Nanoindentation

Basic principles and applications

Content

- Principles
- Shapes of indenter tips
- Data analysis
- Analytic models
- Comparison of sharp and blunt tips
- Factors affecting nanoindentation
- CSM
- Possibility of DSI

What does it mean?

- **Nanoindentation** is called as:
  = depth sensing indentation (DSI)
  = instrumented indentation technique (IIT)
  = univerzal hardness test (UHT)

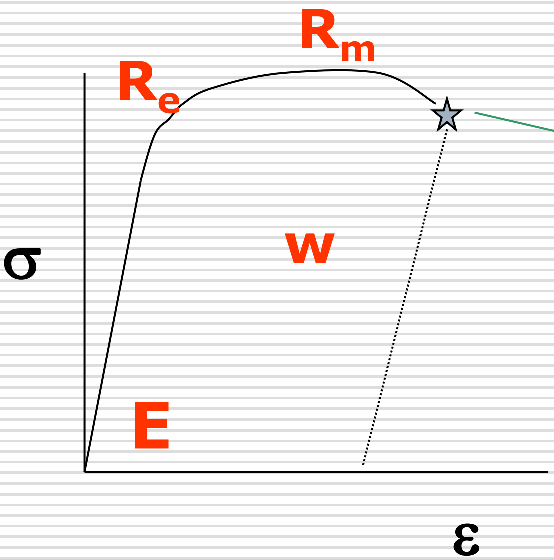
Standardized by **ISO 14577-1**

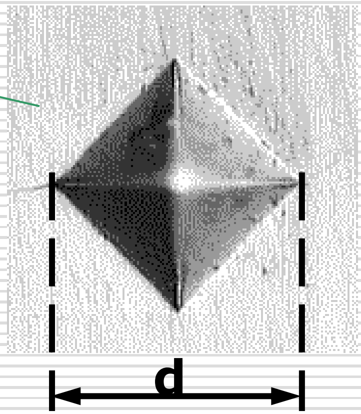
Table 1 — Ranges of application

Macro range	Micro range	Nano range[a]
2 N $\leq F \leq$ 30 kN	2 N $> F$; $h >$ 0, 2 µm	$h \leq$ 0, 2 µm

Inštrumentovaná tvrdosť (DSI) - úvod

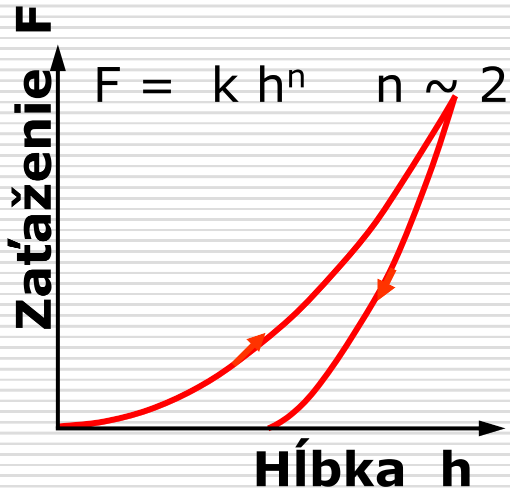
„Klasicky“





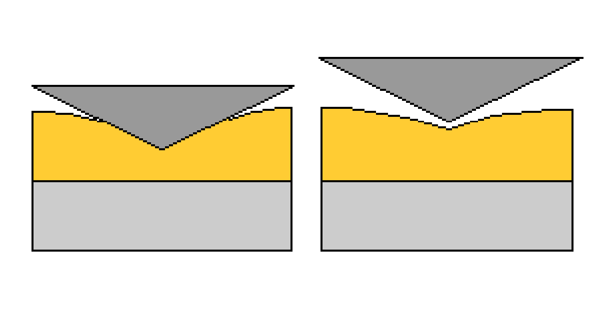
$$HV = 1{,}8544\ \frac{F}{d^2}$$

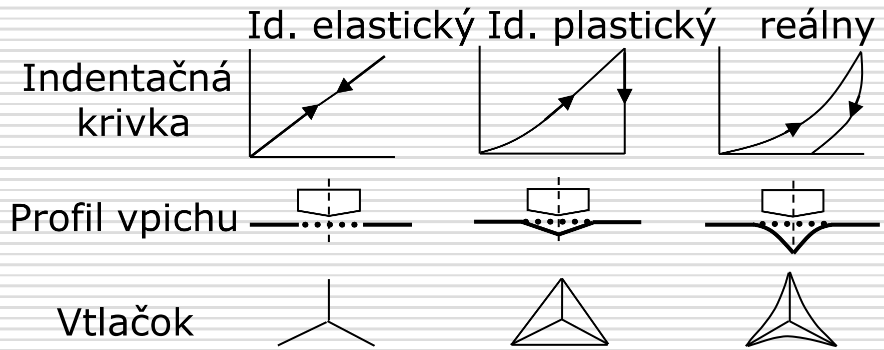
elastické a plastické vlastnosti vzorky určujú tvar indentačnej krivky



Zaťažovanie: statické
dynamické (kmitanie, rázy)

zaťaženie odľahčenie



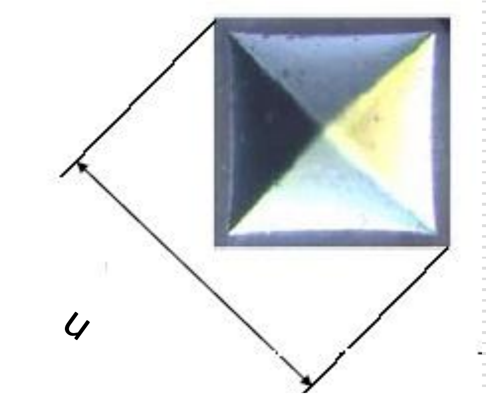


DSI vs. Clasic hardness tests

- **Vickers hardness**

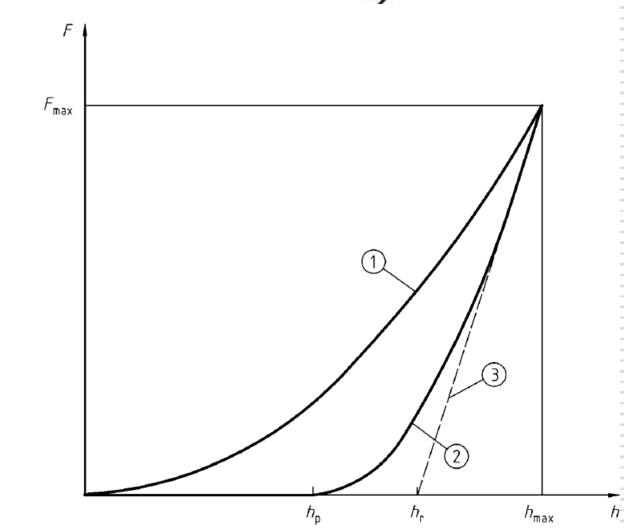
  area of imprints measured optically from surface

