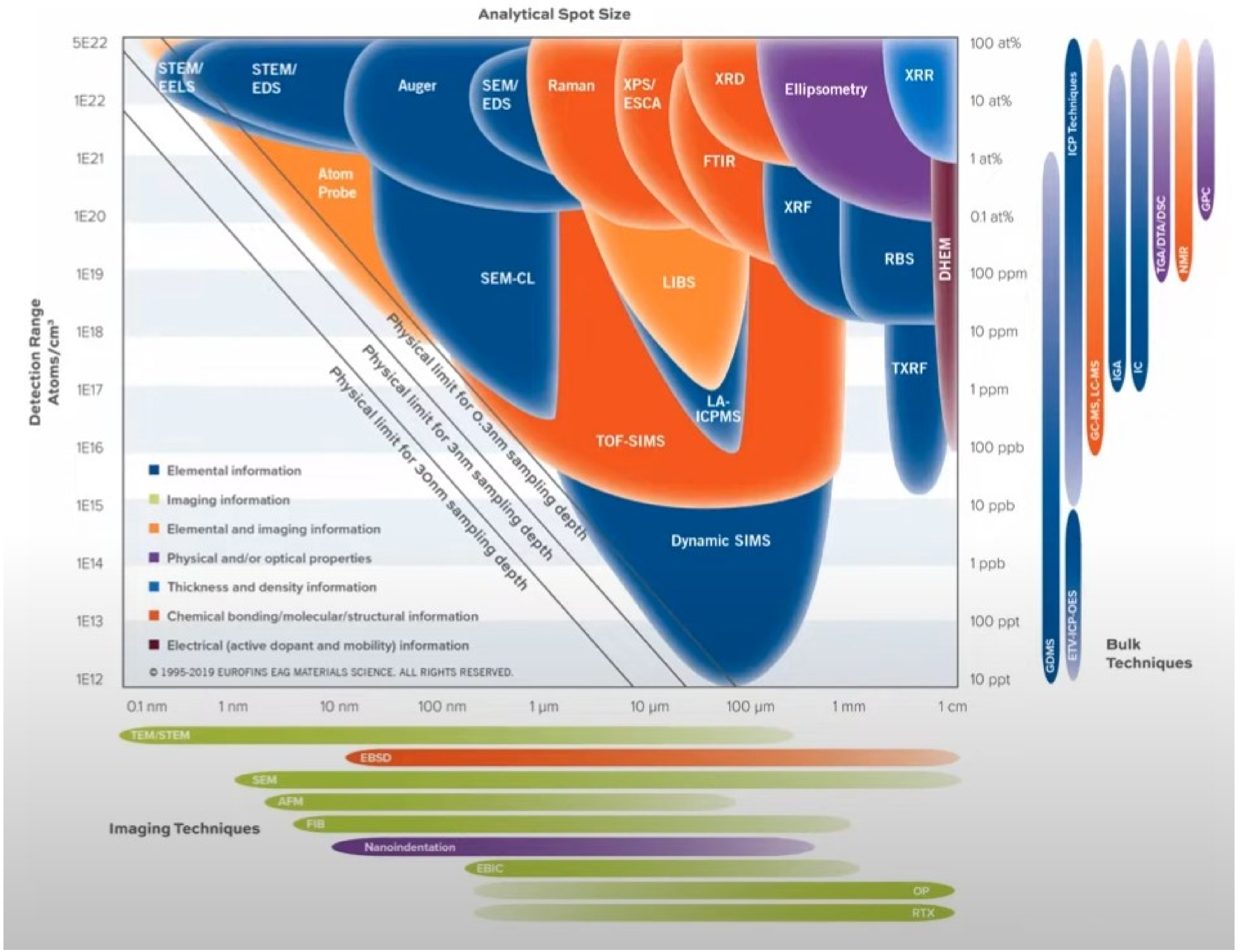
Nanoindentace

praqolab

Ing. Štěpán - Adam Havlíček

Spot size



Analytical Spot Size
Detection Range Atoms/cm³
5E22
1E22
1E21
1E20
1E19
1E18
1E17
1E16
1E15
1E14
1E13
1E12
100 at%
10 at%
1 at%
0.1 at%
100 ppm
10 ppm
1 ppm
100 ppb
10 ppb
1 ppb
100 ppt
10 ppt
0.1 nm
1 nm
10 nm
100 nm
1 µm
10 µm
100 µm
1 mm
1 cm
STEM/ EELS
STEM/ EDS
Auger
SEM/ EDS
Raman
XPS/ ESCA
XRD
Ellipsometry
XRR
Atom Probe
FTIR
XRF
SEM-CL
LIBS
RBS
DHEM
TXRF
LA-ICPMS
TOF-SIMS
Dynamic SIMS
Physical limit for 0.3nm sampling depth
Physical limit for 3nm sampling depth
Physical limit for 30nm sampling depth
Elemental information
Imaging information
Elemental and imaging information
Physical and/or optical properties
Thickness and density information
Chemical bonding/molecular/structural information
Electrical (active dopant and mobility) information
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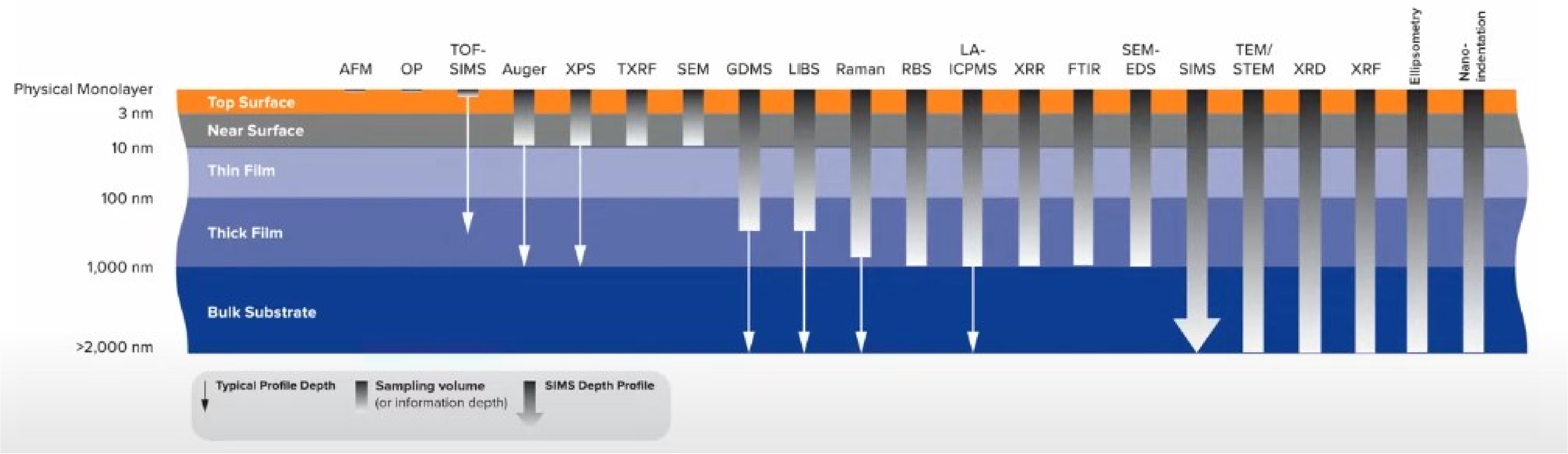
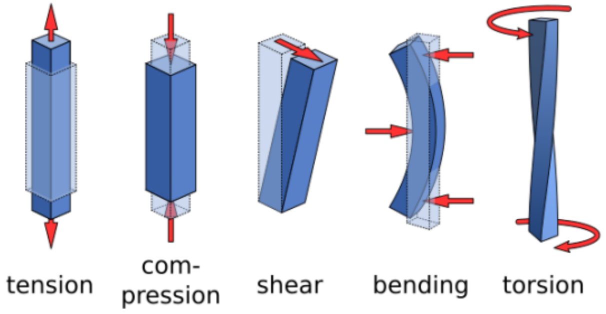
ICP Techniques
GC-MS, LC-MS
IGA
IC
TGA/DTA/DSC
NMR
GPC
GDMS
ETV-ICP-OES
Bulk Techniques
TEM/STEM
EBSD
SEM
AFM
FIB
Nanoindentation
EBIC
OP
RTX
Imaging Techniques

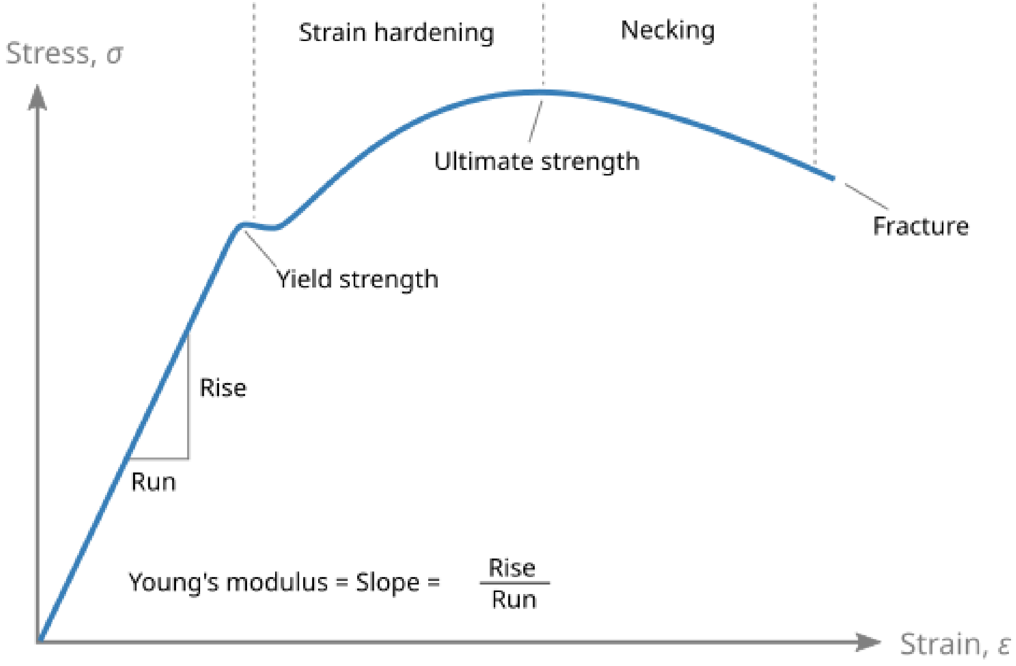
Hloubka penetrace

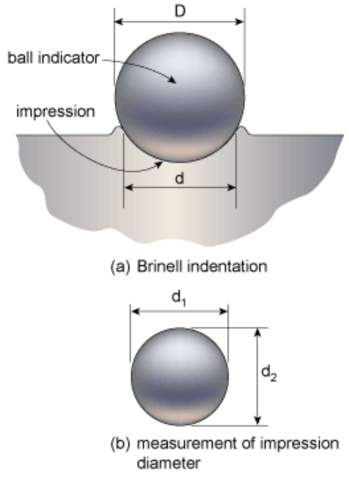


Proč nanoindentace?

