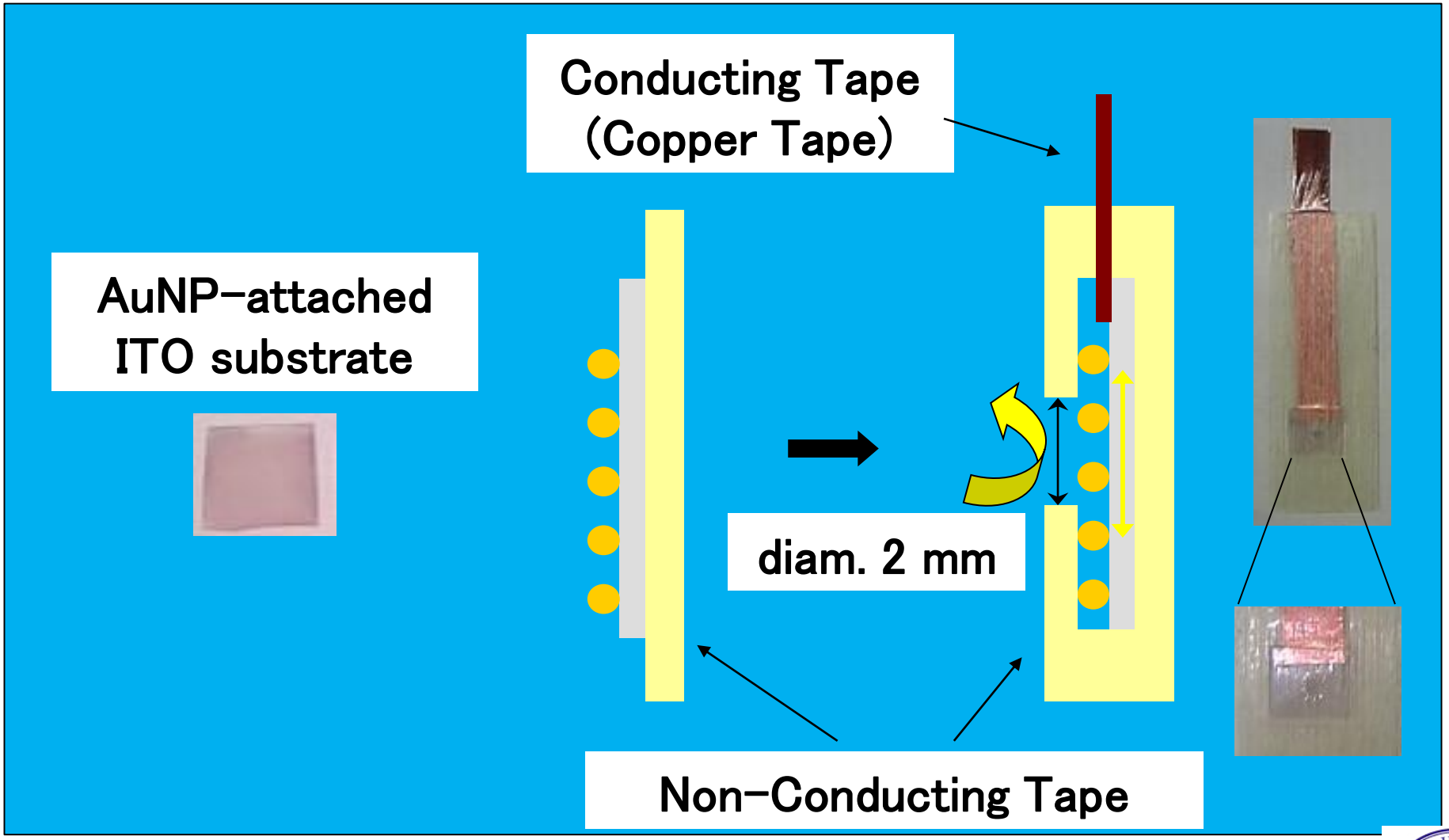
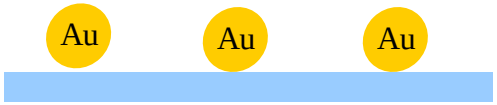
Fabrication of Electrodes

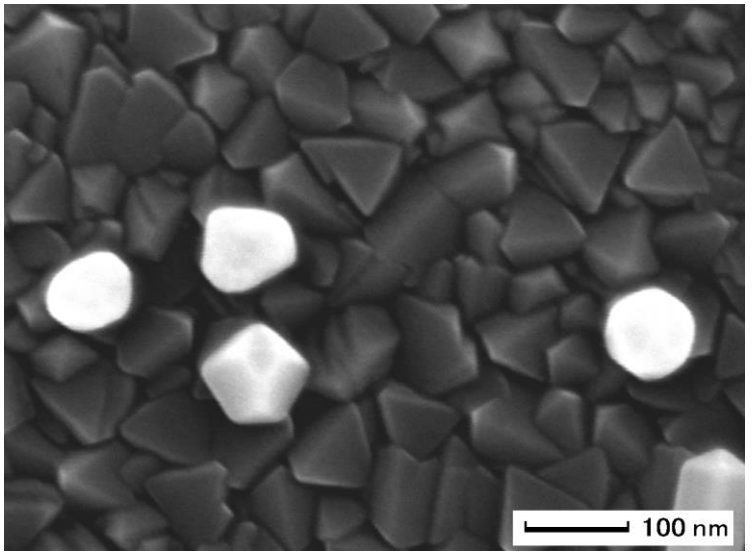


Surface Images of AuNPs

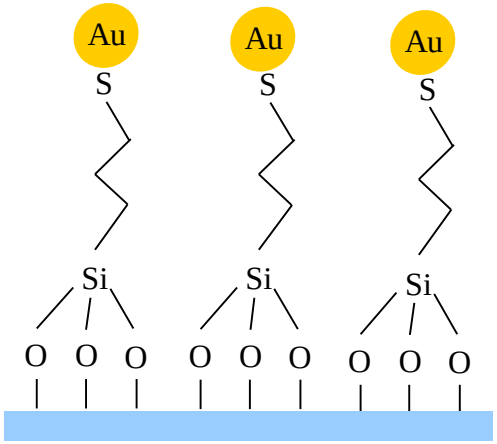


Growth Solution

for 24 hours

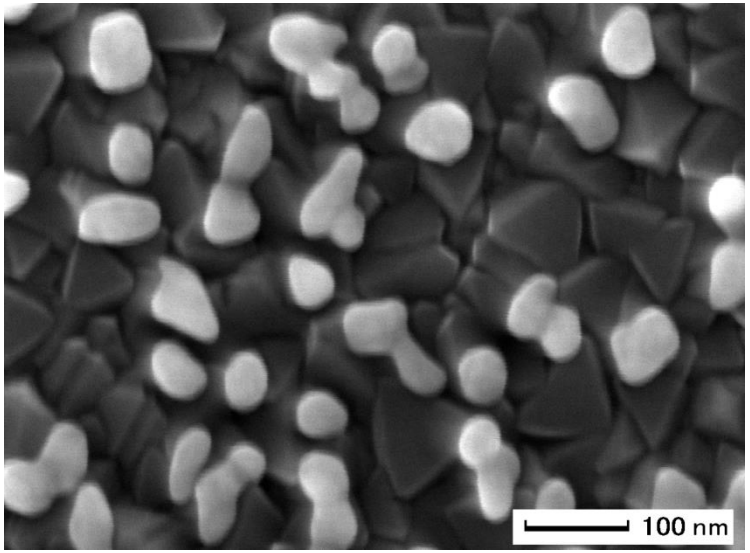


Crystal Growth



Growth Solution

for 24 hours



Amorphous

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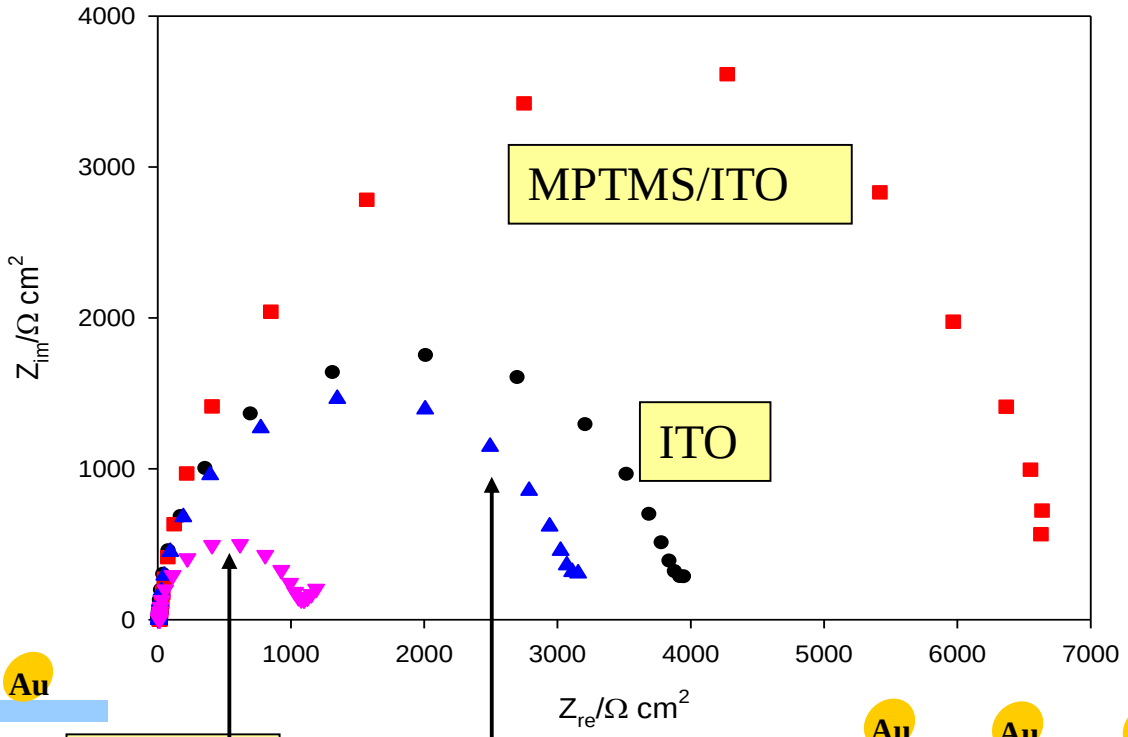
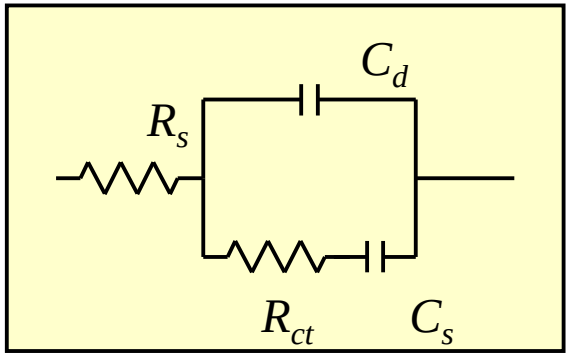
Electrochemical Impedance



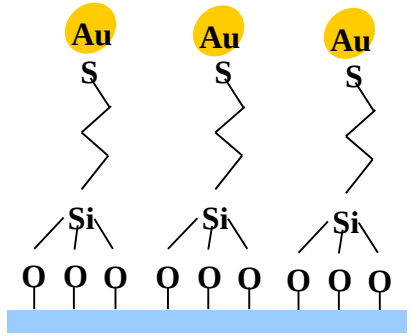


Au/ITO

R_{ct} = 1044 Ω cm^2

Au/MPTMS/ITO

R_{ct} = 3074 Ω cm^2



in 0.1 M PBS (pH7.4) containing 1 mM $Fe(CN)_6^{4-}$ and 1 mM $Fe(CN)_6^{3-}$.

Zhang, Kambayashi, Oyama, *Electrochem. Commun.*, **6**, 683 (2004).

Graduate School of Engineering, Kyoto University, Japan

Dr. Jingdong Zhang's Contributions

Oct. 2003 – Mar. 2005 in Kyoto

Dr. Jingdong Zhang
Professor
Huazhong University of Science and Technology
Wuhan, China

EIS, ascorbic acid, uric acid etc.

Electrochem. Commun., **6**, 683 (2004). **Times Cited: 104**

Electroanalysis, **17**, 408 (2005).

hemoglobin *Electrochim. Acta*, **50**, 85 (2004). **Times Cited: 118**

myoglobin *J. Electroanal. Chem.*, **577**, 273 (2005). **Times Cited: 97**

NO detection *Anal. Chim. Acta*, **540**, 299 (2005). **Times Cited: 82**

TiO_2-AuNPs *Electrochem. Solid-State Lett.*, **8**, E49 (2005).

Collaboration with Professor Goyal

Dr. Rajendra N. Goyal
Professor Emeritus
Indian Institute of Technology
Roorkee, India.

uric acid *Indian J. Chem. A*, **44,** 945 (2005).

paracetamol *Electrochem. Commun.*, **7**, 803 (2005). **Times Cited: 204**

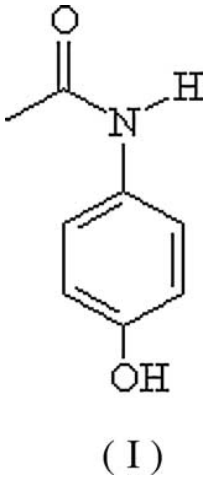
atenolol *Electrochem. Commun.*, **8**, 65 (2006). **Times Cited: 169**

nandrolone *Talanta*, **72**, 3140 (2007).

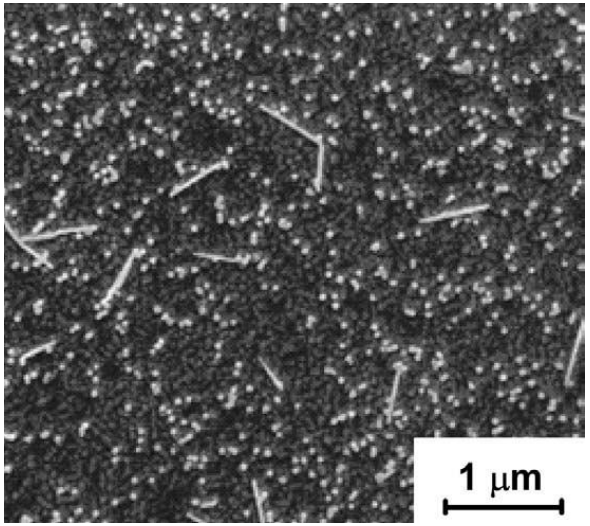
methylprednisolone acetate *J. Pharm. Biomed. Anal.*, **44**, 1147 (2007).

