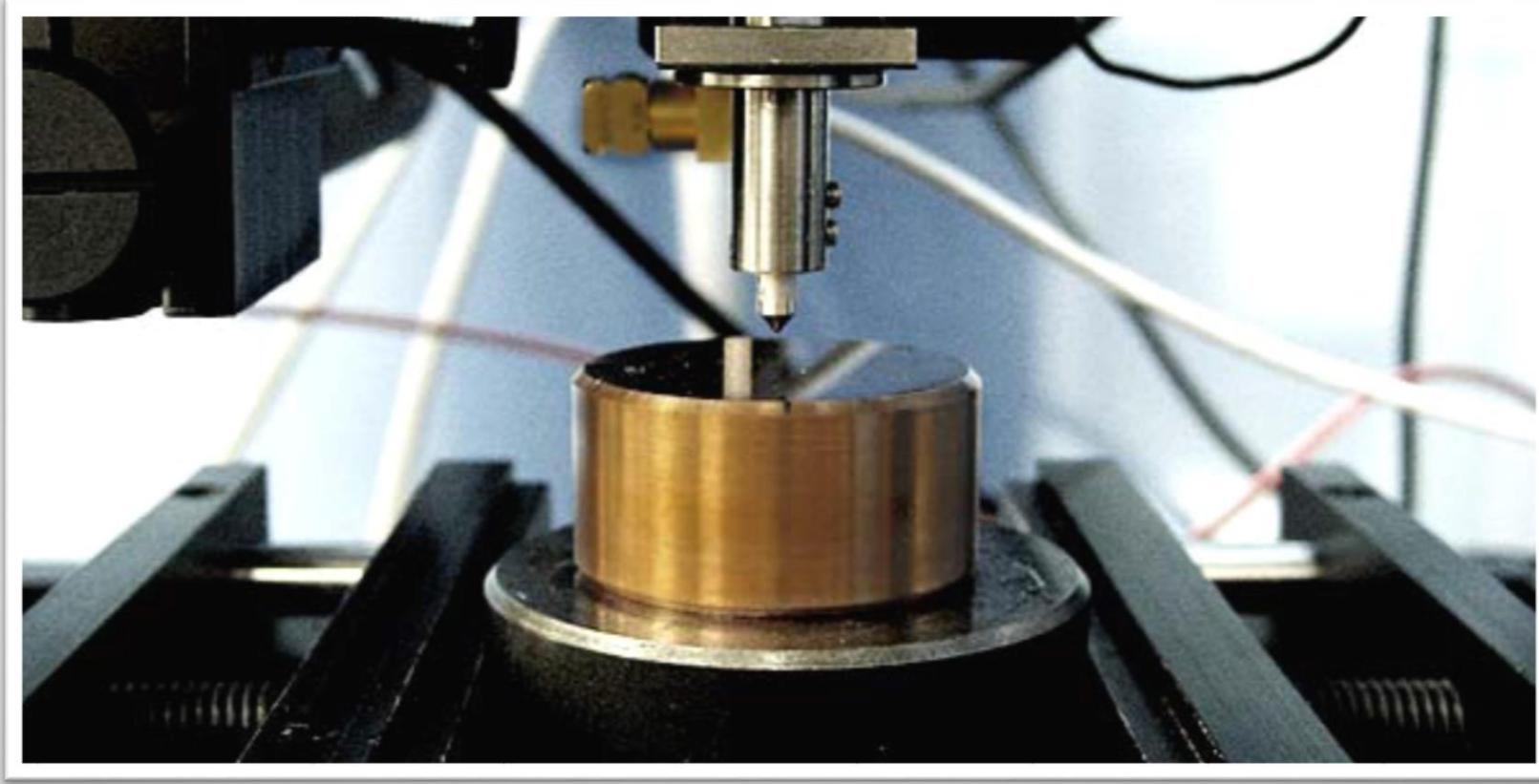


Nano Sertlik Deneyi

(Nanoindentation)



OSMAN SAYGINER

www.sayginer.com

Sunum Planı

I.BÖLÜM

1. Temel Bilgiler

- a) Giriş
- b) Neden Nano Sertlik Testi?
- c) Çalışma Boyutları
- d) Çalışma Ortamı
- e) Kullanılan Cihazlar

2. Nanoiz

- a) Genel bilgiler
- b) Hesaplama Yöntemleri
- c) Atomik Kuvvet Mikroskobu (AFM)
- d) Berkovich tipi uç
- e) Uç Çeşitleri
- f) Oliver-Pharr Metodu