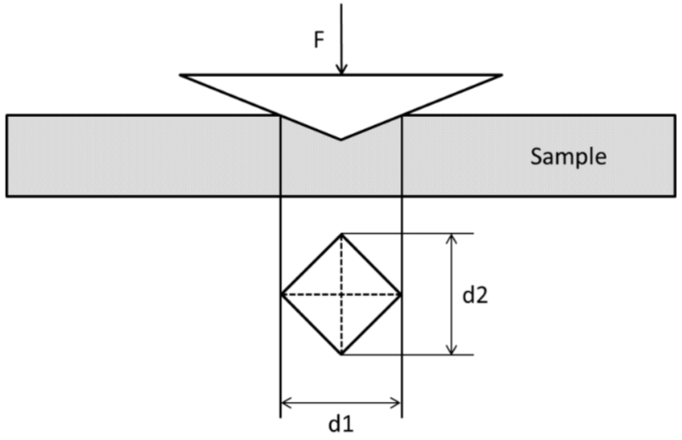
Makroskopické mechanické zkoušky





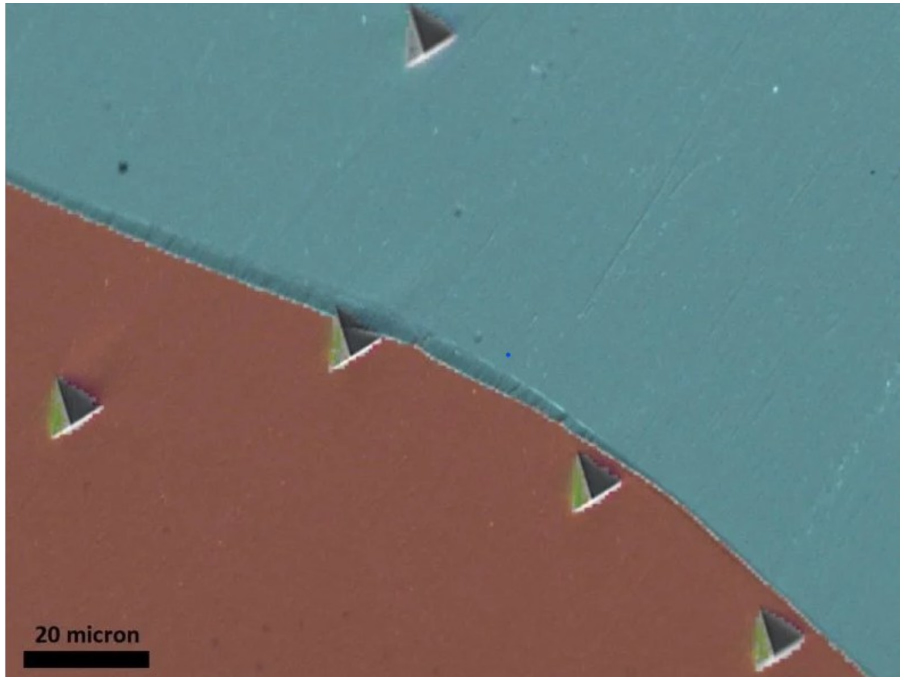


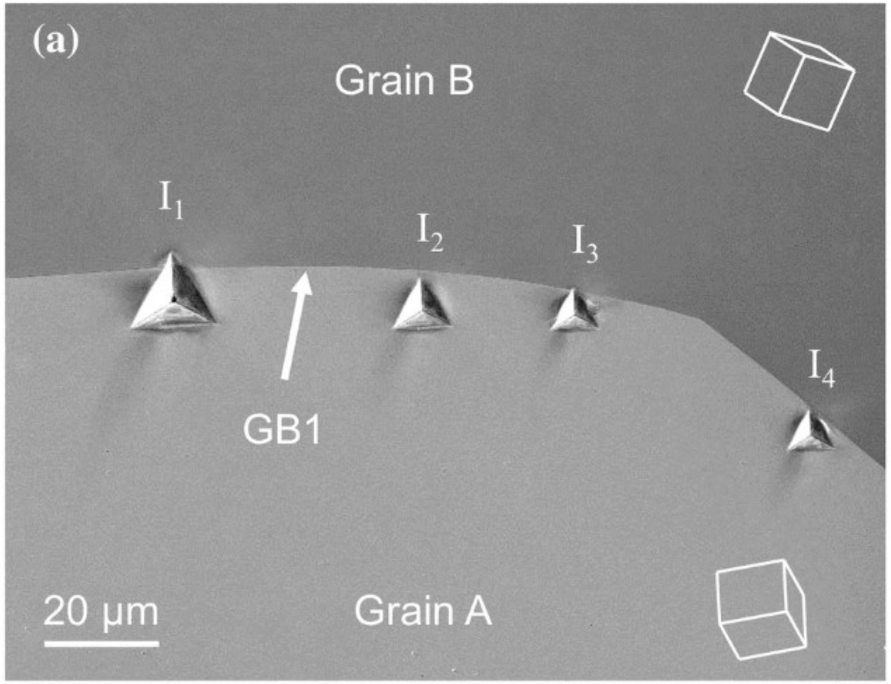
Mikroskopické mechanické zkoušky

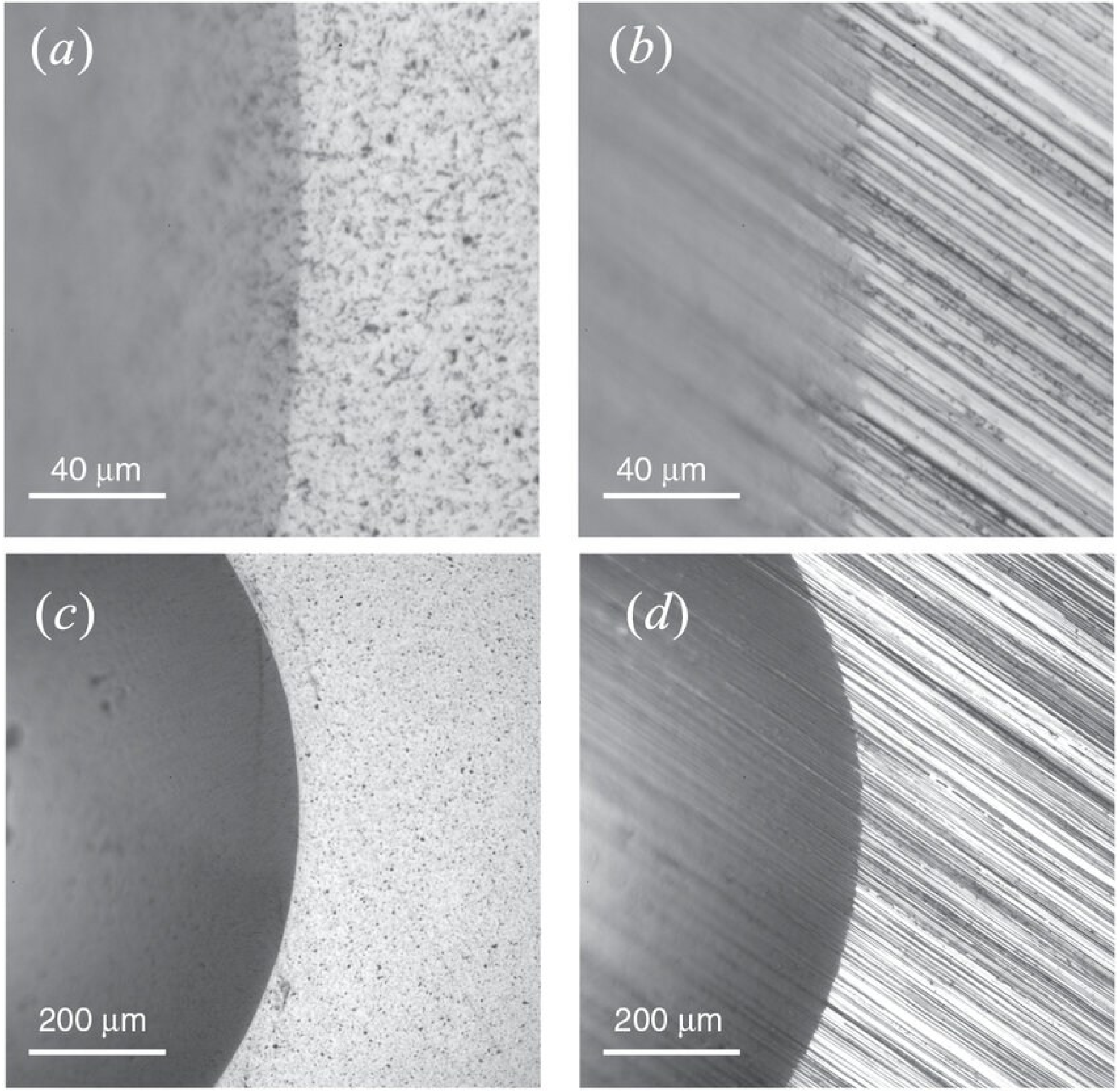


$$d = \frac{d_1 + d_2}{2}$$

$$HV = 0.102 * \frac{2 * F * \sin\frac{136°}{2}}{d^2} = 0.1891 * \frac{F}{d^2}$$

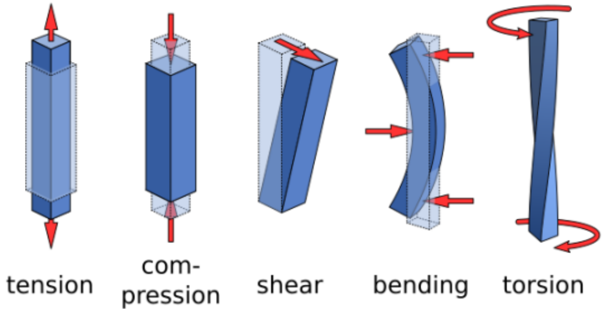

20 micron

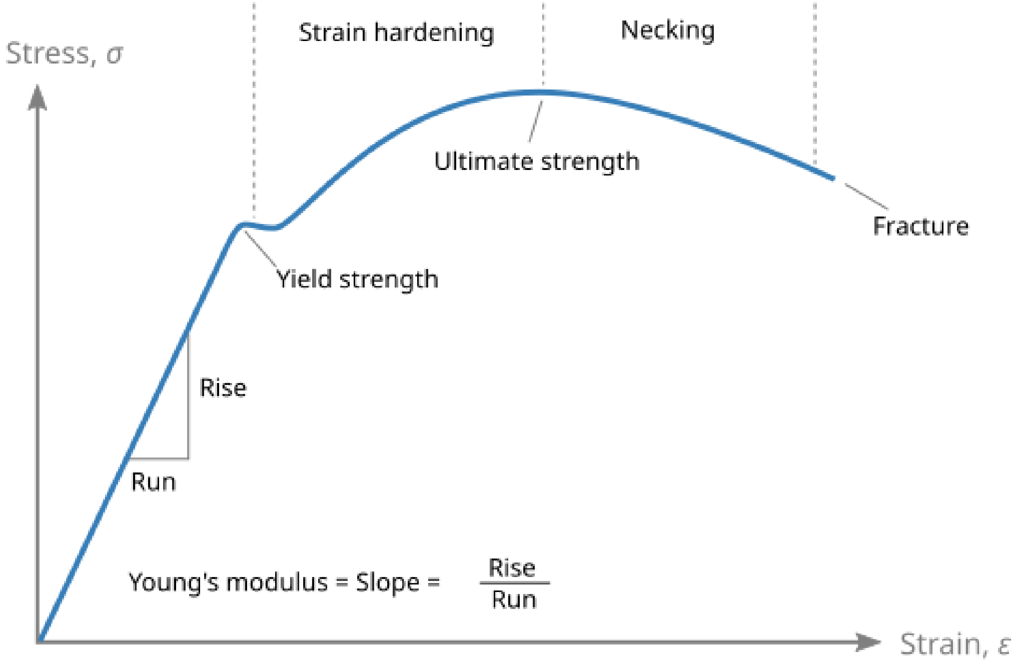

(a)
Grain B
I_1
I_2
I_3
I_4
GB1
20 μm
Grain A

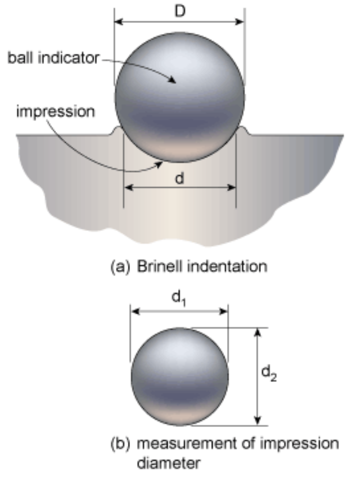

(a)
40 μm
(b)
40 μm
(c)
200 μm
(d)
200 μm

Proč nanoindentace?

