The current status and perspective on low force metrology in Korea

Small force workshop

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Korea Research Institute of Standards and Science

IMEKO

IMEKO 2010 TC3 Conference

Metrology in modern context

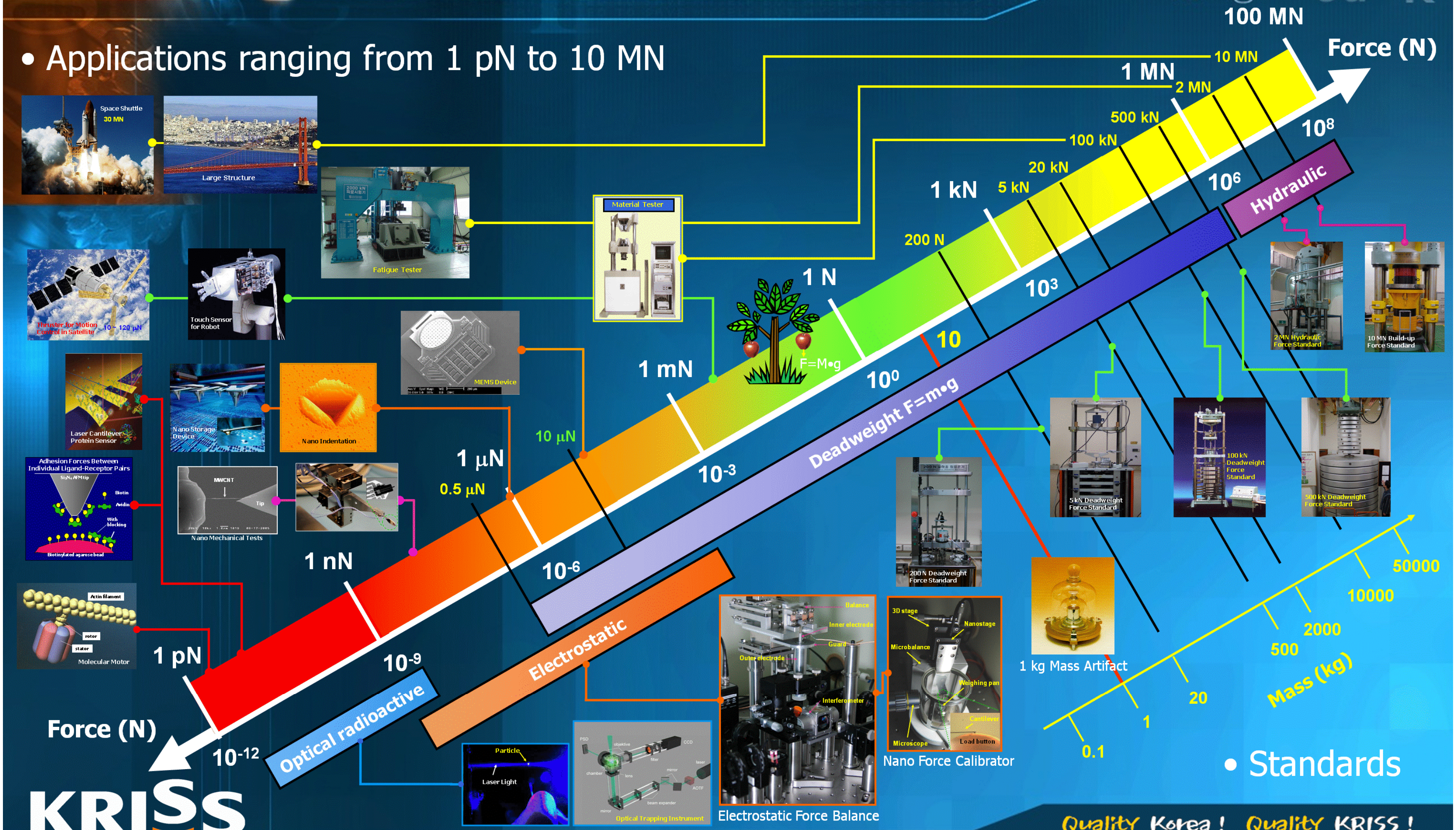
Pattaya, Thailand, Nov. 21-25, 2010

KRISS

- Outline -

Force Applications and Standards

- Applications ranging from 1 pN to 10 MN

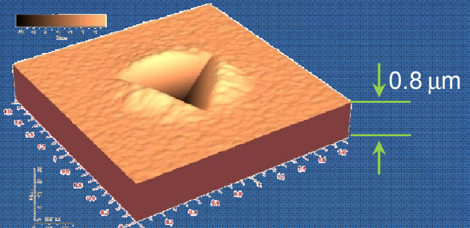


- Standards

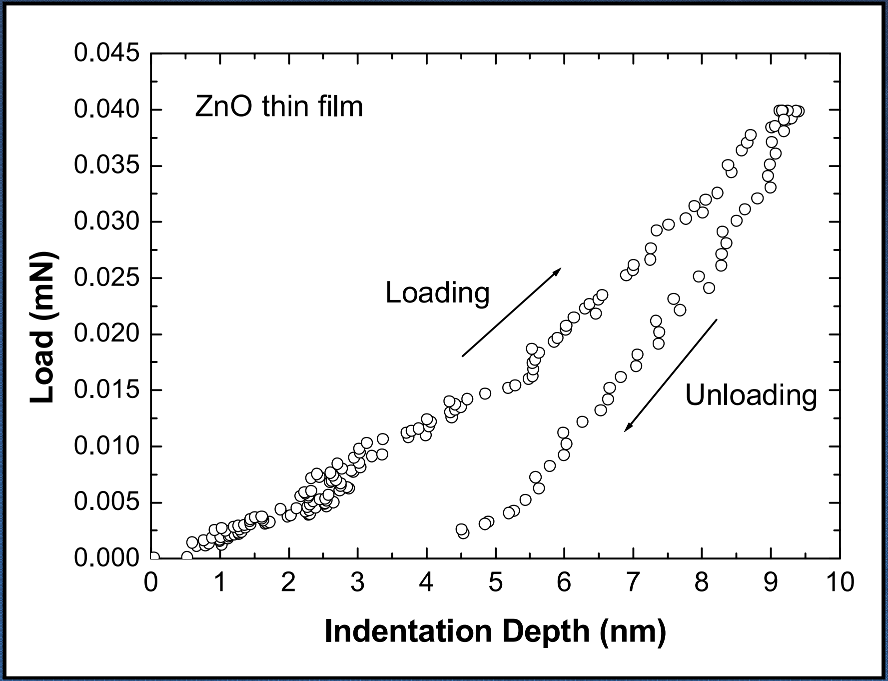
Micro- or nano mechanical testing

Nanoindentation experiments in KRISS

Nanoindenter (MTS)



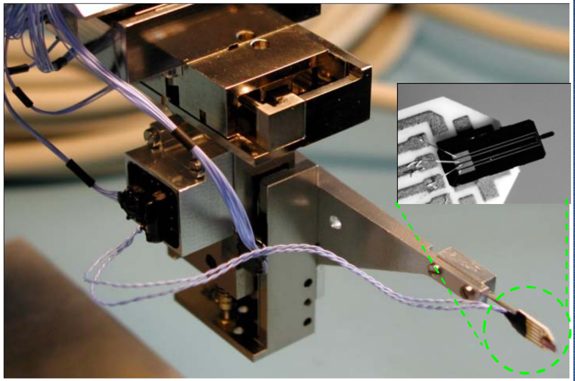
Specimen (ZnO thin film, 0.8 µm)



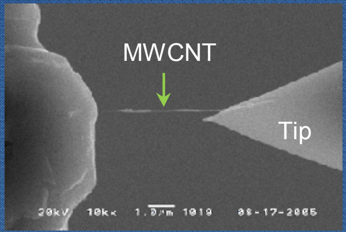
By courtesy of Dr. Jun-Hee Hahn

Micro- or nano mechanical testing

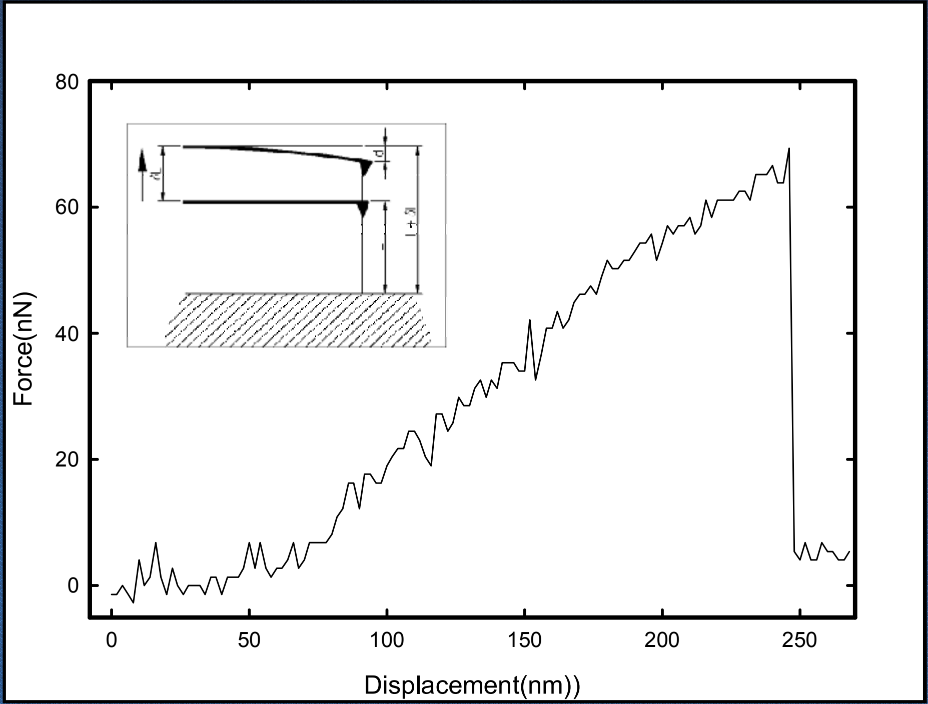
Measurement of tensile properties of carbon nanotube in KRISS