$$HV = k\frac{F}{u^2}$$

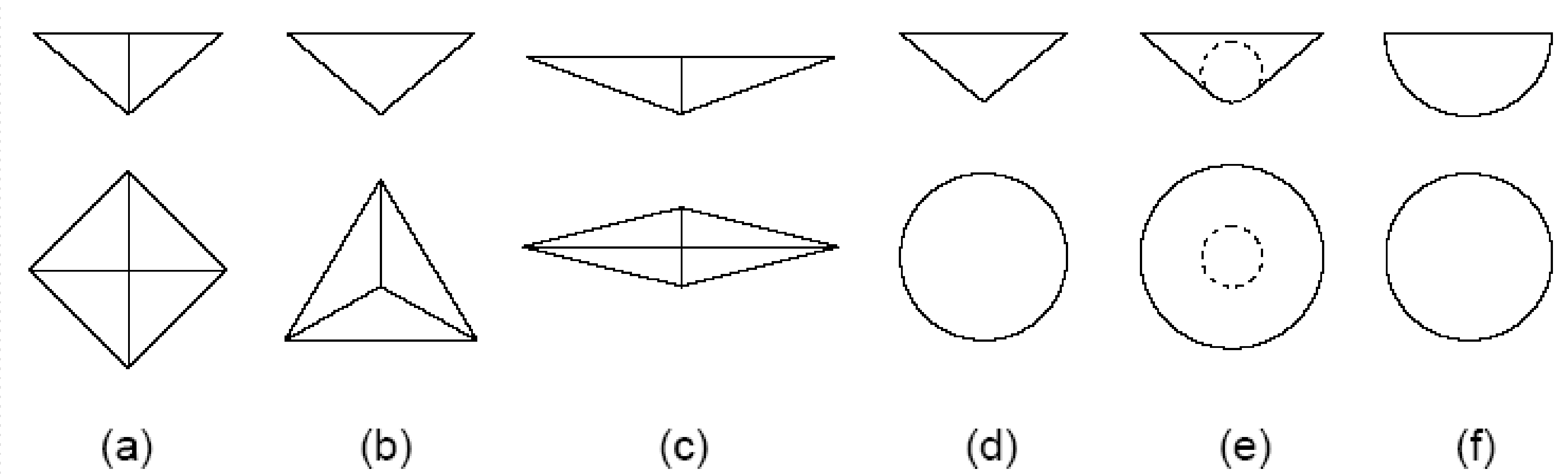


- **DSI**

  area is determined from indentation depth



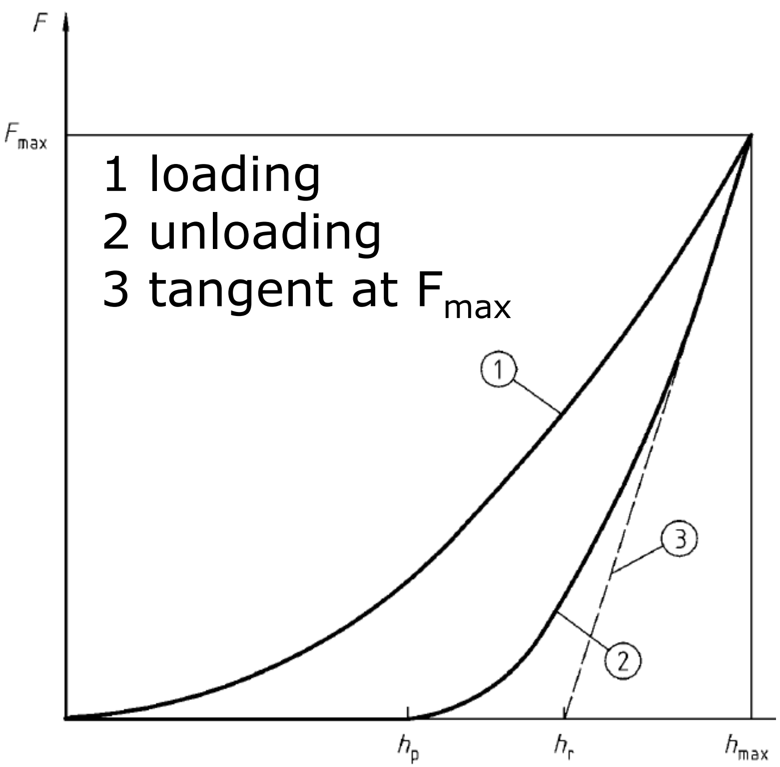
Schematics of indenter tips

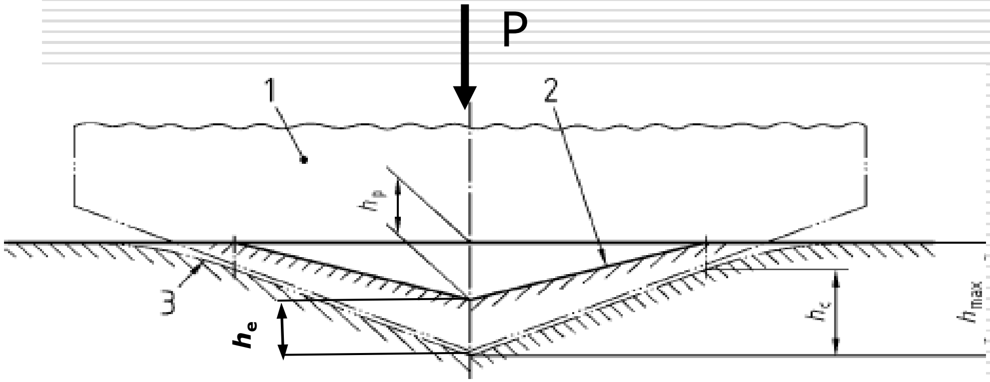


Vickers Berkovich Knoop Conical Rockwell Spherical

Data ananlysis

P applied load
h indenter displacement
h_p plastic deformation after load removal
h_e surface displacement at the contact perimeter





Analytical model-Basic concept

- **First developed by workers at the Baikov Institute of Metallurgy in Moscow during the 1970's (see Bulychev and Alekhin). The basic assumptions of this approach are**
  - **Deformation upon unloading is purely elastic**
  - **The compliance of the sample and of the indenter tip can be combined as springs in series**

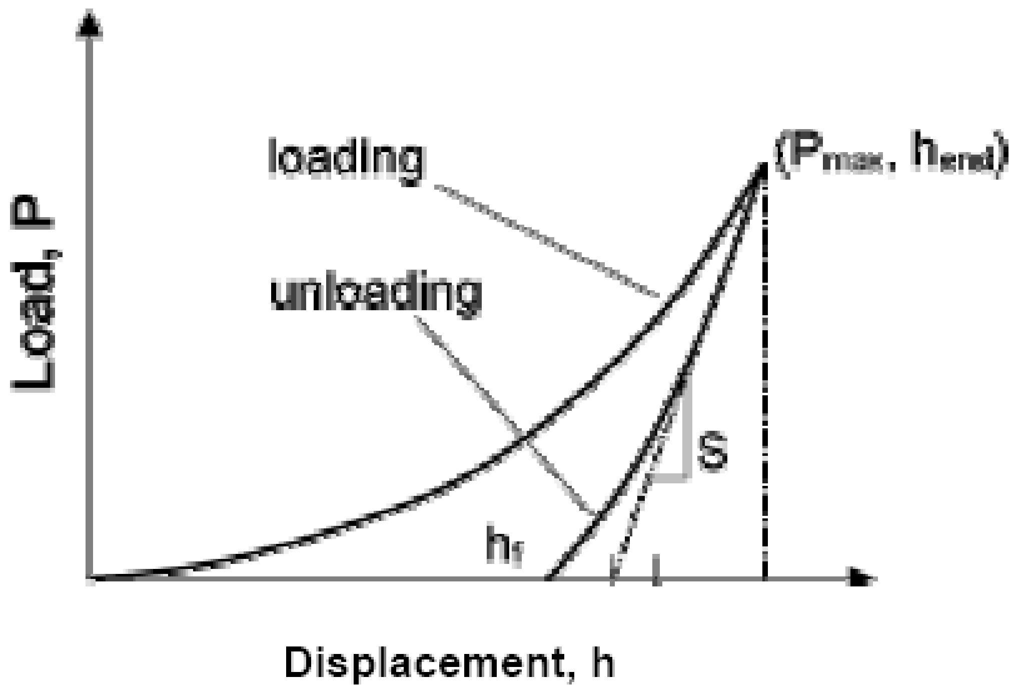
$$\frac{1}{E_r} = \frac{1-\upsilon_i^{\,2}}{E_i} + \frac{1-\upsilon_s^{\,2}}{E_s}$$