Makroskopické mechanické zkoušky







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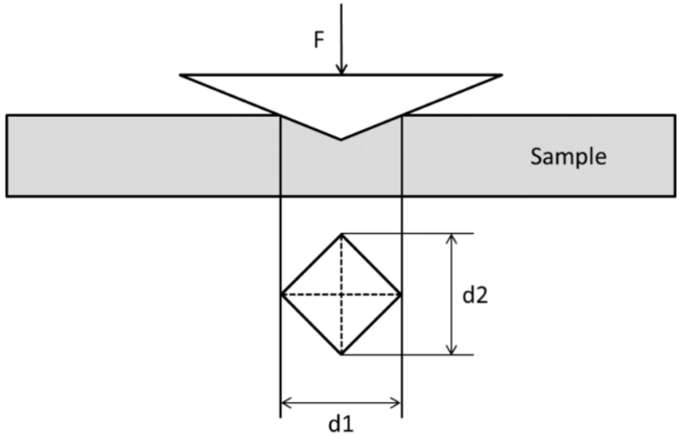


$$d = \frac{d_1 + d_2}{2}$$

$$HV = 0.102 * \frac{2 * F * \sin\frac{136°}{2}}{d^2} = 0.1891 * \frac{F}{d^2}$$

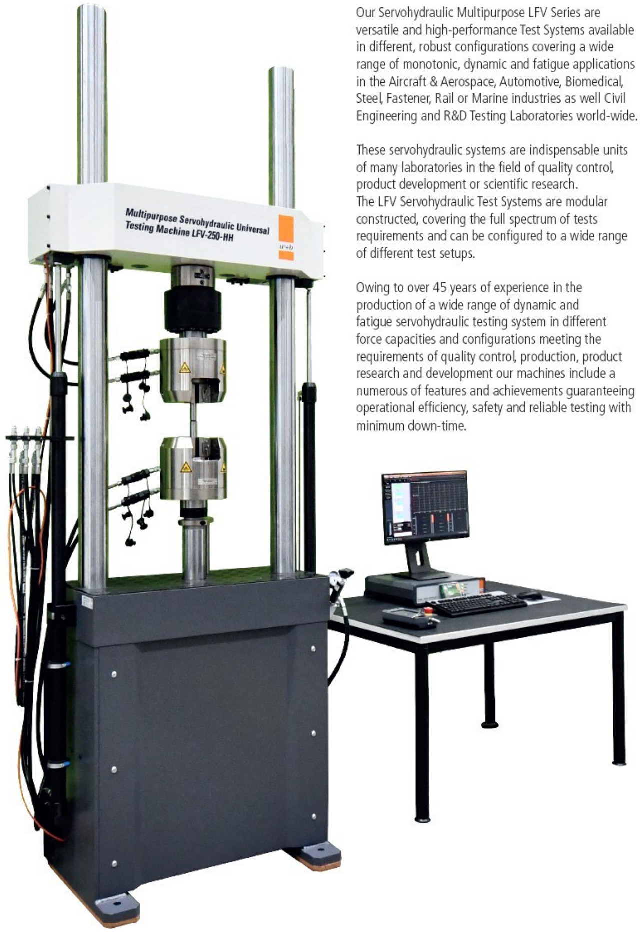
w+b **Materials Testing Systems**

Servohydraulic Multipurpose Fatigue Test System Floor Standing with Actuator in lower base

Series LFV up to 630 kN

w+b

These Tests systems meet the full spectrum of dynamic, fatigue and monotonic applications including TMF (Thermo-Mechanical Fatigue), LCF (Low-Cycle Fatigue), Fracture Mechanics, HCF (High-Cycle Fatigue), High-Strain Rate and Component Testing.



Our Servohydraulic Multipurpose LFV Series are versatile and high-performance Test Systems available in different, robust configurations covering a wide range of monotonic, dynamic and fatigue applications in the Aircraft & Aerospace, Automotive, Biomedical, Steel, Fastener, Rail or Marine industries as well Civil Engineering and R&D Testing Laboratories world-wide.

These servohydraulic systems are indispensable units of many laboratories in the field of quality control, product development or scientific research.
The LFV Servohydraulic Test Systems are modular constructed, covering the full spectrum of tests requirements and can be configured to a wide range of different test setups.

Owing to over 45 years of experience in the production of a wide range of dynamic and fatigue servohydraulic testing system in different force capacities and configurations meeting the requirements of quality control, production, product research and development our machines include a numerous of features and achievements guaranteeing operational efficiency, safety and reliable testing with minimum down-time.

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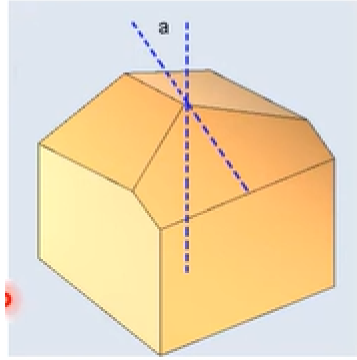
Presi HZ 50-4
Vickers + Knoop

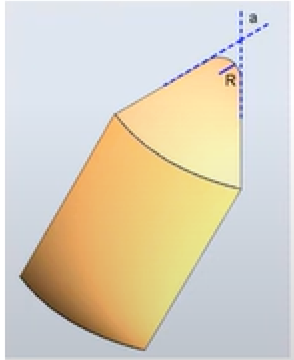
Presi Macrotech VX7

Druhy hrotů

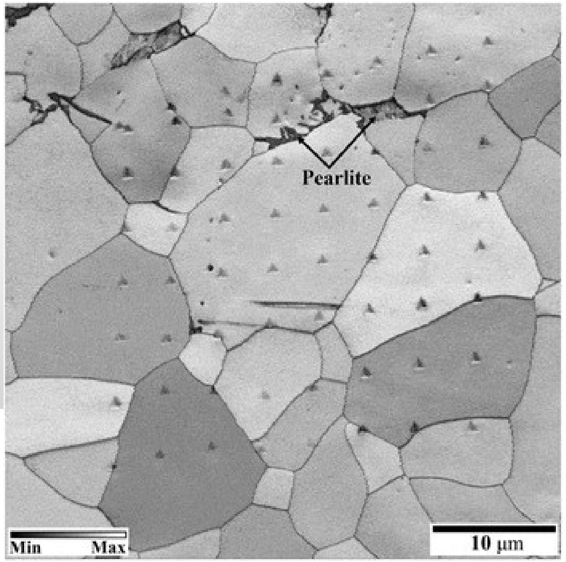


Berkovich Tip
- 3-sided pyramidal tip
- a = 65.35° (half angle)
- Multi-purpose tip
- Thin films >100 nm
- AIF: 150 nm radius of curvature
- **Liquid cell tip** also available

Cube Corner Tip
- 3-sided pyramidal tip
- a = 35.26° (half angle)
- Fine tip
- Ultra thin films & nanostructures
- Sample fracture
- Higher resolution imaging
- AIF: 40 nm radius of curvature



Cono-spherical Tip
- a = 60 - 120° (included angle)
- Very soft materials
- Lower resolution imaging
- AIF: 90°, 1 μm radius of curvature



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(Makroskopické zkoušky) Mechanické zkoušky

1. Materiálové zkoušky

2. Testování komponent

3. Testování stavebních materiálů

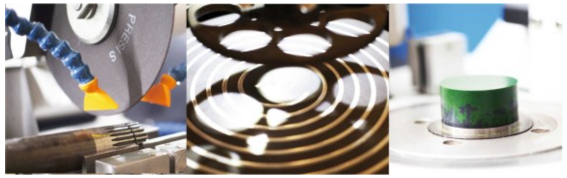
4. Kompletní a komplexní řešení

5. Speciální řešení

6. Modernizace, upgrade přístrojů pro mechanické testování

walter+bai Testing Machines

Mikroskopické zkoušky



1. Krájení

2. Zalévání

3. Broušení a leštění

4. Nanoindentace

Thermo SCIENTIFIC

5. XRD prvková analýza

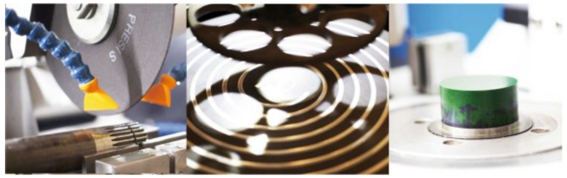
6. Měření tvrdosti

7. Odborné zaškolení a aplikační podpora

Spotřební materiál

pragolab PRESI

Mikroskopické zkoušky



1.

ěštění

4. Nanoindentace

5. XRD prvková analýza

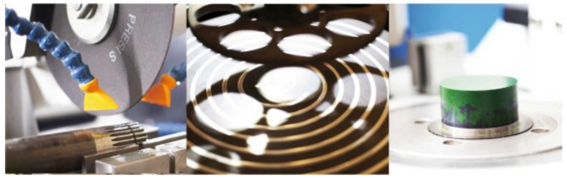
6. Měření tvrdosti

7. Odborné zaškolení a aplikační podpora

Spotřební materiál

pragolab PRESI

Mikroskopické zkoušky



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4. Nanoindentace

5. XRD prvková analýza

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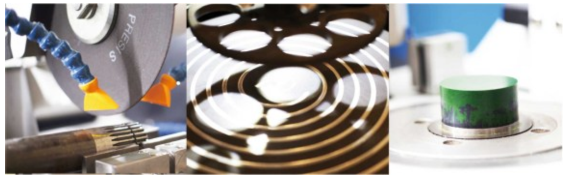
7. Odborné zaškolení a aplikační podpora

Spotřební materiál

Mikroskopické zkoušky



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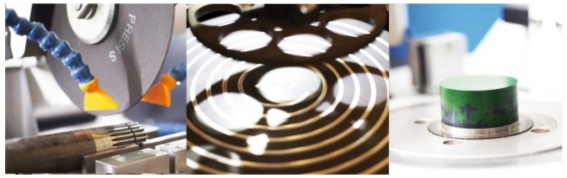
Spotřební materiál

Mikroskopické zkoušky



1. Krájení

2. Zalévání

3. Broušení a leště

5. XRD prvková analýza

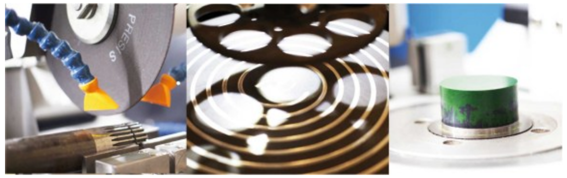
6. Měření tvrdosti

7. Odborné zaškolení a aplikační podpora

Spotřební materiál

pragolab PRESI

Mikroskopické zkoušky



1. Krájení

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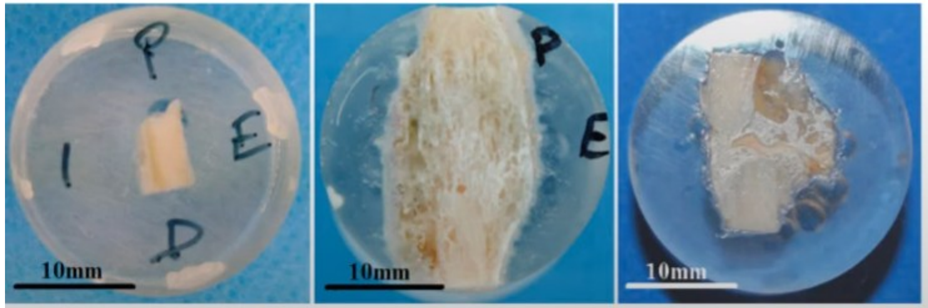
Spotřební materiál

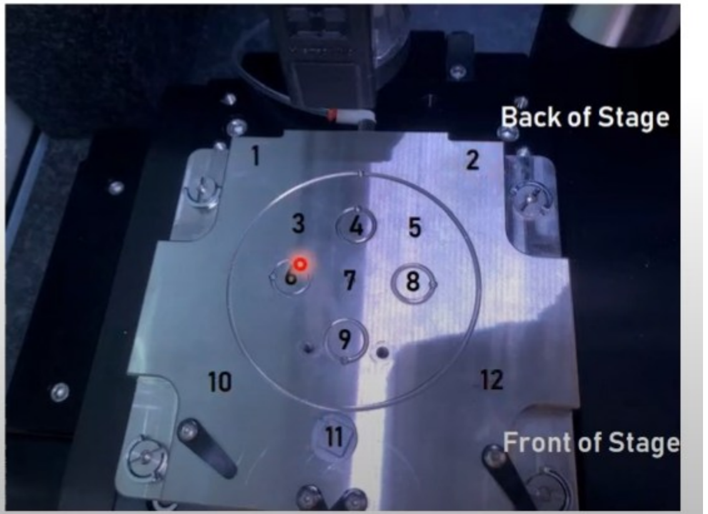
Příprava vzorků

Loose objects/samples are generally not good enough to put in the nanoindenter

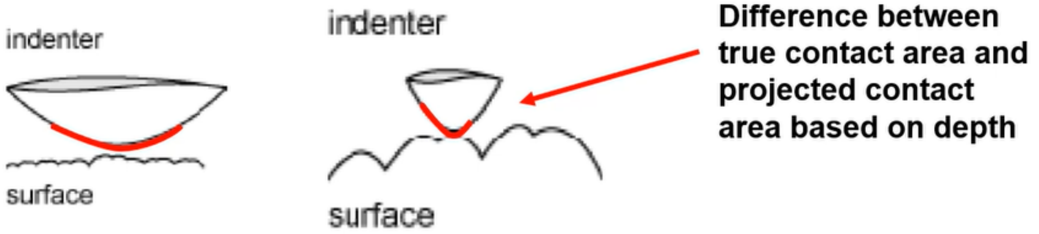
- **Recommended:** Super glue the sample to a magnetic AFM specimen disk (shown right)
- Super glue to a glass slide or clean wafer
- Smooth samples (wafers, glass slide) can be adhered via air suction without gluing