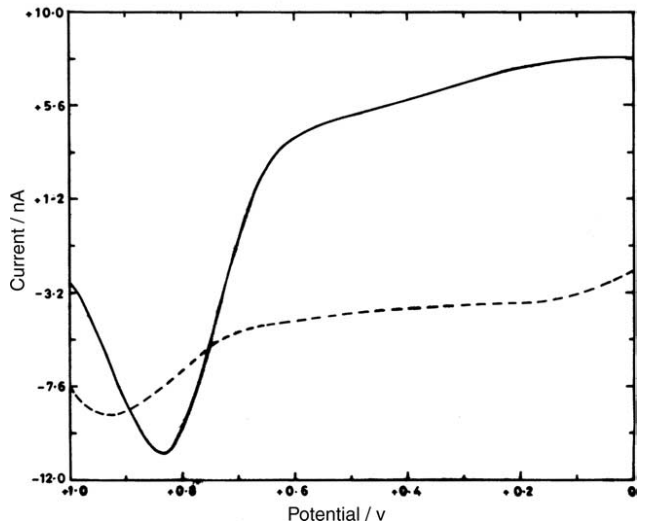
Collaboration with Professor Goyal

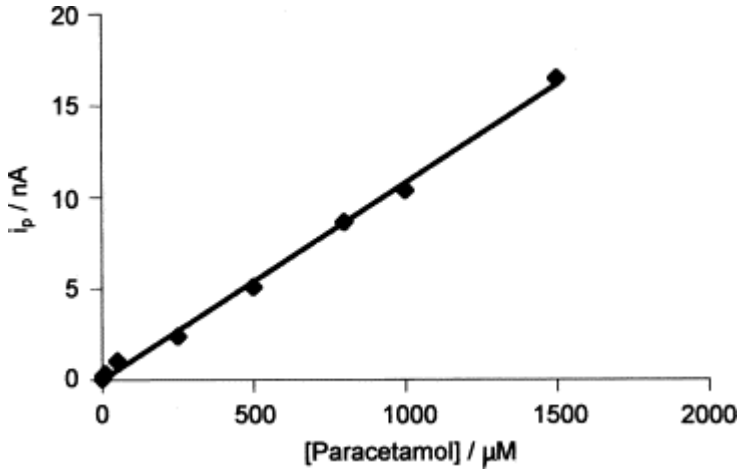
paracetamol



(I)







Goyal, Gupta, Oyama, Bachheti
Electrochem. Commun., **7**, 803 (2005).

Collaboration with Professor Goyal

guanosine and GTP *Anal. Chim. Acta*, **581**, 32 (2007).

adenosine and ATP *Electroanalysis*, **19**, 575(2007) .

adenosine and guanosine *Talanta*, **71**, 1110 (2007). **Times Cited: 157**

dopamine and serotonin *Talanta*, **72**, 976 (2007). **Times Cited: 196**

salbutamol *J. Electroanal. Chem.*, **611**, 140 (2007).

5-hydroxytryptamine and 5-hydroxyindole

Sens. Actuators B, **134**, 816 (2008). **Times Cited: 179**

prednisolone *Bioelectrochemistry*, **74**, 272 (2009).

dopamine and ascorbic acid *J. Electroanal. Chem.*, **631**, 58 (2009).

norepinephrine *Sens. Actuators B*, **153**, 232 (2011).

paracetamol and epinephirine *Anal. Chim. Acta*, **693**, 35 (2011).

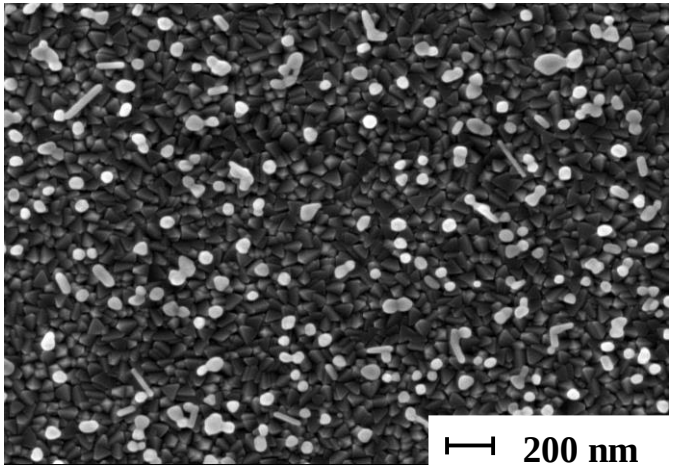
tryptophan *Talanta*, **85**, 2626 (2011).

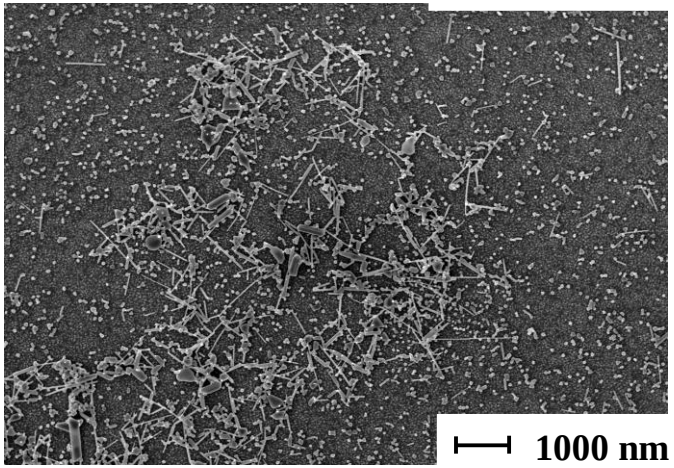
Dr. Chang Gang's Contributions

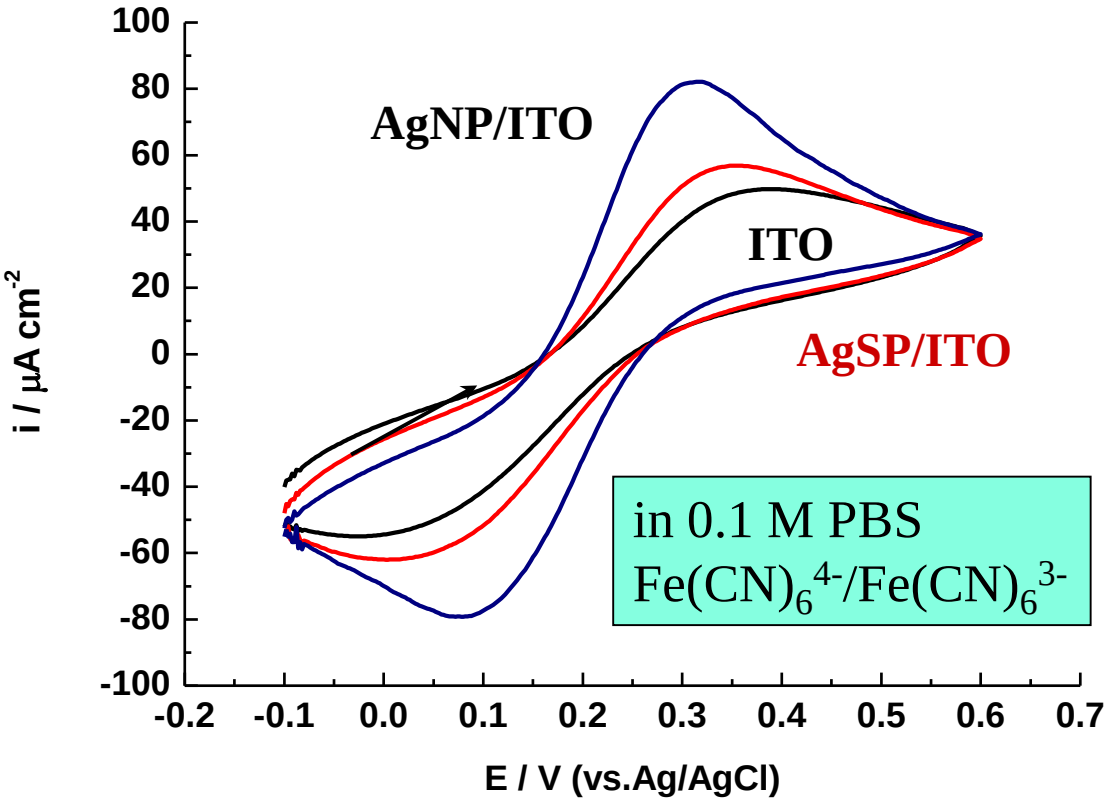
Dec. 2003 – Sep. 2006 in Kyoto

Dr. Gang Chang
Professor
Hubei University
Wuhan, China

Silver Nanoparticle-Attached ITO



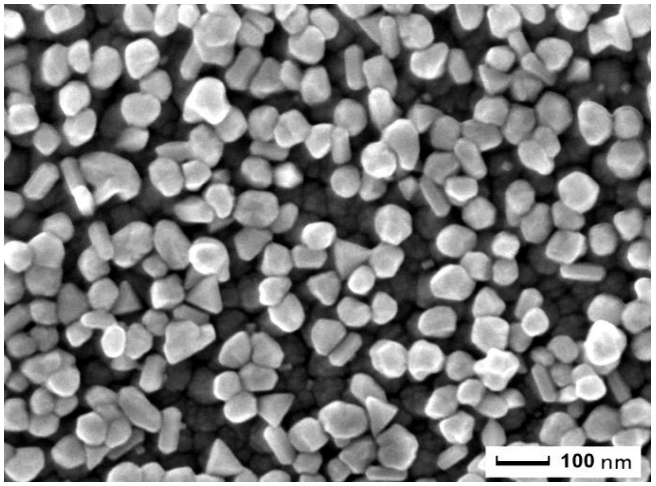


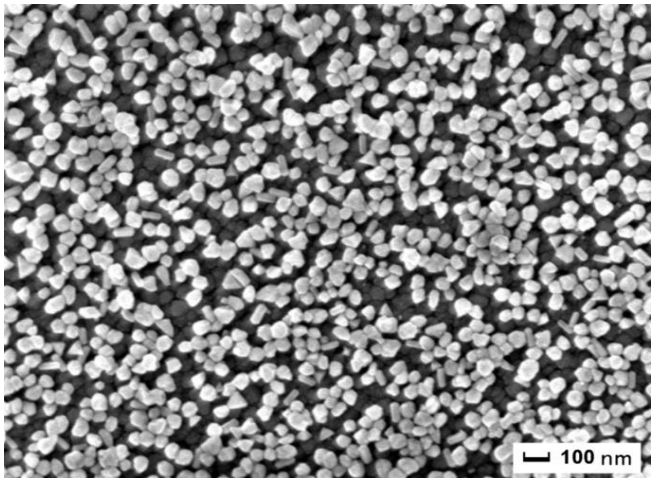


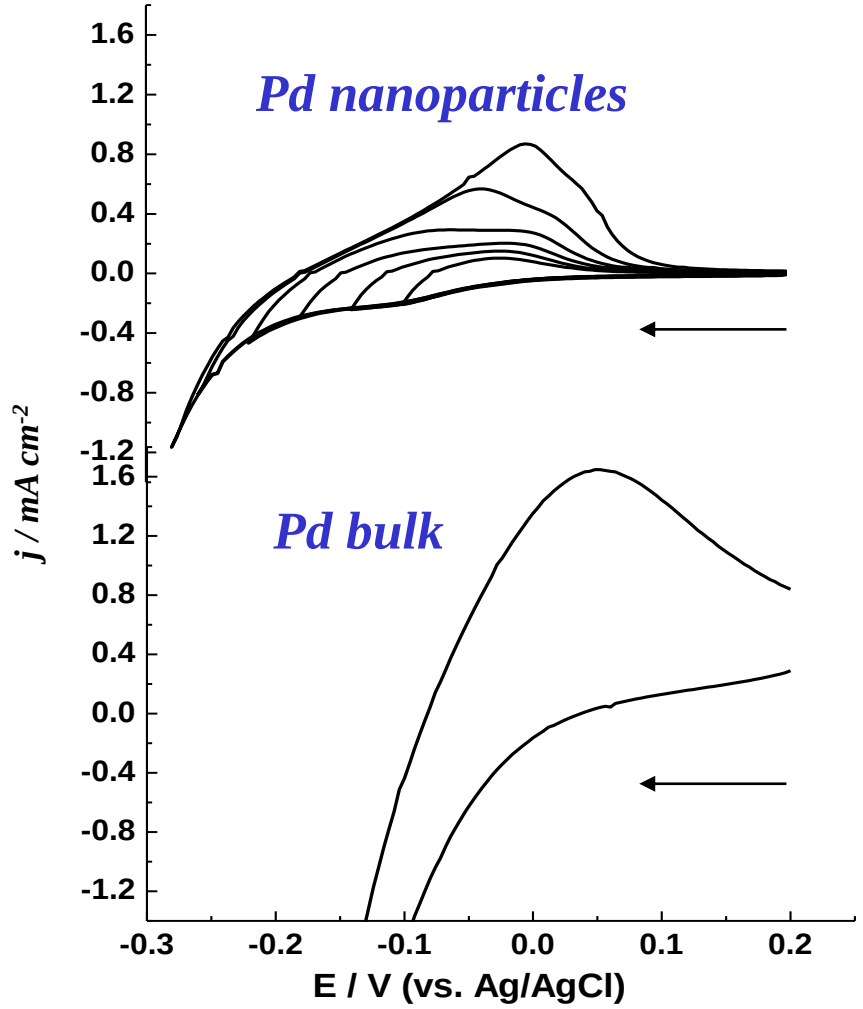
Chang, Oyama, Hirao, *J. Phys. Chem. B,* **109**, 1204 (2005).

Graduate School of Engineering, Kyoto University, Japan

Palladium Nanoparticle-Attached ITO



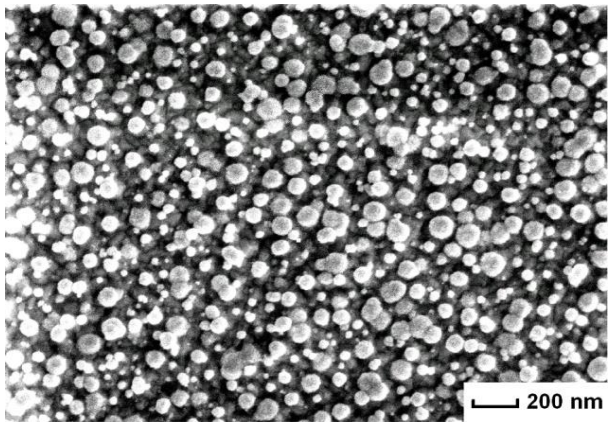


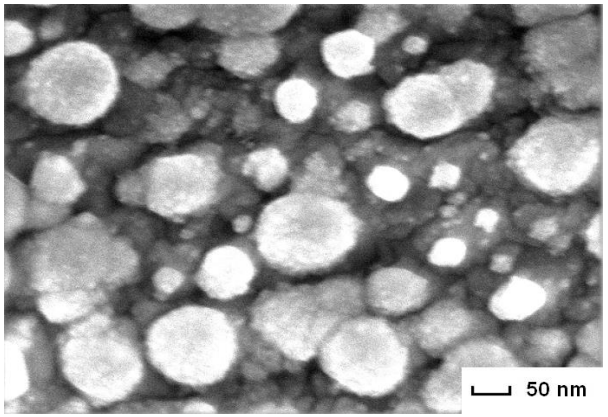


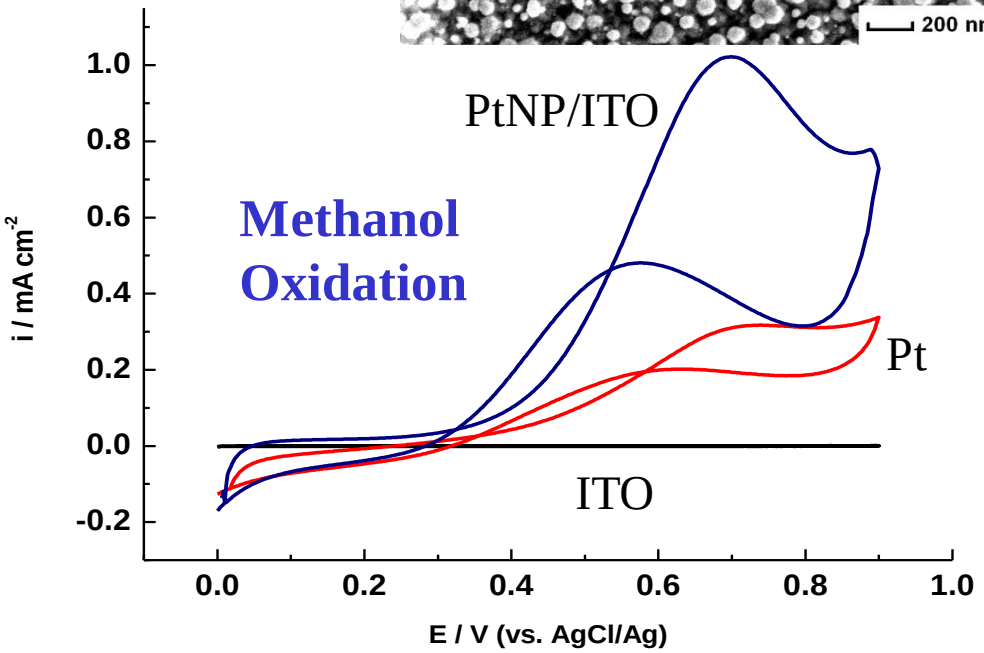
Chang, Oyama, Hirao, *J. Phys. Chem. B,* **110**, 20362 (2006).

