

Calibration method for nanometer-scale step height with crystal periodicity: Investigation and validation using a metrological AFM

Satoshi Gonda, Kazuto Kinoshita, Kentaro Sugawara and
Ichiko Misumi

National Metrology Institute of Japan, AIST

Background

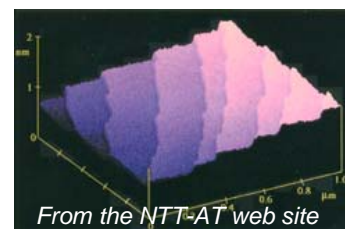
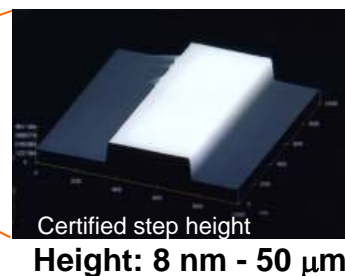
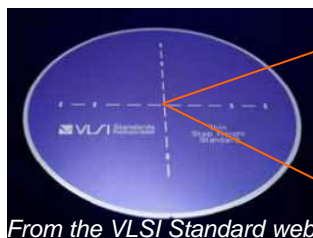
- Shape and size parameters of nanodevices are increasingly important. Reliability of SPM calibration has become significant to guarantee the results. Decreasing of the device size is prompting much smaller size of reference materials for metrology in SPM.

Step height controlled by etching process

- Established fabrication process
- Absence of size below 8 nm
- Significantly large uncertainty
- (1 - 2 nm)
- High-cost standard

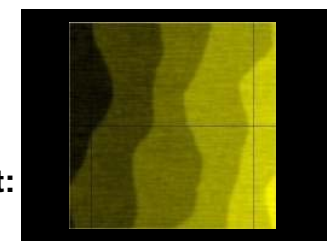
Atomic step/terrace structure on single crystal surface

- Close link to lattice parameters
- Sub-nm height (a little too small)
- Difficult to control the step edge lines
- Challenge to certify using metrological means



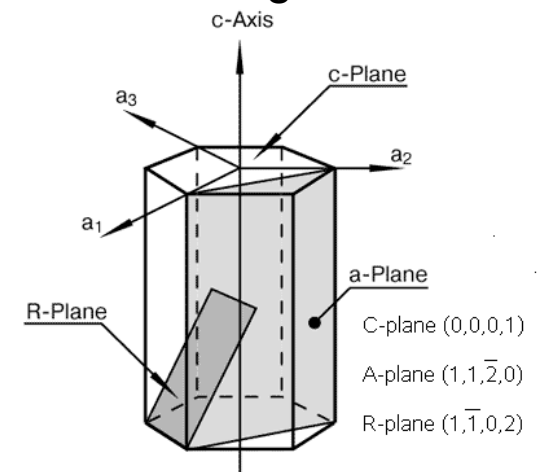
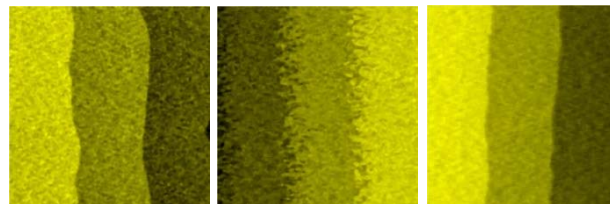
Si(111) /step height:
0.314 nm