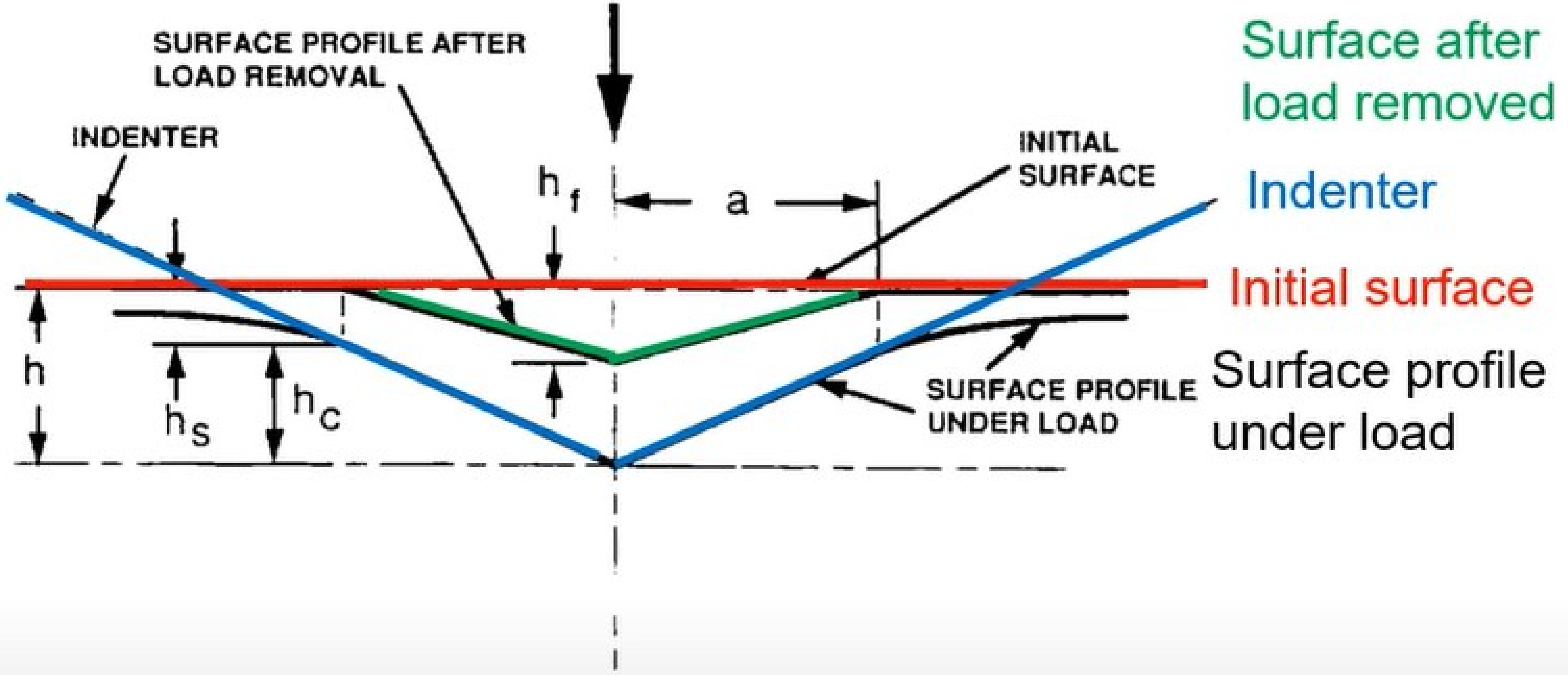


Source: Mora-Macias et. al. (2017)
https://doi.org/10.1016/j.jmbbm.2017.05.031
JMBBM Vol. (Oct 2017) Vol. 74, pp.236-244



Profil povrchu a penetrační hloubka





h = *penetration depth*
h_c = **contact depth** = vertical distance along which contact is made
h_s = surface displacement at perimeter of contact (**sink-in**)

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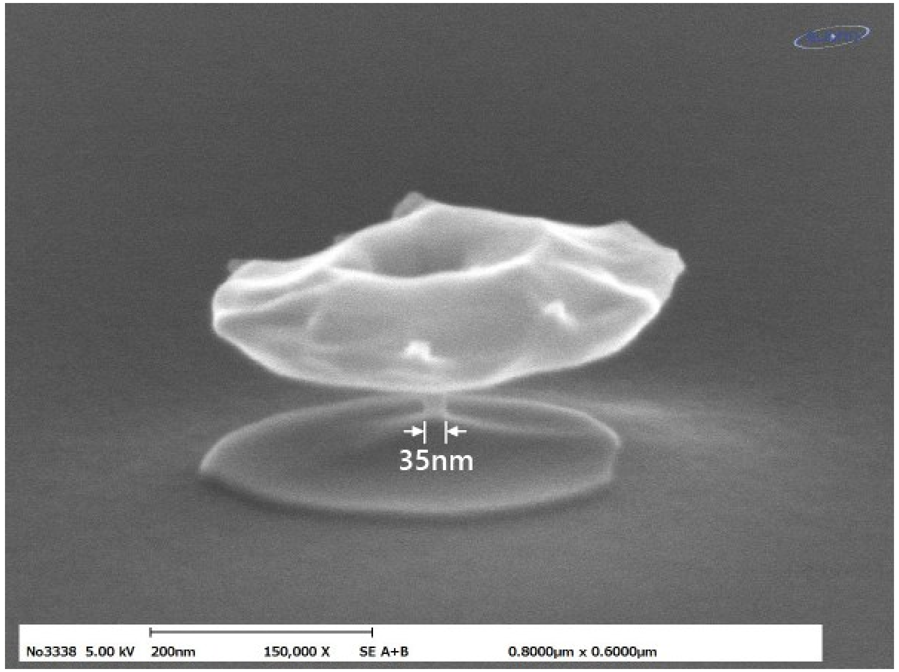
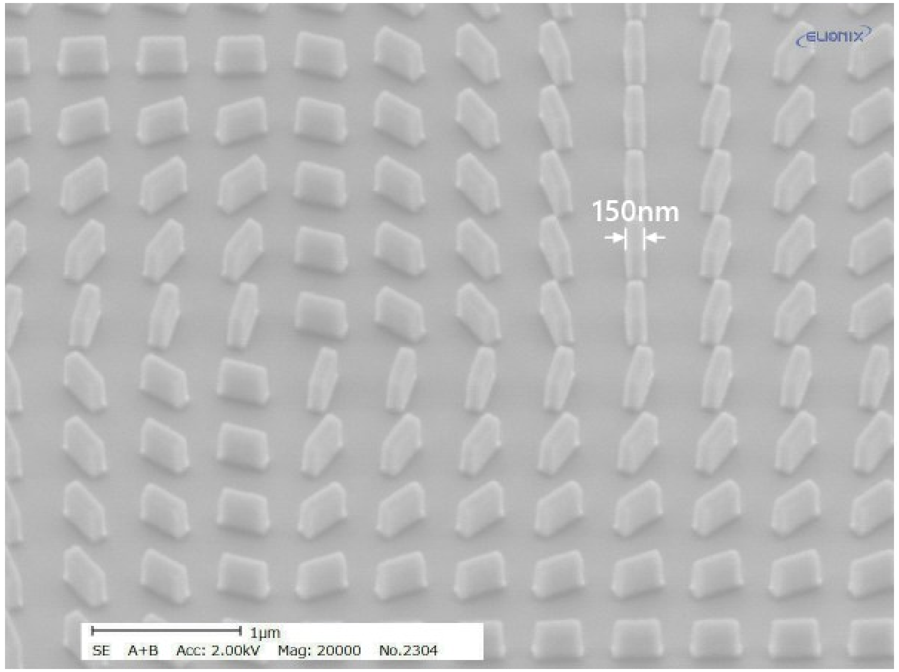
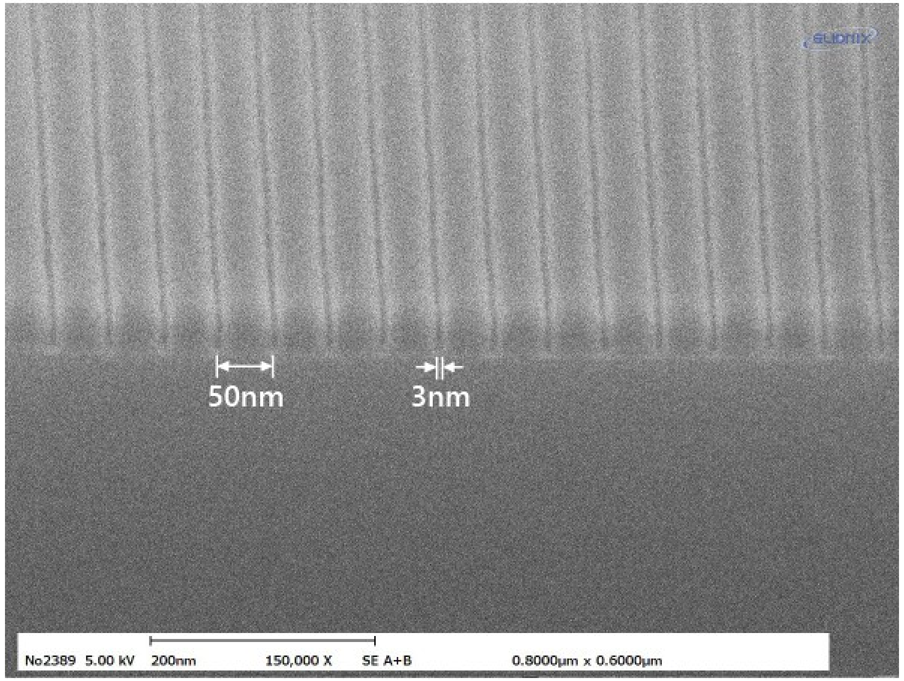
ELIONIX


35nm
No3338 5.00 kV 200nm 150,000 X SE A+B 0.8000µm x 0.6000µm


150nm
1µm
SE A+B Acc: 2.00kV Mag: 20000 No.2304


50nm
3nm
No2389 5.00 kV 200nm 150,000 X SE A+B 0.8000µm x 0.6000µm

NANO INDENTATION TESTER

ENT-5

ELIONIX
Action for Innovation

Nanoindentation tester

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Features

1.Temperature control

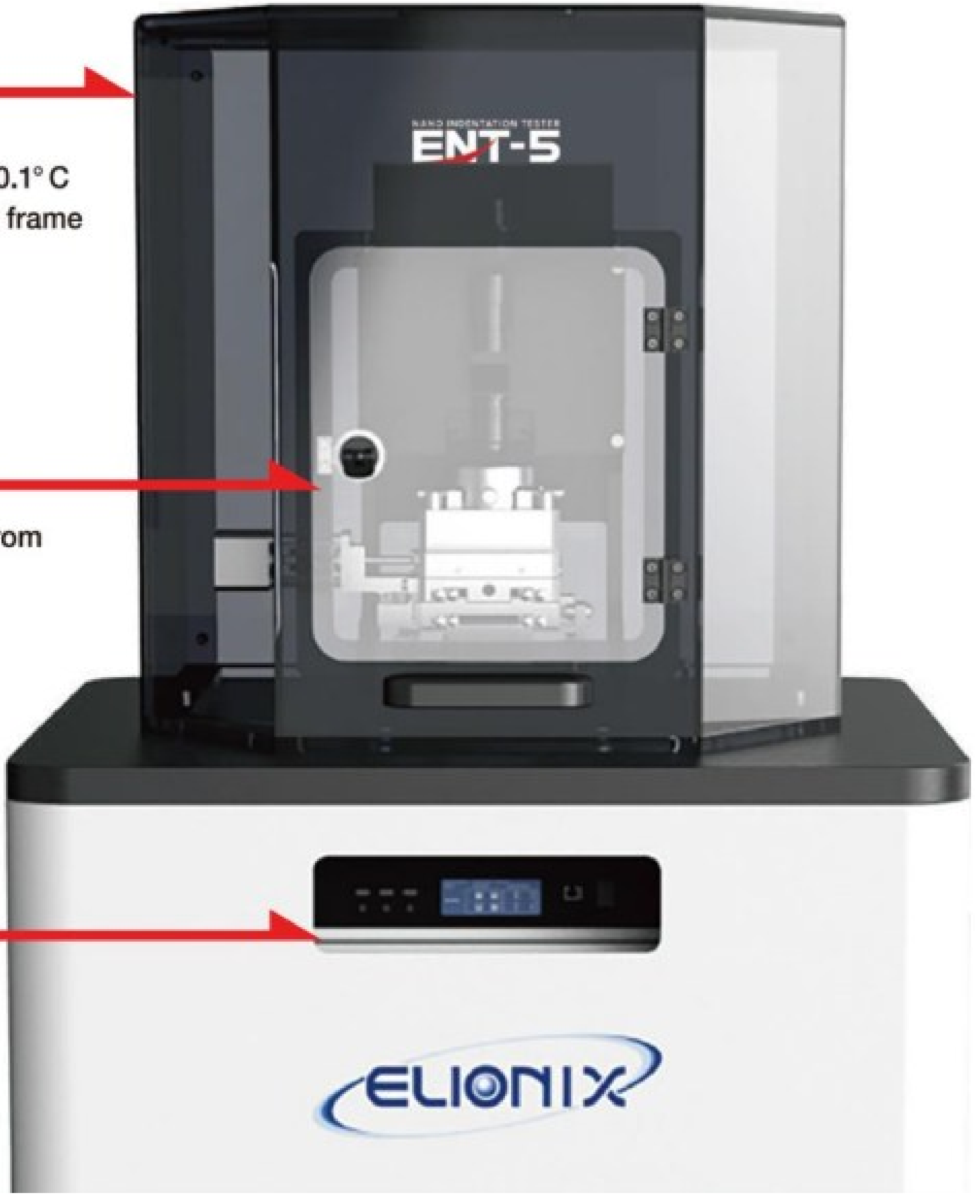
- Prevent thermal expansion of samples and testing machines
- Temperature control in the environmental isolation shield at ± 0.1° C
- Low thermal expansion material (Nobinite) is used for machine frame and Sample holder /stage
- Reduce impact from environmental airflow