Nano-manipulator & force sensor



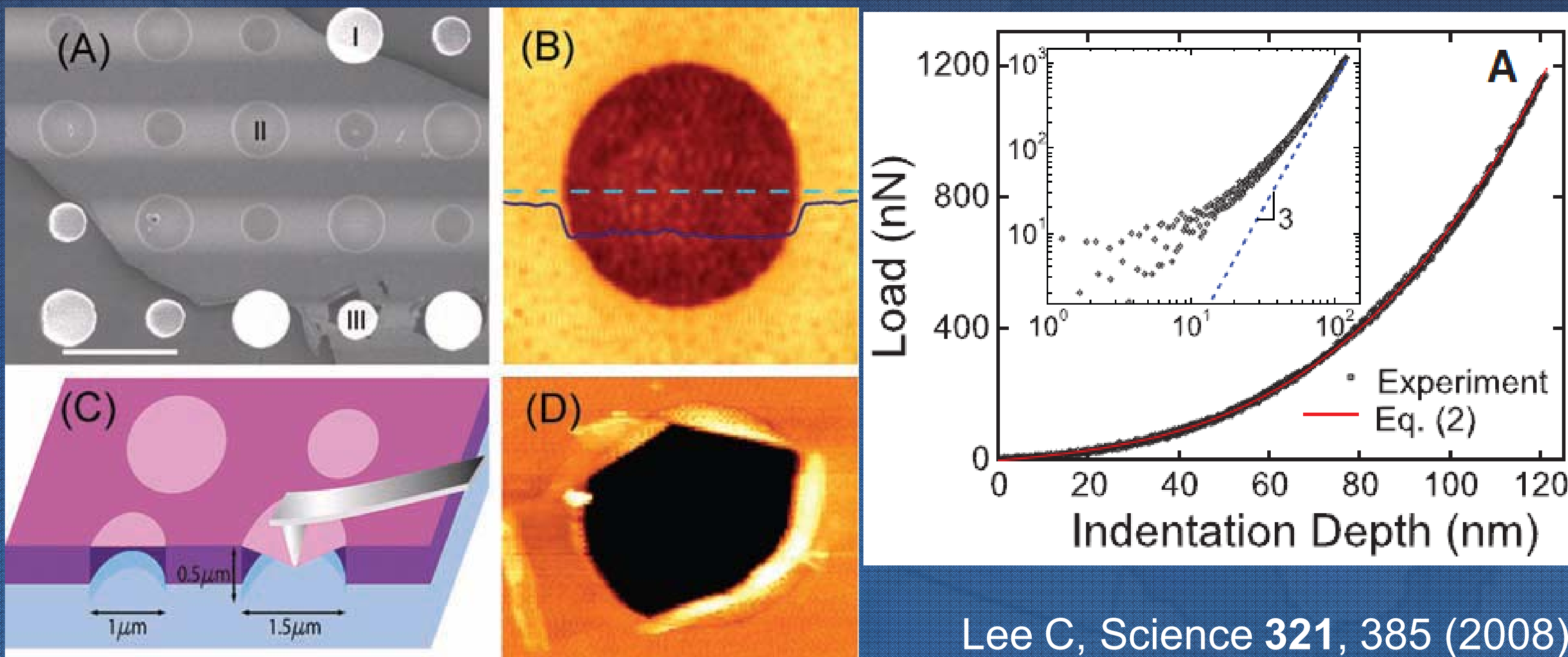
Specimen (MWCNT)



By courtesy of Dr. Seung-Hun Nam

Micro- or nano mechanical testing with AFM

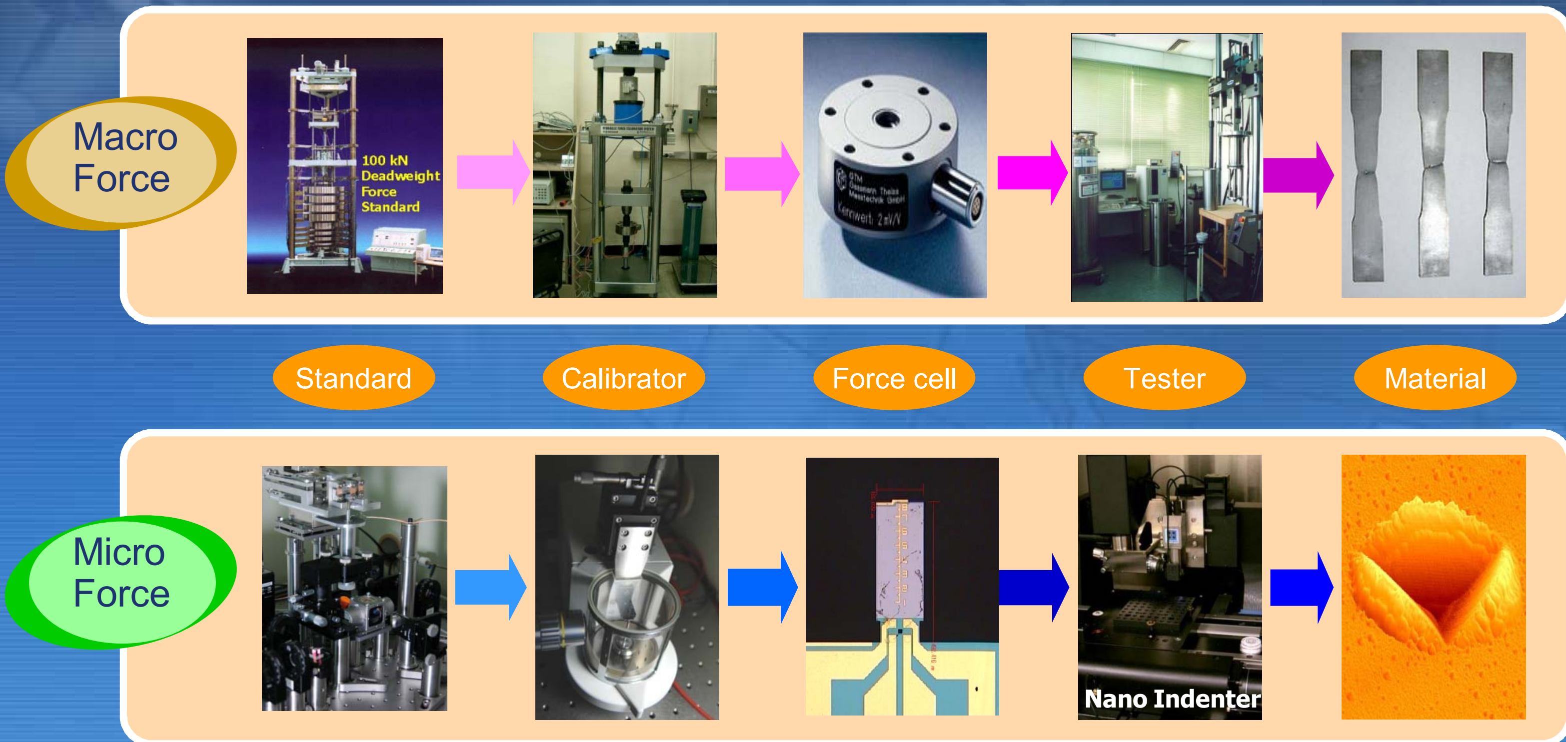
Mechanical testing of graphene



Lee C, Science **321**, 385 (2008)

Traceability

Hierarchy of traceability at macro- and micro-force level

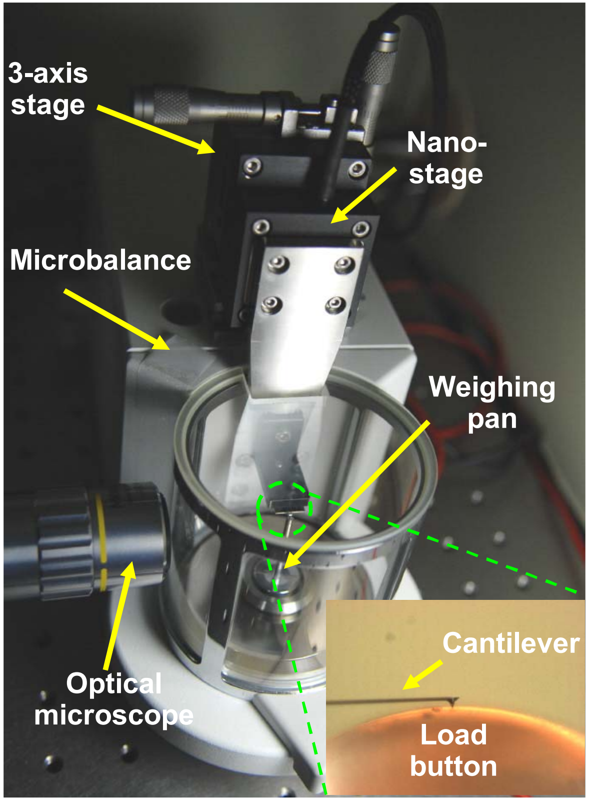


- Outline -

1. Introduction
2. Low force calibration machine
3. Low force sensor
4. International comparison
5. Challenges and Outlook

Low force calibration machine in KRISS

Nano Force Calibrator (NFC)

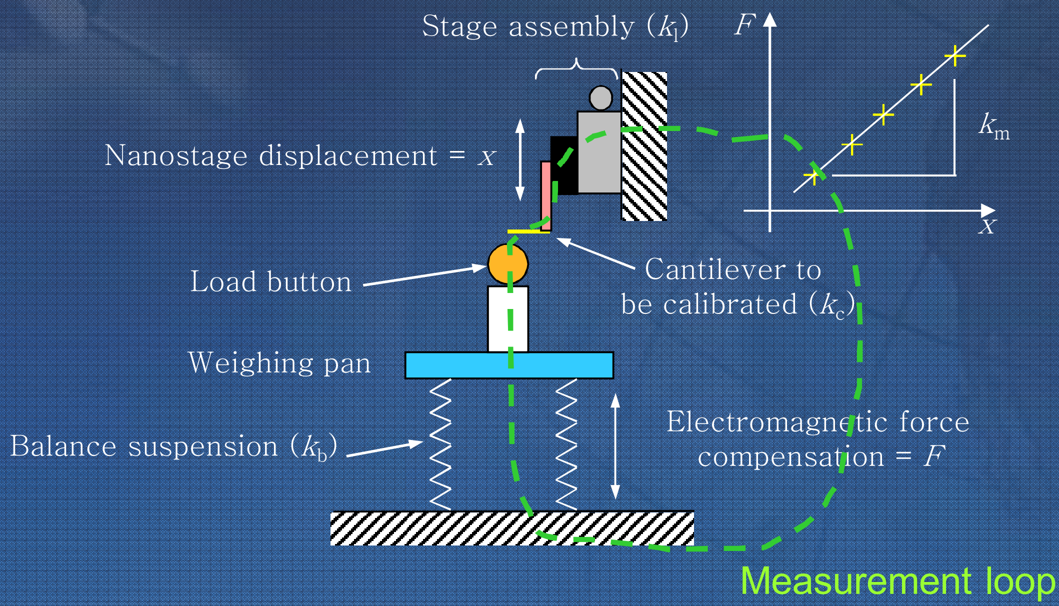


Features

1. Compact and Robust Design
2. Range: (0.5 ~ 20 000) μN
3. CMC: less than 2%(k=2)
4. Traceable to SI
5. Fast and Repeatable Calibration
6. Easy and Automated Operation

Low force calibration machine in KRISS – Cont'd

Calibration principle



$$\frac{1}{k_c} = \frac{1}{k_m} - \frac{1}{k_b} - \frac{1}{k_l}$$

$k_b = (1585 \pm 100)\ \mathrm{N\ m^{-1}}$

For a 100 N m^{-1} cantilever, approximately 6% correction is made for the measured stiffness