Al_2O_3 /step height:
0.206 nm



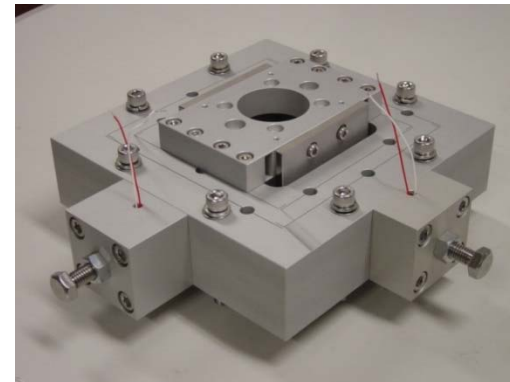
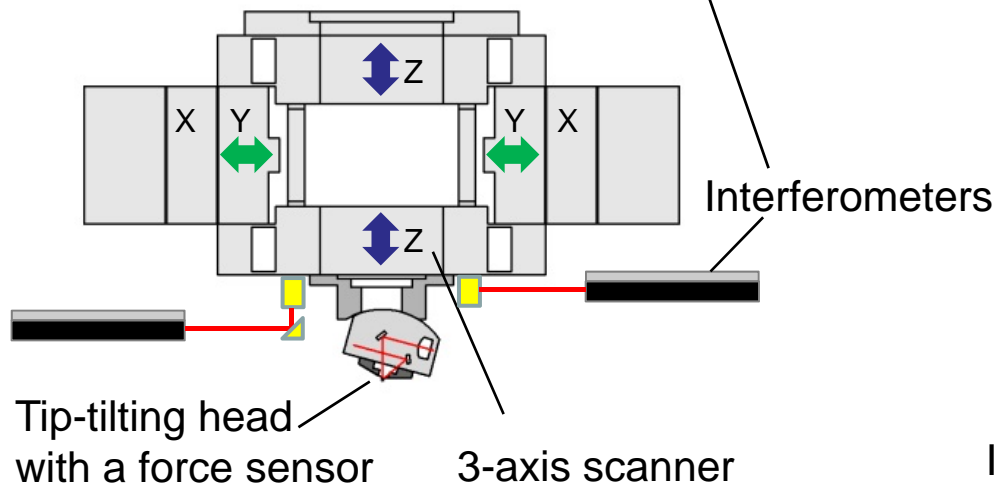
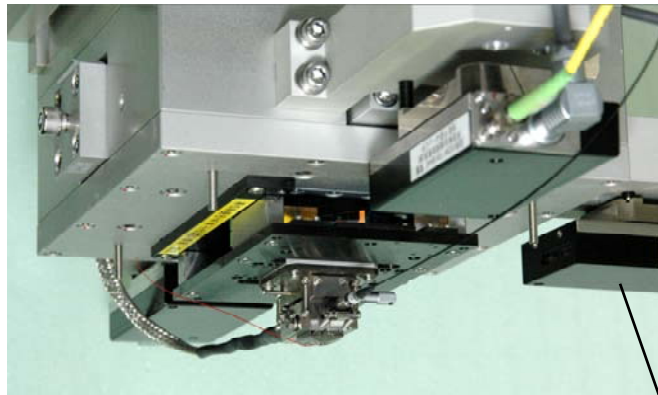
Background - continued

- Address at Consulting Committee for Length under CIPM
 - The development of traceable physical standards for step height (and lateral dimension) at nanometer size
- Bi-lateral comparison
 - Comparison of atomic step height using metrological AFMs between NIST and AIST
 - Artefact: step height of atomic step and terrace structure of single crystals
 - AIST provides Al_2O_3 : c(0001), a(11-20), r(1-102)
 - Found good agreement in preliminary results



Highly-stable tip scanner with 3-axis interferometer

- Tip-tilting mechanism can change the direction of feedback within +/- 16 degrees.
- Structural consideration for eliminating mechanical noise and thermal drift
- Highly-stable laser interferometers monitors 3-axis displacement use them for feedback control

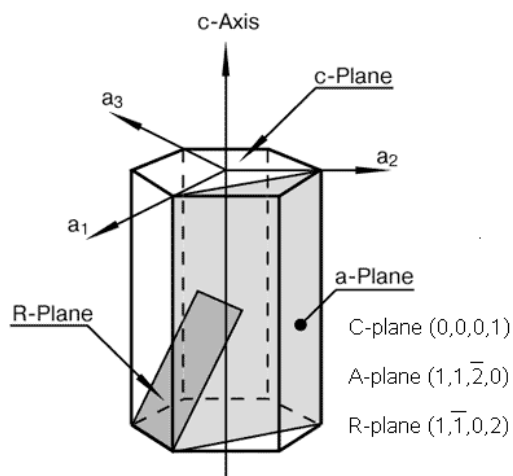


3-axis parallel spring structure fabricated with wire-cutting

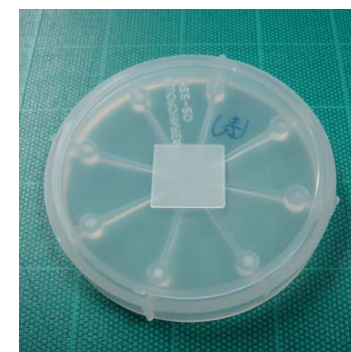
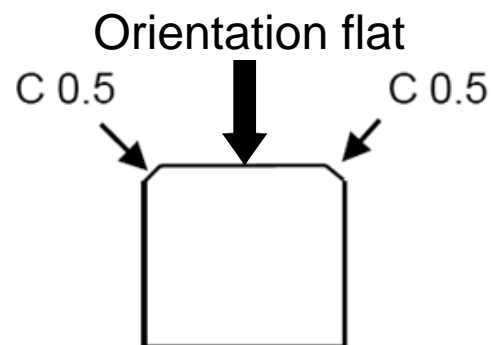


