AFM probe characterization and nano-roughness measurement

Hiroshi Itoh, Chunmei Wang, J. Niitsuma (AIST)
Geroge Orji, Ronald. Dixson (NIST)

Project Agreement between AIST and NIST (Nano Form)

1. PROJECT theme

Nanotechnology standardization: size of nano structure

2. PROJECT abstract

NIST and AIST intend to collaborate on the development of geometric characterization of nano-scale probes using physical standards

3. Participating units and representatives

For each project, each party will designate a responsible laboratory and a project representative and notify of it in writing to the other party.

AWI 13095 (JISC) Procedure for in situ characterization of AFM probe shank profile used for nanostructure measurement

IS 13095 (JISC H. Itoh, 2014 Aug.)

We would like to thanks to Dr. G. Orji. for many important comments and experimental supports

Recent progress : ISO TC201 SC9 (Surface chemical Anaylisis, Probe Microscopy)

WG5（Calibration of probes）

FDIS 11775 (BSI) Determination of cantilever normal spring constants

1 IS 13095 (JISC) Procedure for in situ characterization of AFM probe shank profile used for nanostructure measurement

TR13096 (JISC) Guide to describe AFM probe properties

WG6(E-SPM)

DIS13083 (KATS) Standard on the definition and calibration of spatial resolution of scanning spreading resistance microscopy and scanning capacitance microscopy for two dimensional dopant imaging

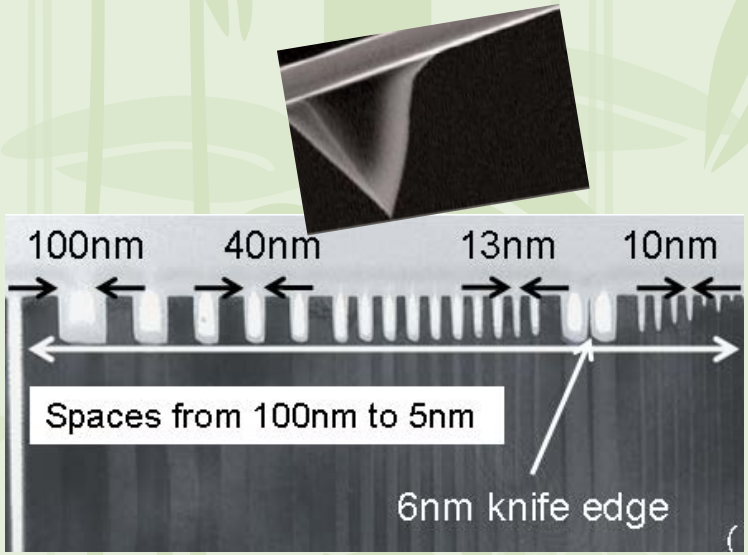
2 Nano Roughness SG in TC201 SC9 was started (JISC H. Itoh)

VAMAS TWA2

3 A18: International Round Robin Test for Carrier concentration Characterization in semiconductor materials by Scanning Capacitance Microscopy

JISC Proposal

IS 13095 Procedure for in situ characterization of AFM probe shank profile used for nanostructure measurement



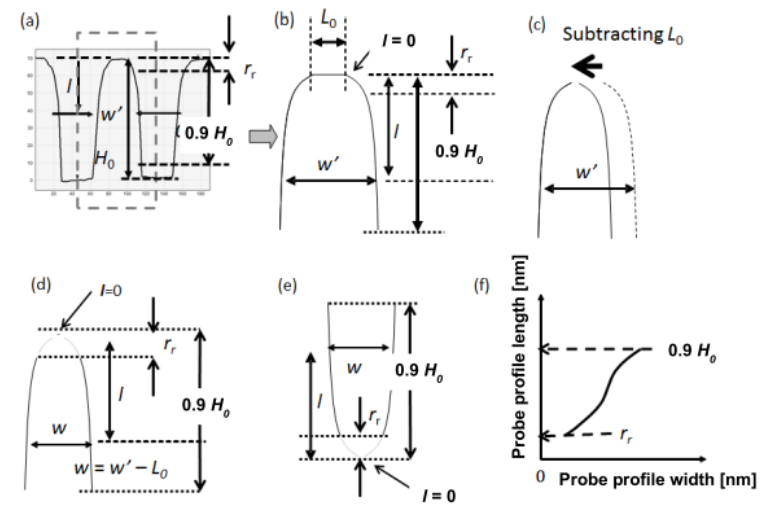


Figure 3 — Determination of PPP and PSC using a narrow-ridge structure. (a) Measured data. (b) The profile of the ridge. (c) Subtracting L_0. (d) Probe profile after removing the ridge width. (e) Inversion of (d) giving the PPP. (f) PSC.