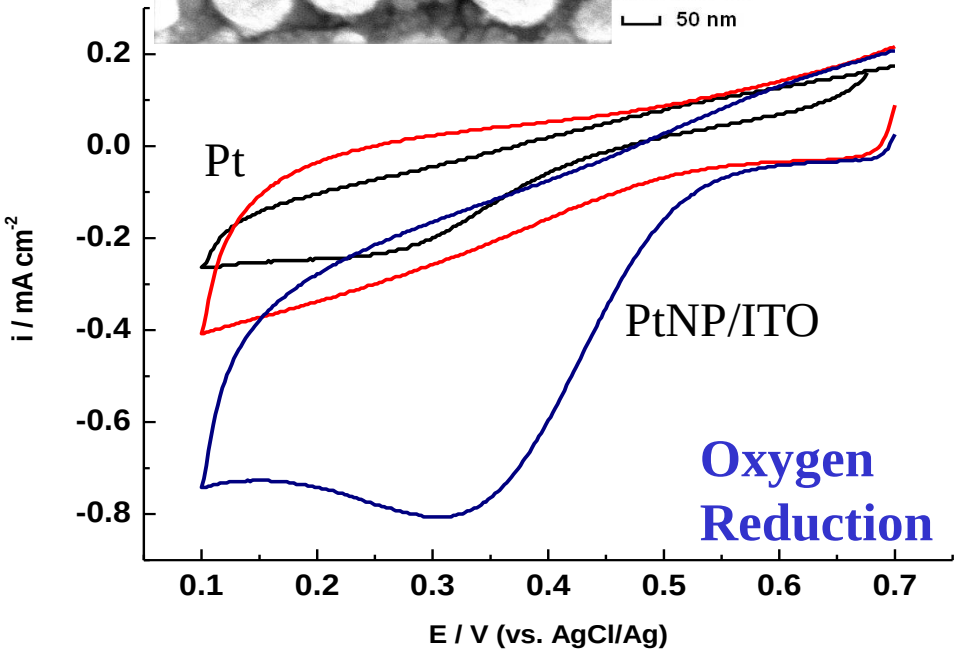
Graduate School of Engineering, Kyoto University, Japan

Platinum Nanoparticle-Attached ITO









Chang, Oyama, Hirao, *J. Phys. Chem. B,* **110**, 1860 (2006).

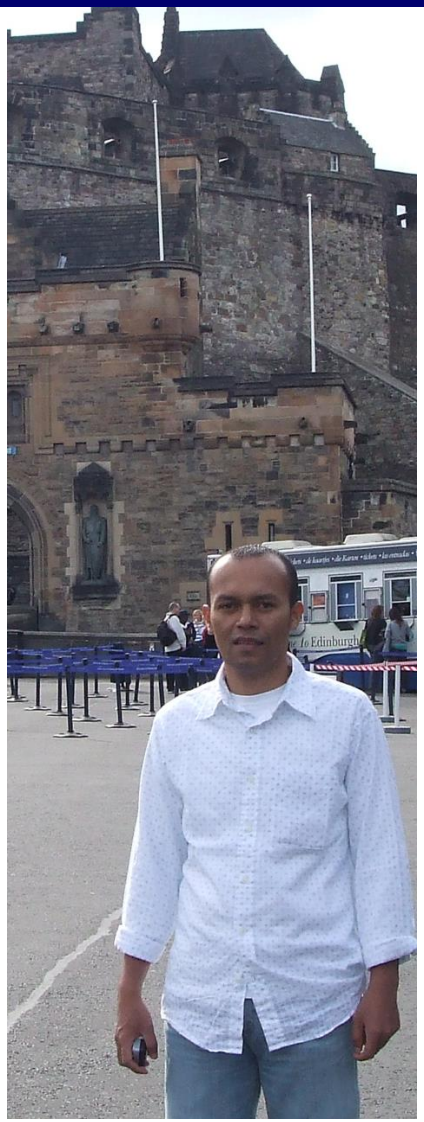
Graduate School of Engineering, Kyoto University, Japan

At Huazhong University of Science & Technology Wuhan China, May 2017

Prof. Gang Chang
Hubei University, Wuhan, China

Prof. Jingdong Zhang
Huazhong University of Science & Technology (HUST), Wuhan, China

Dr. Ali Umar's Contributions



May 2004 – Mar. 2007 in Kyoto

Dr. Akrajas Ali Umar

Associate Professor

Institute of Micro-Engineering and Nanoelectronics (IMEN)

Universiti Kebangsaan Malaysia (UKM)

Malaysia

Dr. Nouneh Khalid's Contributions

Sep. 2007 – May. 2009 in Kyoto

Dr. Khalid Nouneh

Assistant Professor

Universite Ibn Tofail

Morocco

Growth and optical features of AgNPs on ITO

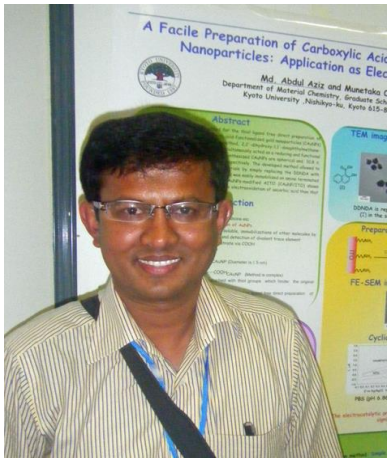
Journal of Alloys Compounds, **509**, 2631 (2011).

Synthesis and optical features of NiNPs on ITO

Journal of Alloys Compounds, **509**, 5882 (2011).

Dr. Md. Abdul Aziz's Contributions

Nov. 2009 – Oct. 2011 in Kyoto



Dr. Md Abdul Aziz
Research Scientist
King Faud University of Petroleum and Minerals
Saudi Arabia

Thermal-driven attachment of AuNPs on ITO
Journal of Nanoparticle Research, **15**, 1618 (2013).

Preparation monodispersed carboxylate-functioalized AuNPs
Gold Bulletin, **47**, 127 (2014).

Dr. Xiaomei Chen's Contributions

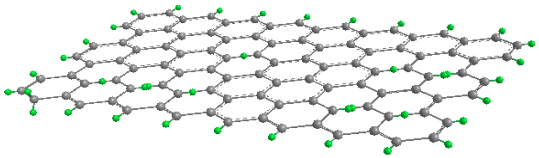
Nov. 2012 – Oct. 2014 in Kyoto

Dr. Xiaomei Chen

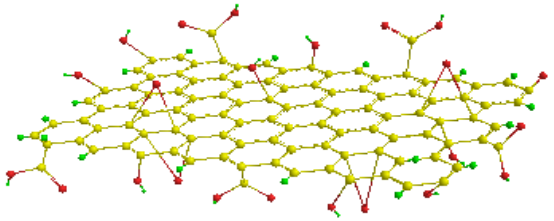
Professor

Jimei University

China

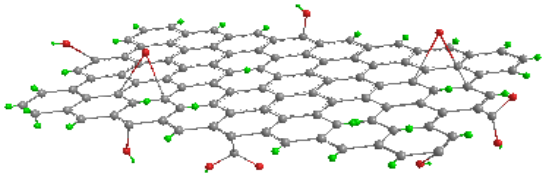


Graphene



GO

↓ **Reduction** + **Metal Ions**



RGO

Graduate School of Engineering, Kyoto University

Dr. Xiaomei Chen's Contributions

Nov. 2012 – Oct. 2014 in Kyoto

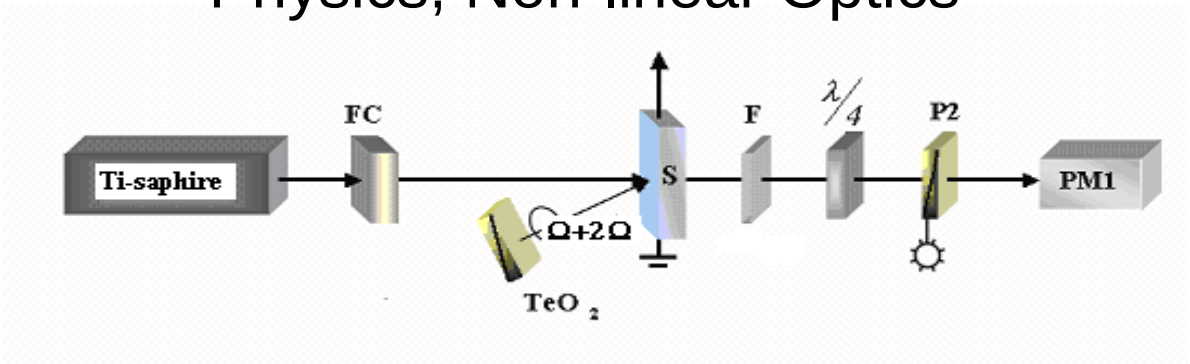
PdNPs+GO, H_2O_2	Electrochim. Acta, **97**, 398 (2013).
PtNPs+RGO, Oxalic Acid	Nanoscale, **5**, 5779 (2013).
PtPdNCs+RGO, MeOH	Carbon, **66**, 387 (2014).
PtPdNPs+RGO, EtOH	J. Mater. Chem. A, **2**, 315 (2014).
PtPdNCs+RGO, Glucose	Microchim. Acta, **181**, 783 (2014).
AuPdNPs+RGO, 4-nitrophenol	J. Mater. Chem. A, **2**, 5668 (2014).
AuNPs+RGO, 3,3,5,5-TMB	Dalton Trans., **43**, 7449 (2014).
PtPdNDs+RGO, 3,3,5,5-TMB	Sens. Actuators B, **201**, 286 (2014).
PdNPs+RGO, 4-nitrophenol	Chem. Lett., **43**, 919 (2014).

Graduate School of Engineering, Kyoto University

Collaboration with Professor Kityk

Prof. Ivan Kityk
Czestochowa University
of Technology,
Poland

Physics, Non-linear Optics



Graduate School of Engineering, Kyoto University

Collaboration with Professor Kityk

Circularly polarized light-induced electrogyration
Physica E, **2005**, 27**,** 420.

Acoustical circularly polarized gyration
Physica E, **2005,** 28**,** 178.

Nonlinear optical properties
Nanotechnology, **2005**, 16, 1687.

AuNPs

Control of the plasmon absorption with a two-color excitation
Journal of Applied Physics, **2005,** 98**,** 084304.

Circular acoustogyration effect
Applied Optics, **2005**, 44, 6905.

Kinetics of photoinduced changes
Philosophical Magazine Letters, **2005**, 85, 549.

Non-linear optical properties
Physica E, **2006**, 31, 38.

AgNPs

Collaboration with Professor Kityk

Luminescence of erbium doped AgNPs

Applied Surface Science, **253**, 1626 (2006).

Acoustically induced nonlinear optics of PdNPs

Physica E, **35**, 121 (2006).

Second order optical effects of PdNPs

Journal of Modern Optics, **55**, 187 (2008).

Nonlinear optical properties of NiNPs

Optical & Laser Technology, **40**, 499 (2008).

Nonlinear optical properties of AuNPs on ZnO

Nanotechnology, **19**, 18709 (2008).

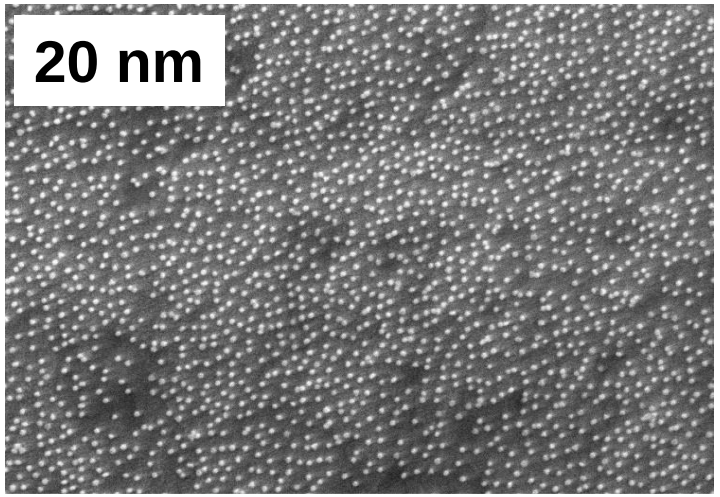
Journal of Nano Research, **2**, 31 (2008).

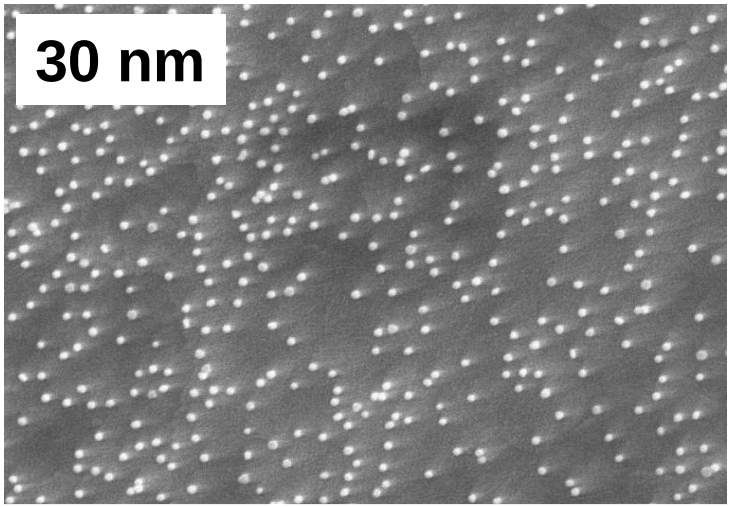
Laser treatment of AgNPs

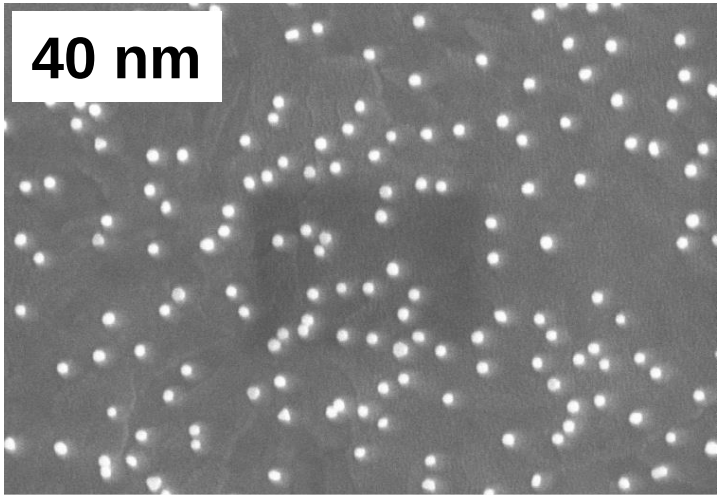
Materrals Chemistry and Physics, **113,** 187 (2009).