Interferometer optics with double-pass, homodyne detection

Artefacts provided by AIST



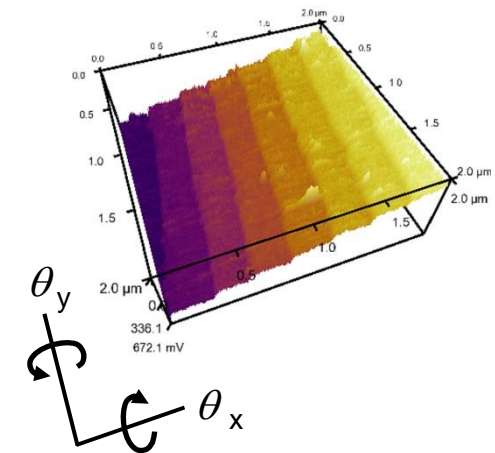
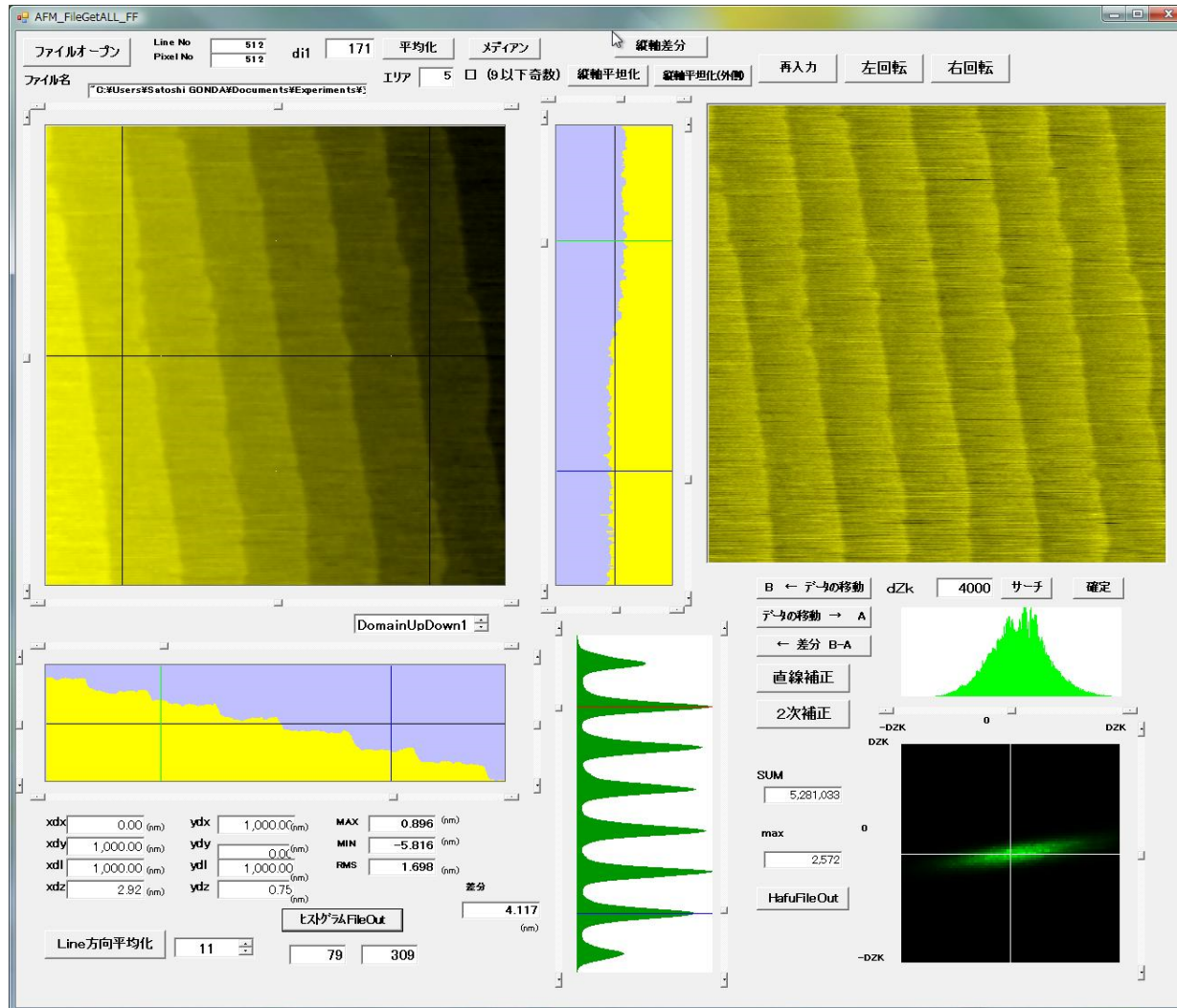
Hexagonal
a=4.758 Å
c=12.991 Å