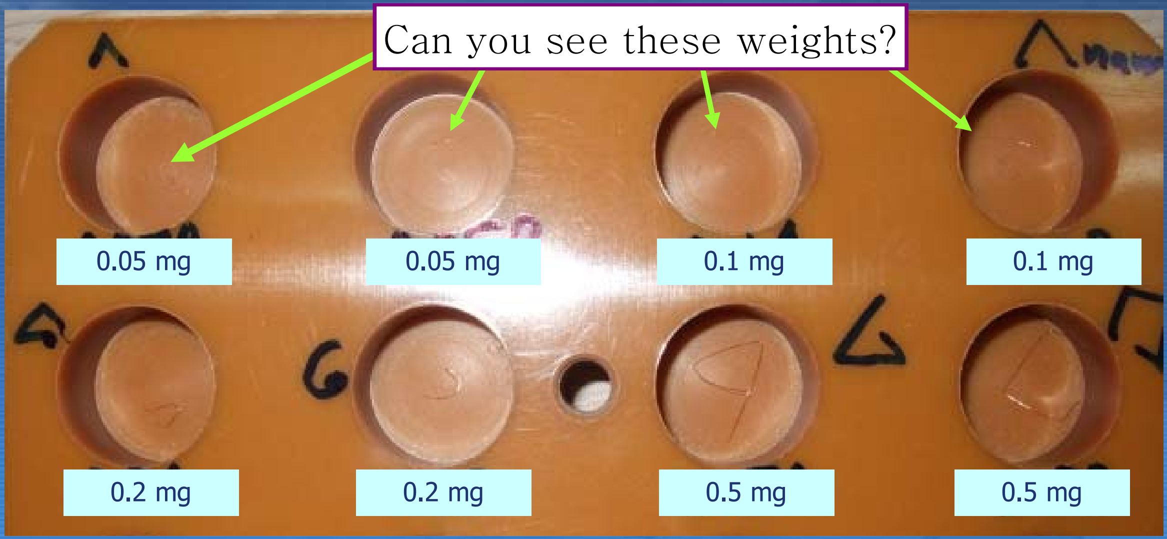
Low force calibration machine in KRISS – Cont'd

Micro weight set for force traceability

- Force range required to calibrate cantilevers: (0.5 ~ 500) μN
- Corresponding mass: 0.05 mg ~ 10 mg
- Usually, 1 mg is the minimum available mass
- From 0.05 mg ~ 0.5 mg, we made weights using 40 μm-gold wire
- Calibrate these weights by comparison and solving weighing equation

Calibration results of the micro weight set

Mass Indication (mg)	1	0.5	0.2	0.2	0.1	0.05	0.05
Calibrated value (mg)	1.0000	0.4974	0.1988	0.1988	0.1003	0.0503	0.0510
Uncertainty, k=1, μg	0.5	0.3	0.2	0.2	0.2	0.2	0.2
Relative Uncertainty, %	0.05%	0.06%	0.1%	0.1%	0.2%	0.4%	0.4%

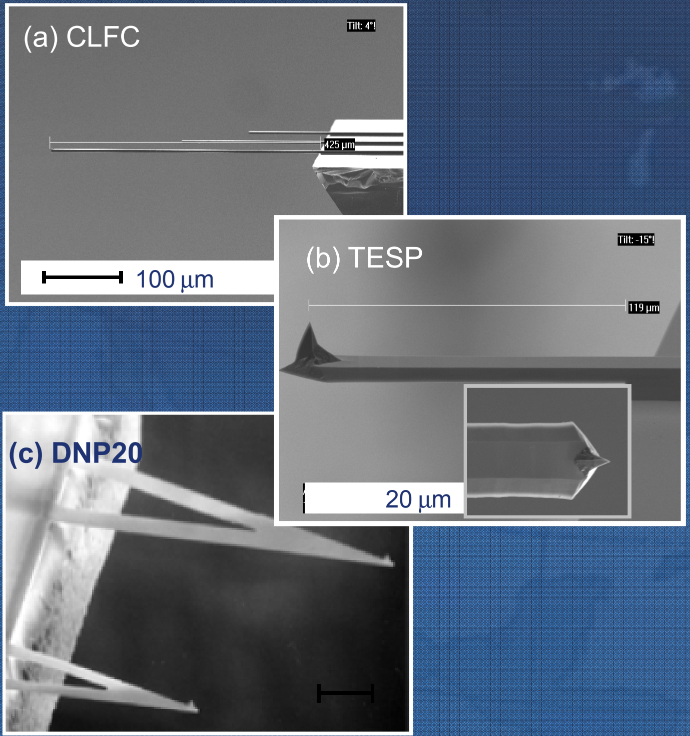


Can you see these weights?
0.05 mg
0.05 mg
0.1 mg
0.1 mg
0.2 mg
0.2 mg
0.5 mg
0.5 mg

Calibration Capability of Nano Force Calibrator

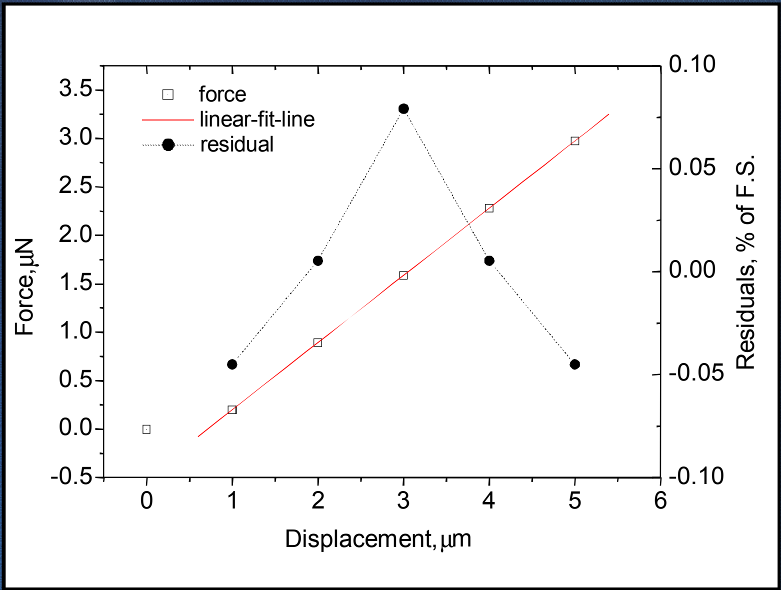
AFM Cantilever Calibration with NFC

Features	DNP20	CLFC	TESP
Operation mode	Contact	Reference	Non-contact
Spring Constant (Nominal)	0.06 N m^{-1}	0.711 N m^{-1}	42 N m^{-1}
Length (Nominal)	196 μm	450 μm	125 μm
Width (Nominal)	23 μm	35 μm	30 μm
Thickness (Nominal)	0.6 μm	4 μm	4 μm
Material	Silicon nitride	n-doped silicon	
Manufacturer	Veeco Inc.		

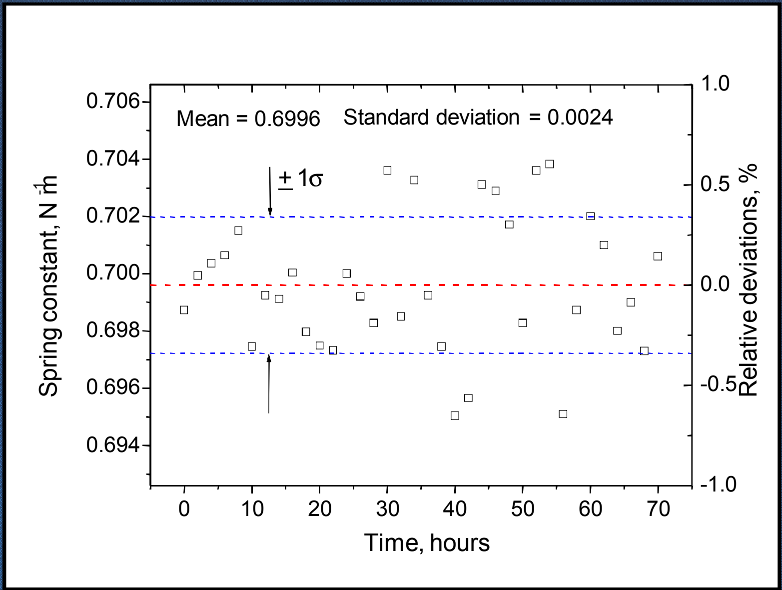


Calibration Capability of NFC – Cont'd

Calibration results – CLFC cantilever, $k_{nom} = 0.711\ N\ m^{-1}$



Force-displacement curve

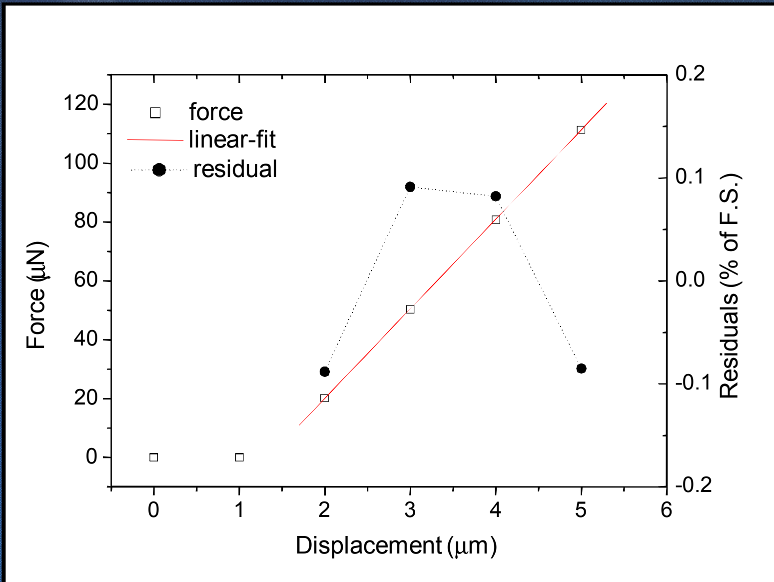


Scatter of measured spring constants

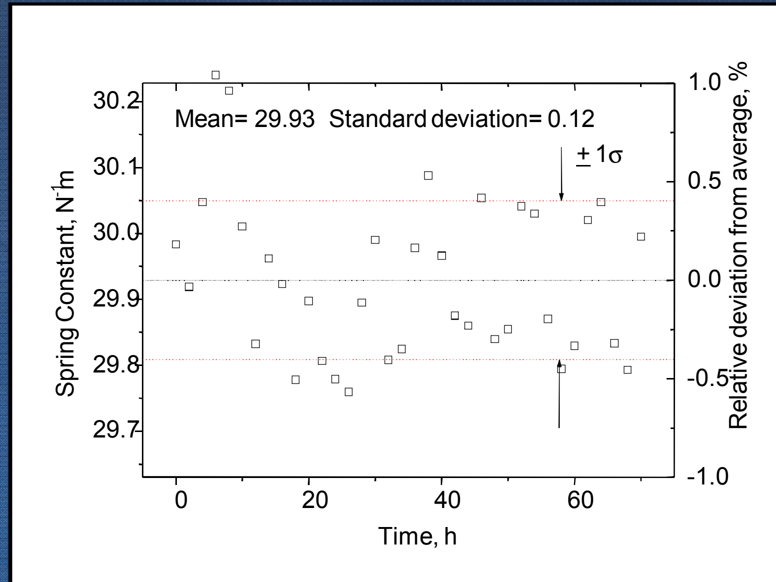
- The relationship between force and displacement is quite linear: residuals < ±0.1%
- Long term repeatability is good: 1 standard deviation < 0.5%