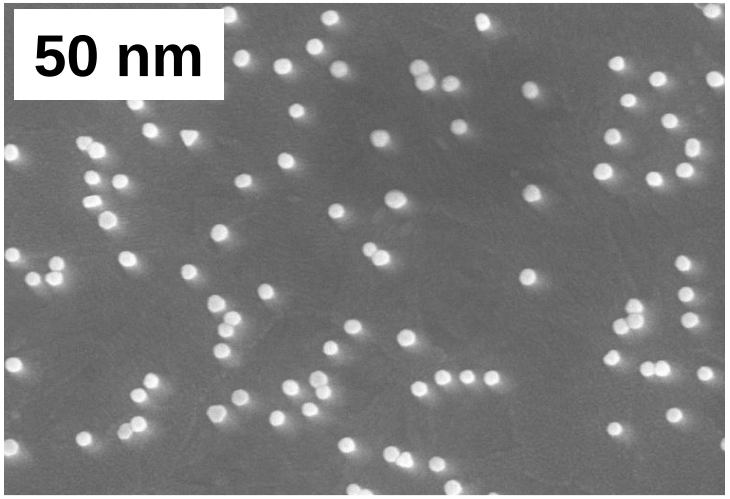
Superlattices and Microstructures, **46**, 637 (2009).

Collaboration with Professor Kityk









Collaboration with Professor Kityk

Optical features of AuNPs on ITO

Optical Communications, **284**, 245 (2011).

Photoinduced absorption of AgNPs on ITO

Journal of Alloys Compounds, **509S**, S424 (2011).

Picosecond laser treatment of AgNPs on ITO

Journal of Alloys Compounds, **509**, 9663 (2011).

Pump-probe third harmonic generation of AuNPs on ITO

Materials Letters, **74,** 226 (2012).

Laser stimulated optical features of AuNPs on ITO

Physica E, **44**, 1182 (2012).

Collaboration with Professor Kityk

Laser operated optoelectronic devices of AuNPs on FTO
J. Mater. Sci: Materials in Electronics, **24**, 2422 (2013).

Second harmonic generation in LiB_3O_5 in AgNP/ITO
J. Mater. Sci: Materials in Electronics, **24**, 4204 (2013).

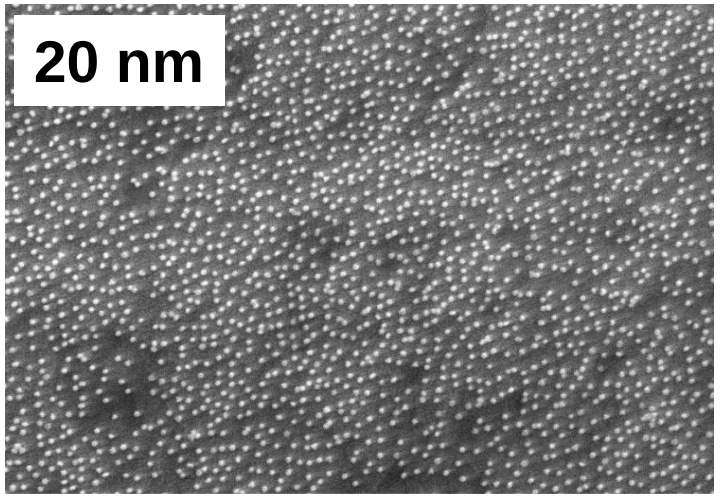
AuNPs aggregation in laser induced anisotropy of ITO
Journal of Alloys Compounds, **585**, 393 (2014).

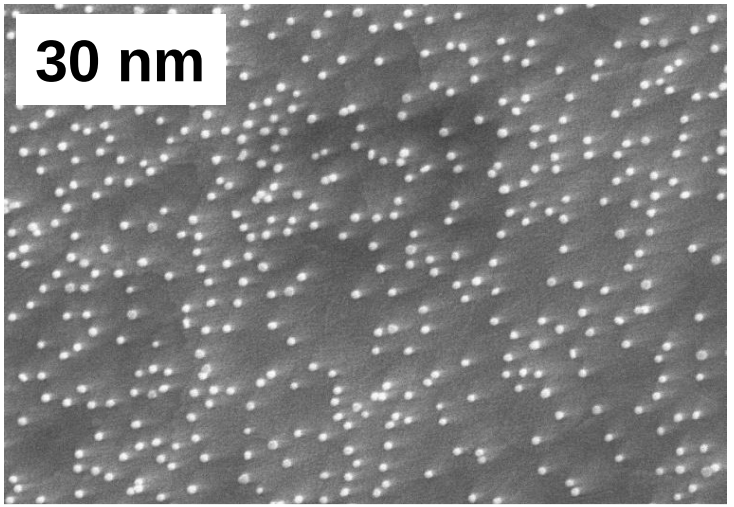
Light reflection reduction of AgNPs attached Al-doped ZnO
Physica E, **56**, 283 (2014).

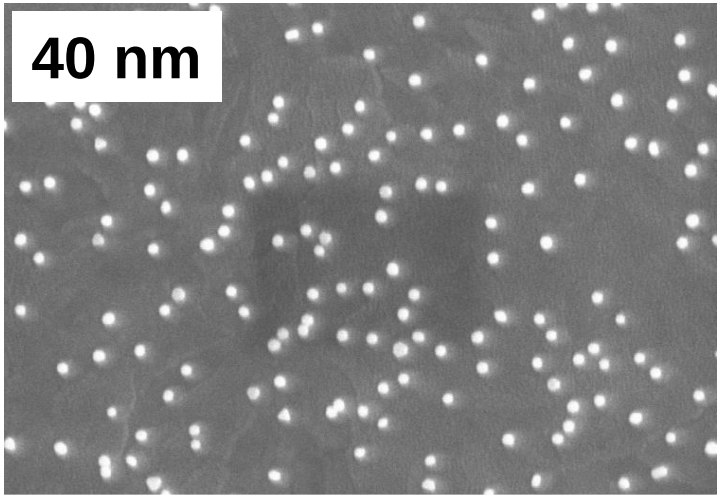
Nonlinear optical features of BiB_3O_6/PVA on Al-doped ZnO
Physica E, **64**, 1 (2014).

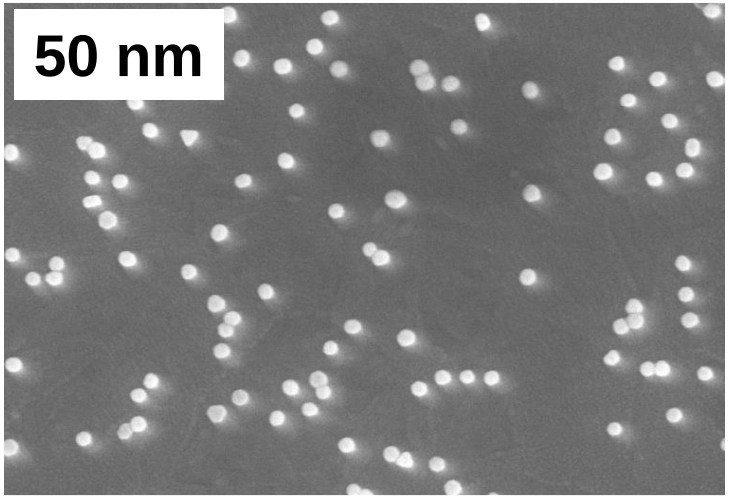
Third harmonic generation of AuNPs on Al-doped and Ga-doped ZnO
Physica E, **71**, 91 (2015).

AuNPs/ITO prepared by Dr. Aziz



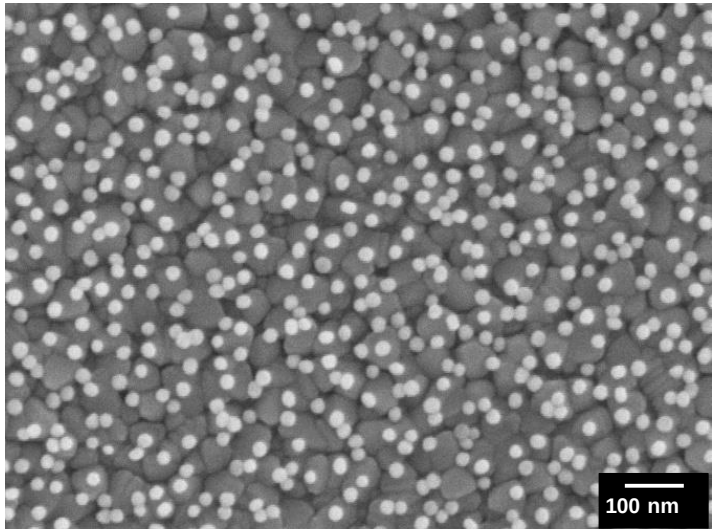


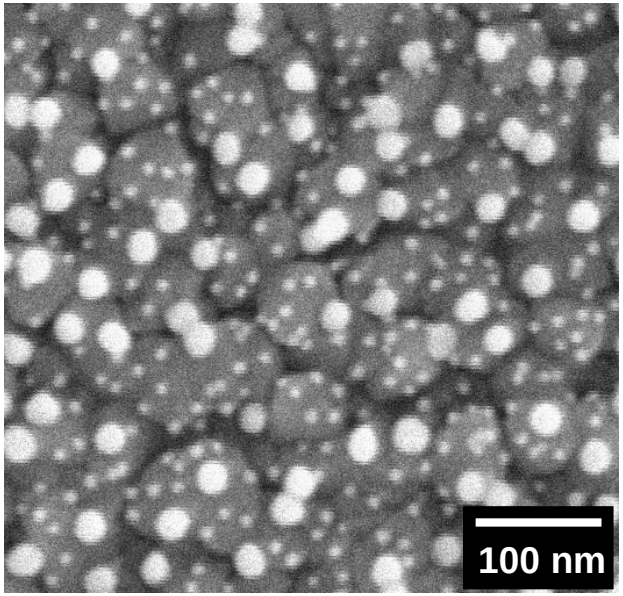




AuNP-Modified ITO Electrodes

5, 10, 20 nm AuNPs + APTMS + ITO





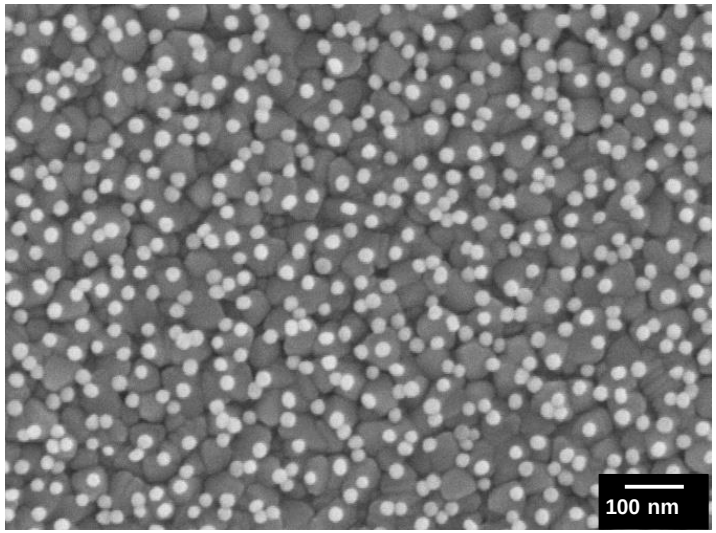
Competitive attachment of commercially available AuNPs

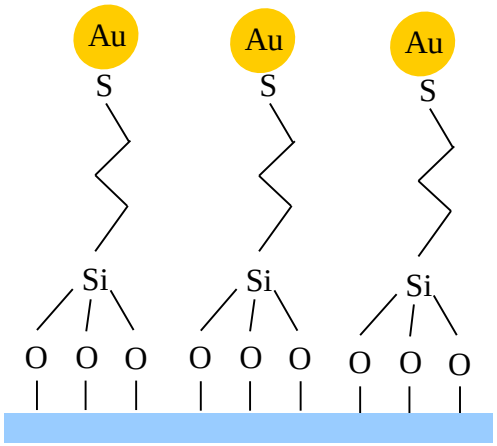
Oyama, Fujita, *Anal. Sci.*, **31**, 597 (2015).

Graduate School of Engineering, Kyoto University, Japan

AuNP-Modified ITO Electrodes

5, 10, 20 nm AuNPs + APTMS + ITO





Competitive attachment of commercially available AuNPs

Oyama, Fujita, *Anal. Sci.*, **31**, 597 (2015).

Graduate School of Engineering, Kyoto University, Japan

Failures in Modifications

Gold nanoparticles (AuNPs)

on indium doped tin oxides (ITO)

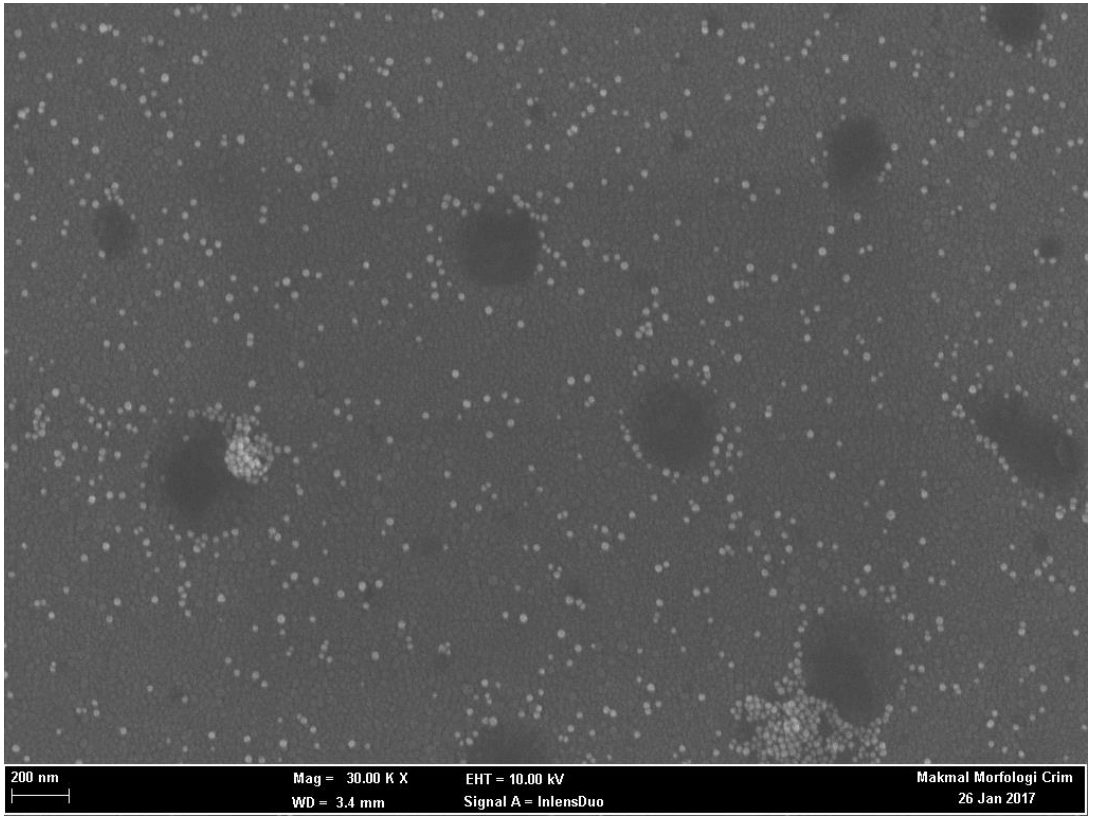
Progress since Oct. 2016

Failure of the preparations

Lower density attachment

Strange islands of linker molecules

Gathering



Progress since Oct. 2016

Jan. 2017 - Jun. 2017

As struggles of trials,
the effects of the kinds of ITO substrates and
the concentrations of linker molecules, APTMS.