S/N	Plane orientation	Size (mm)	Expected step height (nm)
S9079.001	c(0001) /OF(11-20)	15x15x0.5t	0.22
S9080.001	a(11-20) /OF(0001)	15x15x0.5t	0.25
S9180.001	r(1-102) /OF(11-20)	15x15x0.5t	0.35

Manufacturer: Shinkosha Co., Ltd.
http://www.shinkosha.com/english/sehin/2_10.html

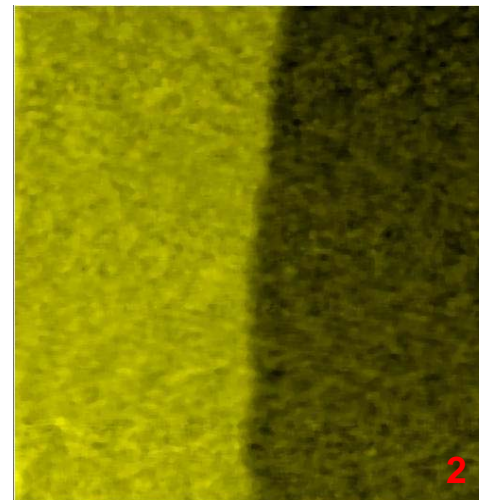
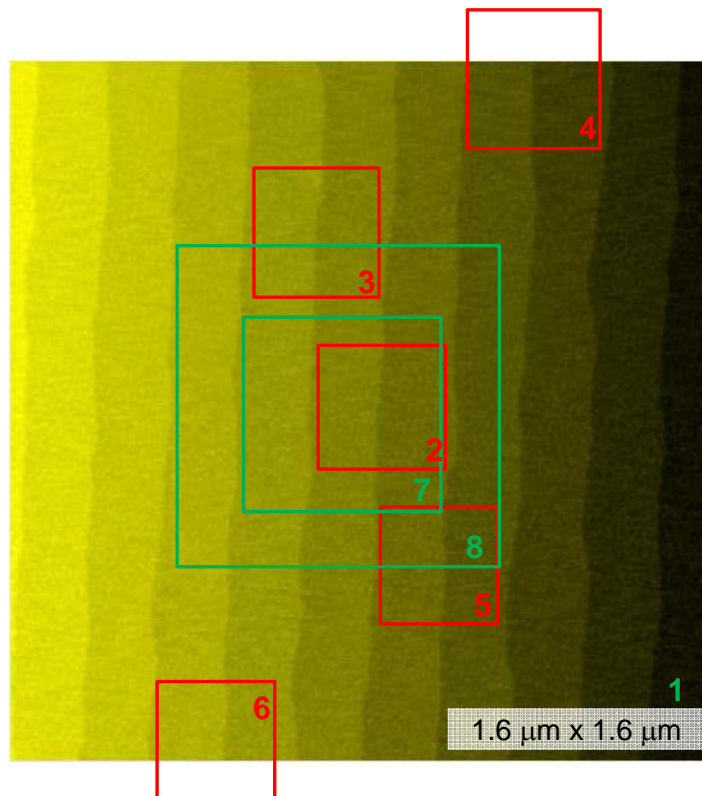
Height calculation based on height histogram method



r-plane ($1\bar{1}02$), nominal height: 0.35 nm

Observations:

- ✓ *Well defined step edges*
- ✓ *Approximately 160 nm in average of terrace width, relatively narrow for data analysis by height histogram.*
- ✓ *Low roughness on the terraces.*



r-plane (1-102)