Calibration Capability of NFC – Cont'd

Calibration results – TESP cantilever, k_{nom} = 42 N m^{-1}



Force-displacement curve

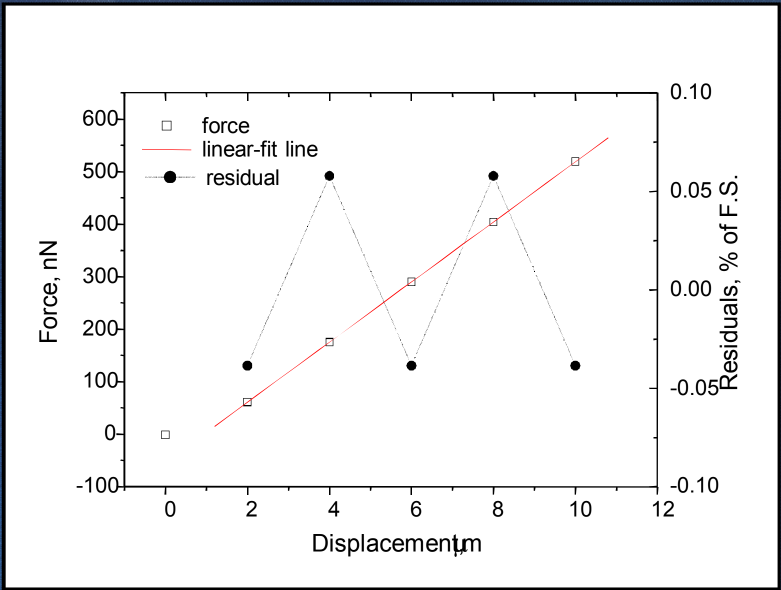


Scatter of measured spring constants

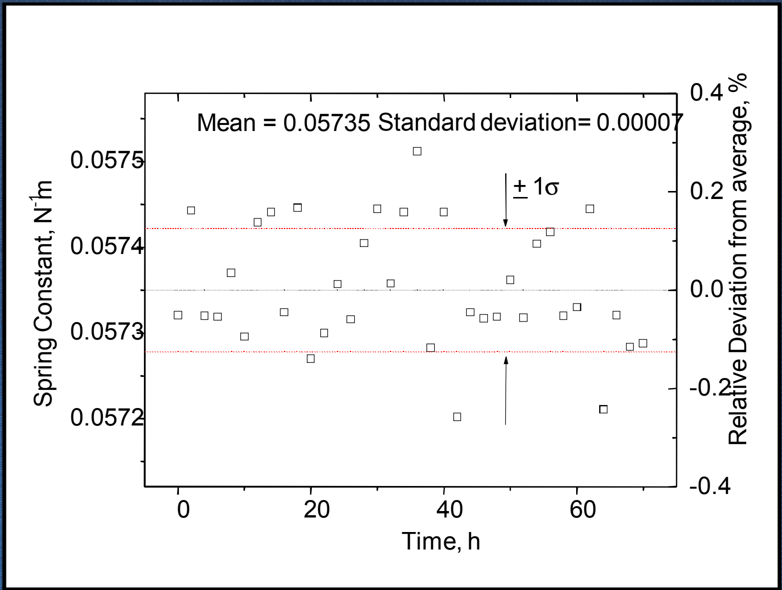
- ➔ The relationship between force and displacement is quite linear: residuals < ±0.2%
- ➔ Long term repeatability is good: 1 standard deviation < 0.5%

Calibration Capability of NFC – Cont'd

Calibration results – DNP20 cantilever, k_{nom} = 0.06 N m^{-1}



Force-displacement curve



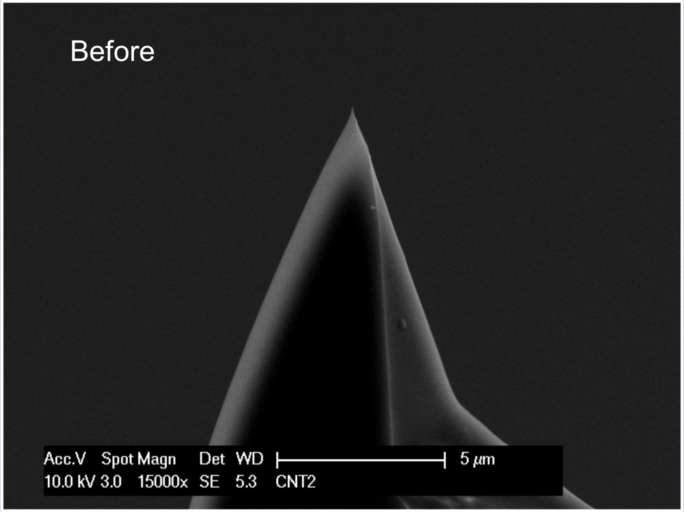
Scatter of measured spring constants

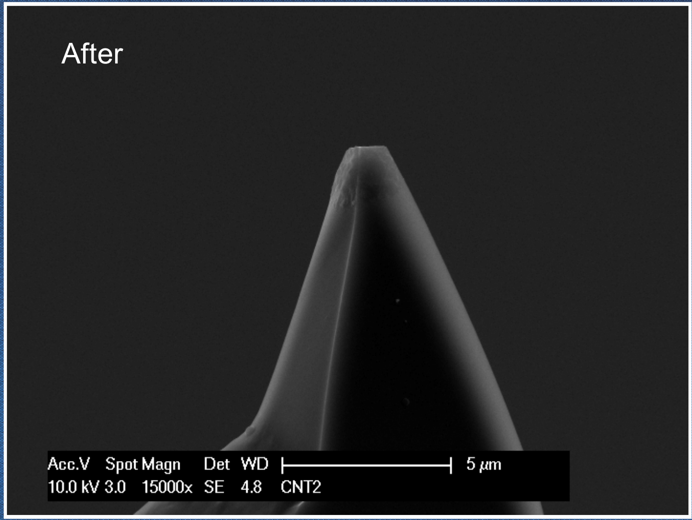
- ➔ The relationship between force and displacement is quite linear: residuals < ±0.1%
- ➔ Long term repeatability is good: 1 standard deviation < 0.2%

Calibration Capability of NFC – Cont'd

Tip damage problem after calibration with NFC

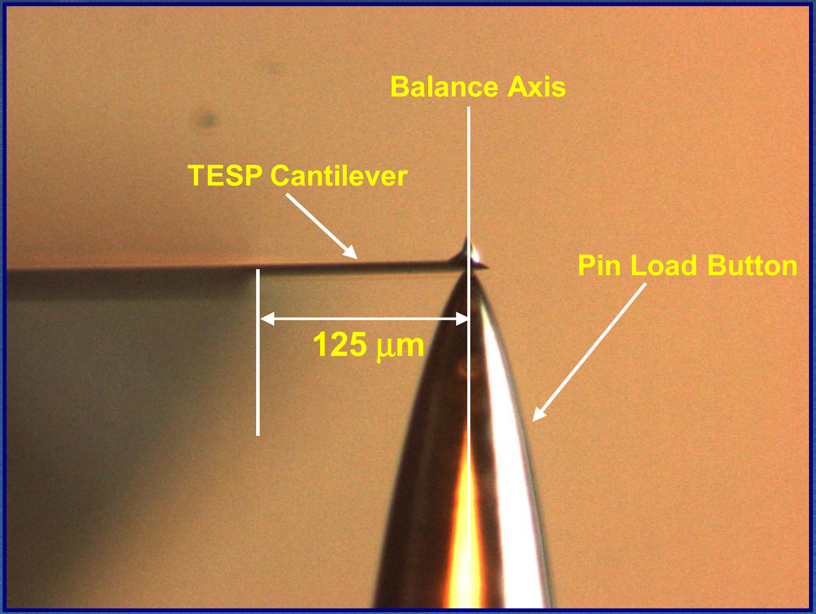
SEM image of the tip of TESP cantilever before and after calibration

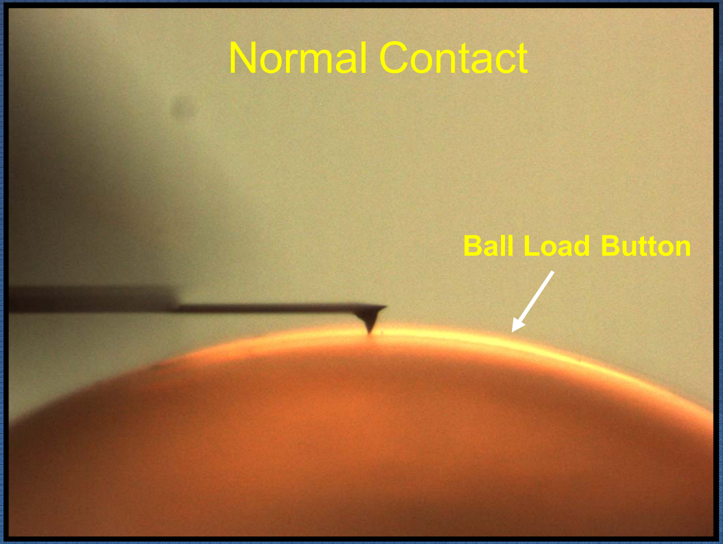




Calibration Capability of NFC – Cont'd

Upside-down contact configuration





$k = (30.13 \pm 0.10)\ \mathrm{N\ m^{-1}}$

$k = (29.93 \pm 0.12)\ \mathrm{N\ m^{-1}}$

- ➔ These results agree with each other to less than 1%
- ➔ Discrepancy between two results is mainly due to the positioning error of 0.3 μm

Calibration Capability of NFC – Cont'd

Uncertainty Budget

Balance was tested using calibrated weights that are traceable to national mass standard of 1 kg weight in KRISS.

Nanostage was tested using a Zygo™ laser interferometer, of which the wavelength of a laser is calibrated in a traceable manner

We can say calibration is traceable to SI and relative uncertainty is conservatively 1%, including unknown uncertainty sources, which is the smallest uncertainty yet attained by any method

Quantity or Influence (Q)		Relative standard uncertainty ($u = dQ/Q$)			Uncertainty type
		CLFC	TESP	DNP20	
Measured value (Nm^{-1})		0.6996	29.93	0.0574	
Repeatability, $u(k_r)$		0.34%	0.4%	0.12%	A
Force	Correction, C_f	-0.01%	-0.004%	1%	B
	$u(f)$	0.05%	0.008%	0.4%	
Displacement, $u(d)$		0.14%	0.14%	0.14%	B
Balance Stiffness	Correction, C_b	0.057%	1.6%	Negligible	B
	$u(b)$	0.004%	0.1%	Negligible	
Non-linearity, $u(n)$		0.1%	0.2%	0.1%	B
Orientation, $u(o)$		0.2%	0.2%	0.2%	B
Contact position, $u(p)$		0.11%	-	-	
Combined relative standard uncertainty, u_c		0.44%	0.52%	0.57%	
Corrected value with combined standard uncertainty ($N m^{-1}$)		0.700 ± 0.003	30.41 ± 0.16	0.0580 ± 0.0003	

Future Plan on Developing a New Balance

Combined electromagnetic and static Force Balance (CFB)