Jul. 2017

As a final trial, we tried to change the source of AuNPs.

Gold Colloid Solutions

Previous ***Sigma***

tannic acid capped (5, 10, 20 nm)

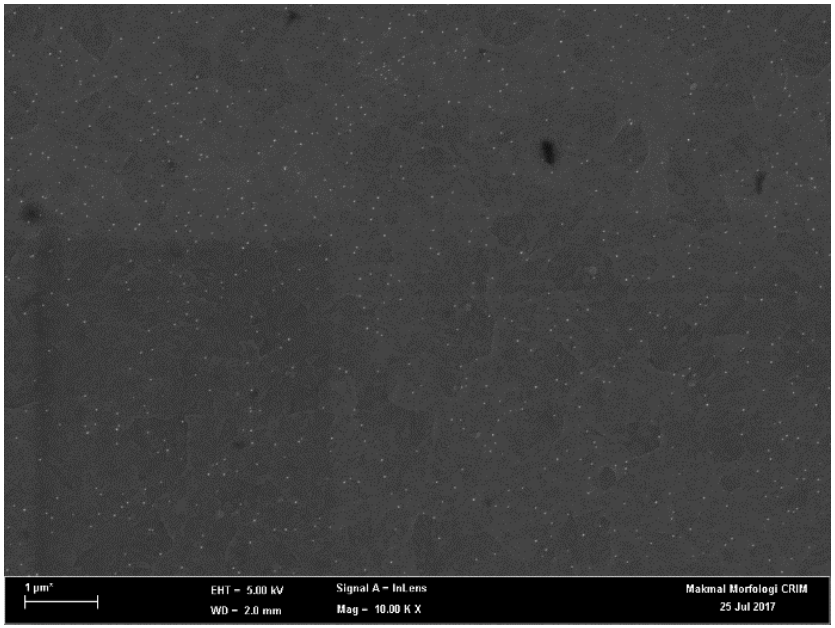
Present ***Sigma-Aldrich*****,** but actually ***Cytodiagnostics*** *(Canada)*

stabilized suspension in citrate buffer
(5, 10, 15, 20, 30, 40, 50, 60, 80, 100 nm)

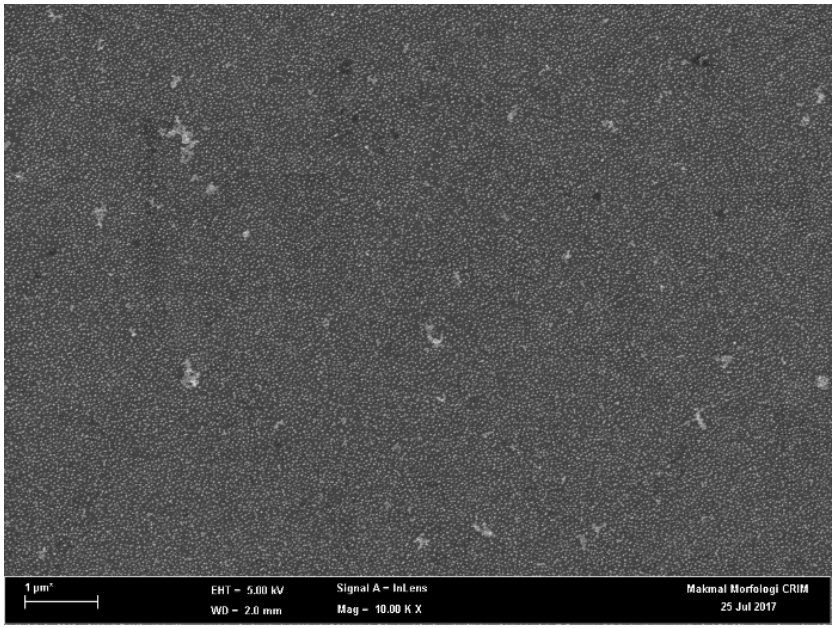
stabilized suspension in 0.1 mM PBS, reactant free
(5, 10, 15, 20, 30, 40, 50, 60, 80, 100, 150, 200,
250, 300, 400 nm)

AuNP-Modified ITO Electrodes

Jul. 2017



stabilized suspension in citrate buffer



stabilized suspension in 0.1 mM PBS, reactant free

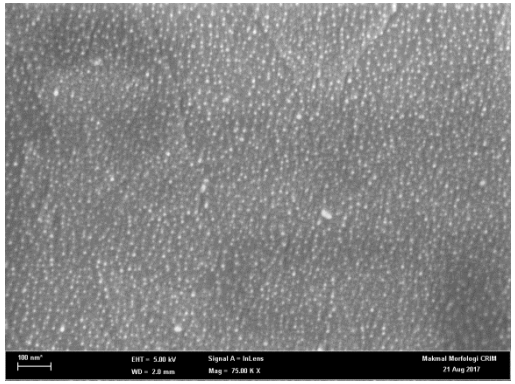
Graduate School of Engineering, Kyoto University, Japan

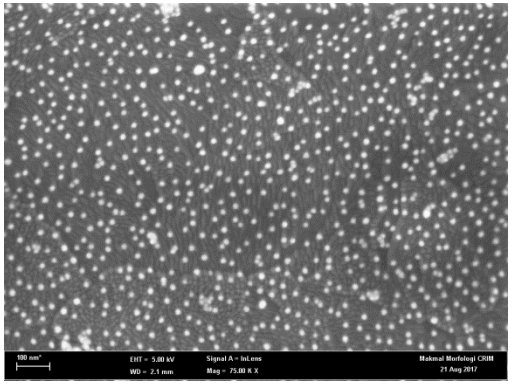
AuNP-Modified ITO Electrodes

Sep. 2017

4% APTMS

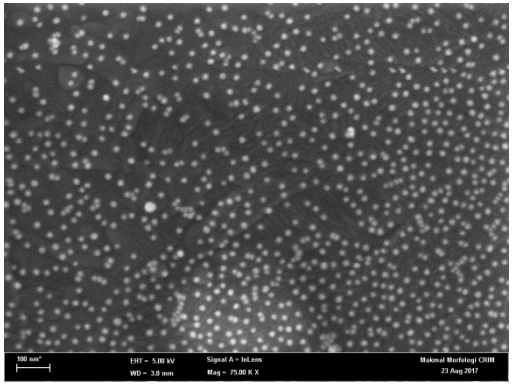
10 nm

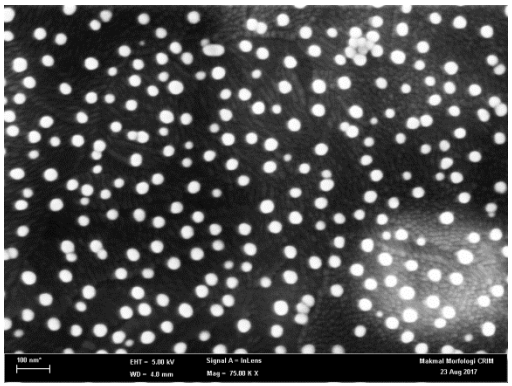




15 nm

20 nm



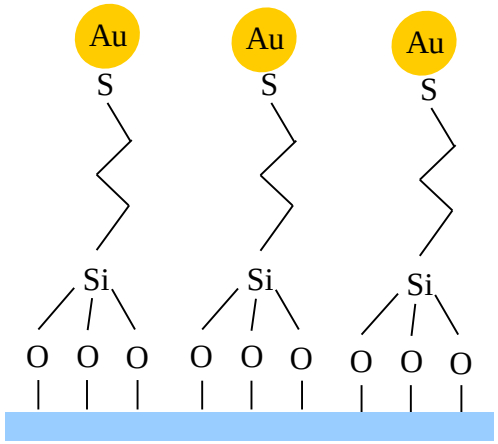


30 nm

AuNPs: stabilized suspension in 0.1 mM PBS, reactant free

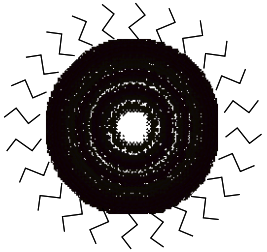
Graduate School of Engineering, Kyoto University, Japan

An Important Point for Modification



polymers

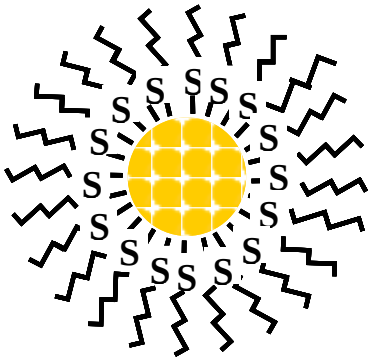
small molecules



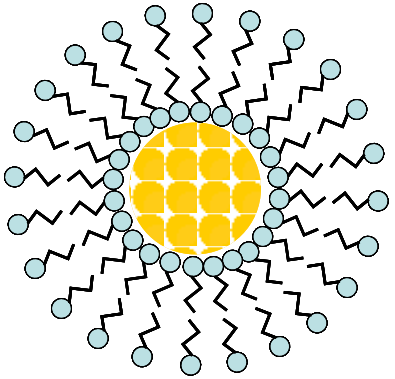
citrate ions

Frens, *Nature*, **1973**, *241*, 20.

thiols



surfactants



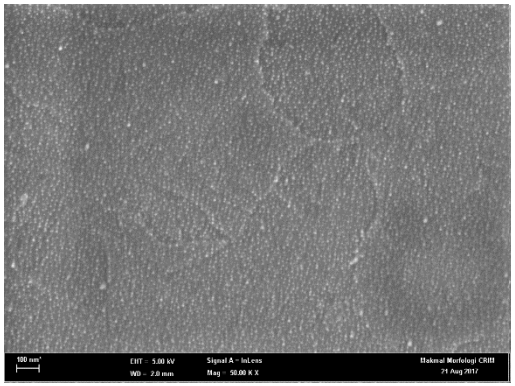
Brust et al., *JCS Chem. Commun.*, **1994**, 801.

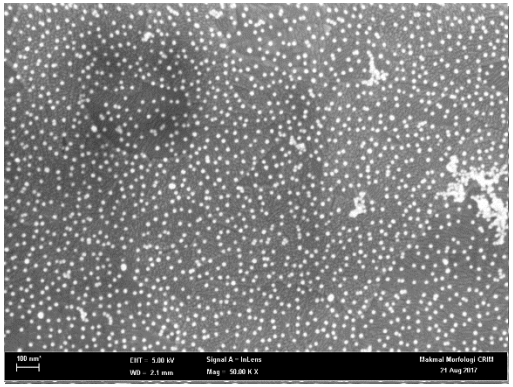
AuNP-Modified ITO Electrodes

Oct. 2017

4% APTMS

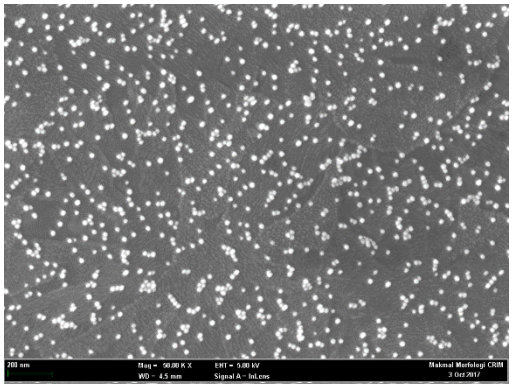
10 nm

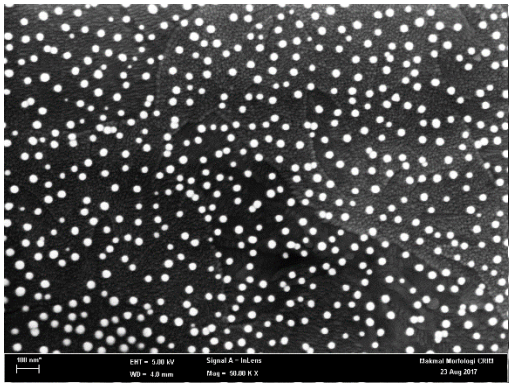




15 nm

20 nm





30 nm

AuNPs: stabilized suspension in 0.1 mM PBS, reactant free

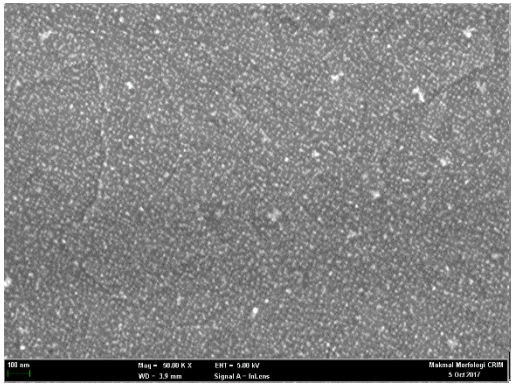
Graduate School of Engineering, Kyoto University, Japan

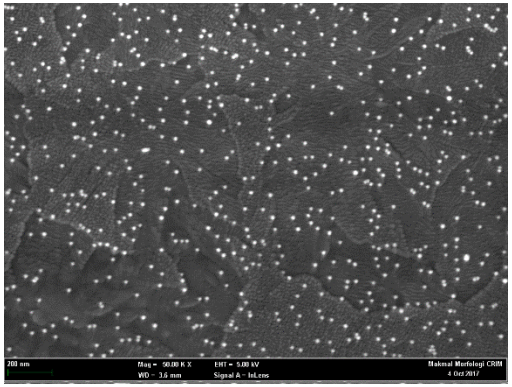
AuNP-Modified ITO Electrodes

Oct. 2017

2% APTMS

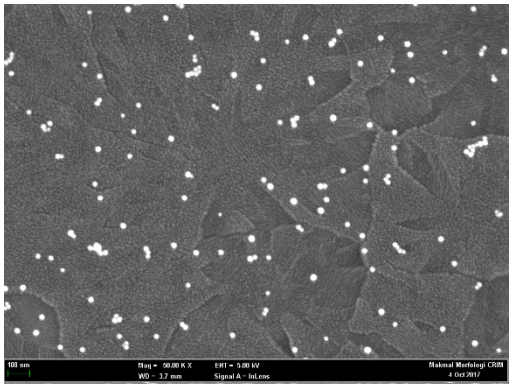
10 nm

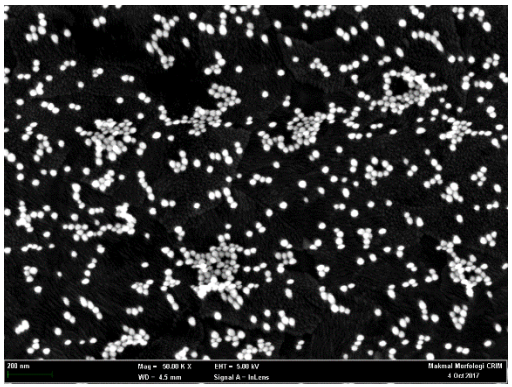




15 nm

20 nm





30 nm

AuNPs: stabilized suspension in 0.1 mM PBS, reactant free

Graduate School of Engineering, Kyoto University, Japan

Preparations for Professor Kityk

Various sized gold nanoparticles (AuNPs)

on fluorine doped tin oxides (FTO)