5 Procedure for probe characterization .....
5.1 Methods for the determination of AFM probe shapes ....
5.2 Reference sample setting .....
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5.4 Measurement of probe shank profile 7
5.4.1 General Remarks 7
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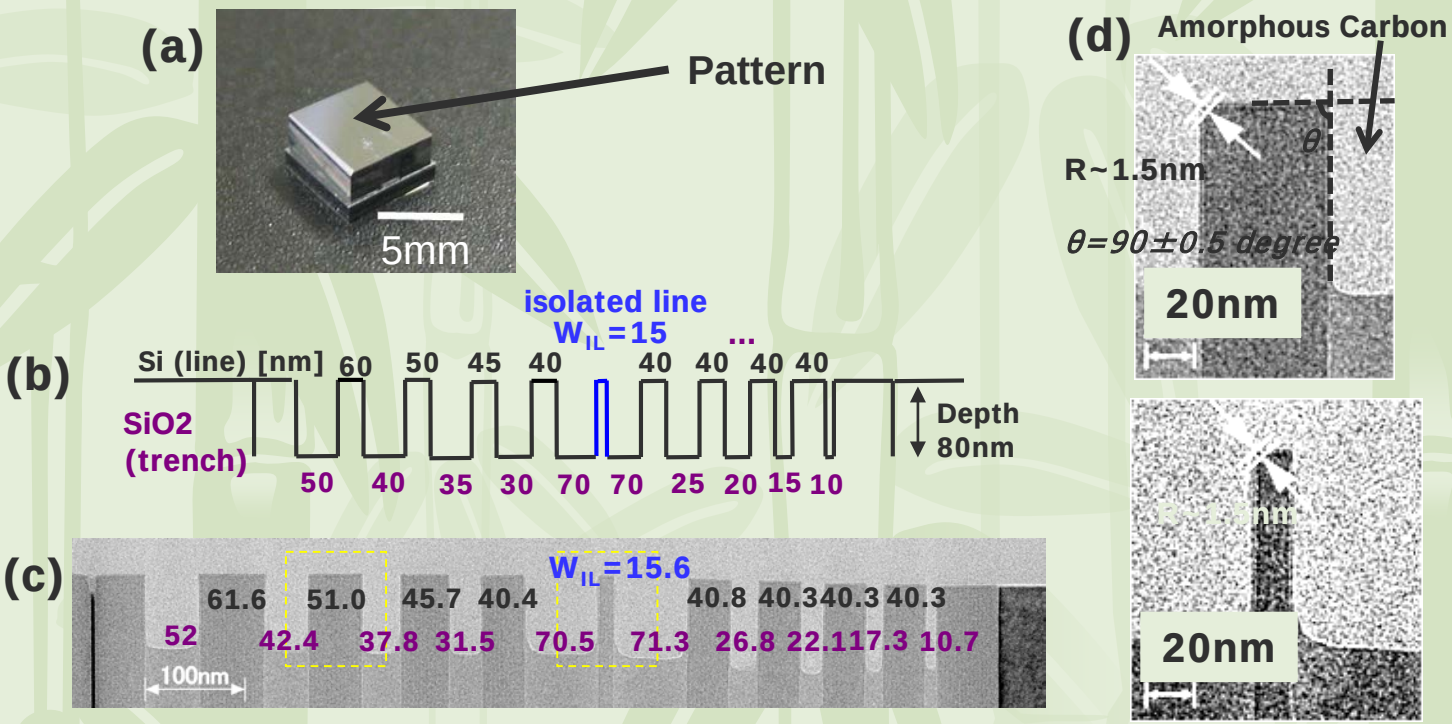
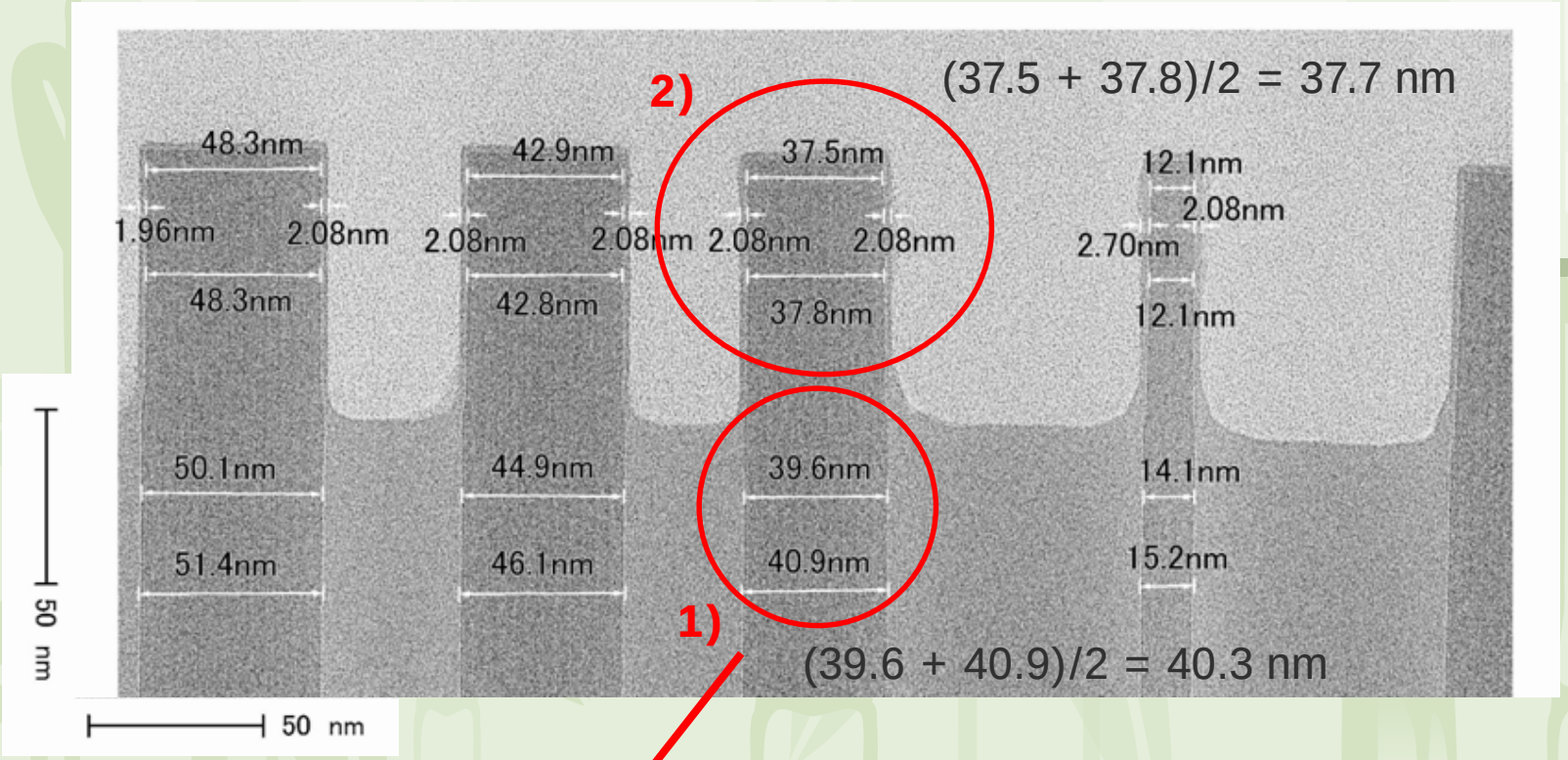
5.5 Uncertainty of the measurement of the probe shank profile 9
5.5.1 Uncertainty of the PPP or PSC measurement 9
5.5.2 Uncertainty of the EPSC measurement.....10