Electromagnetic Force

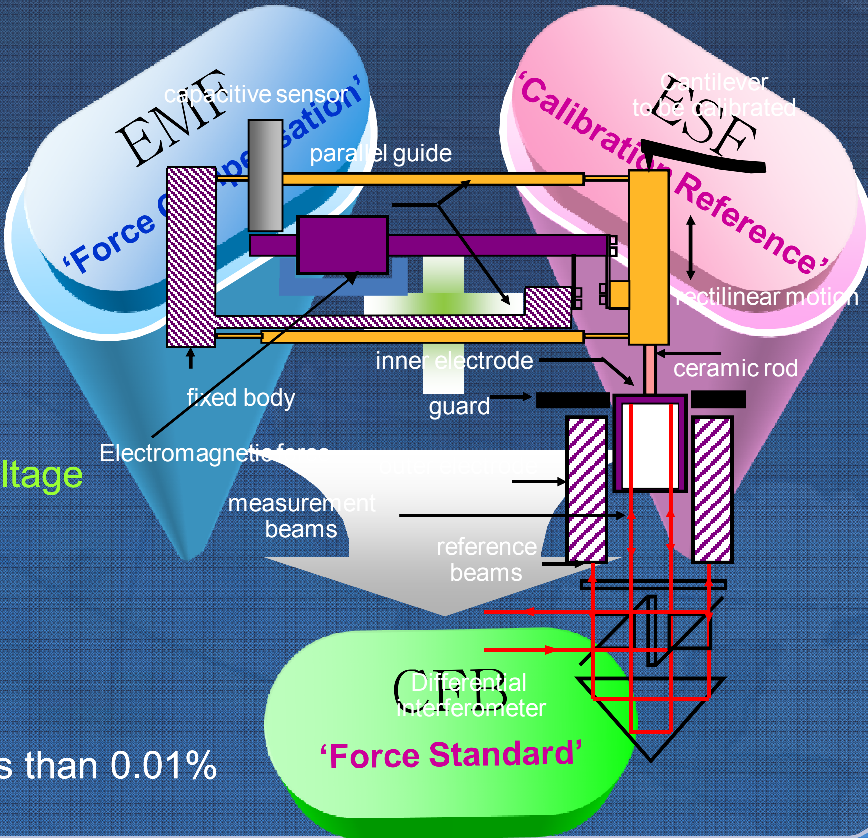
- relatively large
- proportional to the applied current
- difficult to quantify the EMF precisely

Electrostatic Force

- relatively small
- proportional to a square of the applied voltage
- possible to quantify the ESF precisely

Goal

- Range (resolution): 2 mN (20 pN)
- Agreement with 10 mg deadweight to less than 0.01%



- Outline -

1. Introduction
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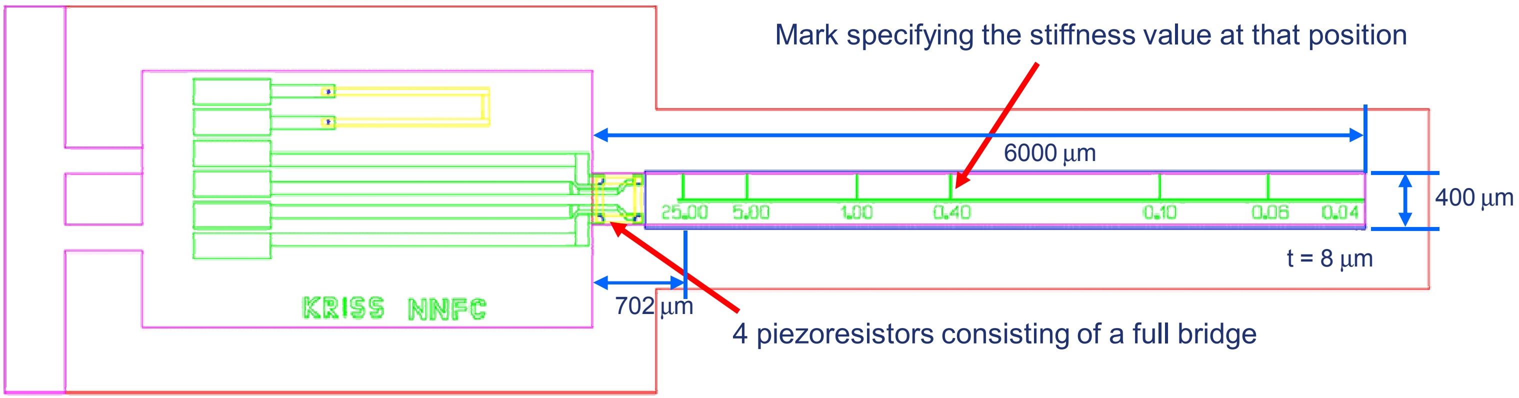
3. Low force sensor
4. International comparison
5. Challenges and Outlook

Low force sensor (transfer standards)

Design Concept

- Adopting cantilever structure: micromachining compatible
- Large dimensions (millimeter level) to reduce positioning error
- Incorporating a ruler on the cantilever
- Calibration range approx. from 0.01 N m^{-1} to 40 N m^{-1}
- Self-detection scheme (on-board piezoresistors)

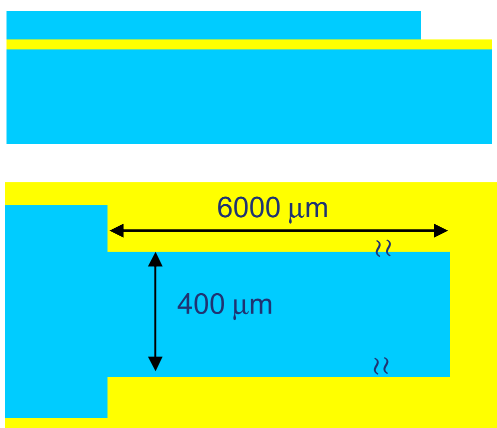


Low force sensor (transfer standards) – Cont'd

Fabrication Process (1)

1. Starting substrate : SOI wafer

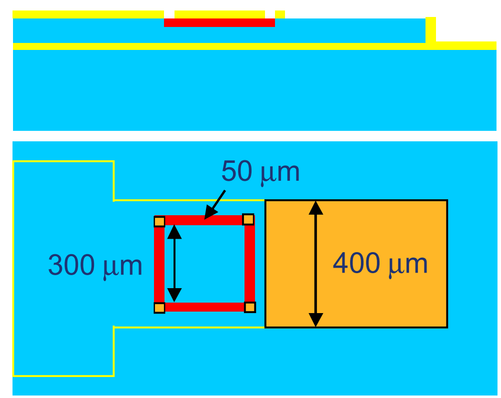
2. Cantilever and leads patterning
3. Cantilever and leads Si etching



4. SiO_2 deposition and Patterning

5. Boron implant for piezoresistor
6. Boron Activation

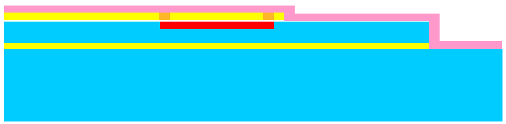
7. SiO_2 deposition & Contact patterning



Low force sensor (transfer standards) – Cont'd

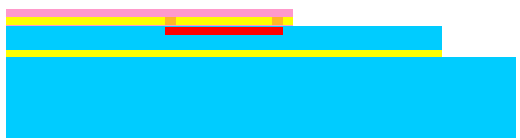
Fabrication Process (2)

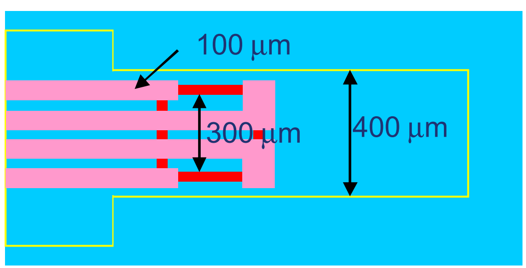
8. Au Deposition



9. PR Patterning for leads

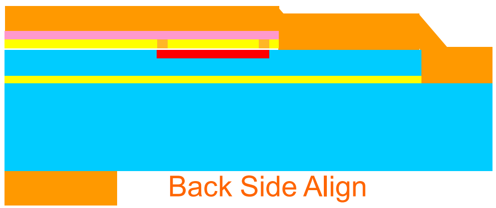
10. Au Etching

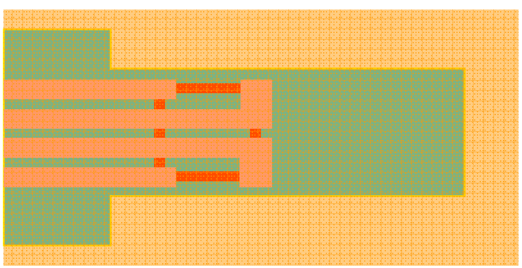




11. Top Side PR Coating for Protect Cantilever

12. PR 10 μm Patterning for Bottom Si Etching



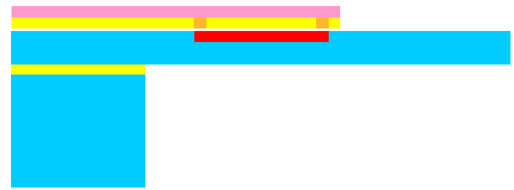


Low force sensor (transfer standards) – Cont'd

Fabrication Process (3)

13. Bottom Si Etching by Deep RIE

15. PR Removal



14. Buried SiO2 Removal by Wet Etching

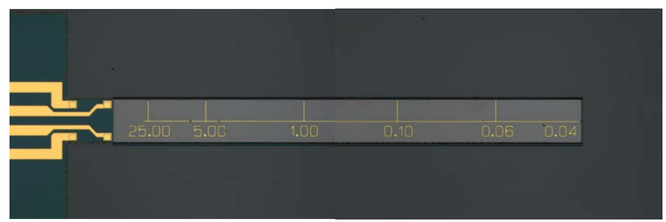
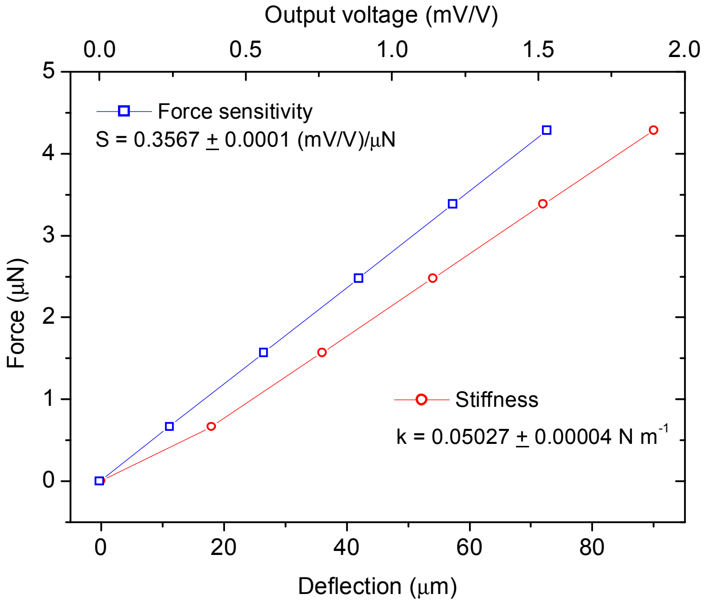
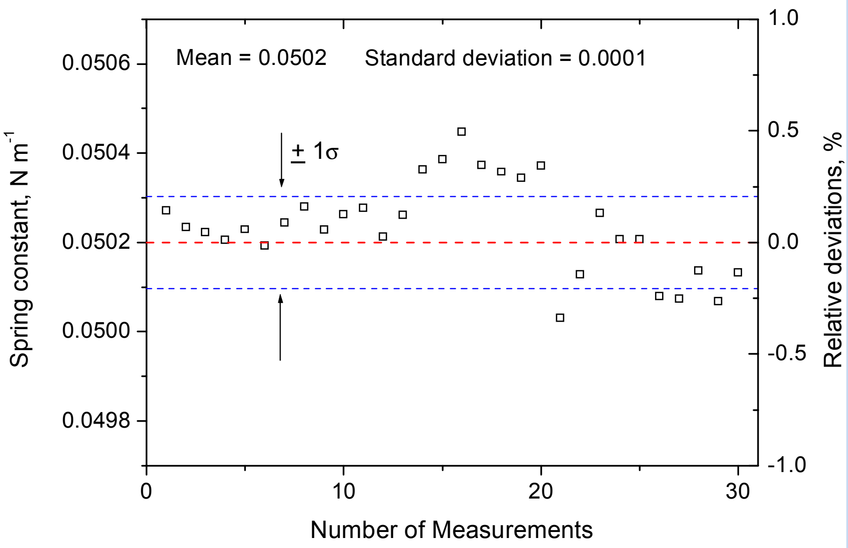