  - **The contact can be modeled using an analytical model for contact between a rigid indenter of defined shape with a homogeneous isotropic elastic half space using**

$$S = \frac{2\sqrt{A}}{\sqrt{\pi}} E_r \qquad Er = \frac{1}{\beta}\frac{dP}{dh}\frac{1}{2}\frac{\sqrt{\pi}}{\sqrt{Ac}}$$

- **where S is the contact stiffness and A the contact area. presented by Sneddon, Pharr, Oliver, Brotzen. The equation applies to tips with a wide range of shapes.**

Analytical Model–Oliver & Pharr



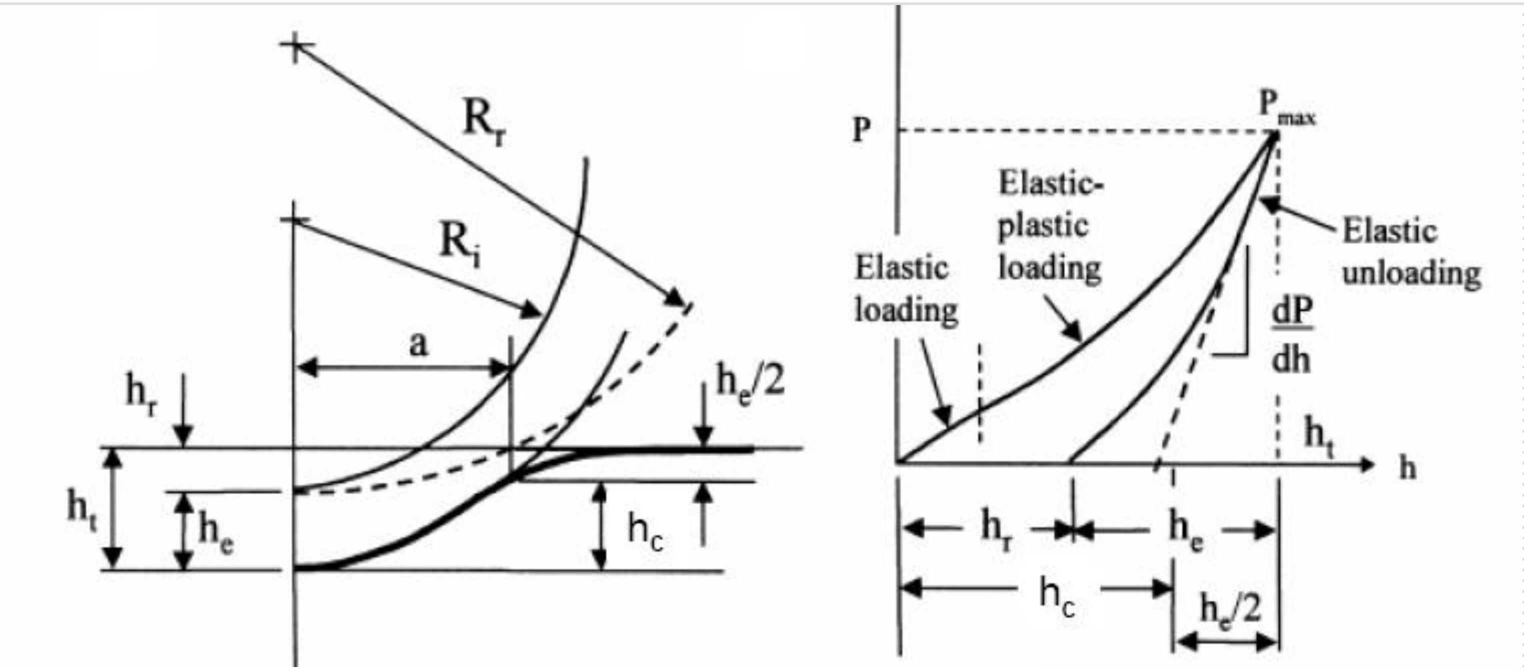
$$P = \alpha (h - h_f)^m$$

$$h_c = h_t - \varepsilon \frac{P}{S}$$

$$A_c = f(h_c)$$

Oliver & Pharr, J Mater Res, 1992

Analytical model – Doerner&Nix



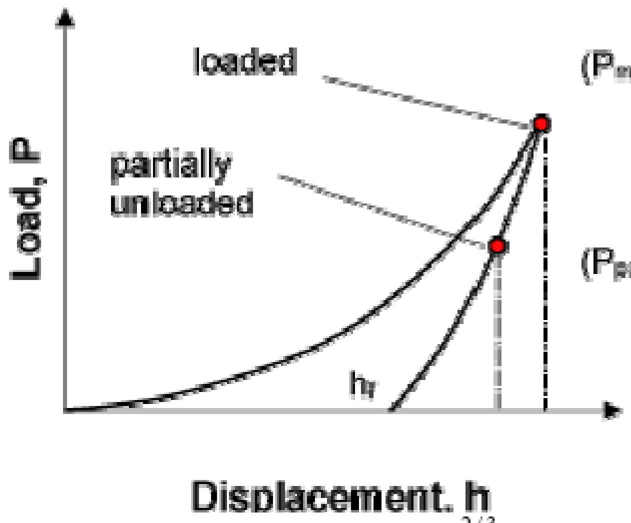
$$A_c = f(h_c)$$

$$H = \frac{P_{max}}{A_c}$$

Doerner, Nix, J Mater Res, 1986

Analytical Model – Field - Swain

Field, Swain, J Mater Res, 1993



$$P = \frac{4}{3} E_r R^{1/2} h^{3/2}$$

$$h_f = \frac{h_{part}\left(\frac{P_{max}}{P_{part}}\right)^{2/3} - h_{end}}{\left(\frac{P_{max}}{P_{part}}\right)^{2/3} - 1}$$

$$a = \sqrt{R(h_{end} + h_f) - \left(\frac{h_{end} + h_f}{2}\right)^2}$$

They treated the indentation as a reloading of a preformed impression with depth hf into reconformation with the indenter

Inštrumentovaná tvrdosť - veličiny

Tvrdosť Martensova

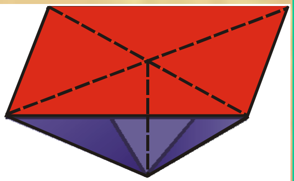
$$HM = \frac{F}{A_s(h)} = \frac{F}{k.h^2}$$