2.High precision positioning stage

- Adopts in-house developed high-precision stage technology from Elionix Electron Beam Lithography system
- 0.1um step in positioning
- Observe the material with magnification of 2,000x and set the measurement point.
  (standard objective lens 20x and digital zoom)

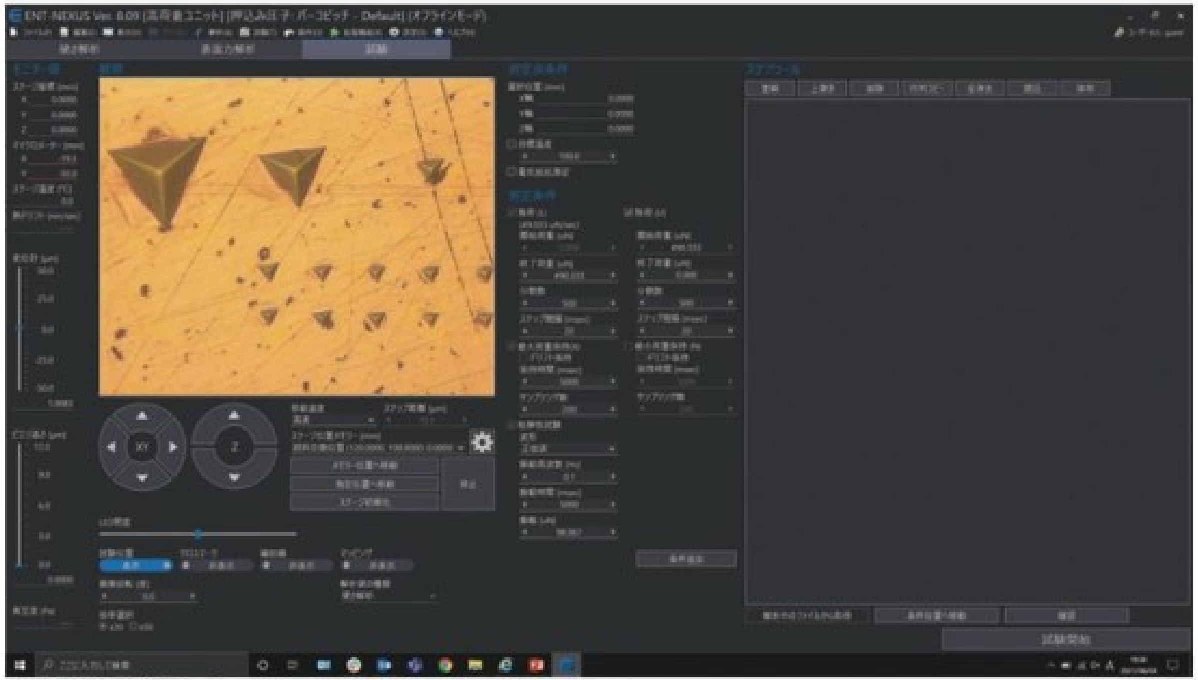
3.Active vibration isolation

- Vibration-resistant wedge-shaped high-precision stage
- Active vibration isolation table



NANO INDENTATION TESTER

ENT-5



※It may differ from the actual product software

4.Software

- Pop-up alerting for daily check : indenter tip correction, temperature drift correction.
- Easy-to-use operability

5.Data reproducibility

- Improved data reproducibility by suppressing the disturbances from vibration and temperature changes
- Stable data even with continuous measurement

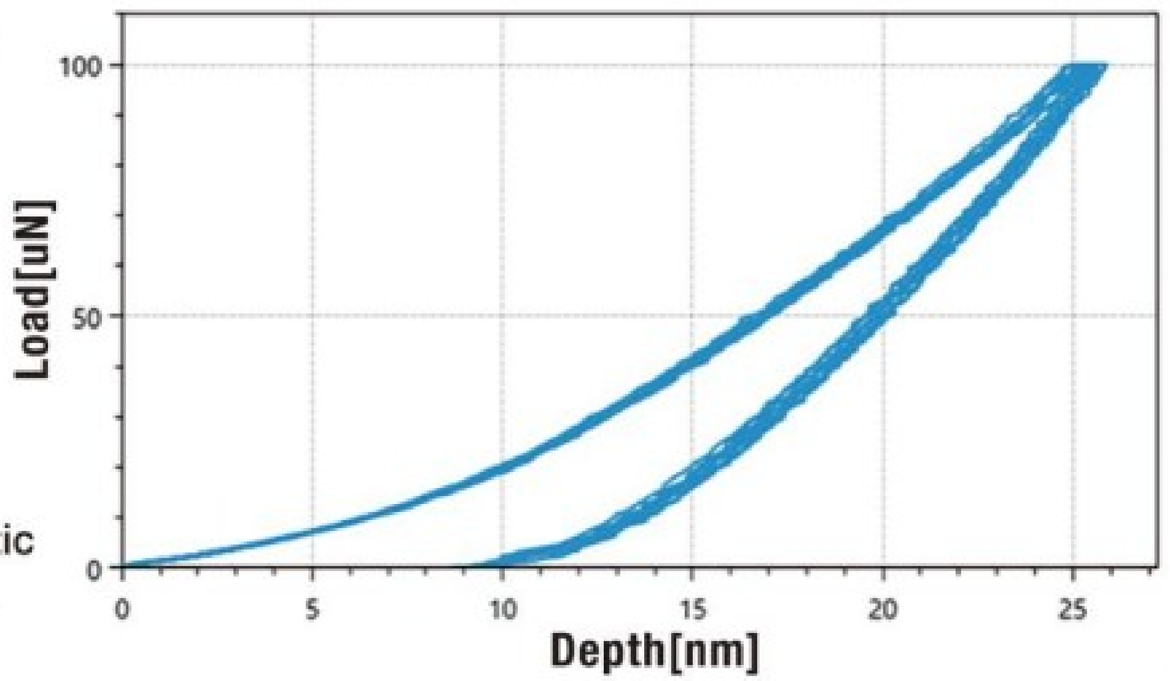
6.Maintainability

- Easy replacement of loading unit
- Minimum downtime with easy-change loading units

Features

● Applicable to variable kind of materials

- Plating, hard-coat thin layer
- Resin, polymer thin film
- Ultra-thin DLC coating
- Functional resin, surface modification layer
- Fine particles and powder materials



● Nano indentation test

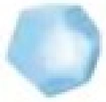
- Mechanical characteristics such as hardness and elastic modulus on thin film or surface layer can be obtained.
- Analyze the load/unload curves to determine the characteristics. No need to observe indentations
- Compliant with ISO14577-1 / JIS Z 2255

● Measurable materials

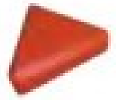

Polymer films


Electronic components (IC, LSI, etc.)


Rubber, Elastomer


Glass, Ceramic

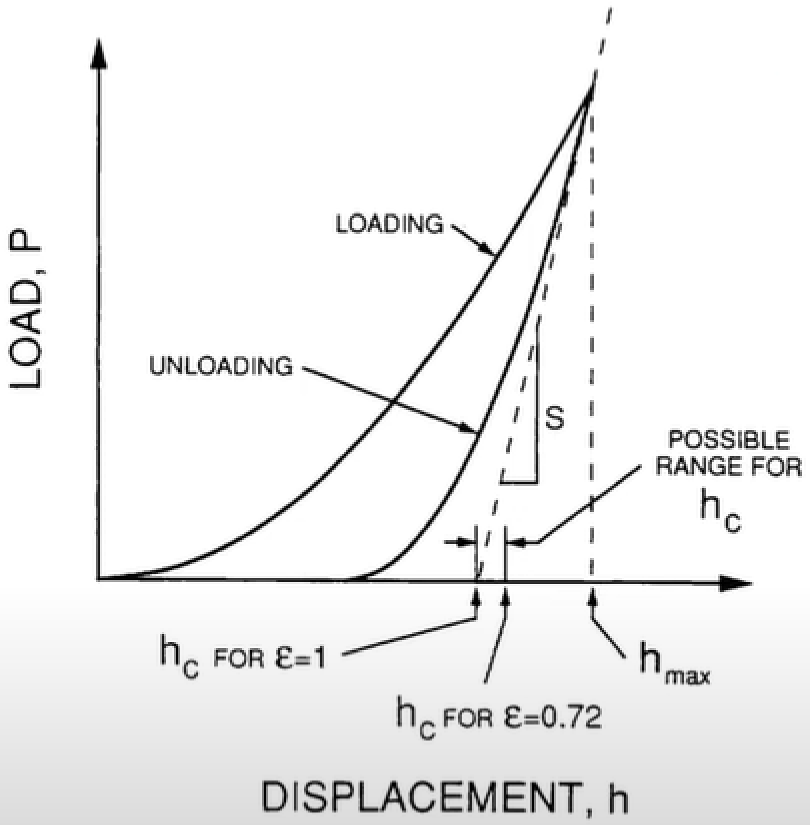

Metal


Plastic


Various fibers, Carbon fiber


Thin film / Coating

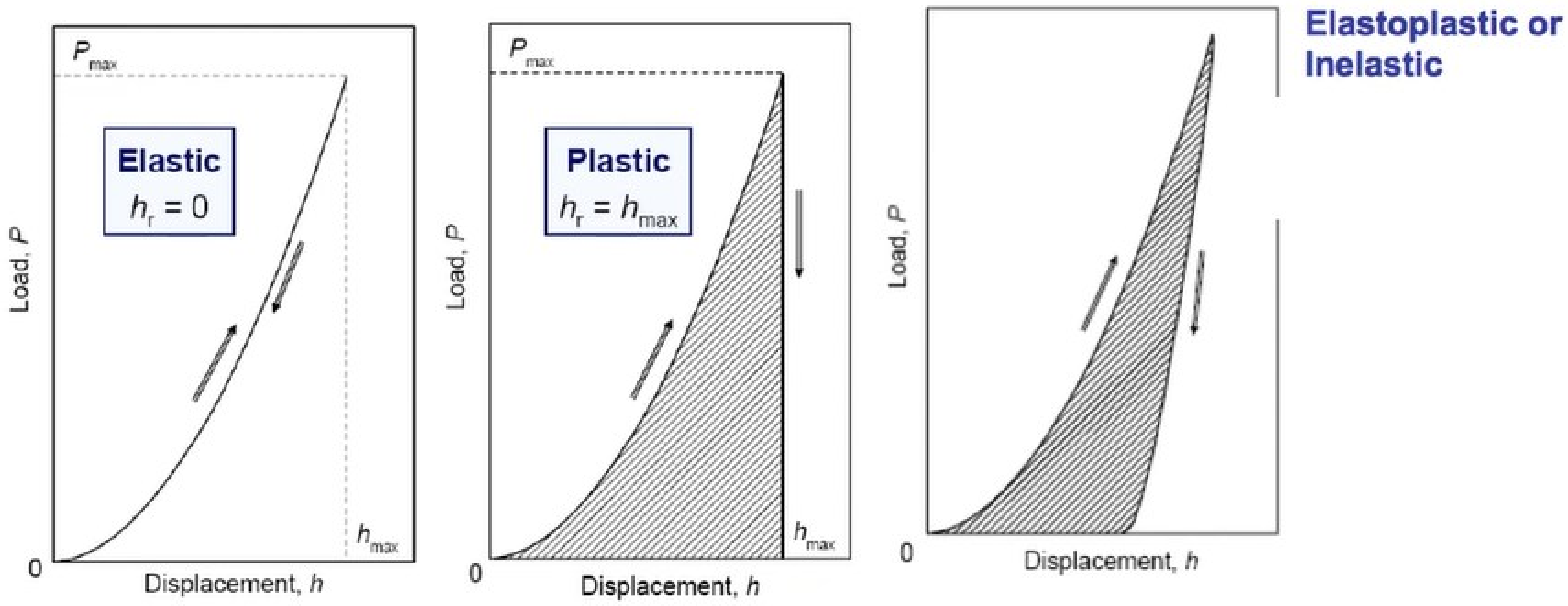
Zatěžová křivka



h_{max} = *penetration depth*
h_c = contact depth = vertical distance along which contact is made
h_s = surface displacement at perimeter of contact (sink-in)