Graduate School of Engineering, Kyoto University, Japan

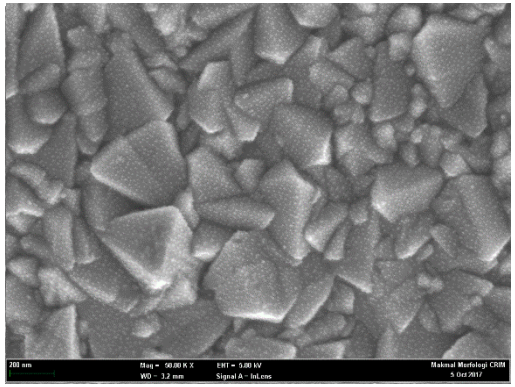
AuNP-Modified FTO Electrodes

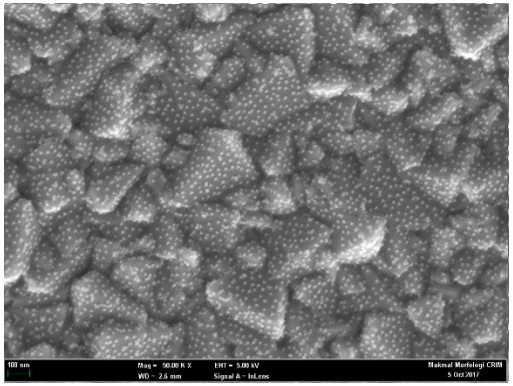
Sigma-Aldrich

Oct. 2017

2% APTMS

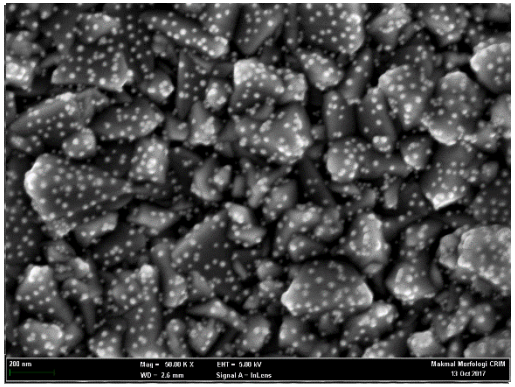
10 nm

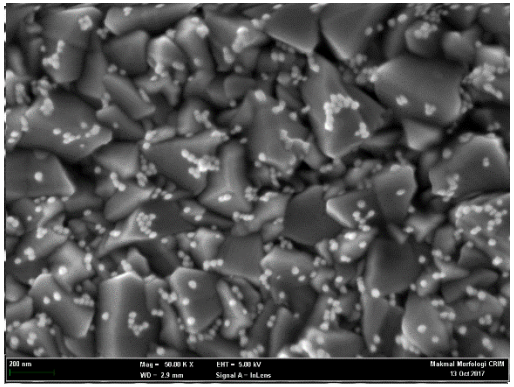




15 nm

20 nm





30 nm

AuNPs: stabilized suspension in 0.1 mM PBS, reactant free

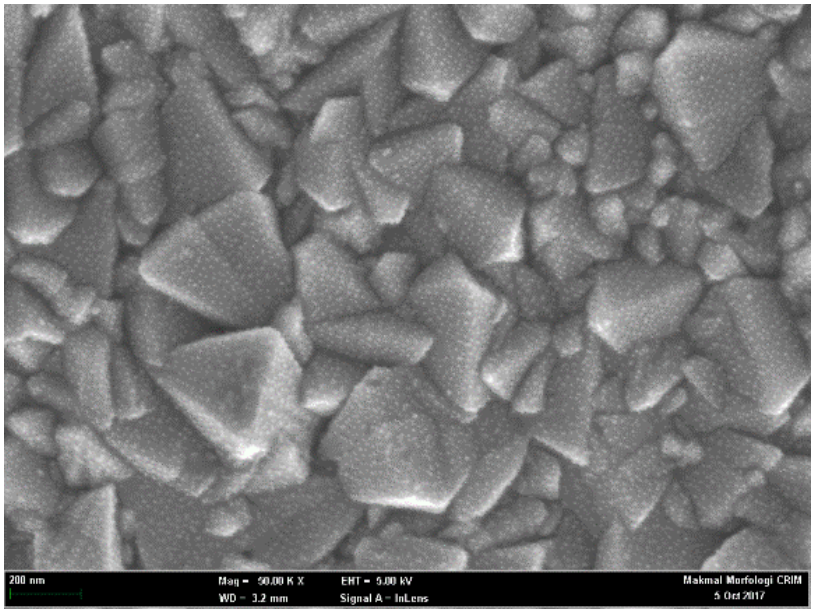
Graduate School of Engineering, Kyoto University, Japan

AuNP-Modified FTO Electrodes

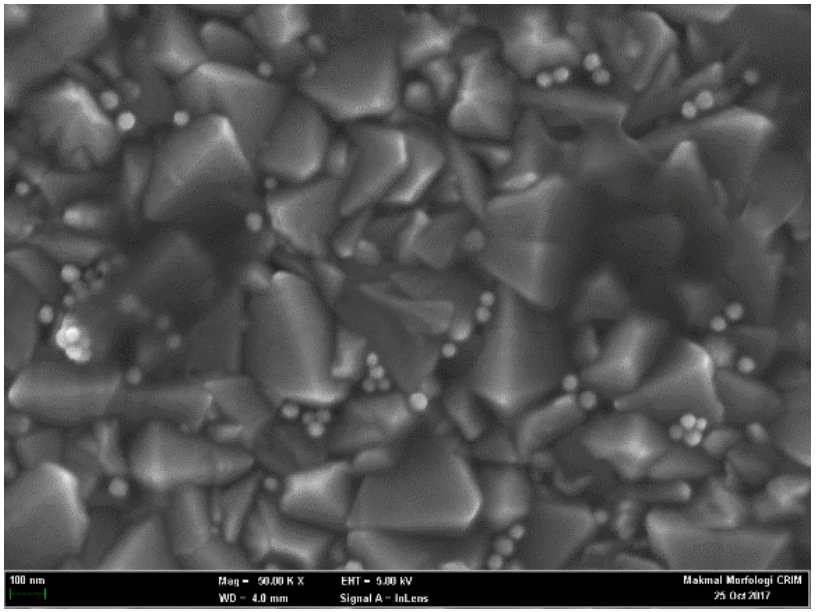
Sigma-Aldrich

Oct. 2017

2% APTMS



10 nm



50 nm

AuNPs: stabilized suspension in 0.1 mM PBS, reactant free

Graduate School of Engineering, Kyoto University, Japan

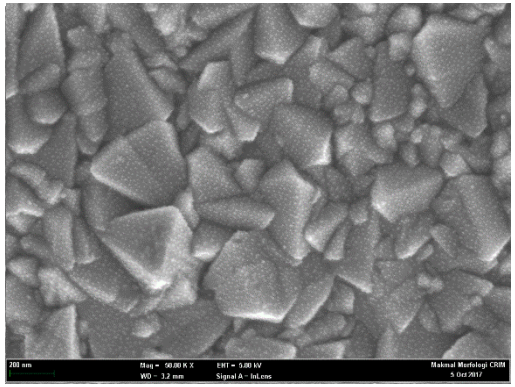
AuNP-Modified FTO Electrodes

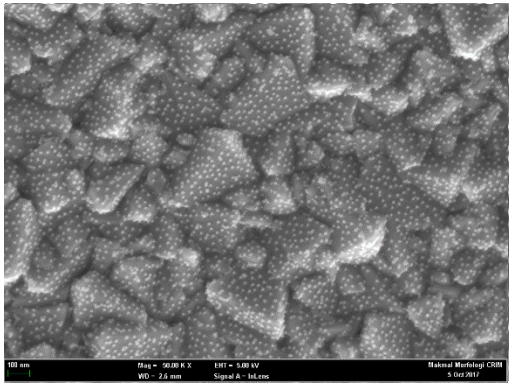
Sigma-Aldrich

Oct. 2017

2% APTMS

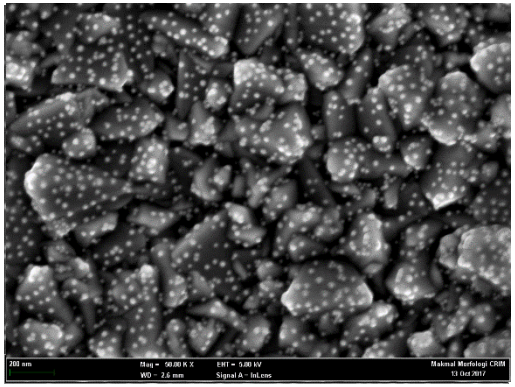
10 nm

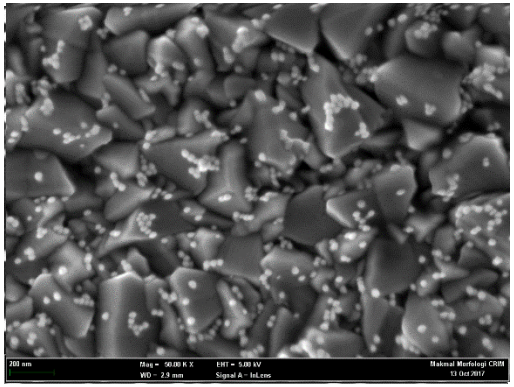




15 nm

20 nm





30 nm

AuNPs: stabilized suspension in 0.1 mM PBS, reactant free

Graduate School of Engineering, Kyoto University, Japan

Preparations for Professor Laskowski

Silver nanoparticles (AgNPs)

on fluorine doped tin oxides (FTO)

Graduate School of Engineering, Kyoto University, Japan

Silver Colloid Solutions

Sigma-Aldrich **(now Merch),** but actually ***nanoComposix*** *(USA)*

0.02 mg/mL in aqueous buffer,

contains sodium citrate as stabilizer

(10, 20, 40, 60, 100 nm)

BBI solutions **(previous, BB international Ltd.)**

Silver nanoparticles (20, 40, 60, 80, 100 nm)

Cytodiagnostics

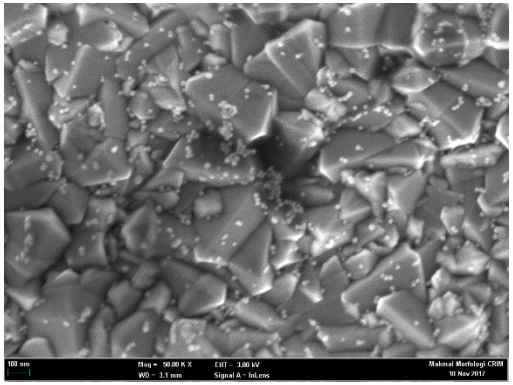
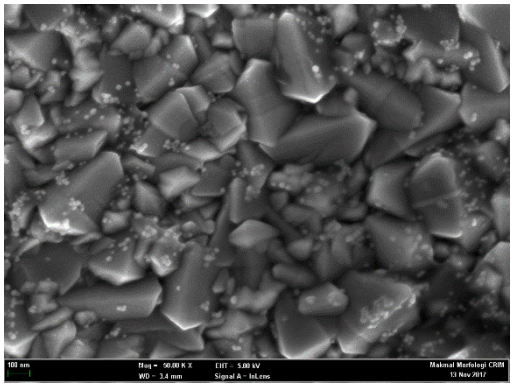
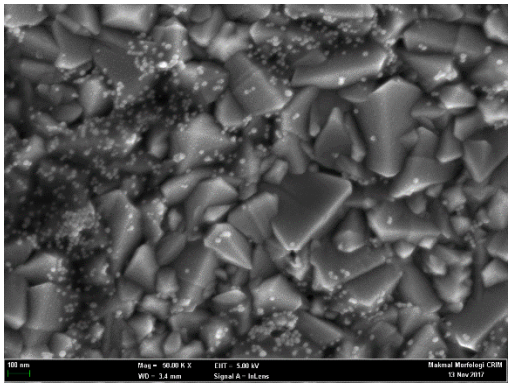
AuNP-Modified FTO Electrodes

From Poland

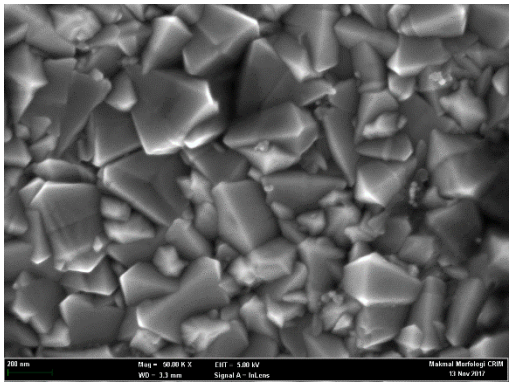
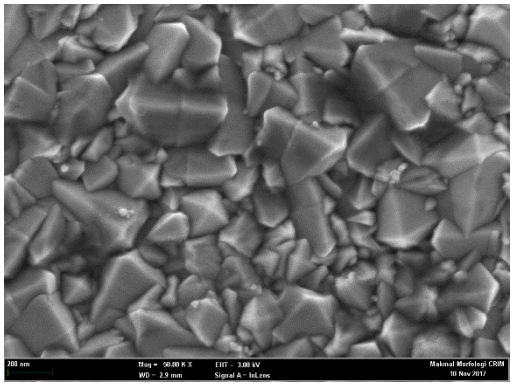
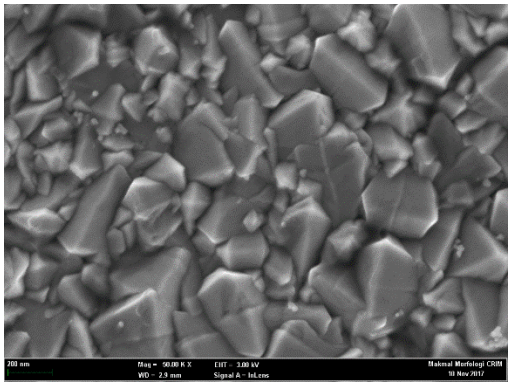
Nov. 2017