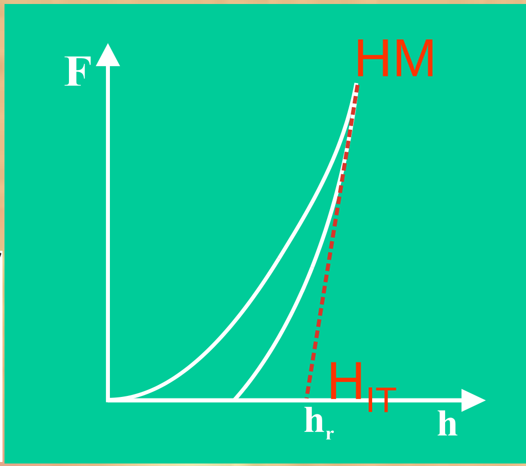
Indentačná

$$H_{IT} = \frac{F_{max}}{A_P(h_r)}$$

$A_P(h)$

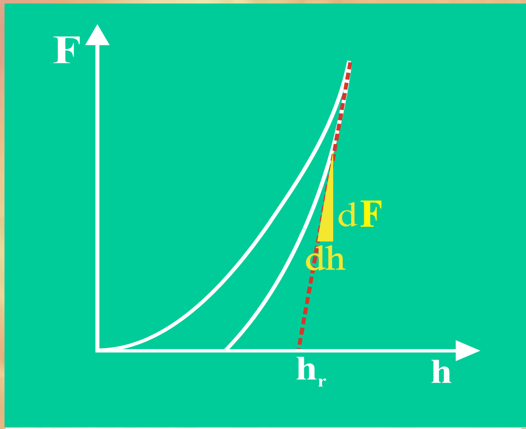
$A_s(h)$





Modul pružnosti

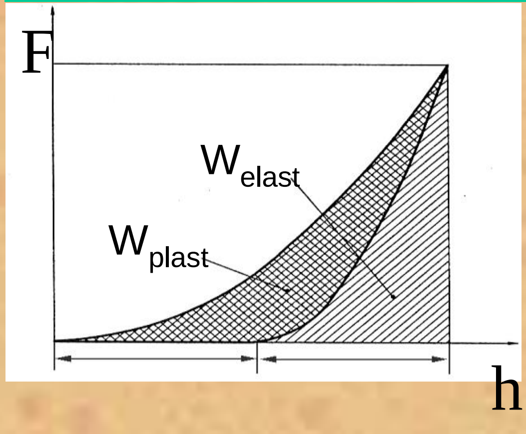
$$\frac{1}{E_r} = \frac{(1-\nu^2)}{E} + \frac{(1-\nu_i^2)}{E_i}$$



Indentačná práca W

$$W_{total} = W_{elast} + W_{plast}$$

$$\eta_{IT} = \frac{W_{elast}}{W_{total}} 100\%$$

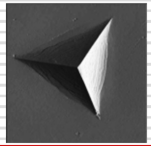
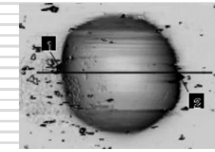


Analysis results

- Reduced modulus $$\frac{1}{E^*} = \frac{1-\upsilon^2}{E} + \frac{1-\upsilon'^2}{E'}$$ E: modulus of specimen
E′: modulus of indenter
- Stiffness $$\frac{dP}{dh} = 2E^* \frac{\sqrt{A}}{\sqrt{p}}$$
- Contact area $$A = 3\sqrt{3}h_p{}^2 \tan^2 65.3 = 24.5h_p{}^2$$ for Berkovich indenter
- Hardness $$H = \frac{P}{24.5h_p{}^2}$$
- Elastic modulus $$E^* = \frac{dP}{dh}\frac{1}{2h_p}\frac{1}{\beta}\sqrt{\frac{\pi}{24.5}}$$

$\beta = 1.034$ for Berkovich indenter

Comparison of indenters

Sharp  Blunt (sphere) 

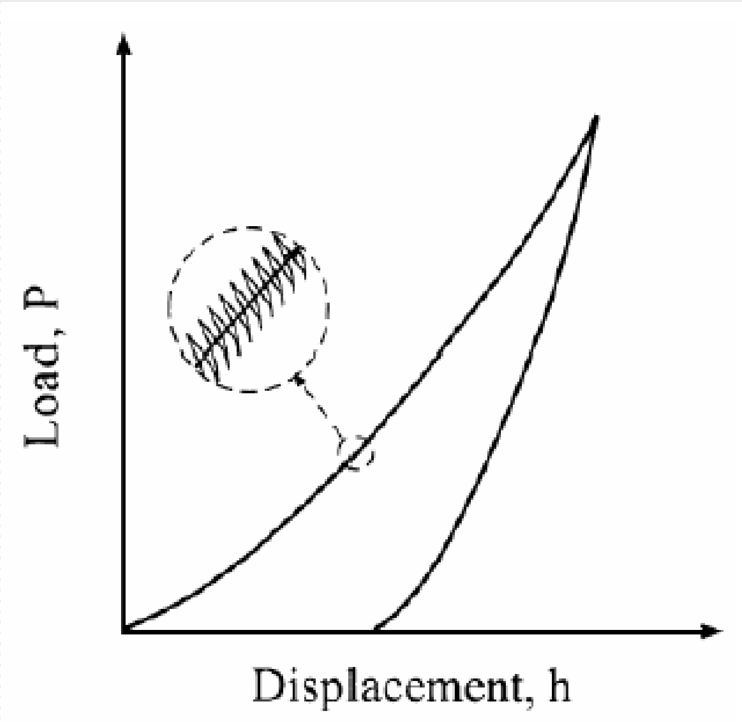
Sharp

- **Advantage**
  - Sharp and well-defined tip geometry
  - Well-defined plastic deformation into the surface
  - Good for measuring modulus and hardness values
- **Disadvantage**
  - Elastic-plastic transition is not clear.

Blunt (sphere)

- **Advantage**
  - Extended elastic-plastic deformation
  - Load displacement results can be converted to indentation stress-strain curve.
  - Useful in determination of yield point
- **Disadvantage**
  - Tip geometry is not very sharp and the spherical surface is not always perfect.

Continuous Stiffness Measurement (CSM)



- During the loading the system simultaneously superimposing an oscillating force with a force amplitude generally several orders of magnitude smaller than the nominal load
- It provides accurate measurements of contact stiffness at all depth
- The stiffness values enable us to calculate the contact radius at any depth more precisely.

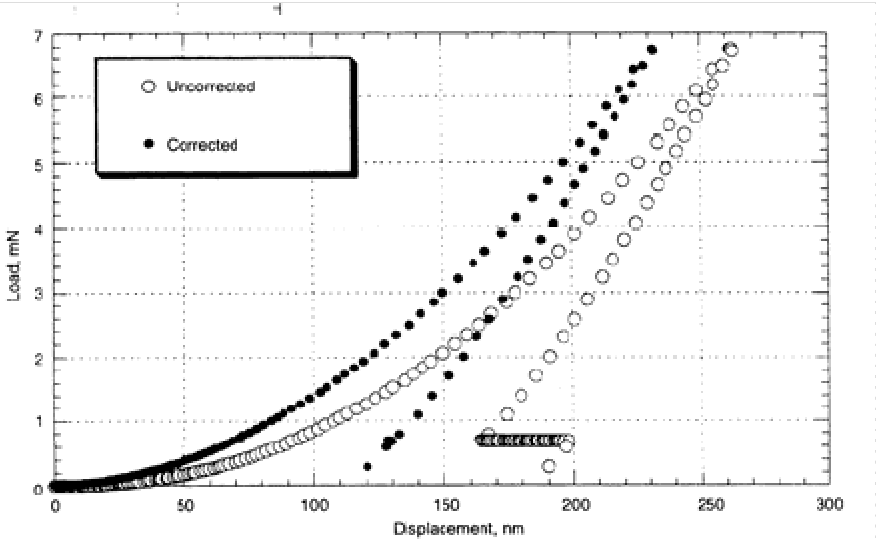
Oliver, Pharr, Nix, J Mater Res, 2004

Factors affecting DSI

- **Thermal Drift**
- Initial penetration depth
- **Instrument compliance**
- Indenter geometry
- **Piling-up and sinking-in**
- Indentation size effect
- **Surface roughness**
- **Tip rounding**
- Residual stress
- Specimen preparation