Correction for sink-in:
S = dP/dh @ P_{max} = stiffness
$h_c = h_{max} - \varepsilon P_{max}/S$
ε = geometric constant = 0.75 (paraboloid)
= 0.72 (conical)
= 1 (flat punch)

Deformace v průběhu zatížení



$h_r = h_f$ = residual / final depth

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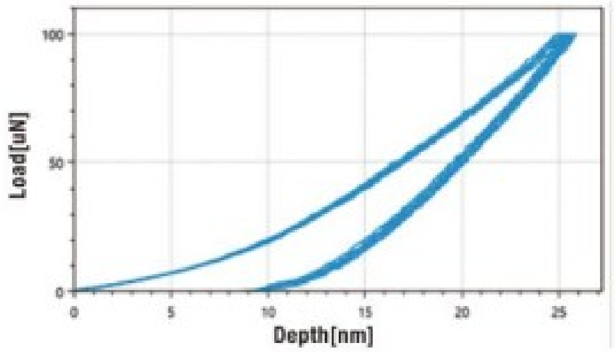
Measurement Example

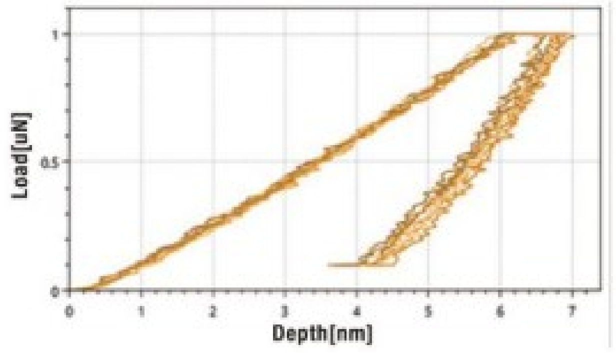
Loading units

High load unit



High load unit Measurement example [Fused silica]
(Load : 100μN Overlay 9times)

Low load unit



Low load unit Measurement example [resist]
(Load : 1μN Overlay 9times)

Measurement example

Dynamic Instrumented Indentation

- This is the method measuring continuous stiffness distribution along the depth. Applying small oscillation during loading course and calculate hardness and elastic modulus.
- Sample
  Fused silica (blue), DLC coating (red)
- Test results
  Fused silica shows almost constant hardness regardless of the indentation depth.
  On the other hand, the DLC sample shows gradual changing in the hardness from the surface to the base material.

Sample holder

Standard sample holder (φ50mm)

Optional holders

Heating holder
*Sample size
10×15×t3mm
*Temperature range
R.T～250℃(High load)
R.T～180℃(Low load)

Cooling holder
*Sample size
10×10×t1mm
*Temperature range
-30℃～100℃

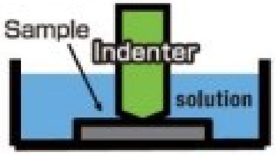
Sample holder for Resin embedding sample
*Sample size
Diameter 25～40mm
Height 9～14mm

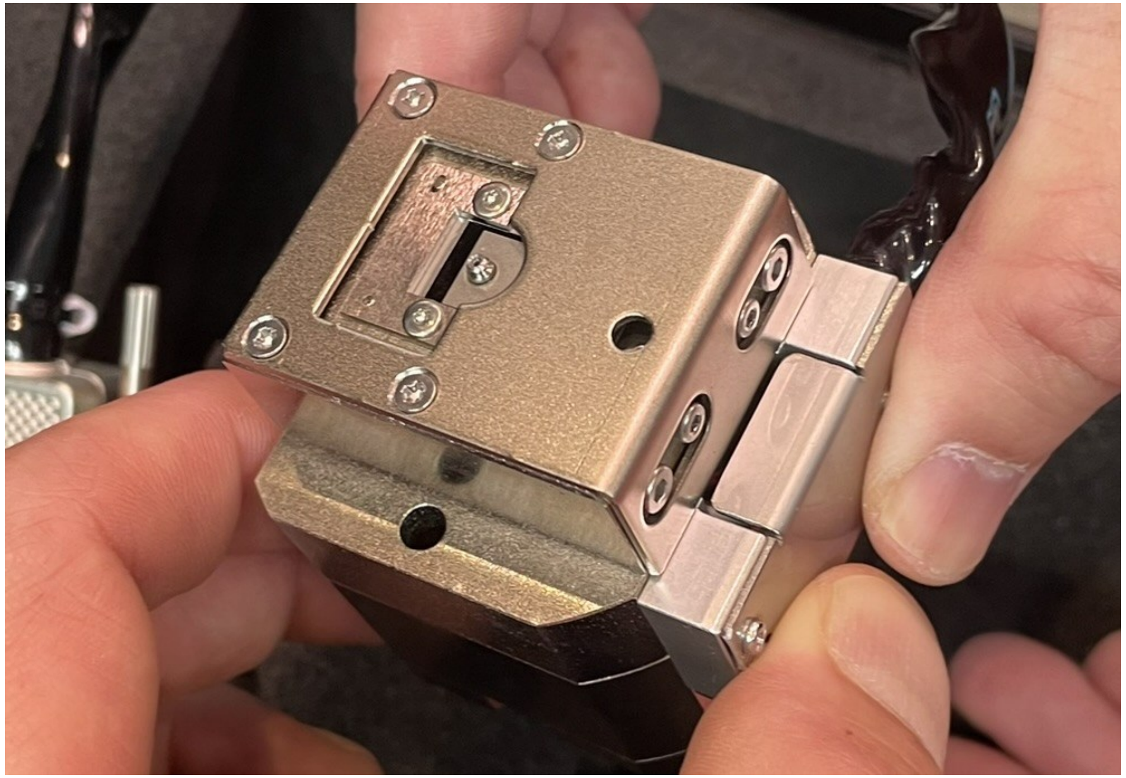
*Customized holders to fix samples with special shapes are available.

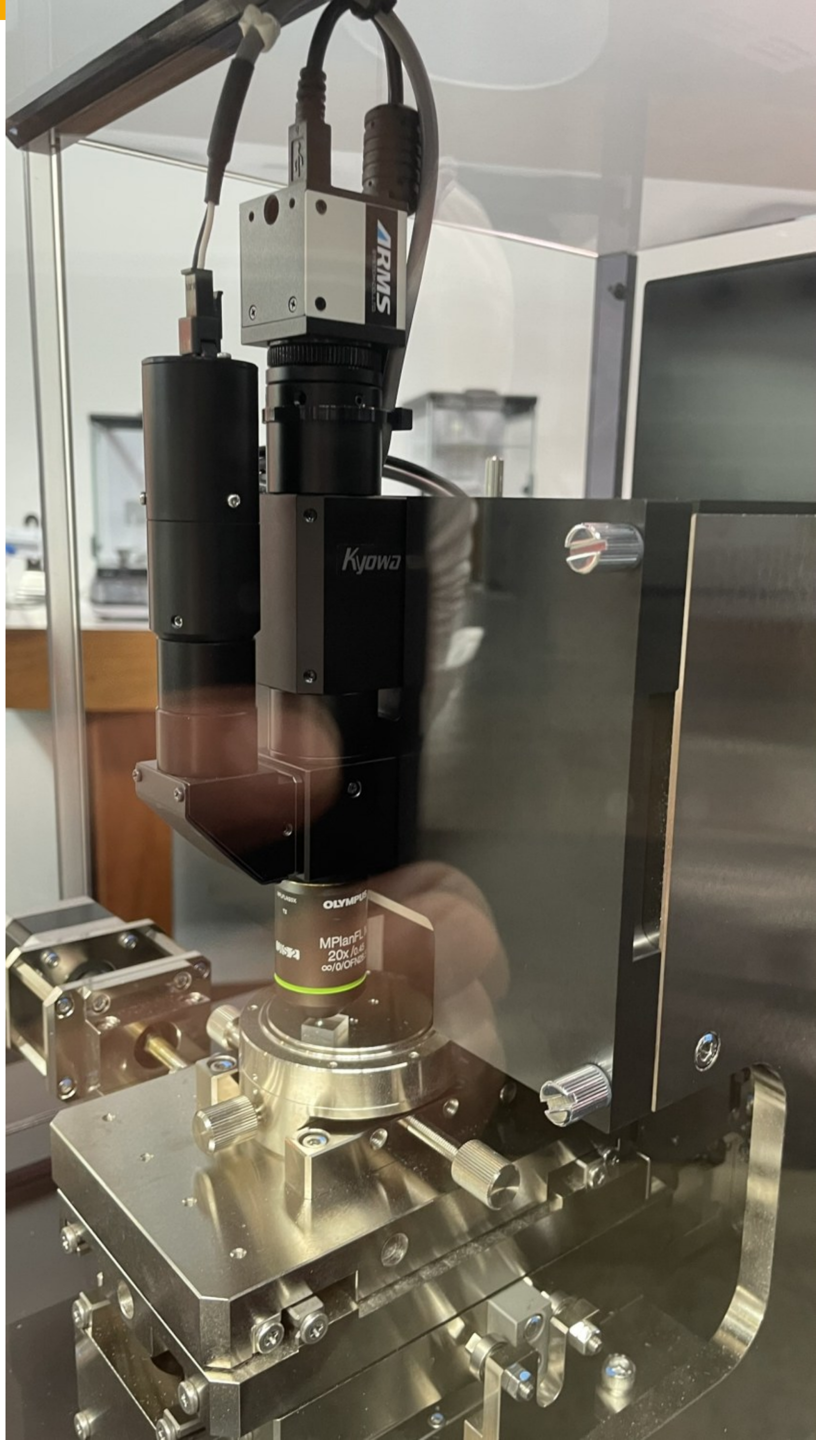
Underwater test (optional)

- Hardness test in the water/solution with immersion objective lens and indenter.

Underwater sample holder
*Sample size
φ20mm×t3mm
*Schematic







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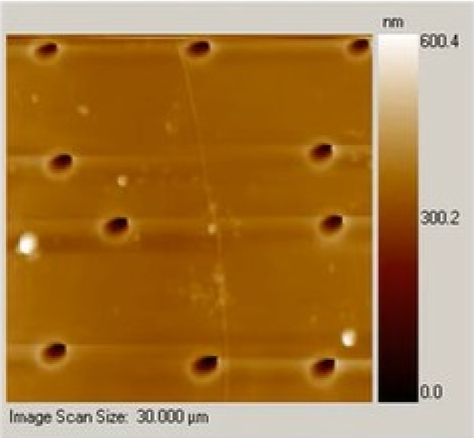
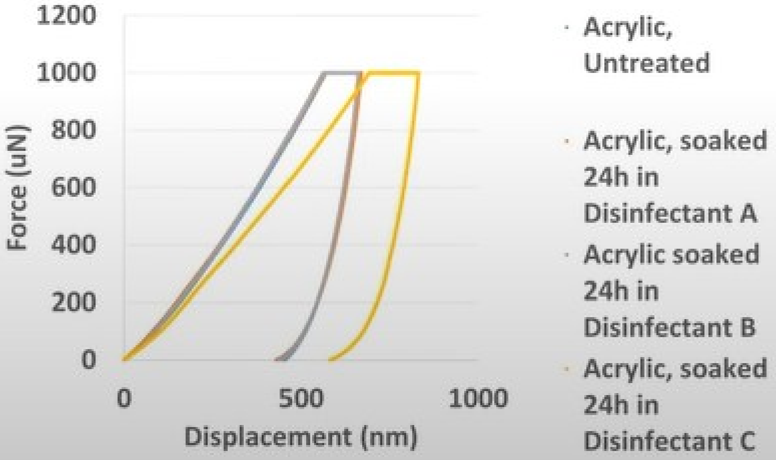
Možné hlubší poznání



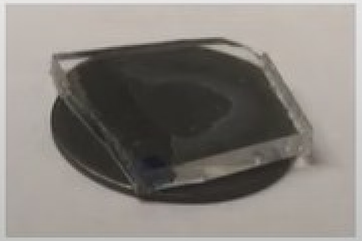
Scanned Probe Microscopy (SPM) Image of acrylic sample post-indentation

	Reduced Modulus (GPa)	Hardness (MPa)
Acrylic Untreated	5.20 GPa ± 0.03	356.5 MPa ± 6.09
Acrylic Disinfectant A	4.97 GPa ± 0.03	335.1 MPa ± 1.53
Acrylic Disinfectant B	5.28 GPa ± 0.05	355.3 MPa ± 3.26
Acrylic Disinfectant C	4.50 GPa ± 0.04	256.0 MPa ± 3.26

Average modulus and hardness measured for each sample



Single force-displacement curve of each sample overlaid as measured in this study



Source:
Strader et al.. **Nano Life;** Vol. 9, Iss. 4, (Dec 2019).