6 Reporting of probe characteristics.....10

Outline of the test sample for probe characterization





1) 40.3nm (Film thickness) + 1.0nm (Oxidation) = 41.3 nm
2) 37.7nm (Si thickness) + 2～4nm (Oxidation) = 39.7nm ～41.7nm

Line width = 40.7 nm ± 1nm

2. Virtual reference probe method for nanoscale roughness

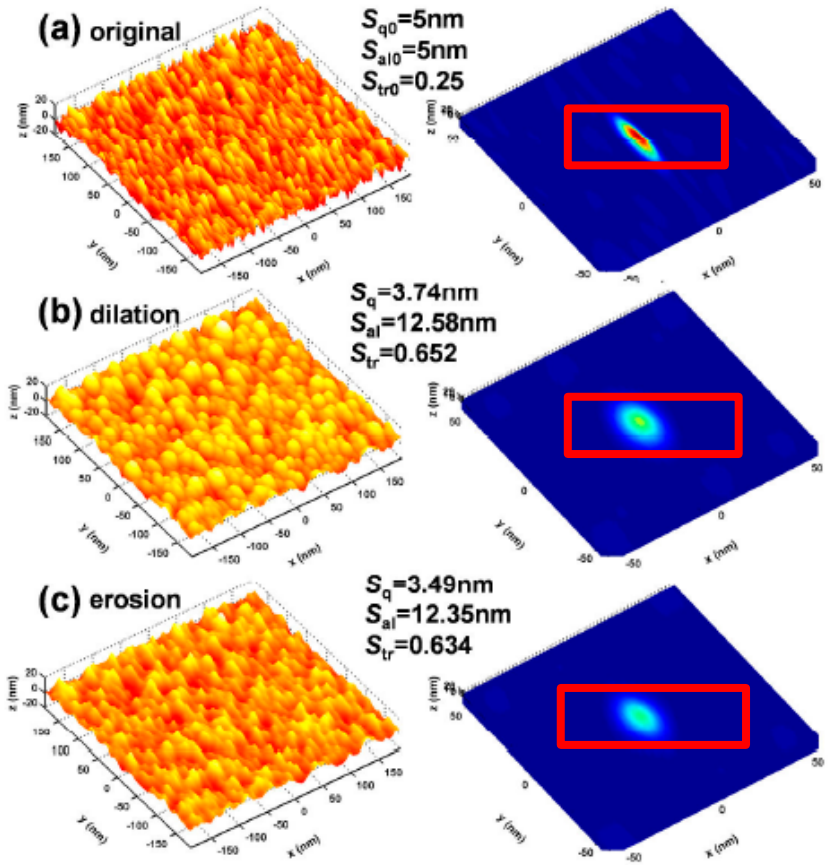


Fig. 2. (a) A simulated Gaussian rough surface (S_{q0}=5 nm, S_{al0}=5 nm, and S_{tr0}=0.25) (left) and its ACF (right). (b) The simulated AFM dilation image obtained by scanning the surface in (a) with a tip with radius R_t=5 nm (left) and its ACF (right). (c) The reconstructed erosion image from the dilation image in (b) (left) and its ACF (right). S_q, S_{al}, and S_{tr} are the measurements of S_{q0}, S_{al0}, and S_{tr0}.

Gaussian rough surfaces

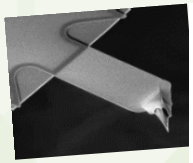
Dilated image due to the AFM probe

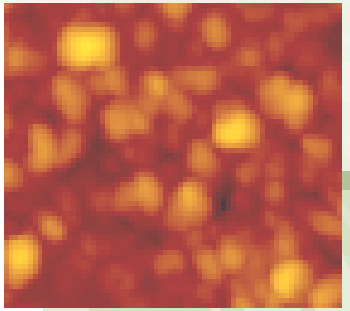
Image reconstruction

C.M. Wang and H. Itoh, JJAP Conf. Proc. **1 (2013) 011005**

Virtual reference probe method

- Removing variation of AFM probes -

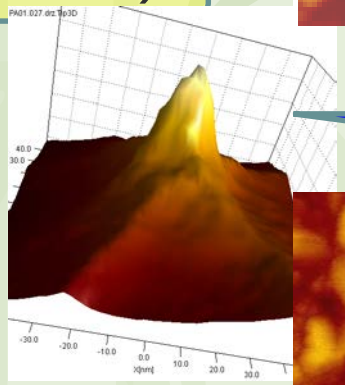




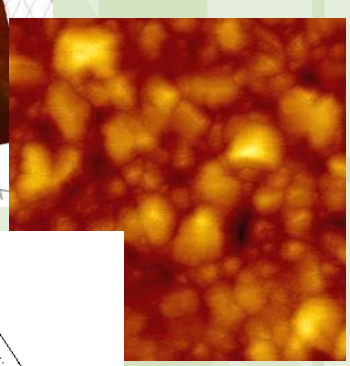
Procedure

1) Measuring surface morphology (roughness)