Photo of the fabricated cantilever

Low force sensor (transfer standards) – Cont'd

Sensor test with NFC

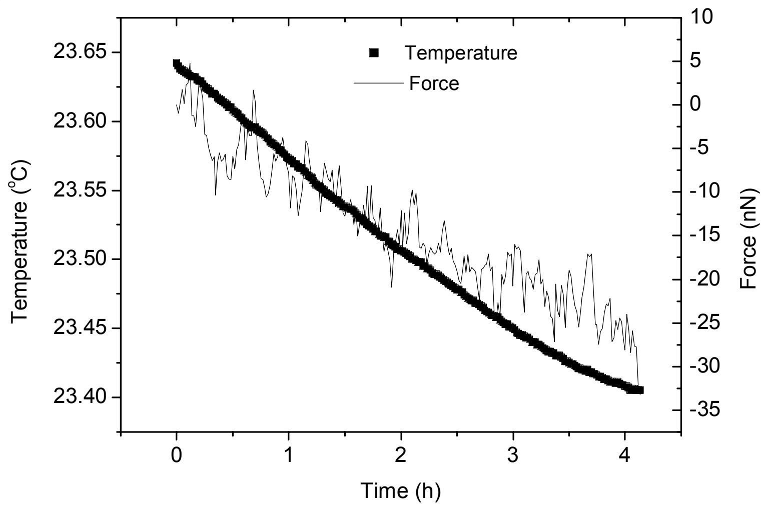


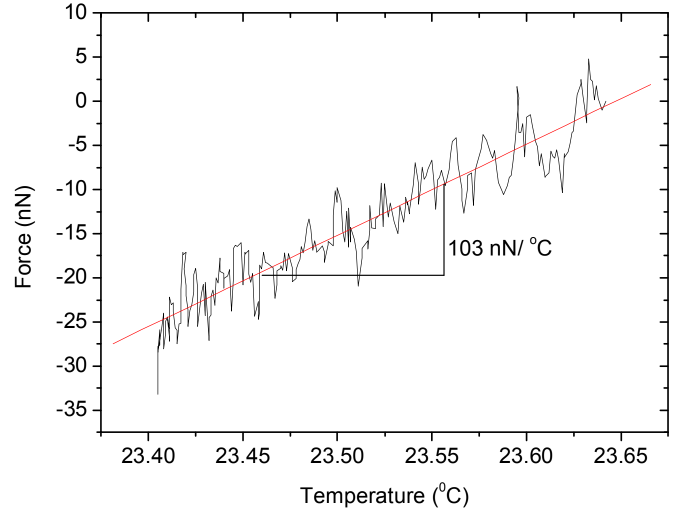


- Force-deflection-voltage relationships are quite linear
- Calibration is repeatable: standard deviation < 0.3%

Low force sensor (transfer standards) – Cont'd

Temperature effect



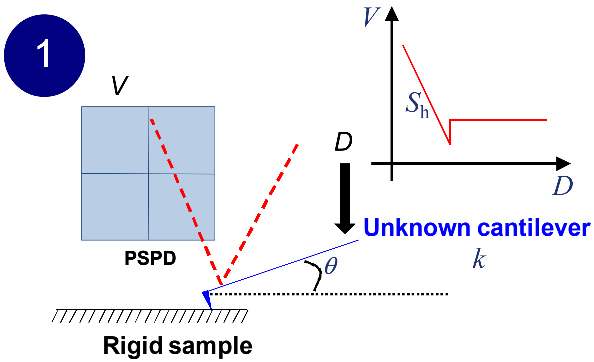


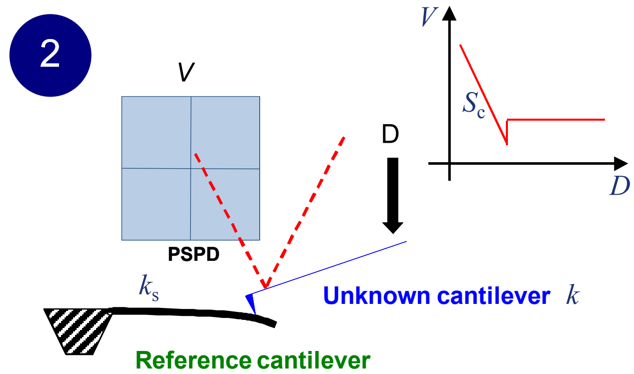
- Voltage output has a dependency on the temperature due to incomplete match of resistors; temperature coefficient $\cong$ 100 nN/K
- Further compensation is possible using a dummy on-board resistor
- Noise level approximately 10 nN

Dissemination to AFM through low force sensors

Cantilever – On – Cantilever Methods

- Principle





$$k = k_s \frac{S_h - S_c}{S_c} \times \cos^2\theta$$

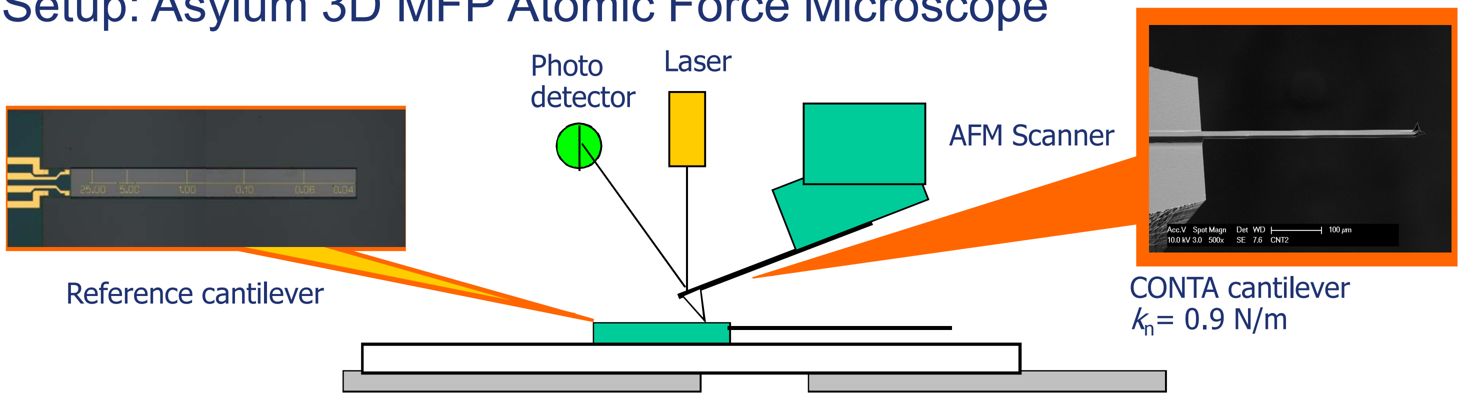
Matching condition: $0.3\,k_s < k < 3.0\,k_s$

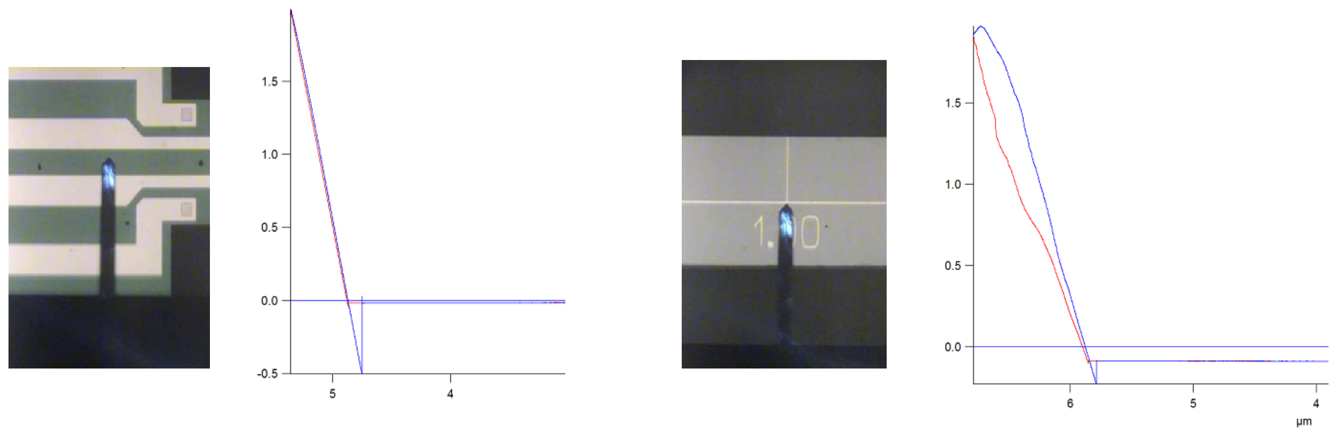
- SI-traceable calibration possible through a calibrated 'Reference' cantilever
- For calibration accuracy, the matching condition should be satisfied
- Accuracy is limited by incomplete positioning and contact mechanics

Dissemination to AFM – Cont'd

Experimental Details

- Setup: Asylum 3D MFP Atomic Force Microscope





Dissemination to AFM – Cont'd

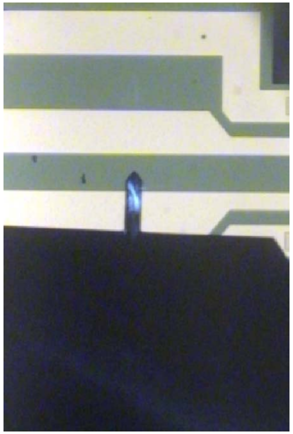
Experimental Results – CONTA Cantilever

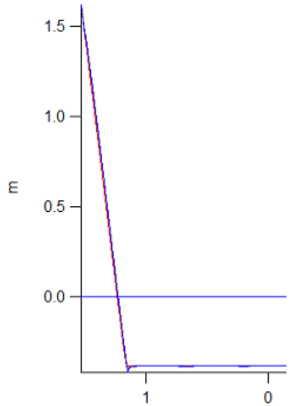
- 10 slopes obtained for each step
- Average two slopes (one is extension and the other is retraction slope)
- F-D curves shows some hysteresis and stick-slip
- All CONTA cantilevers were calibrated with NFC