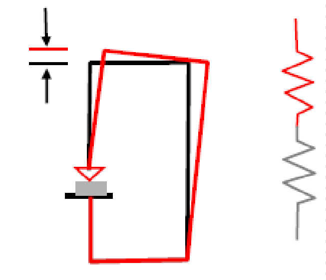
Thermal Drift

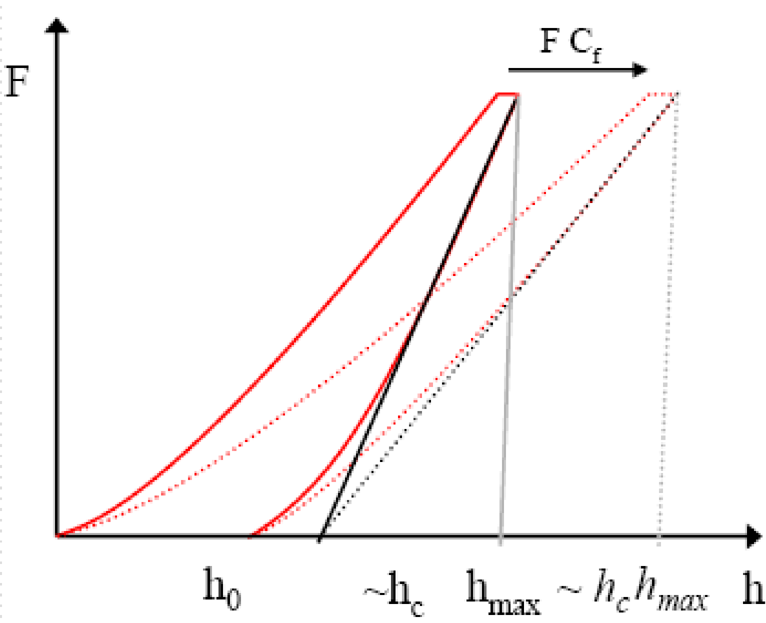
- Thermal drift can be due to
  - Different thermal expansion in the machine
  - Heat generation in the electronic devices
- Thermal drift is especially important when studying time varying phenomena like creep



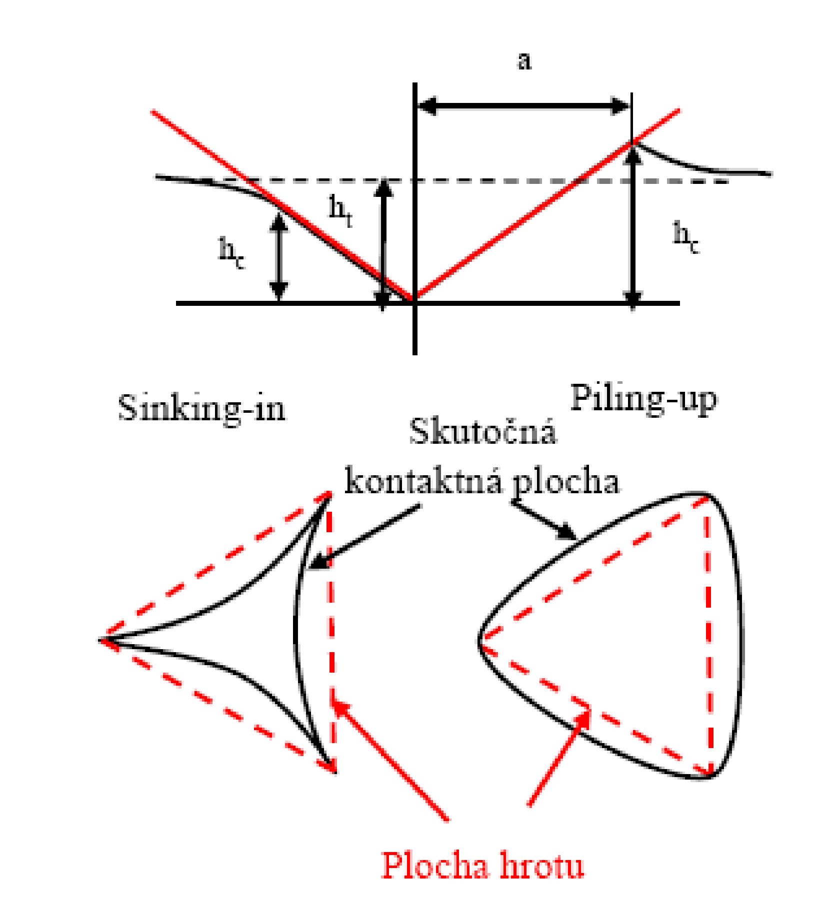
Instrument compliance

- Displacement arising from the compliance of the testing machine must be subtracted from the load-displacement data
- The machine compliance includes compliances in the sample and tip mounting and may vary from test to test