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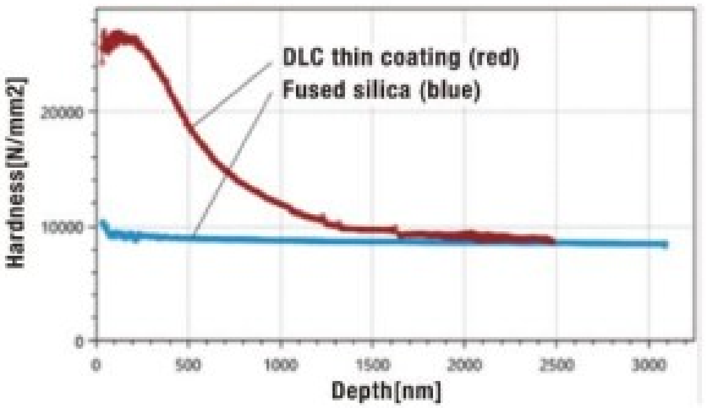
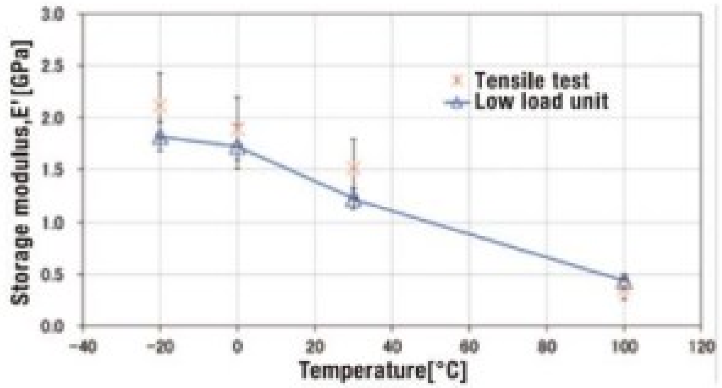
Test (standard function)



Heating / cooling viscoelasticity test (optional)

- **What is a viscoelasticity test?**
  This is the method to obtain elastic modulus (E'), the loss elastic modulus (E"), and the loss coefficient (tanδ). Applying small oscillation at the maximum loading.
- **Sample**
  Ultra High Molecular Weight Poly Ethylene (PE-UHMW)
- **Test results**
  The result with low-load unit shows same tendency as tensile test.

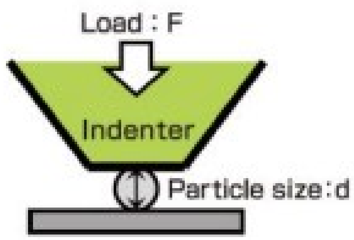


Compressive strength of micro particles test (optional)

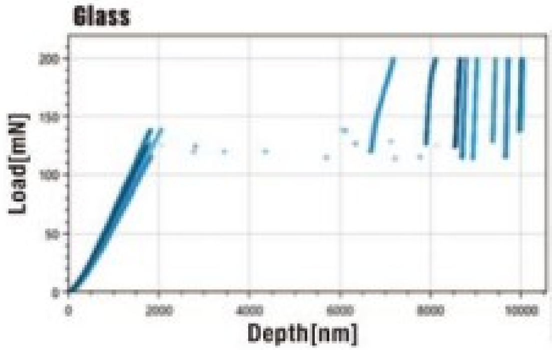
- This is the method to measure the fracture strength /deformation strength of particles in which a load is continuously applied by a flat punch.

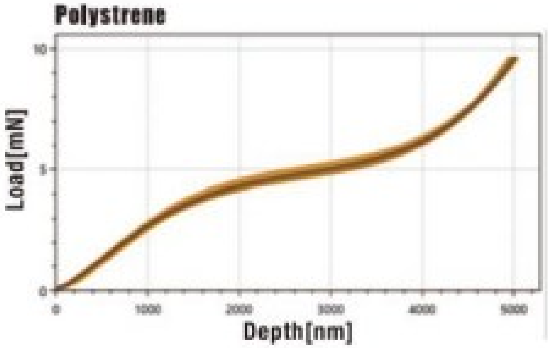
JIS Z 8844:2019 Test method of fracture and deformation strength of a fine particle

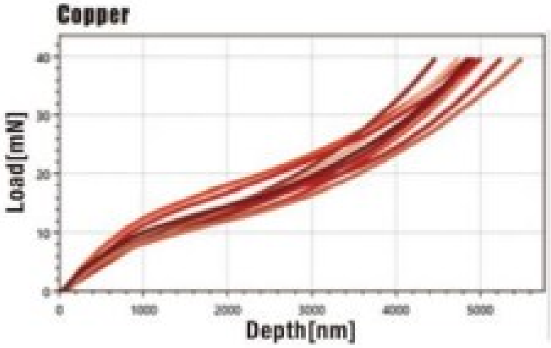
*Schematic



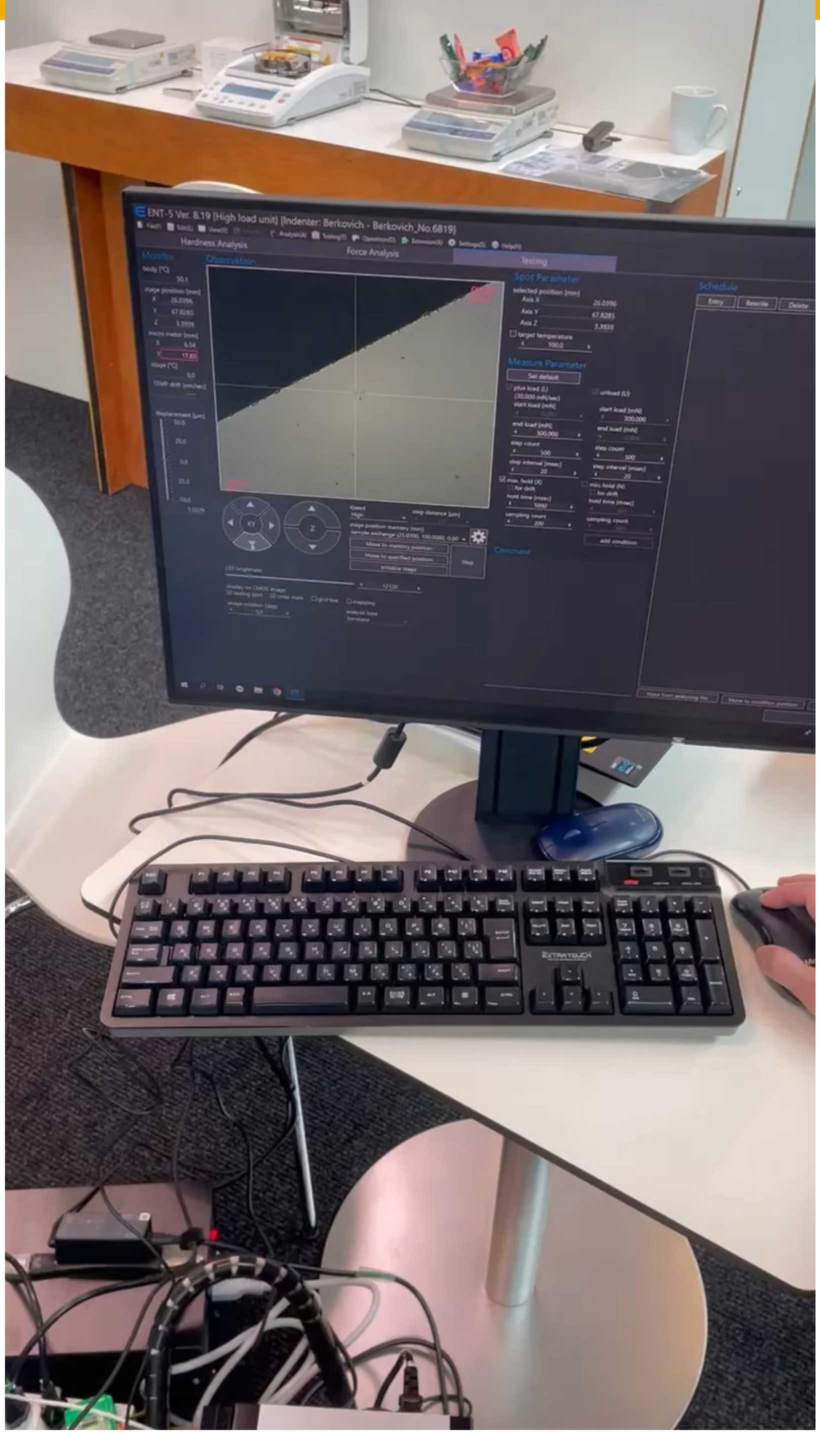
- **Sample**
  Glass(Particle size10μm±0.5μm)
  Polystyrene(Particle size10μm±0.5μm)
  Copper(Particle size10μm±2μm)
- **Test results**
  In ductile polystyrene and copper, the displacement changes continuously with the load.
  In brittle glass, discontinuous curve is seen when the glass is broken.