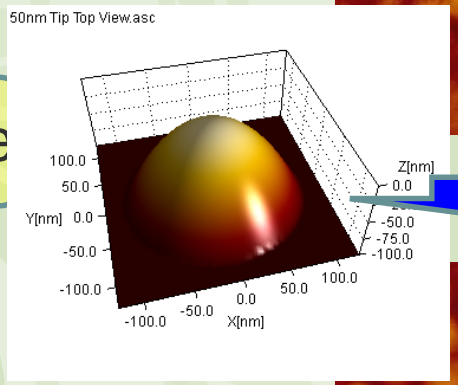
2) Measuring probe shape



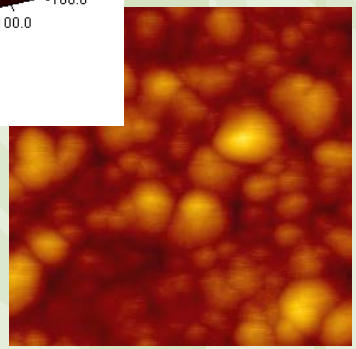
3) Image erosion

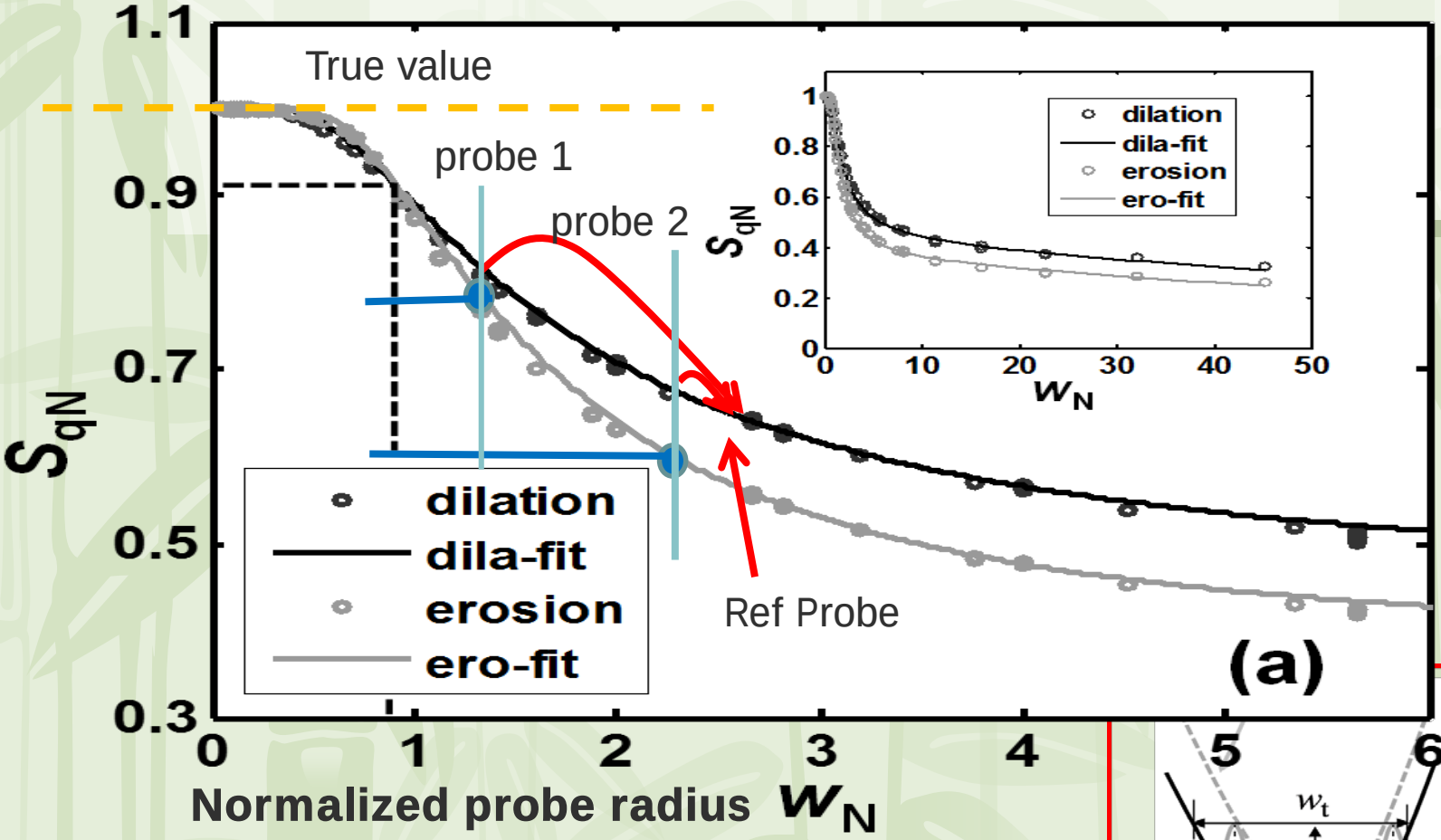


4) Image dilation with reference probe

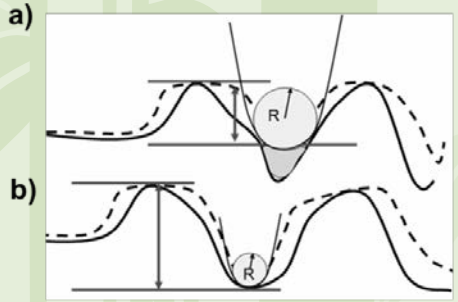


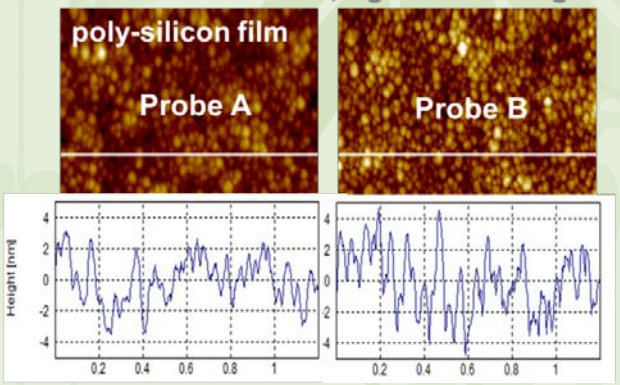
5) Roughness calculation
(ISO 25178 .....)