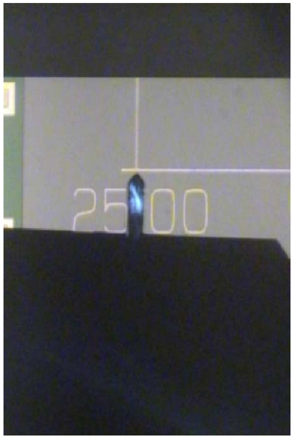
Sample #	Nominal k (N m^{-1})	COC method	COC Std (%)	NFC Calibration	NFC Std (%)	Discrepancy (%)
1	0.9	0.646	4.0	0.683	0.8	-5
2	0.9	0.588	3.3	0.604	0.9	-3
3	0.9	0.665	5.0	0.669	0.9	-1
4	0.9	0.704	5.1	0.609	1.6	16
5	0.9	0.774	3.9	0.758	0.8	2

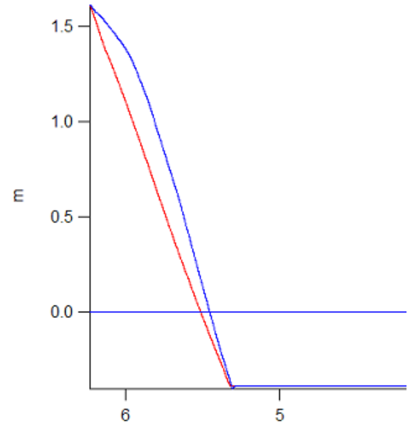
Dissemination to AFM – Cont'd

Experimental Results – TESP Cantilever





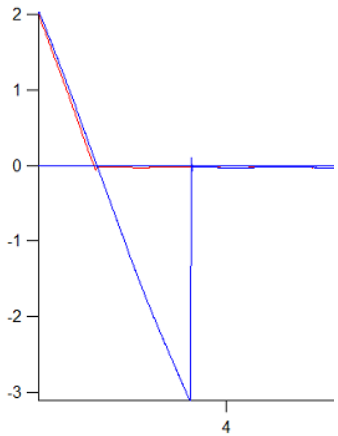


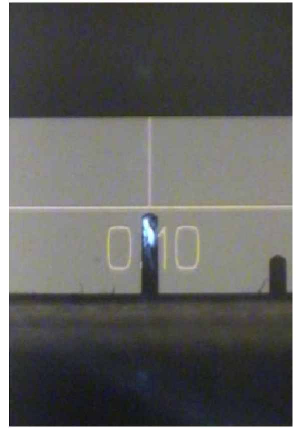


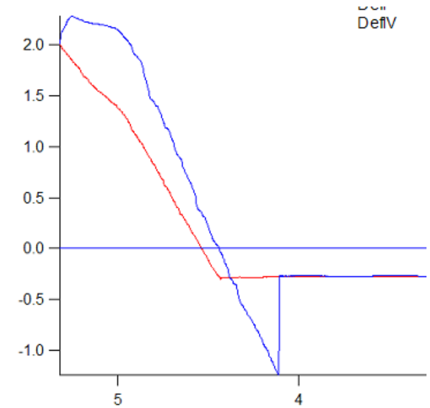
Sample #	Nominal k ($N\ m^{-1}$)	COC method	COC Std (%)	NFC Calibration	NFC Std (%)	Discrepancy (%)
1	42	27.84	1.8	27.21	1.2	2
2	42	29.02	0.5	28.45	1.1	2
3	42	30.70	1.1	29.84	1.4	3
4	42	30.75	1.5	32.17	1.5	-4
5	42	33.21	1.2	33.07	1.6	0

Dissemination to AFM – Cont'd

Experimental Results – ORC8-B Cantilever







Sample #	Nominal k (N m^{-1})	COC method	COC Std (%)	NFC Calibration	NFC Std (%)	Discrepancy (%)
1	0.1	0.072	4.0	0.127	1.5	-43
2	0.1	0.144	3.0	0.130	1.4	11
3	0.1	0.128	4.9	0.127	1.2	1
4	0.1	0.119	1.6	0.127	1.1	-7
5	0.1	0.131	4.7	0.126	1.1	3

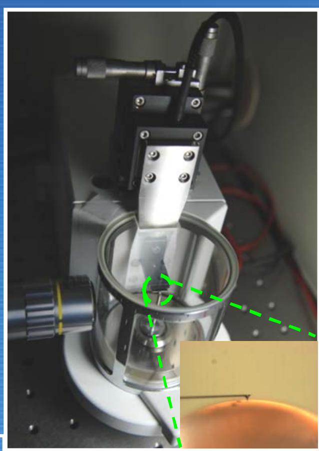
- Outline -

1. Introduction
2. Low force calibration machine
3. Low force sensor
4. International comparison
5. Challenges and Outlook

International Comparison

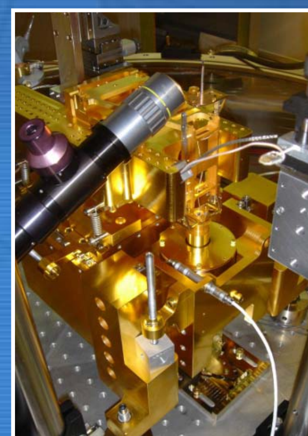
Low force facilities around globe joined in this comparison

- Participating NMIs: KRISS, NIST, PTB, NPL
- Co-organized as a pilot study by KRISS and NIST
- Protocol prepared by KRISS and transfer artifacts provided by NIST
- First international comparison on force at micro-Newton level
- Types of facilities: mass-based, electrostatic-based



KRISS

PTB



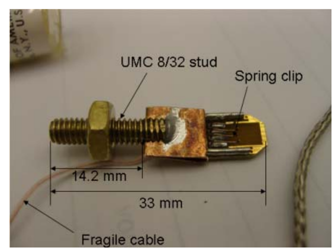
NIST

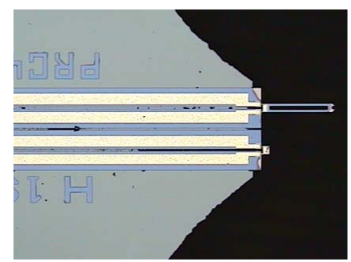
NPL

International Comparison – Cont'd

Transfer Artifacts

- Transfer artifacts
  - Commercial micro force sensors of cantilever type with an amplifier from Kleindeik GmbH
- Four artifacts from two models (FMT- 400 and FMT- 120) were used
  - 3 FMT-400 sensors: (2 ~ 4) N m^{-1}
  - 1 FMT-120 sensor: (30 ~ 40) N m^{-1}
- Amplifier outputs voltage from 0 V to approx. 10 V

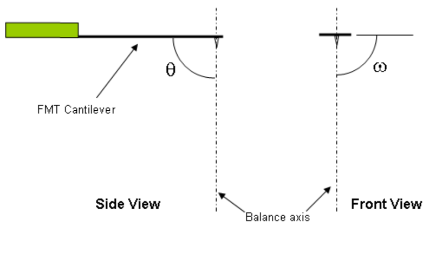




International Comparison – Cont'd

Protocols

- Comparison of the calibration capability on determination of stiffness (N/m) and force sensitivity (V/µN) of the transfer artifact
- Cantilevers are deflected up to the point where voltage output reaches to approximately 10 V with 4 ~ 5 equal steps
- Deflection, force and voltage output are recorded simultaneously at each step
- Linear-regression fits are used to get slopes
- The axis of cantilevers should be perpendicular to the balance axis
- Comparison range:
  - 2 N m^{-1} ~ 80 N m^{-1} in terms of the stiffness
  - 10 µN ~ 50 µN in terms of the force
- No strict rules in the protocol



International Comparison – Cont'd

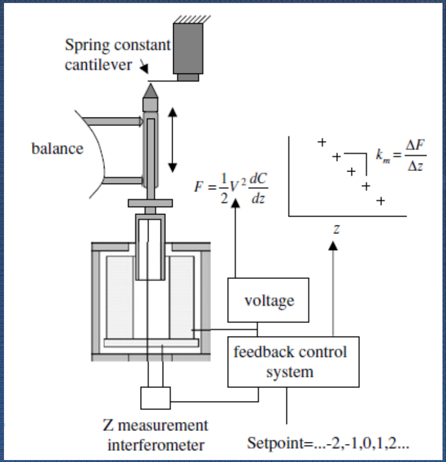
Different procedures, data-processing methods among participants

- Cantilever scan vs. Balance scan (indentation)

$$\frac{1}{k_c} = \frac{1}{k_m} - \frac{1}{k_b} - \frac{1}{k_l}$$

$$\frac{1}{k_c} = \frac{1}{k_m - k_{EFB}} - \frac{1}{k_l}$$

$k_b = (1000 \sim 5000)\ \mathrm{N\ m^{-1}}$ $k_{EFB} = (0.01 \sim 0.03)\ \mathrm{N\ m^{-1}}$

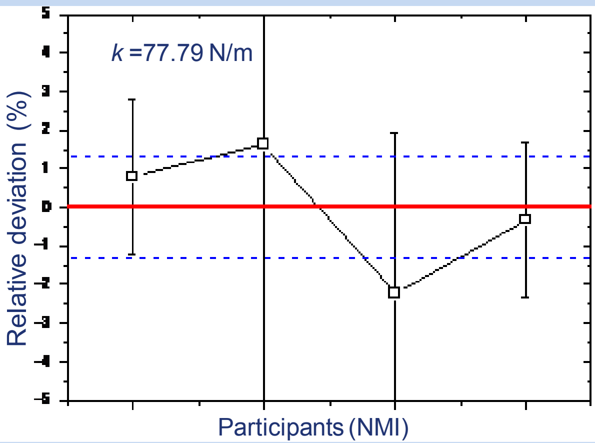


- Force loading directions: use both of ascending and descending force curves (, ,) or just use ascending force curve only ( )
- Uncertainties are estimated with their own ways
  - Ranging from 0.9 % to 6 % for the same artifact among participants
  - Dominant uncertainties are from transfer artifacts

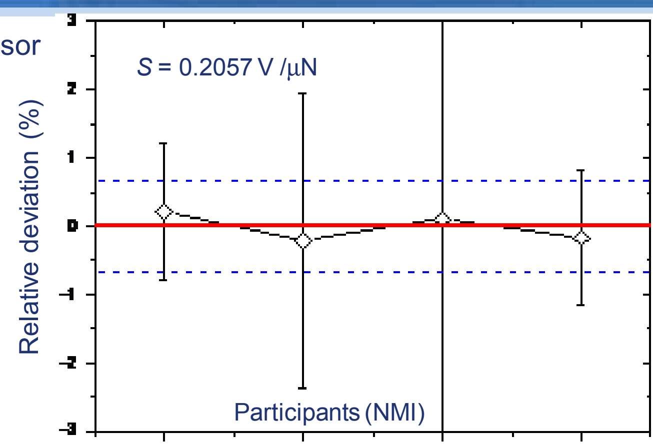
International Comparison – Cont'd

Progress report

- First circulation finished
  - KRISS(Feb. 08'), NIST(May 08'), PTB(Sept. 08'), KRISS(July 09')
- Preliminary results were circulated among KRISS, NIST and PTB on Sept. 09'
- NPL joined comparison and finished measurements of #3 artifact on Feb. 10'
- All results are equivalent to each other within their uncertainties
- Preparing publication