2% APTMS

Sigma-Aldrich

BBI solutions

AgNPs: 20 nm

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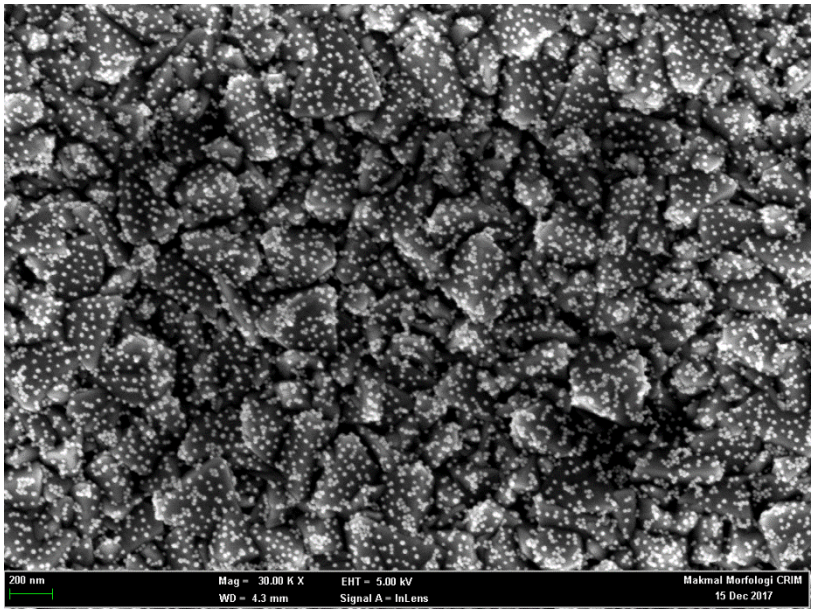
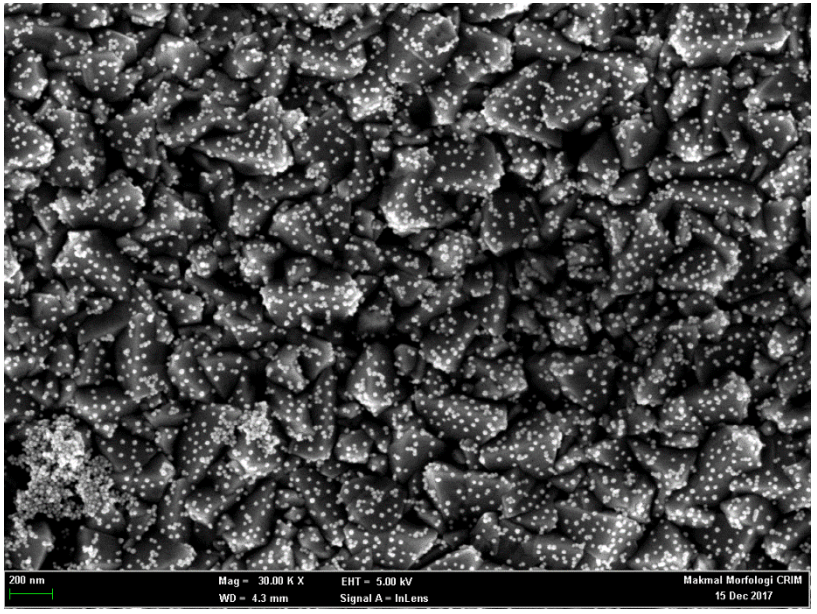
Systematic Comparison of Modification

Dec. 2017

2% APTMS



FTO from Sigma-Aldrich



FTO from Poland

AuNPs: 20 nm, stabilized suspension in 0.1 mM PBS, reactant free

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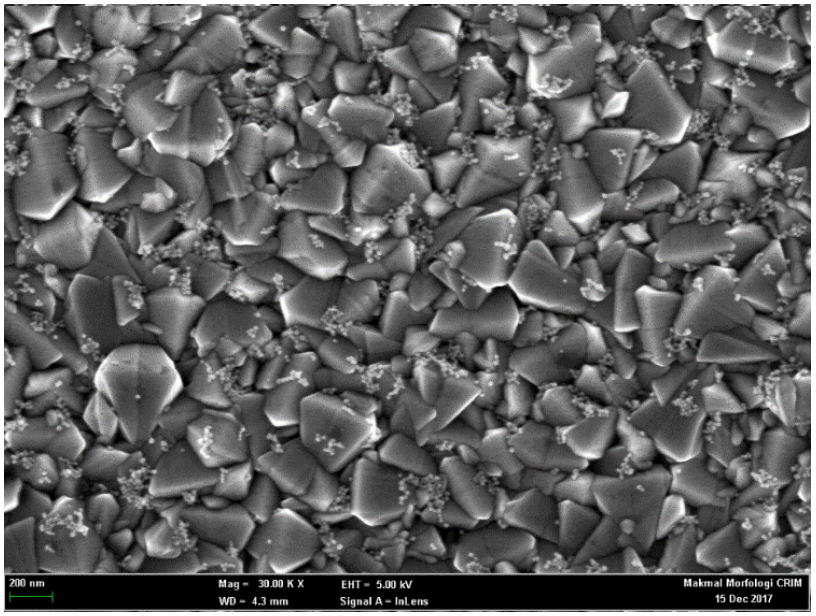
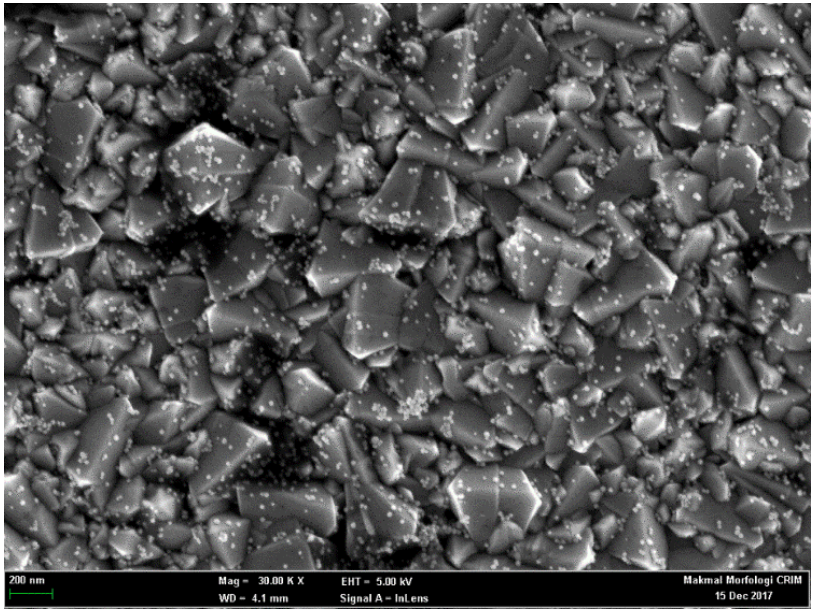
Systematic Comparison of Modification

Dec. 2017

2% APTMS



FTO from Sigma-Aldrich



FTO from Poland

AgNPs: 20 nm, 0.02 mg/mL in buffer, contains sodium citrate as stabilizer

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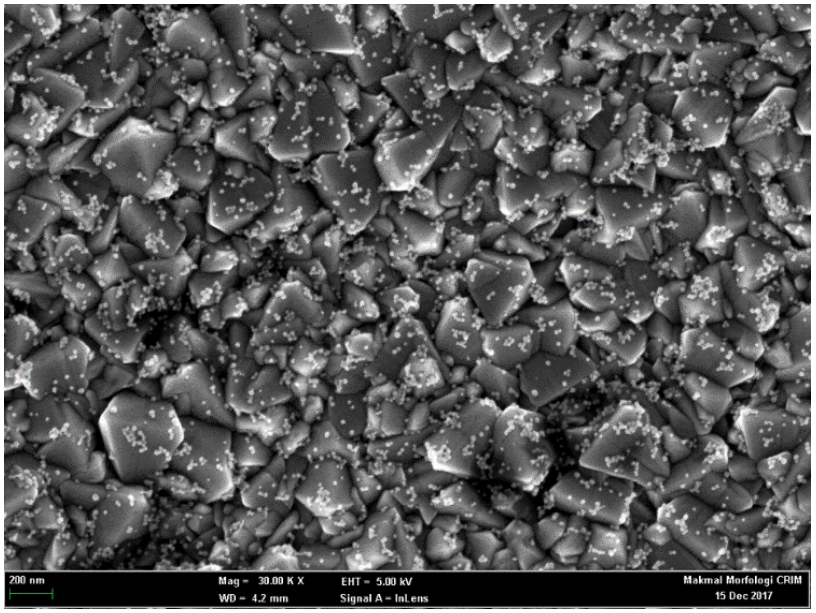
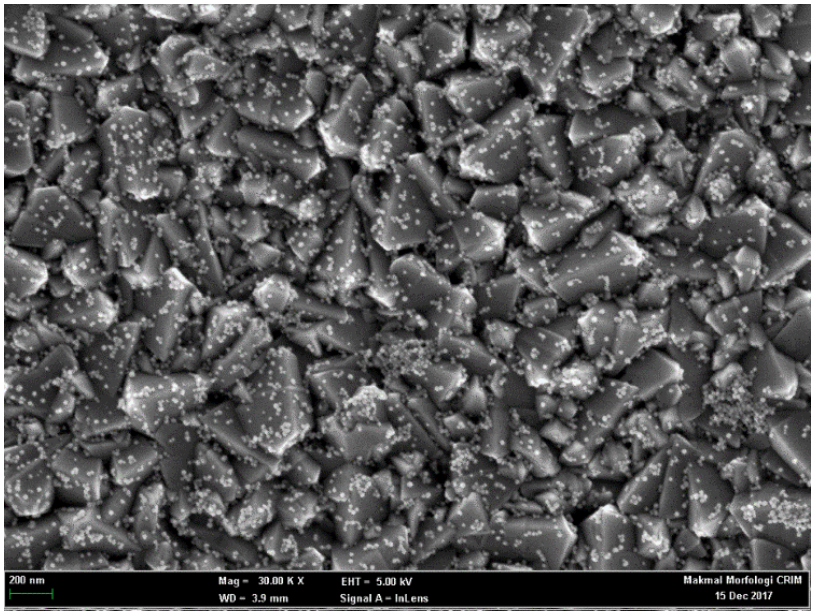
Systematic Comparison of Modification

Dec. 2017

4% APTMS



FTO from Sigma-Aldrich



FTO from Poland

AgNPs: 20 nm, 0.02 mg/mL in buffer, contains sodium citrate as stabilizer

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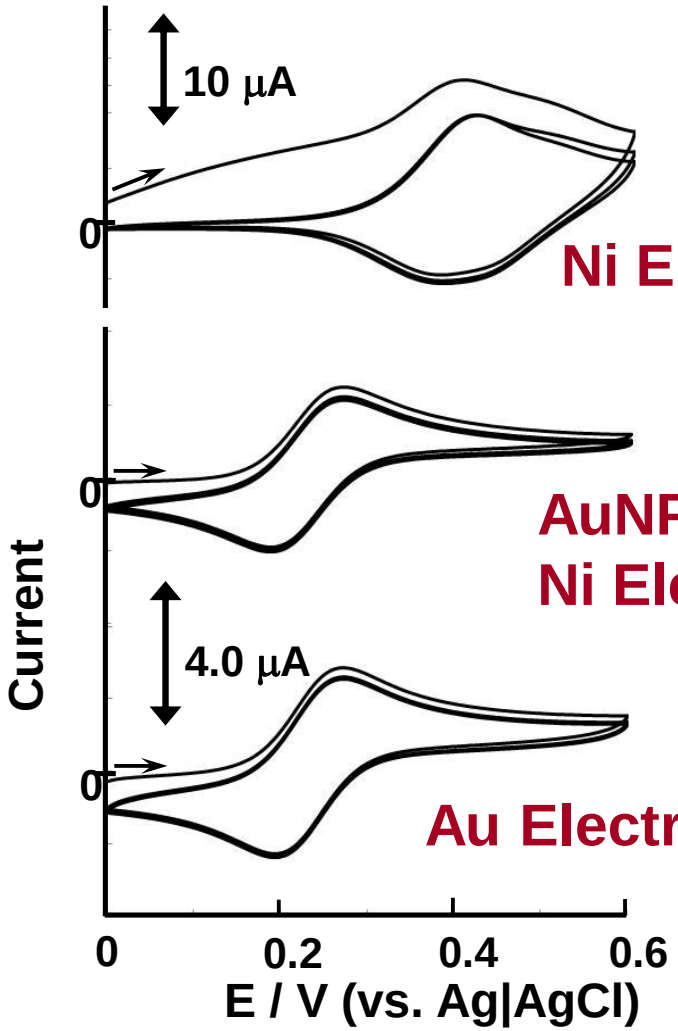
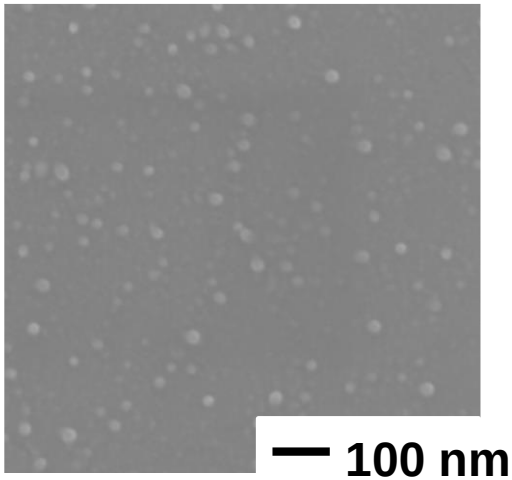
Recent Development of Metal Nanoparticle-Modified Electrodes

Graduate School of Engineering, Kyoto University

AuNP-Modified Ni Electrode



Ni Electrode



Uemoto, Nakayama, Chen, Chang, Oyama, Electroanalysis, **27**, 964 (2015).

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Galvanic Replacement Reaction