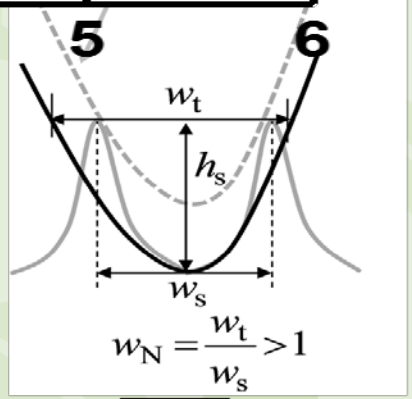




Surface roughness → surface properties such as friction, light scattering…





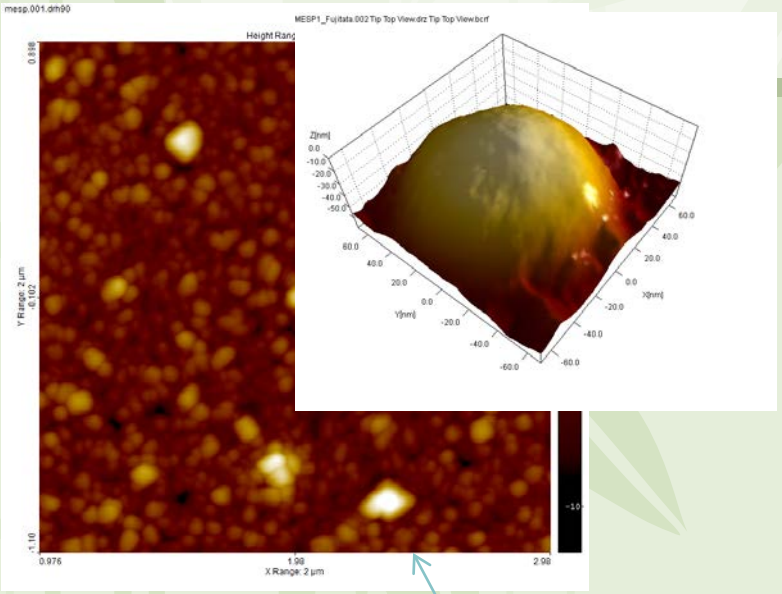


$$w_N = \frac{w_t}{w_s} > 1$$

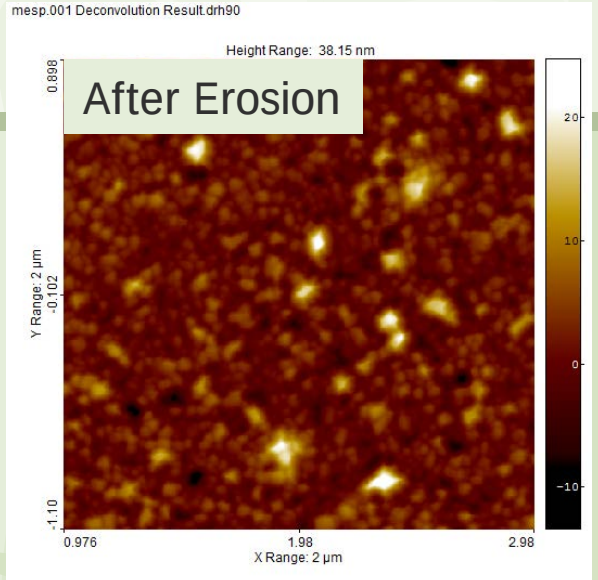
$$w_N = \frac{\sqrt{8 S_{q0} R_t}}{S_{al0}} = \sqrt{\frac{R_t}{R_s}}$$

a, normalized

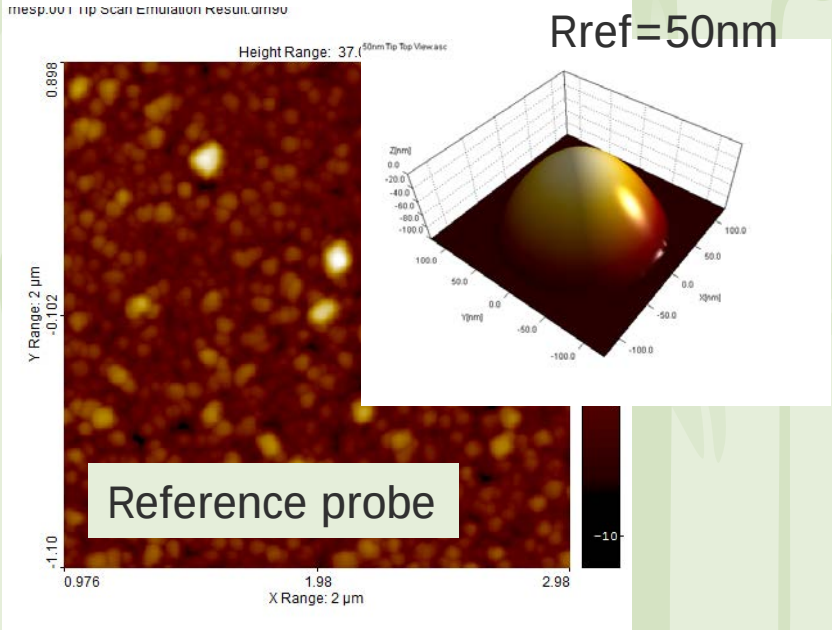
Why image reconstruction (erosion) is not used for roughness ?



Original Image



Rref=50nm



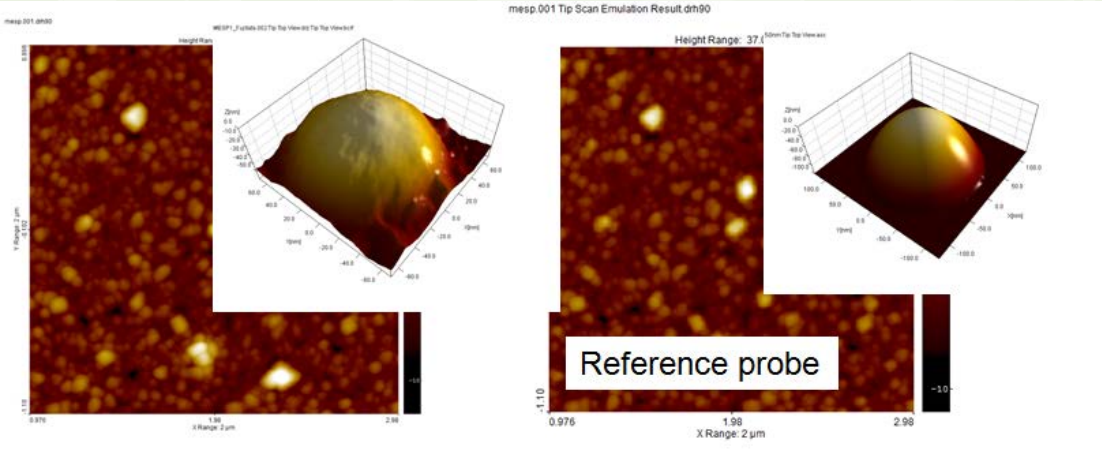
Roughness Ra

Original image	2.7nm
After erosion	**2.4nm**
reference probe	2.6nm

Result of RRT (Continuing in Japan)