Artifact #5, FMT120 sensor



- Outline -

1. Introduction
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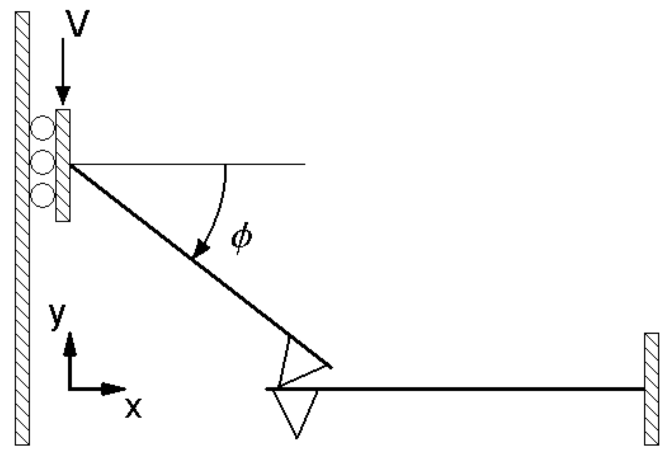
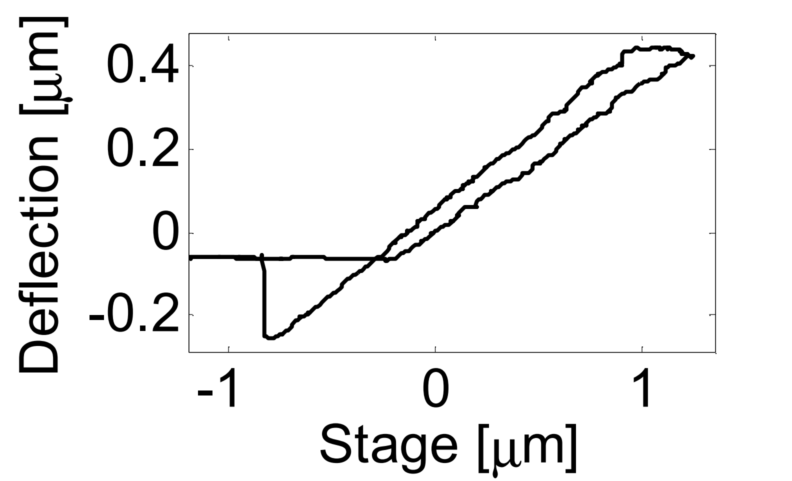
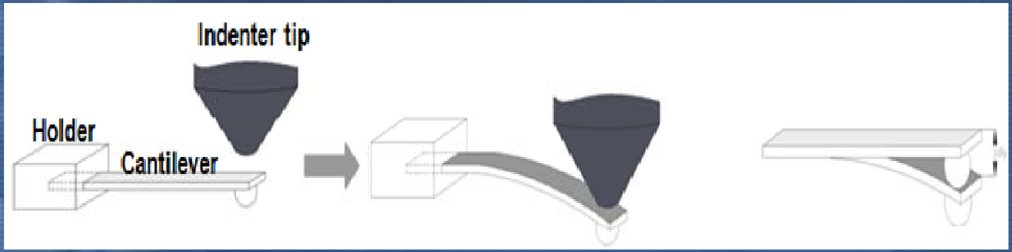
Dissemination Problem

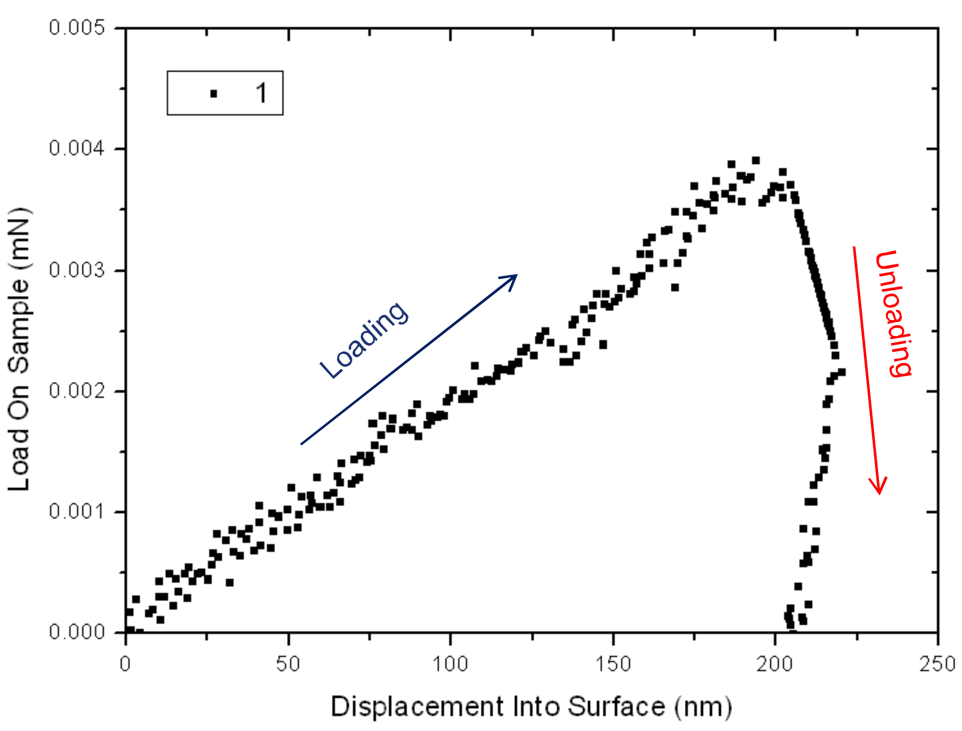
Lack of available transfer standards other than cantilevers

- Cantilever structure
  - Micromachining, High normal force sensitivity 🙂
  - Side loads, Off-axis loads, Sliding during deflection 🙁
- Dissemination to the AFM or Nanoindentor is limited
- Uncertainty better than 1% is not easily achievable even at the calibration with low-force standards





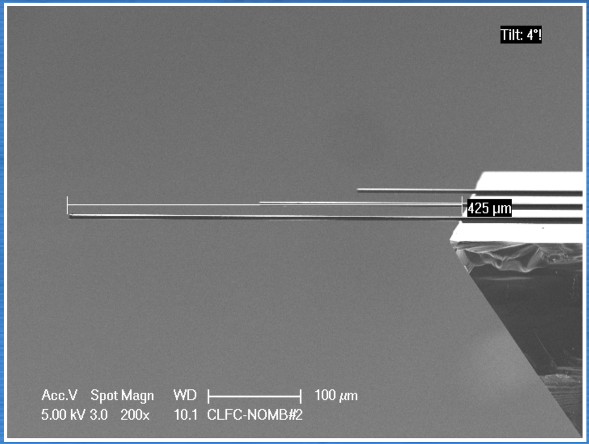

Indenter tip
Holder
Cantilever


0.005
0.004
0.003
0.002
0.001
0.000
1
Loading
Unloading
Load On Sample (mN)
0
50
100
150
200
250
Displacement Into Surface (nm)

Dissemination Problem – Cont'd

Current force calibration artifacts used in AFM and Nanoindentor

- Calibrated weight set
  - (0.5 ~ 10) g weight set is provided
  - Not small enough for nanoindentation using micro-Newton level of forces
- Reference cantilevers for stiffness calibration
  - (0.7 ~ 20) N m^{-1} stiffness range
  - Friction, sliding effect limit the application



Dissemination Problem – Cont'd

Requisites for useful low force transfer standards

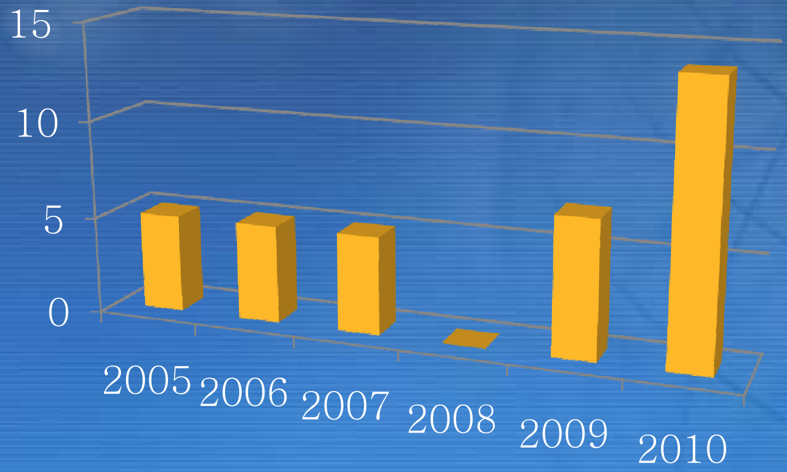
- Insensitive to side and off-axis loads
  - Confine the motion of a spring element of the sensor in one direction
- Insensitive to loading point
  - Deflection of the sensing structure not affected by contact position
- Small size enough to fit into the limited space in AFM and Nanoindentor
- Self-contained displacement metrology
  - Not dependent on displacement metrology of the target instrument
  - Piezoresistive, optical, capacitive sensor integrated in the sensor

Most immediate challenge

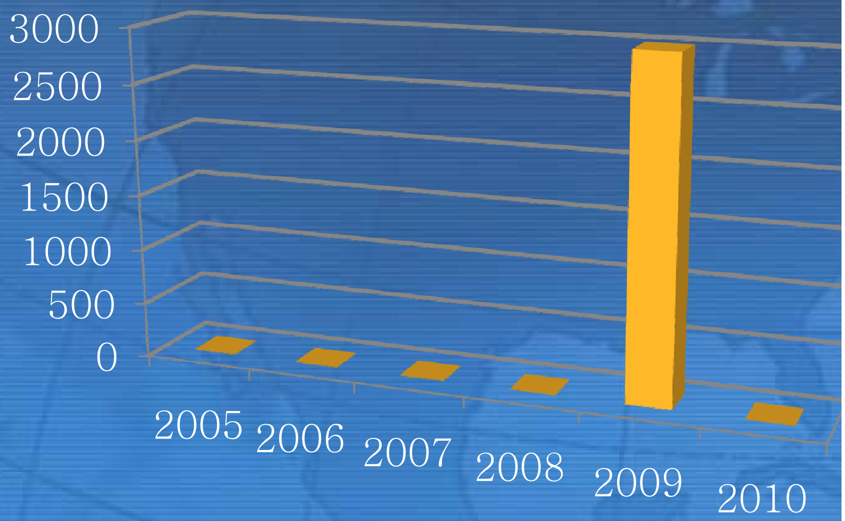
Outlook

When the time has come?

Number of calibration service



Annual income (US $)



Outlook – Cont'd

Perspective on micro force metrology

- High-performance low force transfer standards are necessary to complete hierarchy of SI-traceable force metrology at micro-Newton level
- For the time being, there would be no replacement of cantilevers' technology
- Due to lack of available high-performance transfer standards, both approaches to a primary standards (Mass and electrostatic force balance) can find their own reasons to exist
- AFM and Nanoindentor users want force calibrations and developing high-performance sensors and available dissemination methods can boost industrial demands on micro-force calibration

Reference

- Kim et al., "Atomic force microscope cantilever calibration device for quantified force metrology micro- or nano-scale regime: the nano force calibrator (NFC)," Metrologia 43 (5) (2006) 389–395.
- Kim et al., "SI-traceable determination of spring constants of various AFM cantilevers with a small uncertainty of 1%," Meas. Sci. Technol. **18** (11) (2007) 3351-3358
- Kim and Pratt, "SI-traceability: Current status and future trends for forces below 10 uN," Measurement 43 (2010) 169-182

Thanks for your attention!

Cup Kun Kap!

IMEKO

KRISS