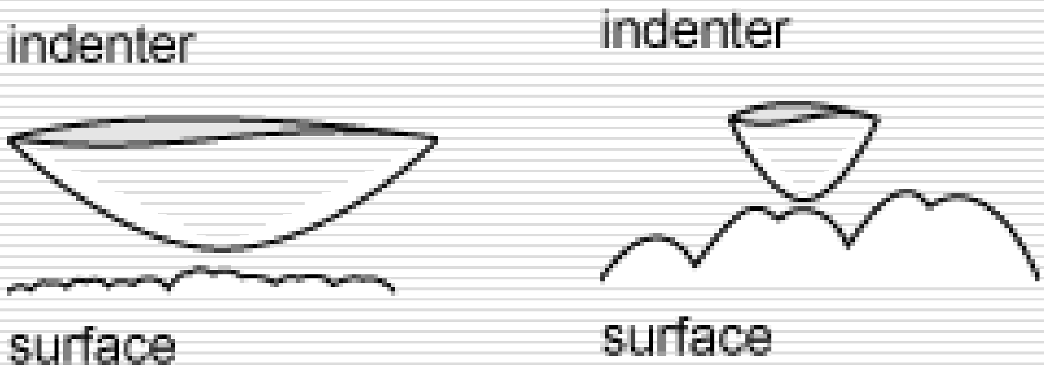




Piling-up and sinking-in

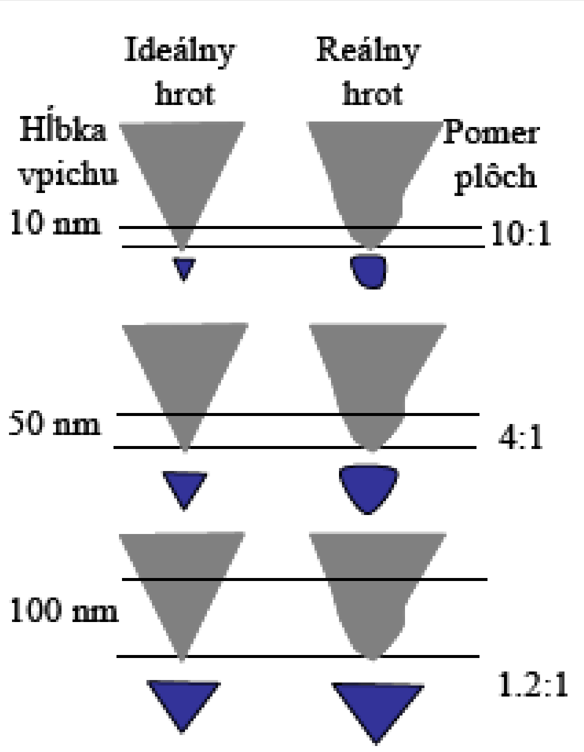


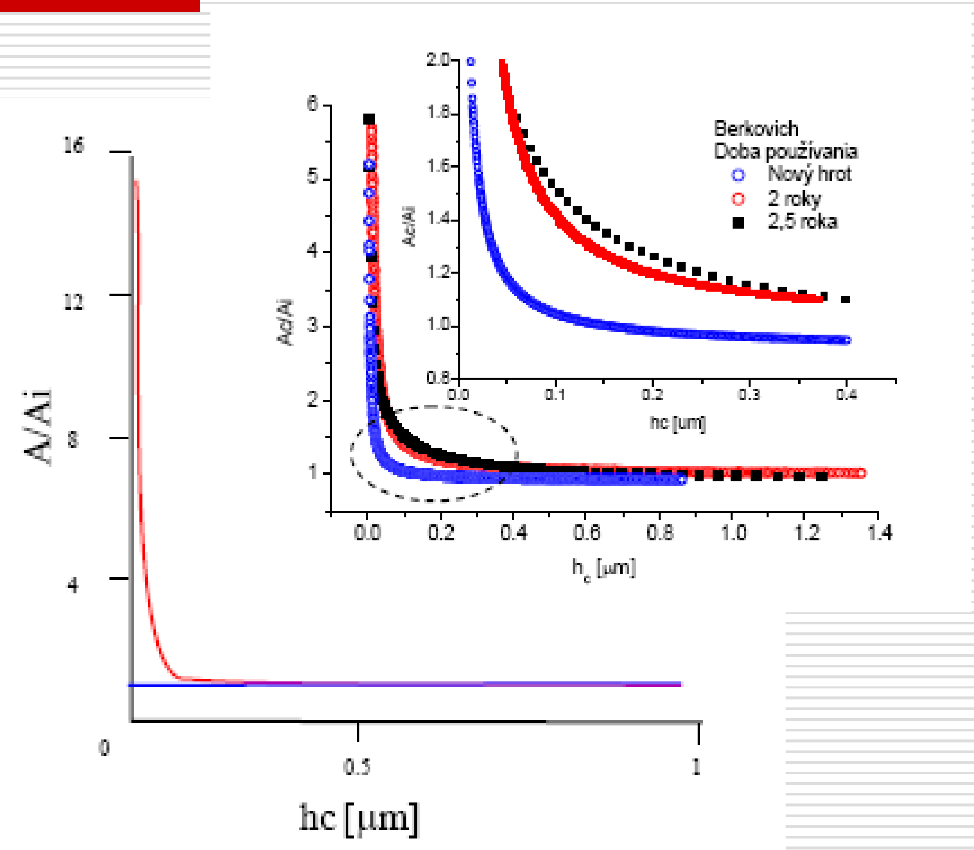
Surface roughness



- As sample roughness does have a significant effect on the measured mechanical properties, one could either try to incorporate a model to account for the roughness or try to use large indentation depths at which the influence of the surface roughness is negligible.
- A model to account for roughness effects on the measured hardness is proposed by Bobji and Biswas.

Tip rounding





DSI

- **Tests**
  - Nanohardness/Elastic modulus
  - Continuous Stiffness Measurements
  - Acoustic Emmisions
  - Properties at Various Temperature
  - Friction Coefficient
  - Wear Tests
  - Adhesion
  - NanoScratch Resistance
  - Fracture Toughness
  - Delamination
  - Shape memory
  - Phase transformation
- **Applications**
  - Fracture Analysis
  - Anti-Wear Films
  - Lubricant Effect
  - Paints and Coatings
  - Nanomachining
  - Bio-materials
  - Metal-Matrix Composites
  - Diamond Like Carbon Coatings
  - Semiconductors
  - Polymers
  - Thin Films Testing and Development
  - Property/Processing Relationships

0,1 mN – 2 N	Zaťaženie	max 10 N
10 μm	Rozsah hĺbky	500 μm
	Presnosť:	
1 nm	Hĺbka	0,01 nm
20 μN	Zaťaženie	0,05 μN

Nanotvrdomery

Indentation curve

Hardness	H
Modulus of elasticity	E
Indentation work	W W_{el} W_{pl}
Relaxation parameter	R_{IT}
Creep parameter	C_{IT}

Derived:

stress – strain curve, $\sigma - \varepsilon$

Yield strength, tensile strength

Fracture toughness K_{IC}

Thermal shock resistance (ceramics)

Adhesion strength, wear

Phase boundary strength

Fatigue properties

Residual stresses σ_{rez}

MOŽNOSTI: Inštrumentovaná tvrdosť (DSI)

OBJEKTY

Zrná a ich hranice
Vrstvy a povlaky
Častice (inklúzie, precipitáty, ...)
Fázy a štruktúrne zložky
Oblasti zvaru
„Bulk“