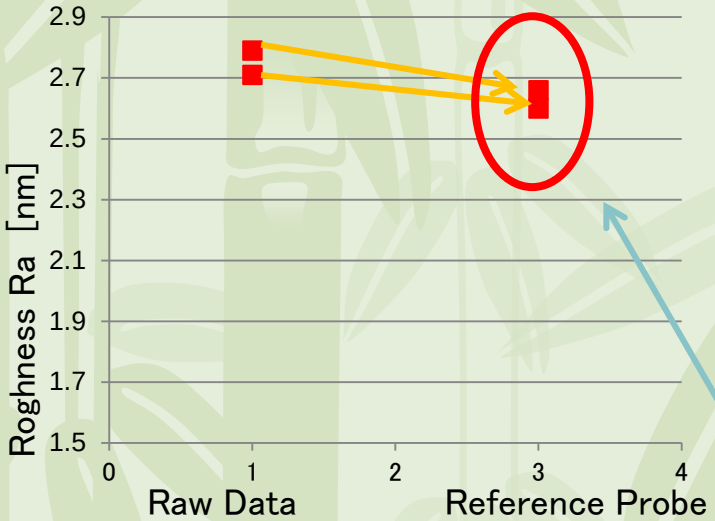
Probe
AC200
MESP

Sample
Ag film onSi(100)

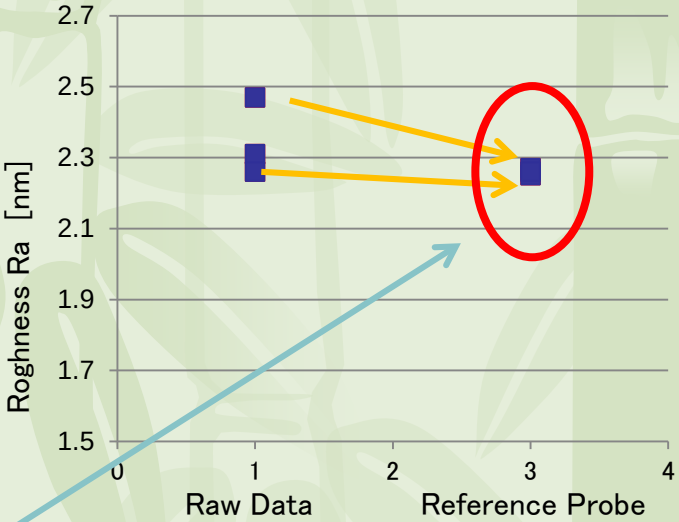


Normalizing AFM image under 50nm reference probe

RRT 1 Ra = 2.63 nm



RRT 2 Ra = 2.28 nm



Virtual reference probe R_0 = 50nm

—> RRT in VAMAS

Virtual reference probe method
- Removing variation of AFM probes -

Procedure

1) Measuring surface morphology (roughness)

2) Measuring probe shape

R= 8nm, 12nm,
7nm, 13nm, ...

3) Image erosion

Removing asymetry of the used probe

4) Image dilation with reference probe

Converting AFM image
to an AFM image
with virtual reference probe

Ex. R_0= 20nm .

5) Roughness calculation
(ISO 25178 .....)

Following previous ISO standard

AFM image with 20nm probe

3. Proposal of RRT Project

VAMAS TWA-2 (Project A18)

International Round Robin Test for Carrier concentration Characterization in semiconductor materials by Scanning Capacitance Microscopy

Project Leader: Mr. Takaya Fujita
JISC, Japan

International RRT is on the progress

VAMAS

VAMAS ACTIVITY INITIATION FORM

1. **Proposed Activity**

☐ New Technical Working Area (TWA)

☒ New Project Existing TWA Number ____TWA2_____

2. **Proposed Theme / Title**

International Round Robin Test for Carrier concentration Characterization in semiconductor materials by Scanning Capacitance Microscopy

3. **Proposed Activity and Objectives**

Scanning Capacitance Microscopy, abbreviated as SCM, is a contact-mode variant of atomic force microscopy (AFM) that is used for two-dimensional imaging of derivative capacitance (dC/dV) on a surface and cross-section of semiconductor materials. AC modulation around 100 kHz is applied to the conductive AFM probe, and the signal from lock-in amplifier is proportional to the derivative capacitance between probe and specimen. It is interpreted that dC/dV signal is depending upon electrical carrier concentration (N) of electrons and holes in the contact area, which is covered with a thin oxide layer beneath the probe. The distribution of the carrier concentration which corresponds to ability of electrical conductance, relates to various electrical properties and reliabilities of integrated semiconductor devices. Then, SCM with nanometer scale resolution has become one of the most important methods for characterization of semiconductor devices in research, quality control of actual devices, and failure analysis of the commercial devices. Out of these backgrounds, it is necessary to establish the standard protocol for the analysis of dC/dV signal by SCM. A calibration curve between logarithmic N and normalized dC/dV signal is proposed for the analysis carrier concentration in unknown samples. The reference sample with criteria of N shall be prepared in advance. The dC/dV signal shall be normalized for fitting the calibration curve in the subsequent process. The objective of this proposal is to establish the standard protocol for the analysis of carrier concentration, to establish how to determine the calibration curve, to exam the data-compatibility among participants, and to test the reproducibility of calibrated SCM data.