$$Ni^{2+} + 2e^- \rightleftarrows Ni(s) \qquad E^0 = -0.24\ V$$

$$AuCl_2^- + e^- \rightleftarrows Au(s) + 2Cl^- \qquad E^0 = 1.15\ V$$

$$AuCl_4^- + 2e^- \rightleftarrows AuCl_2^- + 2Cl^- \qquad E^0 = 0.93\ V$$

$$2AuCl_4^- + 3Ni(s) \rightarrow 2Au(s) + 3Ni^{2+} + 8Cl^-$$

During just immersing an electrode in a solution containing only $HAuCl_4$

Ni Electrode Treated with $HAuCl_4$

with $HAuCl_4$

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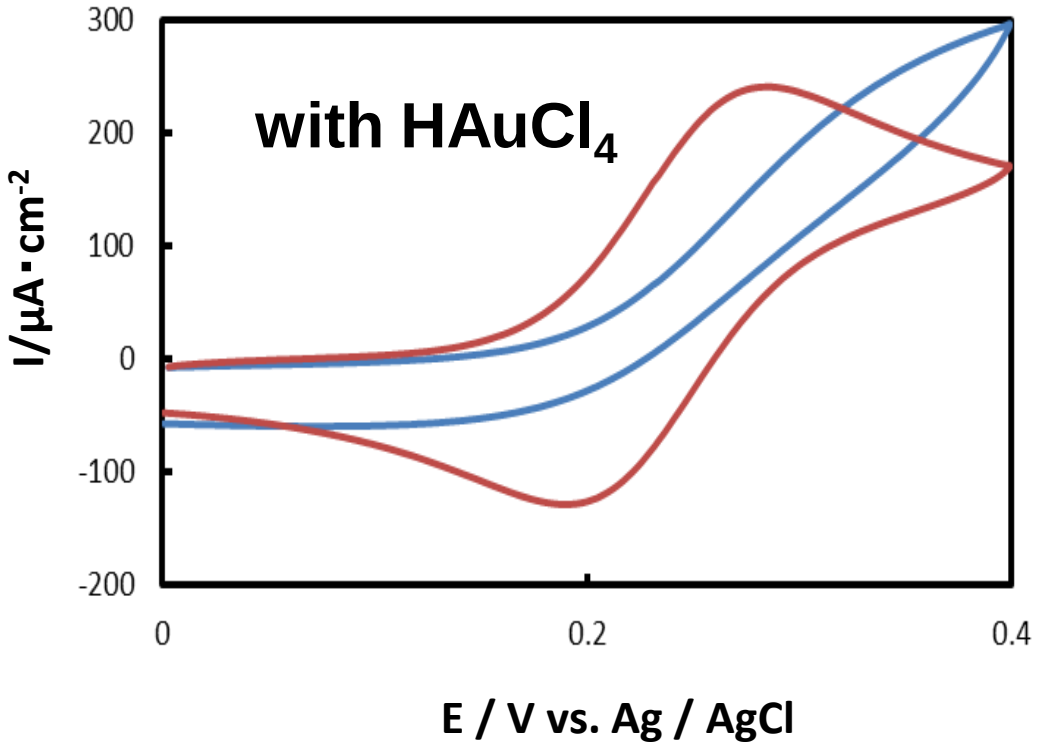
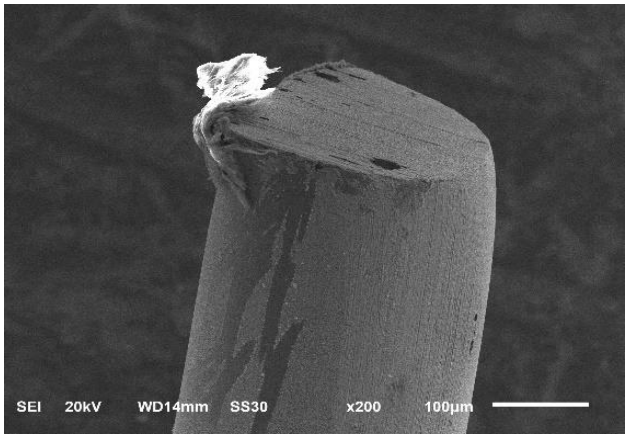
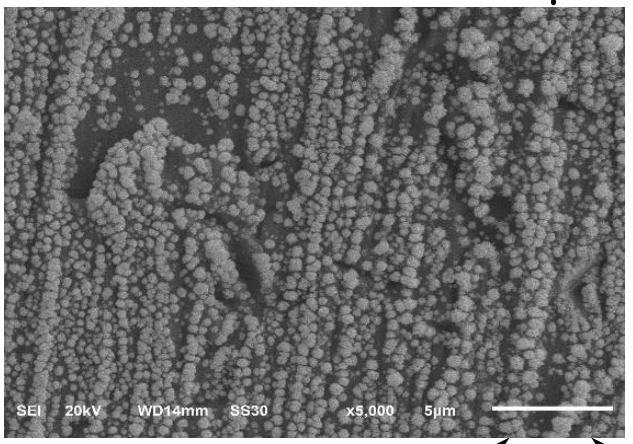
Ni Wire Treated with $HAuCl_4$





100 μm



5 μm

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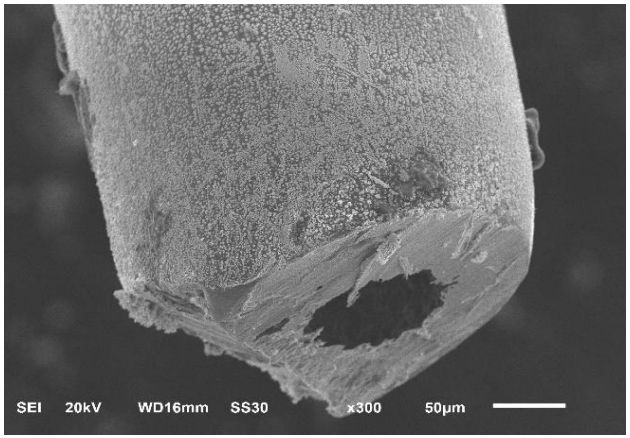
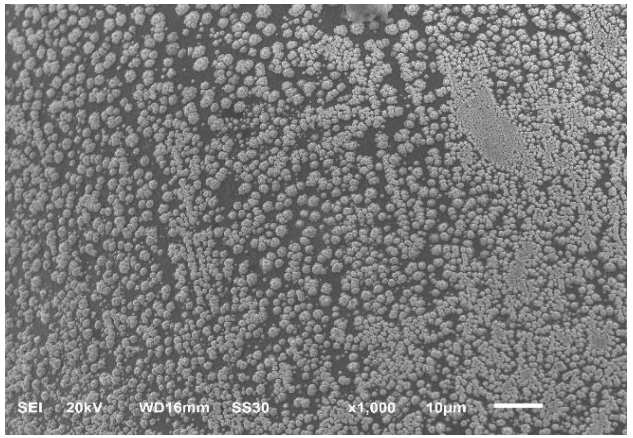
Ni Wire Treated with $HAuCl_4$

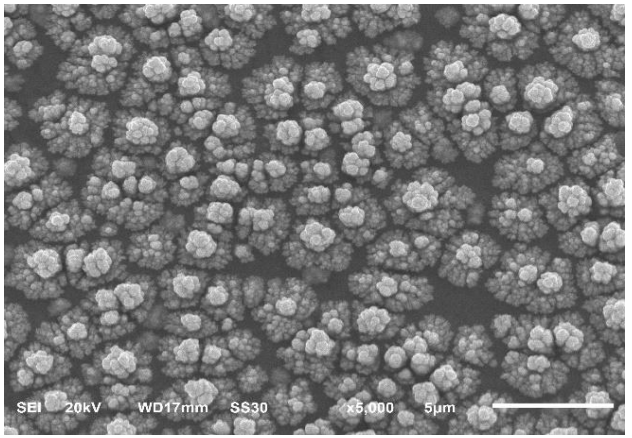
1.0×10^{-3} M $HAuCl_4$ Immersion Time 1 hour



50 μm



10 μm



5 μm

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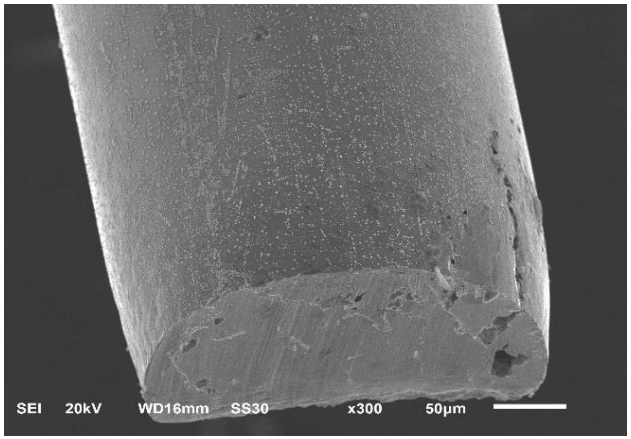
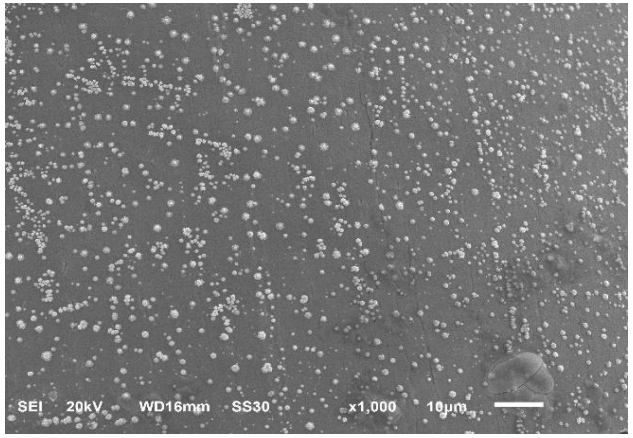
Ni Wire Treated with $HAuCl_4$

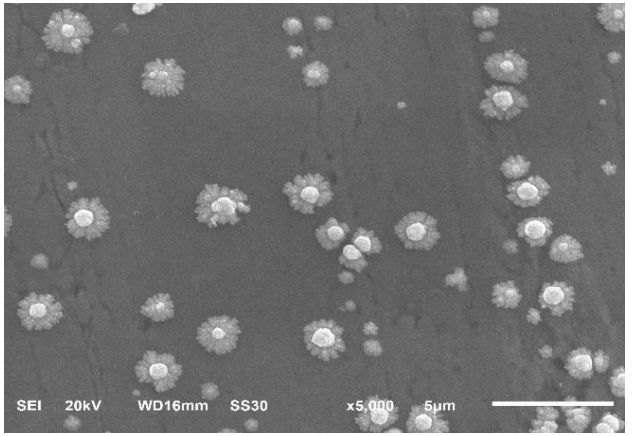
1.0×10^{-4} M $HAuCl_4$ Immersion Time 1 hour



50 μm



10 μm



5 μm

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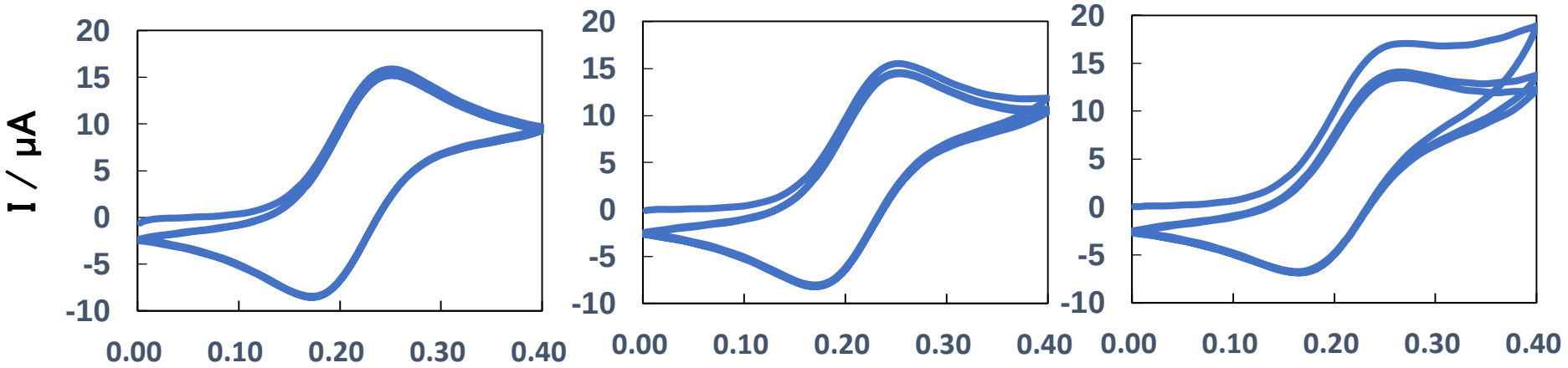
Ni Wire Treated with $HAuCl_4$

Immersion Time: 1 hour

1.0 mM ferrocyanide



1.0×10^{-5} M

1.0×10^{-6} M

1.0×10^{-7} M

$HAuCl_4$

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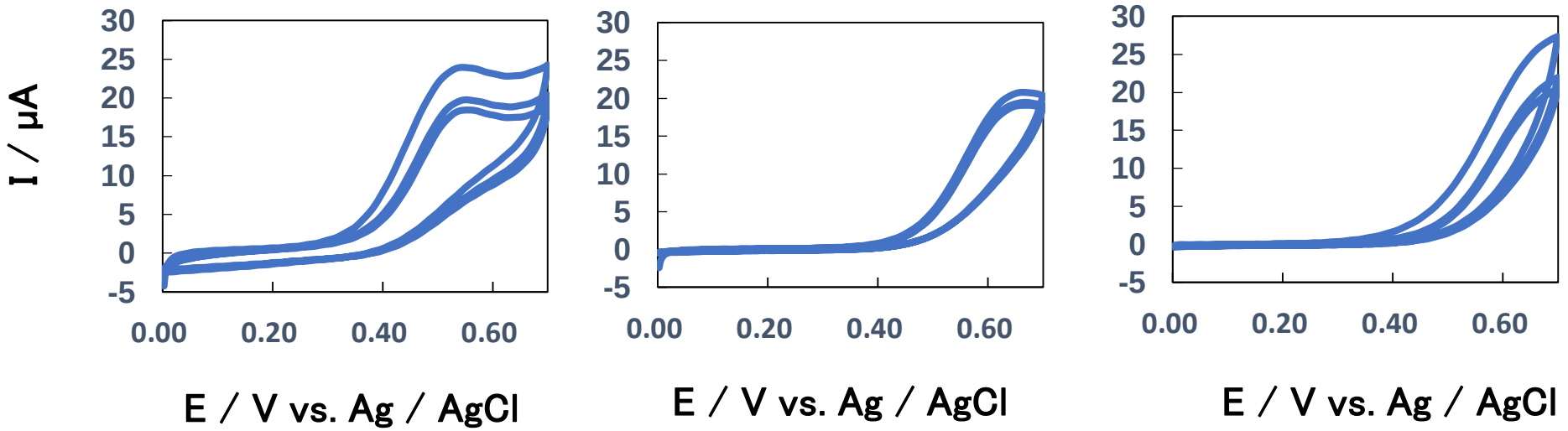
Ni Wire Treated with $HAuCl_4$

Immersion Time: 1 hour

1.0 mM uric acid



1.0×10^{-4} M

1.0×10^{-5} M

1.0×10^{-6} M

$HAuCl_4$

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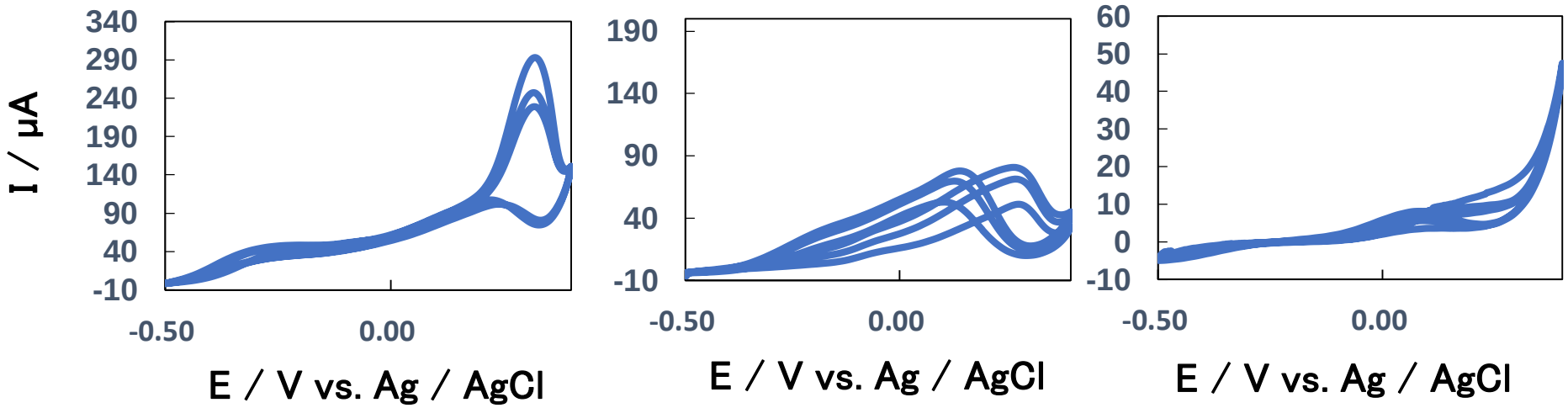
Ni Wire Treated with $HAuCl_4$

Immersion Time: 1 hour

10 mM glucose



1.0×10^{-4} M

1.0×10^{-5} M

1.0×10^{-6} M

$HAuCl_4$

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Summary

Wet Chemical Synthesis of Metal Nanoparticles

Potentials for Nanostructuring

and Electrochemical Applications

Easy Synthesis in Aqueous Solution

at Room Temperature

At moment, we would like to focus the modification of noble metal nanoparticles on Ni or Ti electrodes

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Acknowledgements

All collaborators, coauthors,

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