Video



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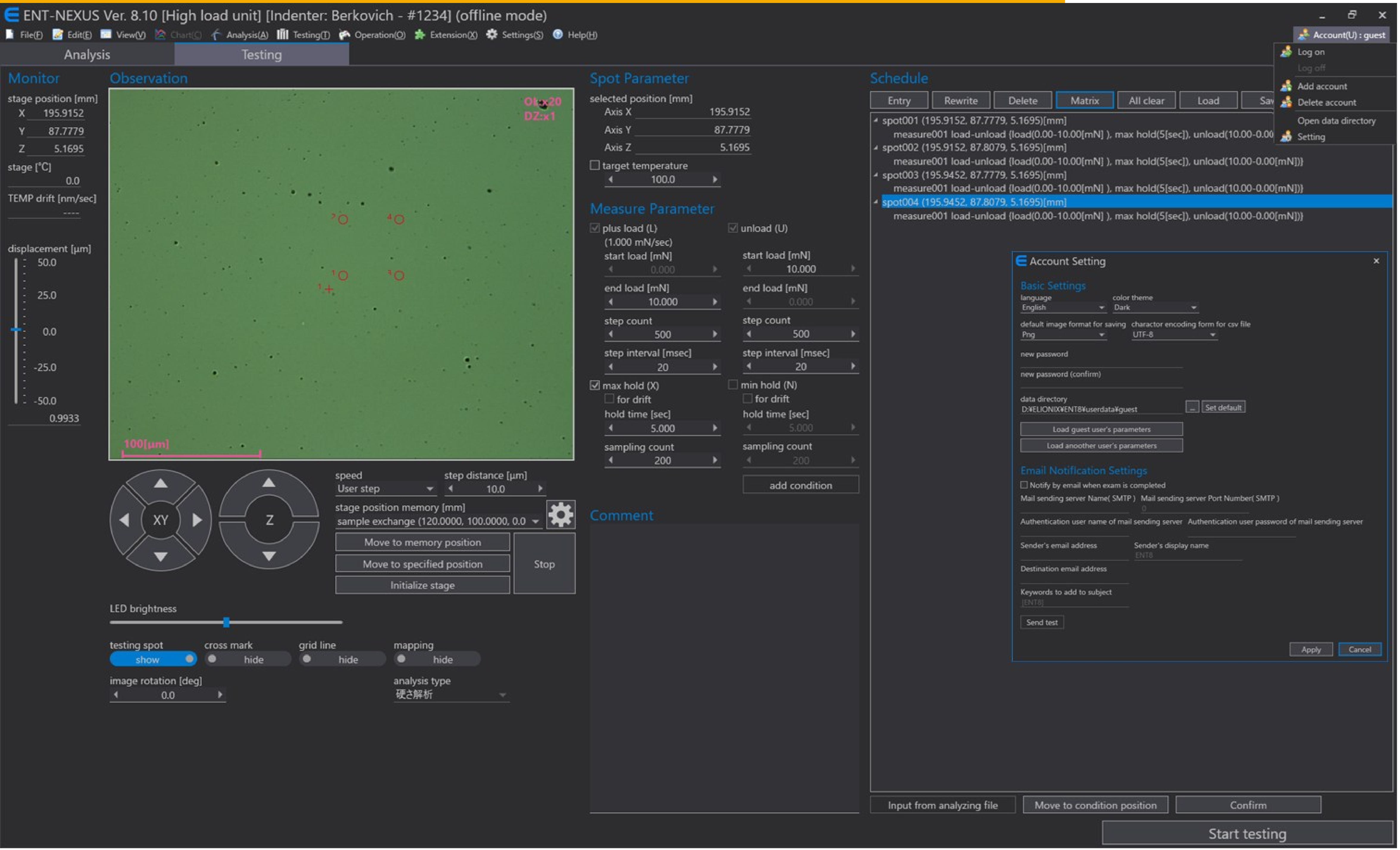
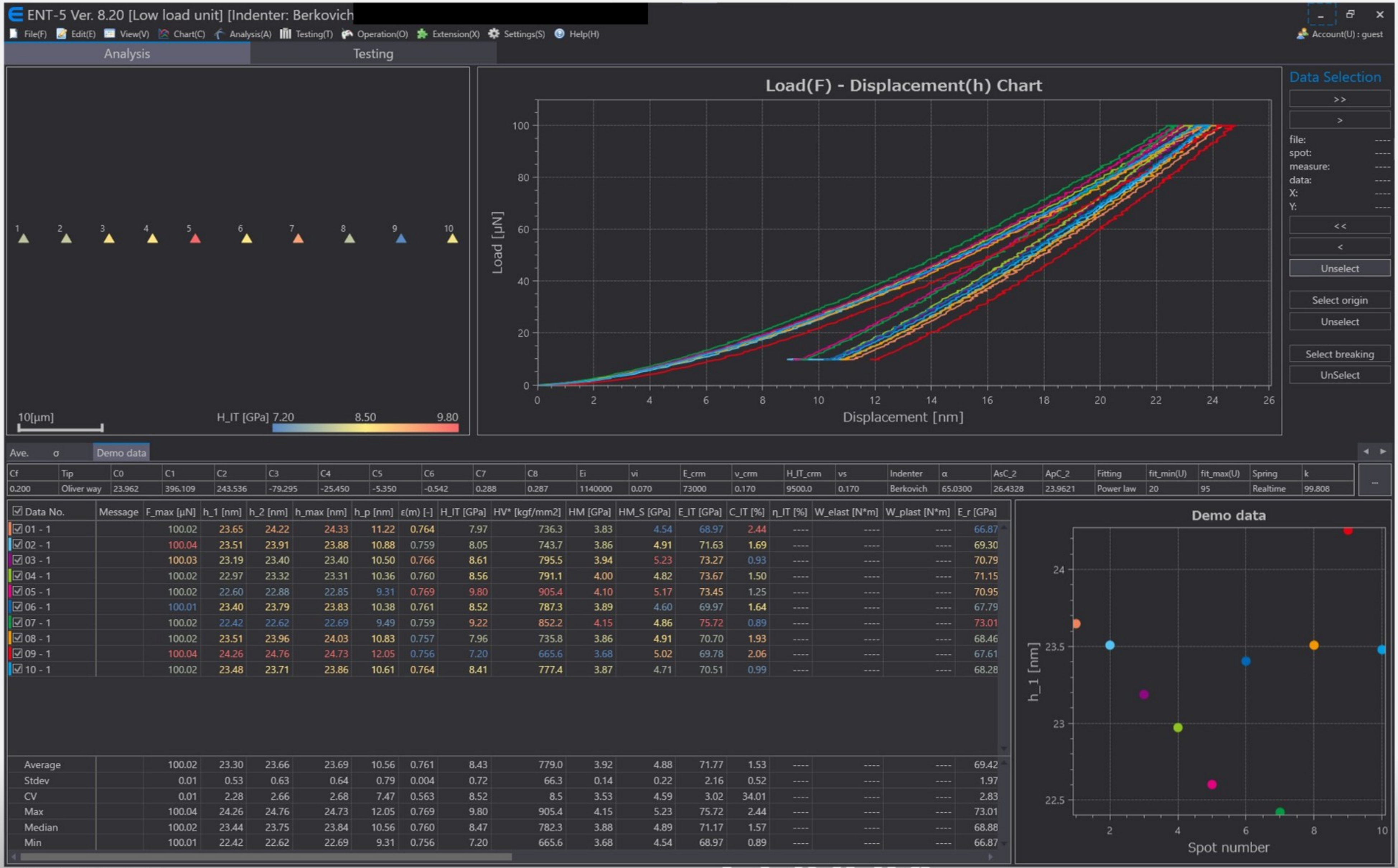
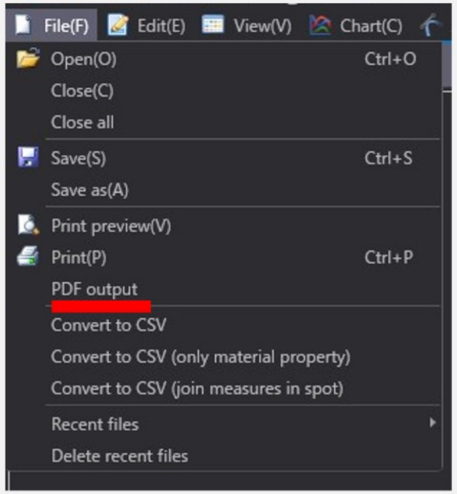

ENT-NEXUS Ver. 8.10 [High load unit] [Indenter: Berkovich - #1234] (offline mode)
File(F) Edit(E) View(V) Chart(C) Analysis(A) Testing(T) Operation(O) Extension(X) Settings(S) Help(H)
Account(U) : guest
Analysis
Testing
Monitor
stage position [mm]
X 195.9152
Y 87.7779
Z 5.1695
stage [℃]
0.0
TEMP drift [nm/sec]
----
displacement [µm]
50.0
25.0
0.0
-25.0
-50.0
0.9933
Observation
Ob:x20
DZ:x1
100[µm]
speed
User step
step distance [µm]
10.0
stage position memory [mm]
sample exchange (120.0000, 100.0000, 0.0
Move to memory position
Move to specified position
Initialize stage
Stop
XY
Z
LED brightness
testing spot
show
cross mark
hide
grid line
hide
mapping
hide
image rotation [deg]
0.0
analysis type
硬さ解析
Spot Parameter
selected position [mm]
Axis X 195.9152
Axis Y 87.7779
Axis Z 5.1695
target temperature
100.0
Measure Parameter
plus load (L)
(1.000 mN/sec)
unload (U)
start load [mN]
0.000
start load [mN]
10.000
end load [mN]
10.000
end load [mN]
0.000
step count
500
step count
500
step interval [msec]
20
step interval [msec]
20
max hold (X)
for drift
min hold (N)
for drift
hold time [sec]
5.000
hold time [sec]
5.000
sampling count
200
sampling count
200
add condition
Comment
Schedule
Entry
Rewrite
Delete
Matrix
All clear
Load
Sav
spot001 (195.9152, 87.7779, 5.1695)[mm]
measure001 load-unload {load(0.00-10.00[mN]), max hold(5[sec]), unload(10.00-0.00
spot002 (195.9152, 87.8079, 5.1695)[mm]
measure001 load-unload {load(0.00-10.00[mN]), max hold(5[sec]), unload(10.00-0.00[mN])}
spot003 (195.9452, 87.7779, 5.1695)[mm]
measure001 load-unload {load(0.00-10.00[mN]), max hold(5[sec]), unload(10.00-0.00[mN])}
spot004 (195.9452, 87.8079, 5.1695)[mm]
measure001 load-unload {load(0.00-10.00[mN]), max hold(5[sec]), unload(10.00-0.00[mN])}
Log on
Log off
Add account
Delete account
Open data directory
Setting
Account Setting
Basic Settings
language
English
color theme
Dark
default image format for saving
Png
charactor encoding form for csv file
UTF-8
new password
new password (confirm)
data directory
D:¥ELIONIX¥ENT8¥userdata¥guest
Set default
Load guest user's parameters
Load anoother user's parameters
Email Notification Settings
Notify by email when exam is completed
Mail sending server Name(SMTP)
Mail sending server Port Number(SMTP)
Authentication user name of mail sending server
Authentication user password of mail sending server
Sender's email address
Sender's display name
ENT8
Destination email address
Keywords to add to subject
[ENT8]
Send test
Apply
Cancel
Input from analyzing file
Move to condition position
Confirm
Start testing

REPORT OUTPUT (Original data)

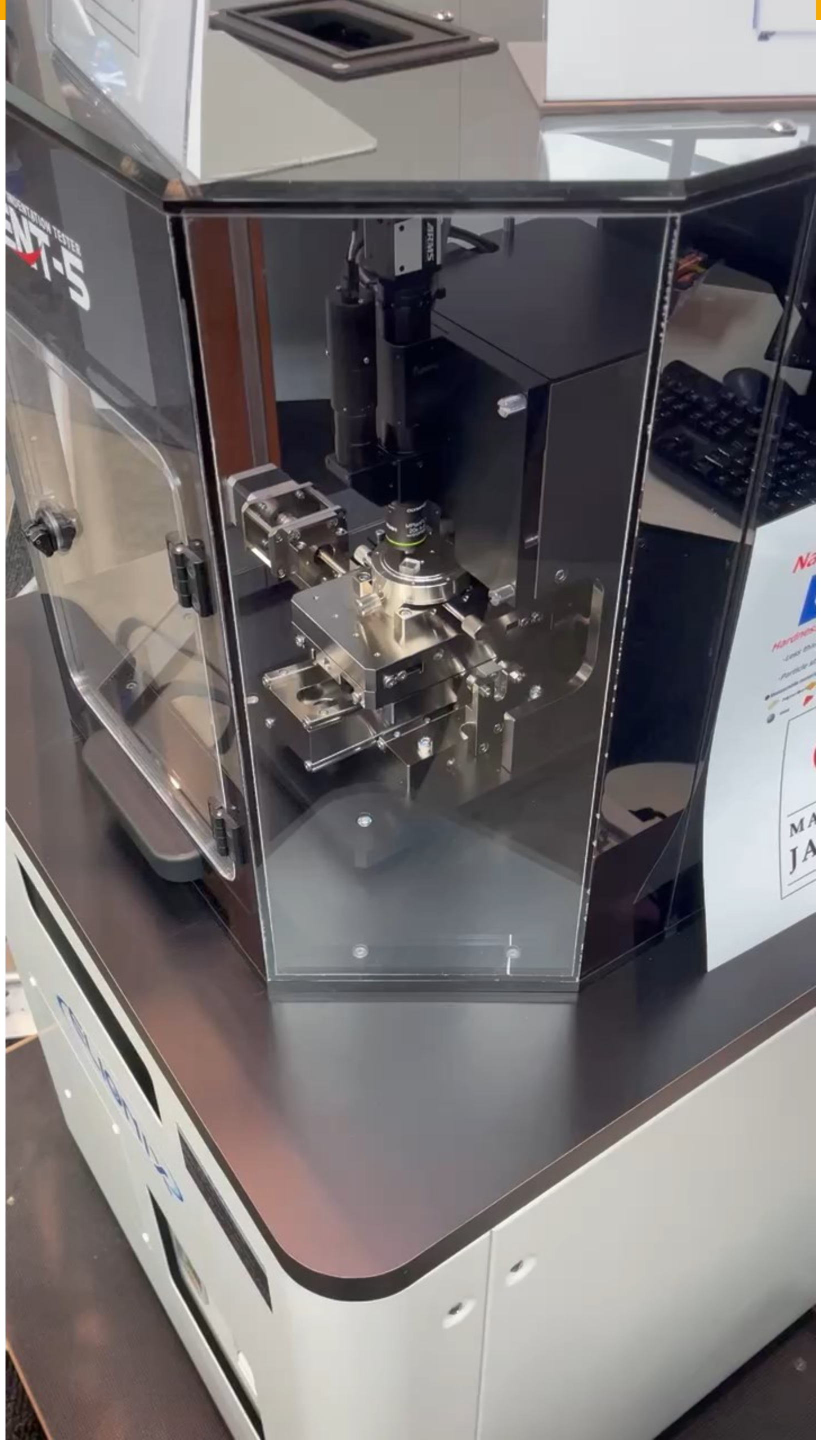
REFERENCE only.
Do NOT Distribute,
Just to show only.



PDF report data can be obtained by function of “PDF out put”.



Video



Dotazy

Děkuji za pozornost

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