4. **Proposer**

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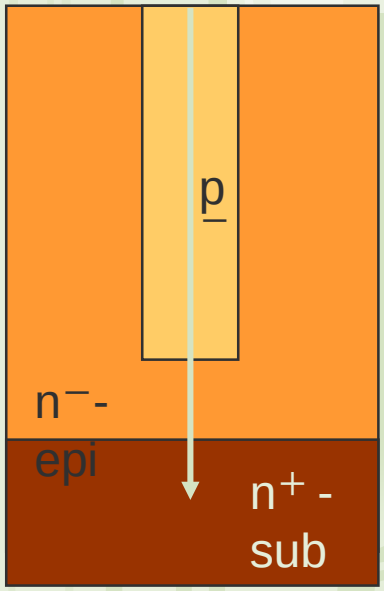
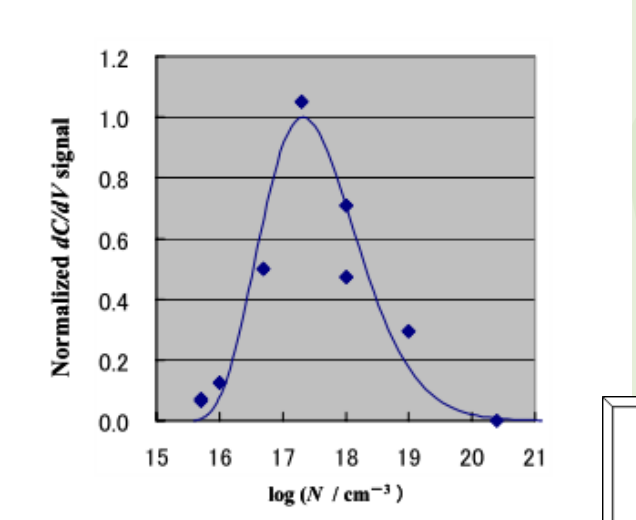
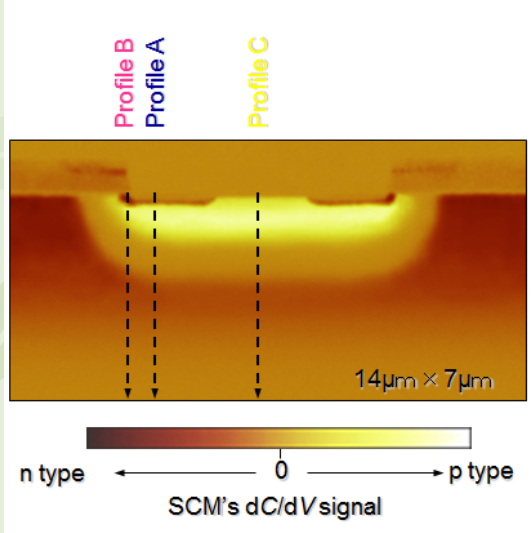
Calibration of Scanning Capacitance Microscopy (SCM)

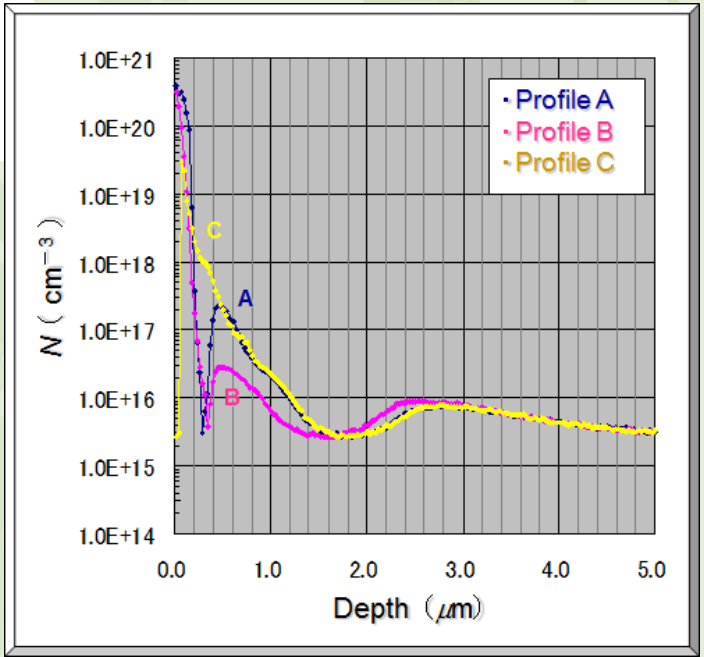


Applicable to other methods such as SNDM, EFM, KFM, etc.





Calibration curve (SCM)



Summary of ISO standardization

- **ISO TC201 SC9**
  - **IS13095 "**Procedure for in situ characterization of AFM probe shank profile used for nanostructure measurement" was published as IS on Aug. 2014.

- **Resolutions (ISO TC201 Meeting 2014 Sep.)**
  - ISO/TC201/SC 9 requests Dr. H. Itoh to circulate a report on his lab tests on nano-scale roughness measurement with different AFM instruments based on the domestic RRT in Japan by 2014-11-30, and initiate international RRT through VAMAS

  - ISO/TC201/SC 9 requests Mr. T. Fujita to report VAMAS TWA2 A18 RRT and submit the NWIP on "Dopant concentration and detection limit of electric SPM (SSRM and SCM)" by 2015-1-31.

Plan → NWIP submission in 2015

Summary

- **AIST and NIST collaboration**
  - IS13095 was published as IS on Aug. 2014. (Many important comments and experimental supports from Dr. G. Orji, Dr. R. Dixson)
  - Nanoscale standards was improved after collaboration
  - Fruitful discussion on VAMAS A18 Project "Dopant concentration and detection limit of electric SPM (SSRM and SCM)

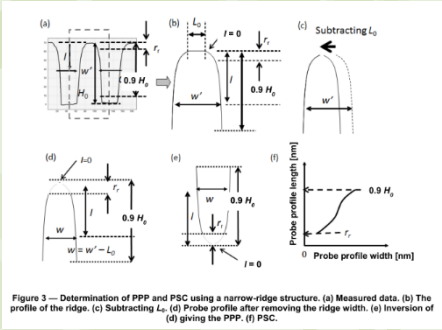
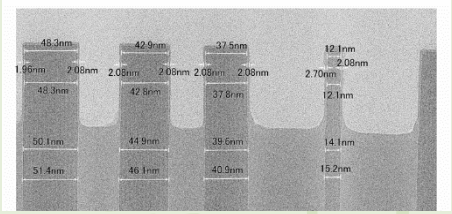
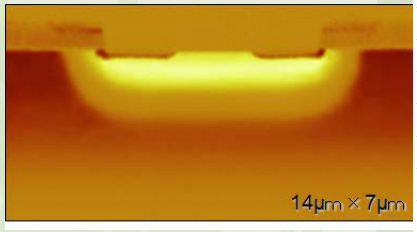
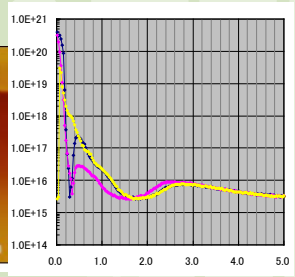