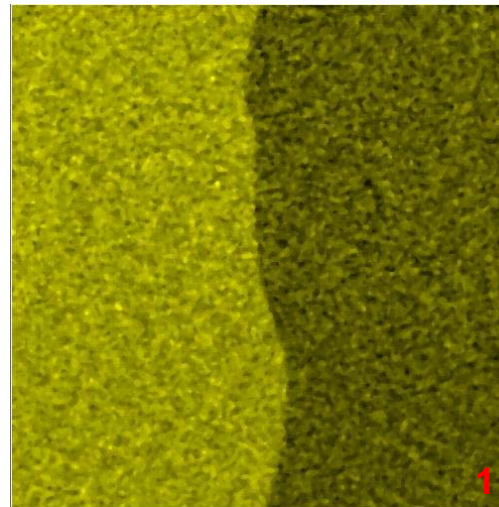
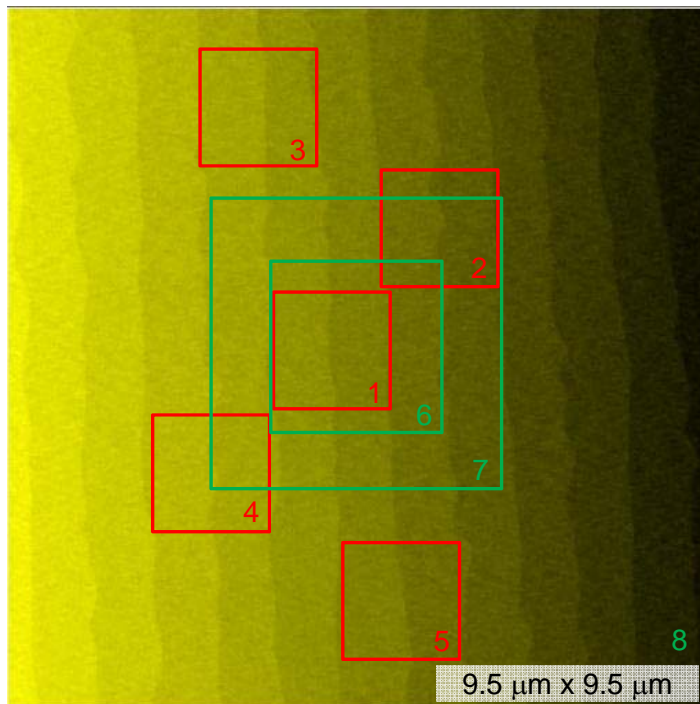
Location	Mean (nm)	Deviation (nm)
1	0.311	-0.016
2	0.331	0.005
3	0.367	0.041
4	0.340	0.014
5	0.323	-0.004
6	0.336	0.010
7	0.332	0.006
8	0.272	-0.055
Average / σ	0.327	0.027

0.327 nm @NMIJ
0.357 nm @NIST

c-plane (0001), nominal height: 0.22 nm

Observations:

- ✓ *Well defined step edges*
- ✓ *Approximately 790 nm in average of terrace width, wide enough for data analysis by height histogram.*
- ✓ *Low roughness on the terraces.*



c-plane (0001)

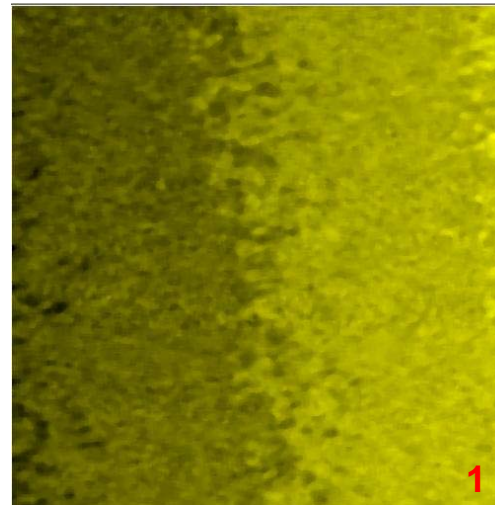
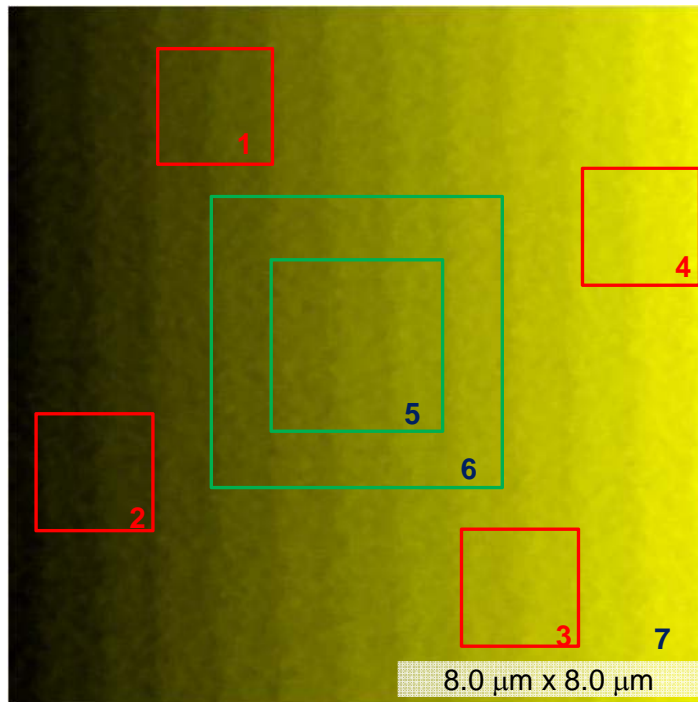
Location	Mean (nm)	Deviation (nm)
1	0.210	-0.001
2	0.224	0.013
3	0.224	0.013
4	0.218	0.007
5	0.198	-0.013
6	0.186	-0.025
7	0.220	0.009
8	0.210	-0.001
Average / σ	0.211	0.013