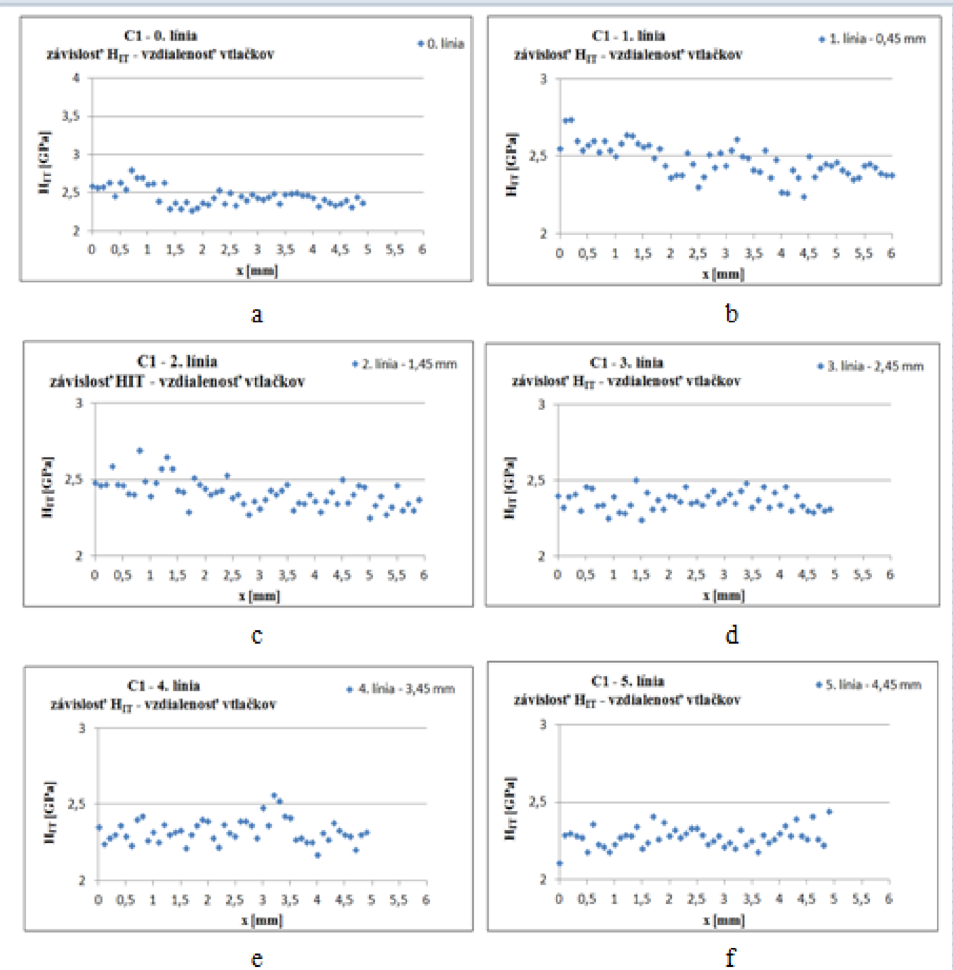
VELIČINY

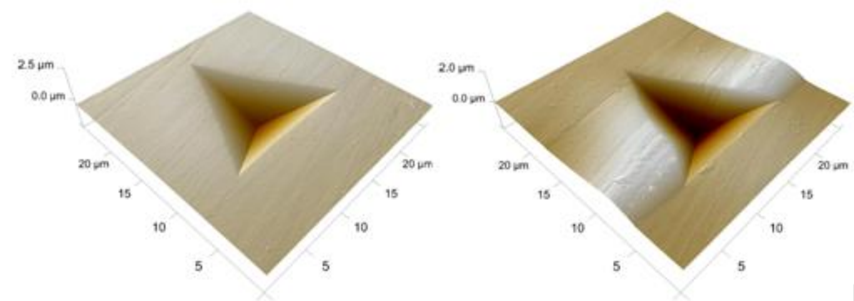
Tvrdosť H
Modul pružnosti E
Medza klzu, pevnosti
Deformačná (indentačná) energia W W_{el} W_{pl}
Lomová húževnatosť K_{IC}
Relaxačné a creepové vlastnosti
Deformačná krivka $\sigma - \varepsilon$
Odolnosť voči tepelným rázom (keramika)
Adhézne vlastnosti – adhézna pevnosť
Pevnosť interfázového rozhrania
Únavové vlastnosti
Zvyškové napätia σ_{rez}

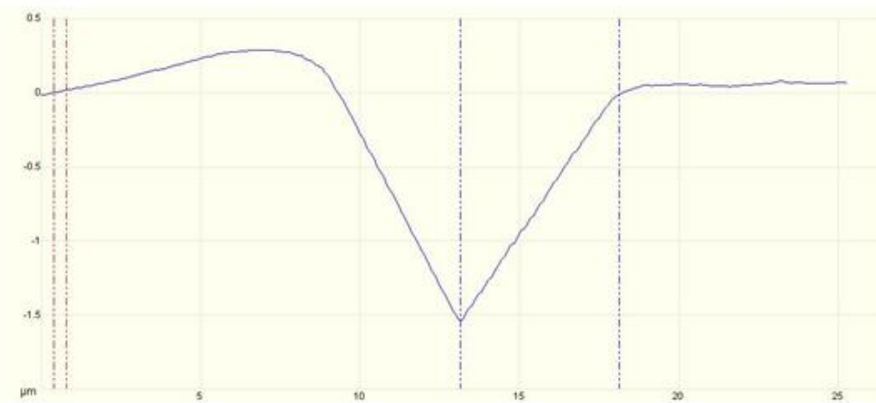
MATERIÁLY

Kovy
Keramika
Gradientne materiály
Plasty (reologické chovanie)
Porézne materiály
Kompozity
Biologické materiály
Cement

EXPERIMENTÁLNA ČASŤ – VZORKA C1

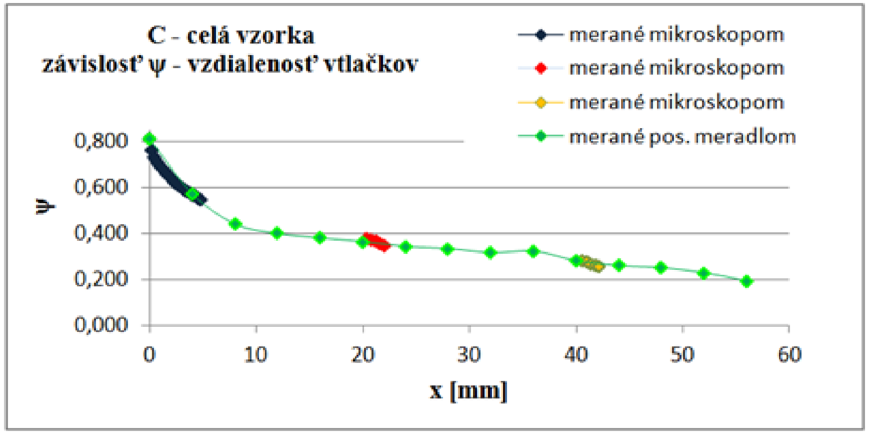


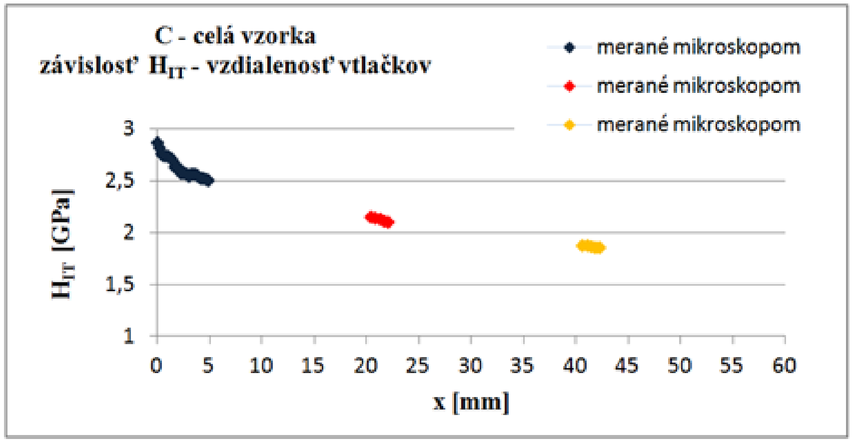


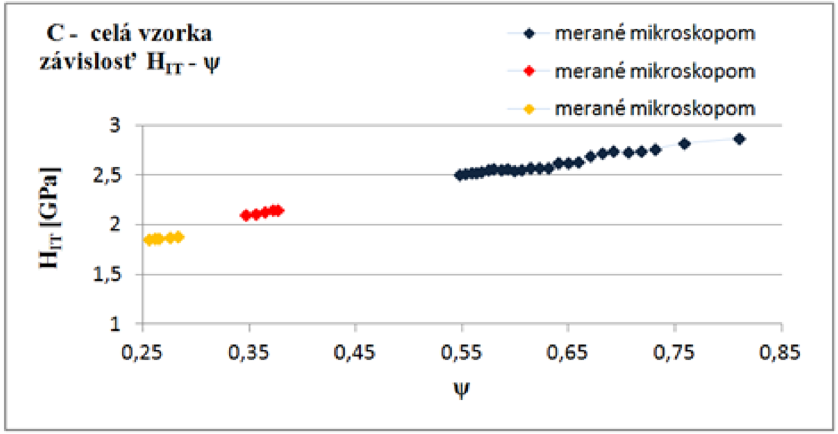


Vzorka C1 (závislosť H_{IT} – x)