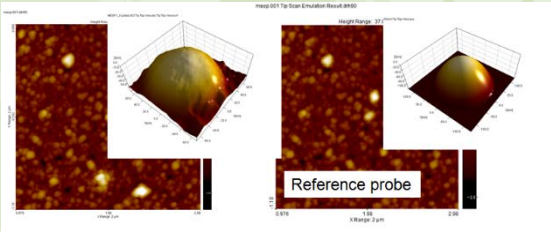
Figure 3 — Determination of PPP and PSC using a narrow-ridge structure. (a) Measured data. (b) The profile of the ridge. (c) Subtracting L_0. (d) Probe profile after removing the ridge width. (e) Inversion of (d) giving the PPP. (f) PSC.







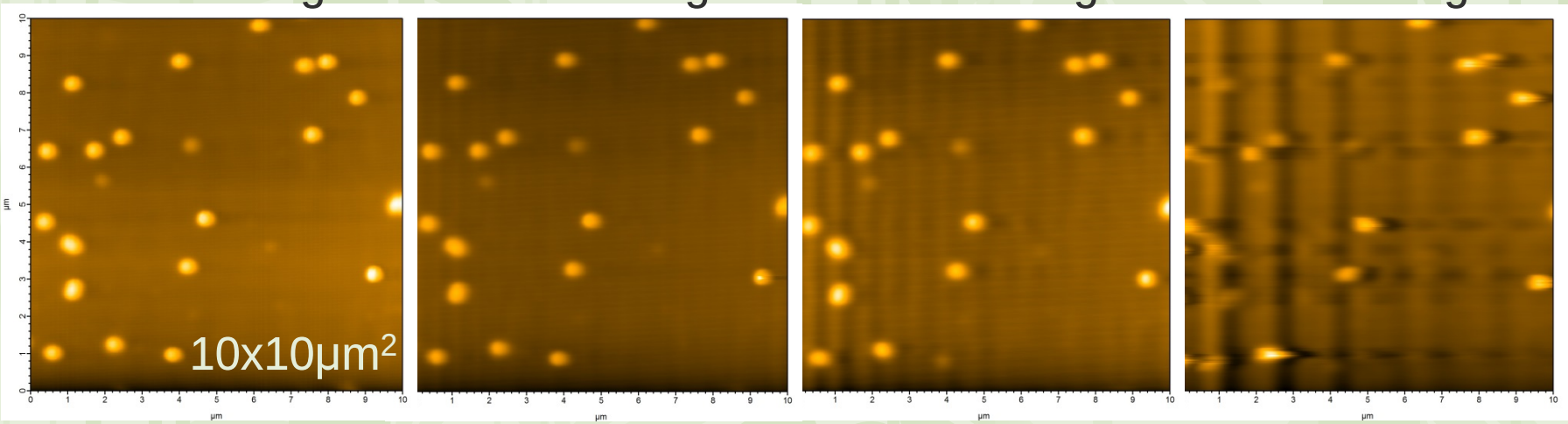
- (Possible) **Future collaboration**
  - Nanoscale roughness characterization
  - VAMAS A18 Project
  - Improving throughput of AFM



Improving Throughput of AFM

Observation of PSL (150nm) with high-speed AFM

60sec/image 30sec/image 20sec/image 10sec/image



500 pixels x 500 lines

20particles/60sec ≈ 1000particles/h

Plan: Calibrate scanner, Improve feedback system

→ Goal: 1s / Frame

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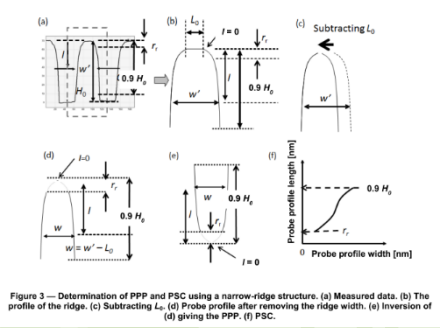

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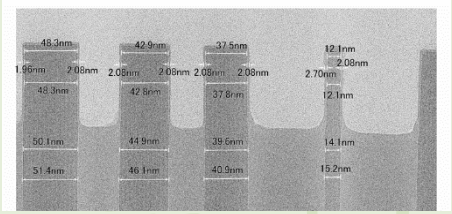

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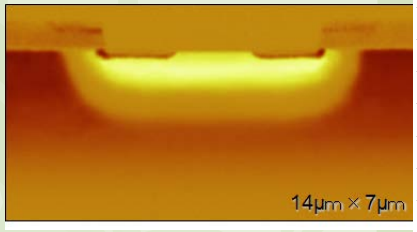
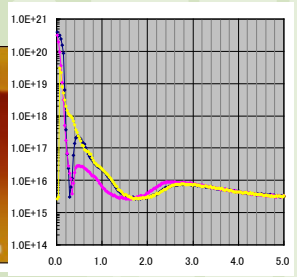
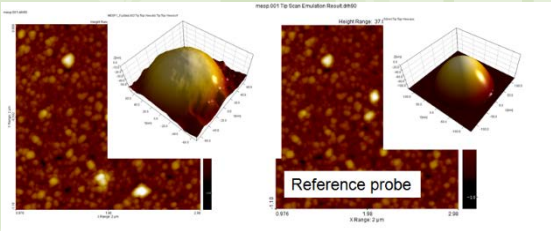
  - VAMAS A18 Project
  - Improving throughput of AFM







Acknoledgement

I would like to thanks to the METI’s support