0.211 nm @NMIJ
0.228 nm @NIST

a-plane $(11\bar{2}0)$, nominal height: 0.25 nm

Observations:

- ✓ *Ambiguous step edges, making linear fit on the terrace difficult*
- ✓ *Approximately 650 nm in average of terrace width, wide enough for data analysis by height histogram.*
- ✓ *High roughness on the terraces.*



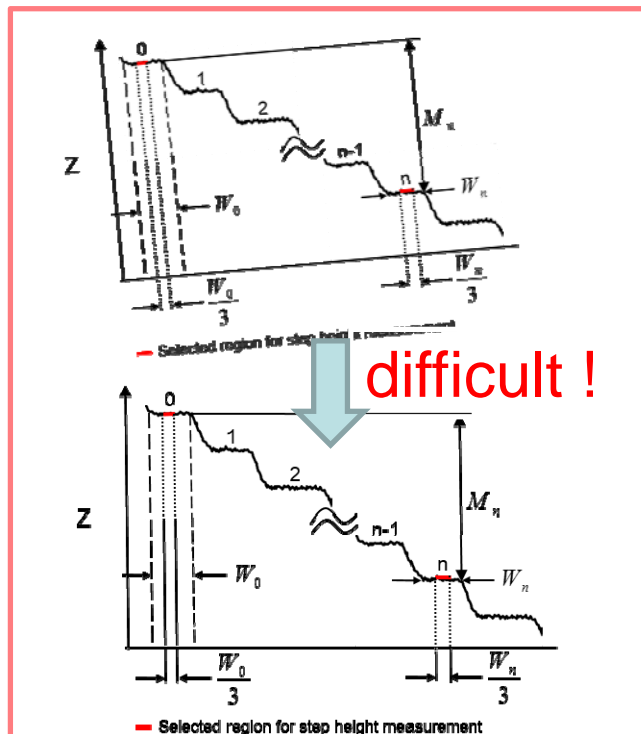
a-plane (11-20)

Location	Mean (nm)	Deviation (nm)
1	0.241	-0.007
2	0.246	-0.002
3	0.253	0.005
4	0.270	0.022
5	0.238	-0.010
6	0.240	-0.008
7	0.245	-0.003
Average / σ	0.248	0.011

0.248 nm @NMIJ

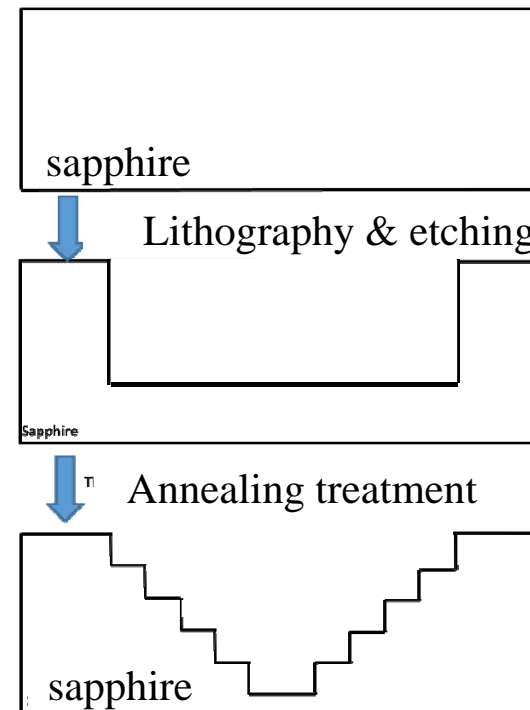
Steps at both sides

- However, stairs-like steps structure is not easy to be measured precisely due to the difficulty of good plane fit treatment on such single base line structure.



difficult !