EXPERIMENTÁLNA ČASŤ – ŤAHOVÁ VZORKA C





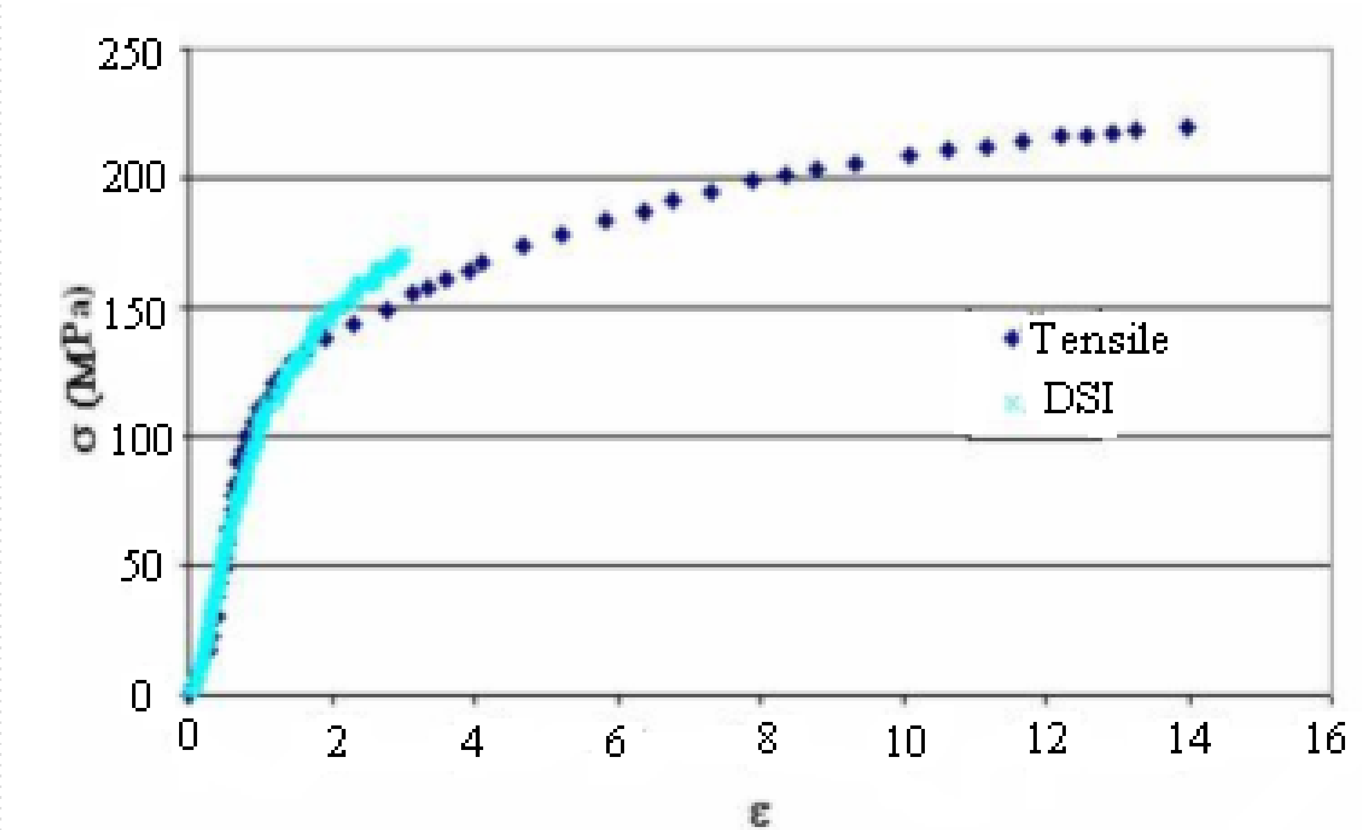


Ťahové teleso C;
závislosť ψ – x , závislosť H_{IT} – x,
závislosť H_{IT} – ψ

Comparison - experiment

(indentation - tensile)

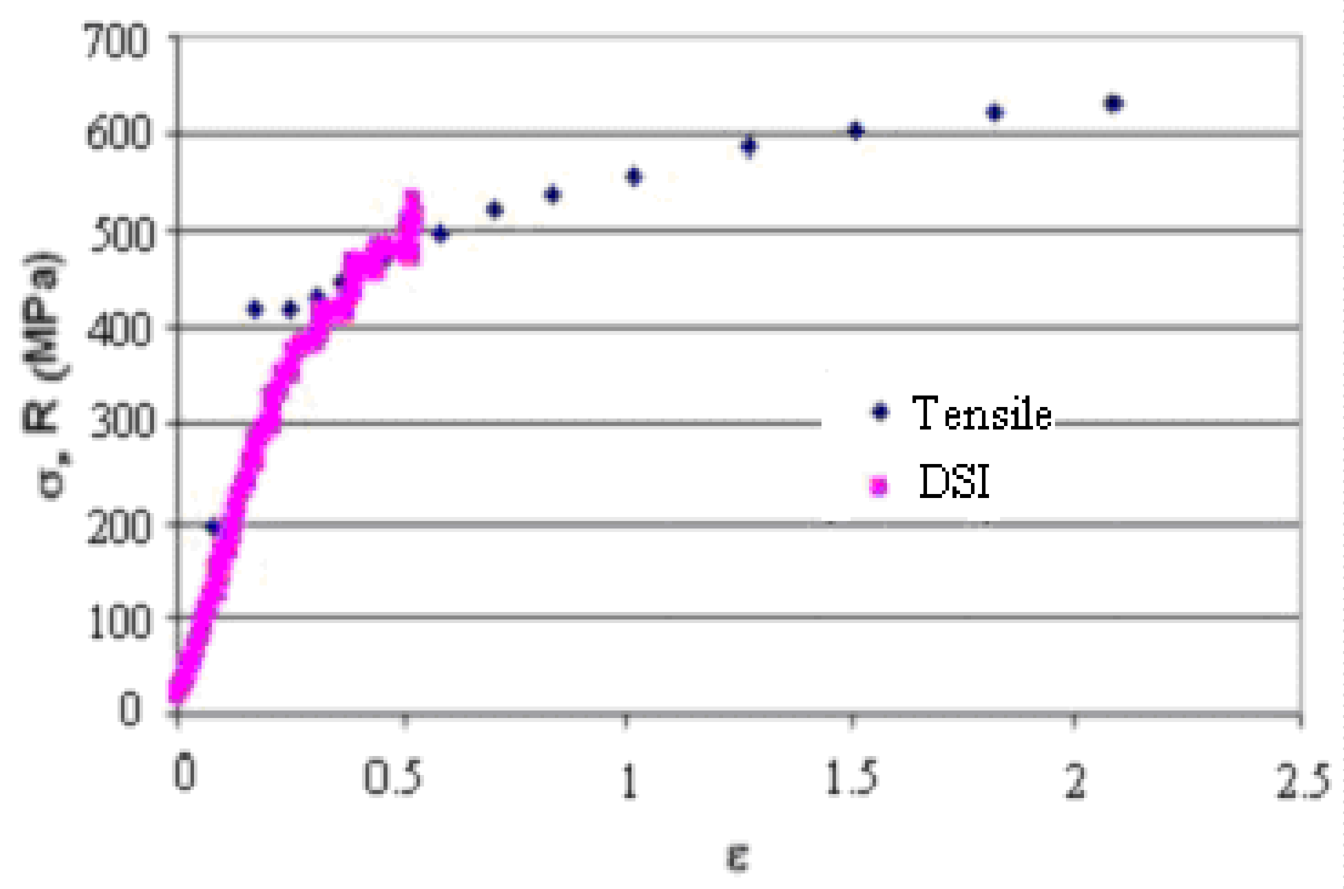
Aluminium alloy AA6063:



$R_{p0,2}$ – 99,5 MPa (Tensile)

– 110 MPa (DSI)

Steel St 52:



R_e - 410 MPa (Tensile)

$R_{p0,2}$ - 450 MPa (DSI)