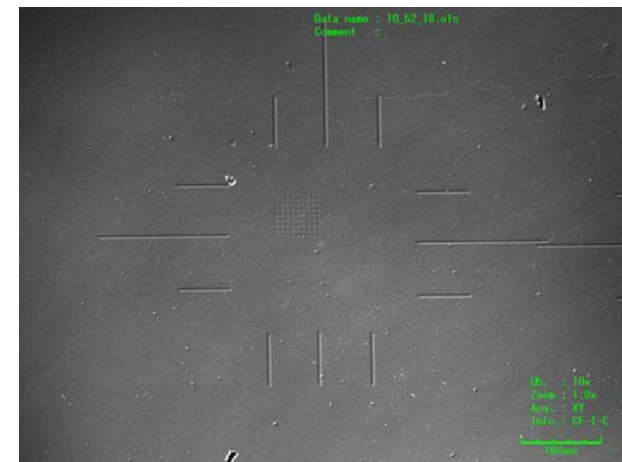
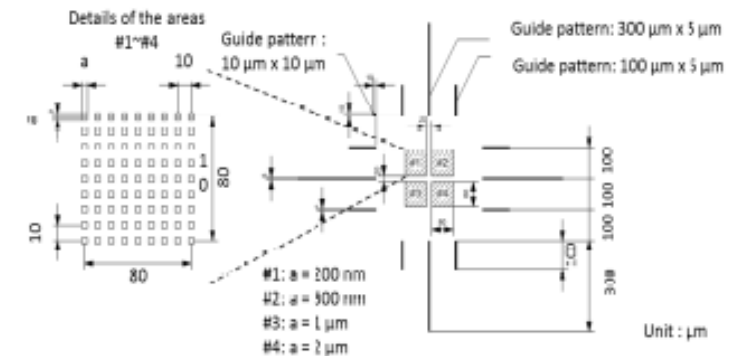
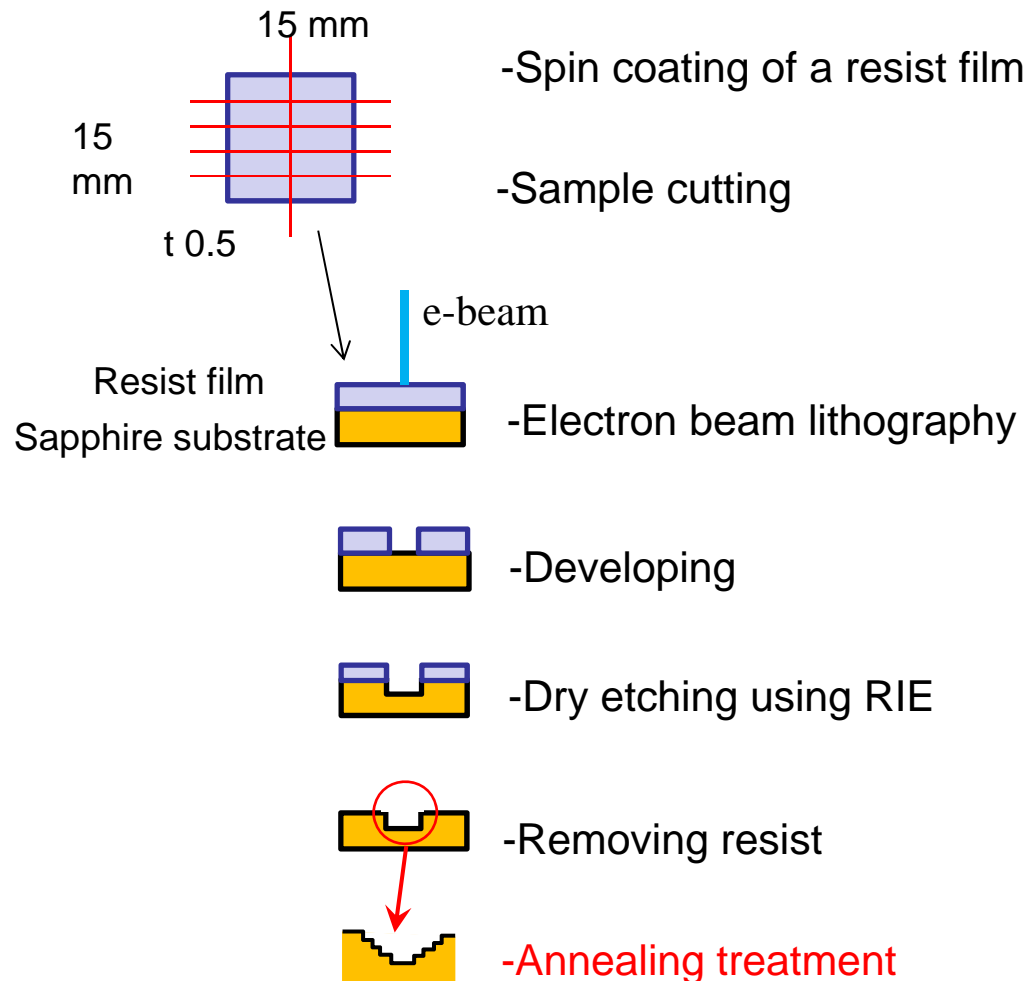
solution



Normal steps & terraces

Steps at both ends

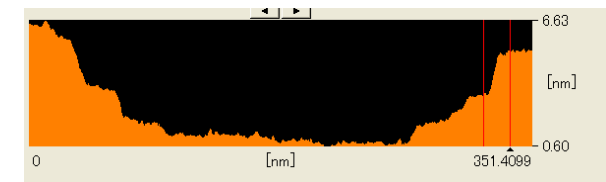
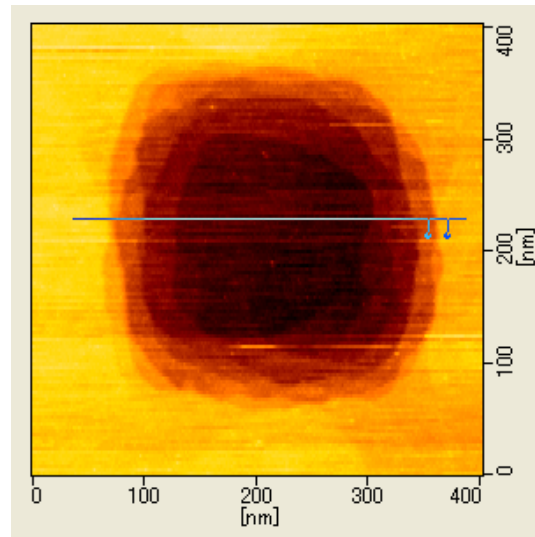
Experimental -sample preparation recipe-



An optical microscope image of
patterned area

Preliminary results

- The sidewall tilt angle decreased from 2.2° to 0.8°
- After annealing, steps are observed on the sidewall of hole structure



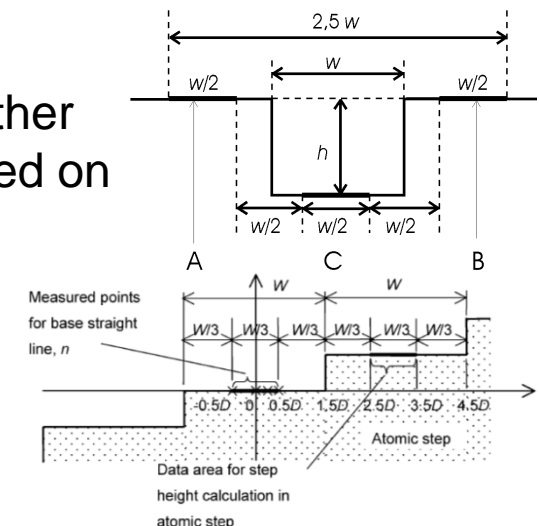
step structure

Annealing temperature	1050 °C	1050 °C	1150 °C
Annealing time	1 hr	10 hrs	10 hrs
sidewall tilt angle	2.2°	2.2°	0.8°

Challenges to spread users

- Alternative material ?
 - ✓ Only Si(111) ?
 - ✓ SiC, Al₂O₃, Diamond, and other crystals, which might be used in special conditions.
- More variation of the size of step height ?
 - ✓ Acquire latest information of larger size > 1 nm by SiC, C₆₀...
- More suitable, or alternative algorithms for the calculation of step height ?
 - ✓ Procedures based on the standards JEITA EM3505 and ASTM Int'l E2530-06. Others ?
- Recommendation of bilaterally symmetric step structure rather than normal stepwise structure. Stable profile analysis based on ISO 5436 is applicable.
- Consistency between the values based on XRD and AFM
 - ✓ Possible RRT under VAMAS or CCL (NMIs)
 - ✓ Length-traceable measurements by NMIs

(Si(111) has already been done by NIST)



Considerations toward NWIP

- The time is right to proceed ...
 - ✓ Increasing demand for the size inspection of 3D nanodevices
 - ✓ Support development of nano-roughness measurements
 - ✓ Encourage material suppliers to develop more various sorts of atomic step & terrace structure for reference materials
- The standard will have following possible sections;
 - ✓ terms and definitions
 - ✓ description of atom step-based reference samples
 - ✓ measurement condition and data acquisition
 - ✓ procedures for image processing and step height analysis
 - ✓ uncertainty consideration
 - ✓ reporting
- It will be worth to establish an ISO standard, compiling existing local standards and upgrading with latest information.

Summary

We have achieved;

- Development of a **metrological AFM** for precision measurements of **atomic step height at single crystal surface**
- Validation of step height for step & lattice comparing with the **lattice constant** which would be a reference value in the standard
- **Bilateral comparison between NIST and NMIJ** with a preliminary good looking results.
- Technical presentation aiming a new work item proposal at **ISO / TC 201 / SC 9**

We will continue;

- To **collaborate closely** in next several years toward next stage of standardization
- To conclude bilateral comparison between NIST and NMIJ as **one of evidences for standardization**
- To draft a preliminary **guideline** for the calibration procedure for height sensitivity of an AFM using atomic steps

Acknowledgement

Kentaro SUGAWARA, Ichiko MISUMI, Kazuto KINOSHITA (NMIJ/AIST)
Bo-Ching He (guest researcher from CMS/ITRI)

This work has been supported by METI.

Thank you for your kind attention !