3. Ek Bilgiler

4. Kaynaklar

II.BÖLÜM

1. Deney Analizi (CoCr & Ti)

- a) Gwyddion Programı
- b) AFM resimleri

2. Deney Sonuçları

- a) Veriler & Grafikler
- b) Tartışma

Tanım & Anlam

Nanoindentation

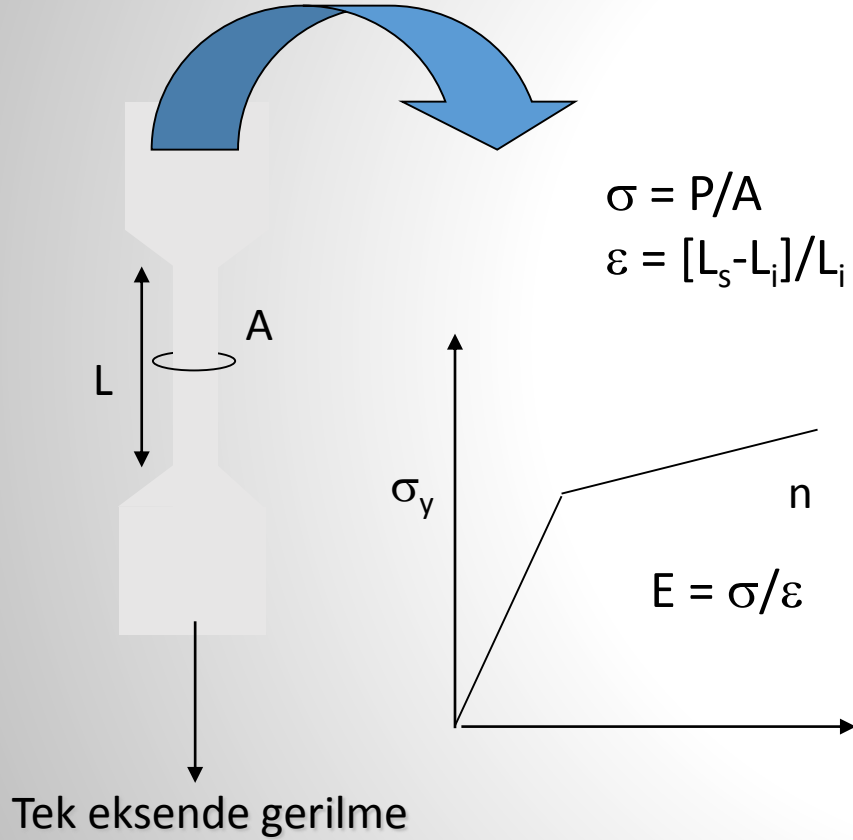
Nano sertlik testi

Nanoiz

Tanım: Malzeme yüzeyinden nano düzeyinde mekanik özelliklerinin *yükleme-derinlik* ile eş zamanlı olarak elde edilmesidir.

Temel Bilgiler: Nedir?

Çekme Deneyi

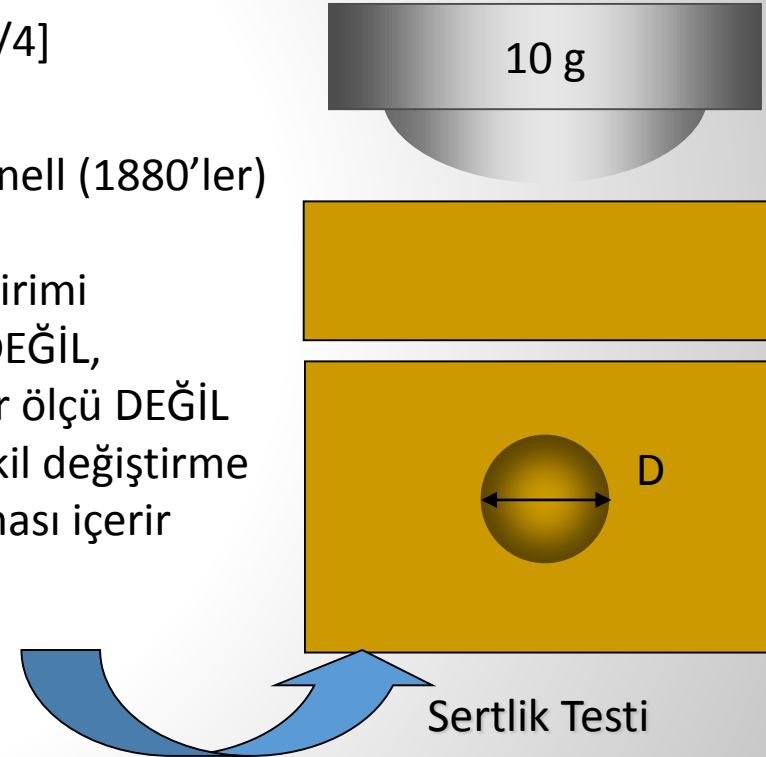


Brinell Sertlik Testi

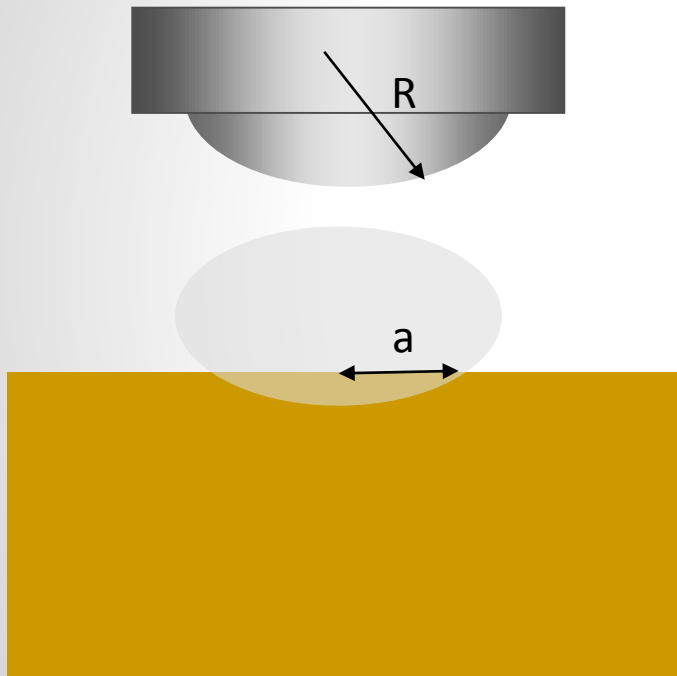
$$P = mg$$
$$H = P/[\pi D^2/4]$$

Johannes Brinell (1880'ler)

- Gerilme birimi
- Gerilme DEĞİL,
- Fiziksel bir ölçü DEĞİL
- Plastik şekil değiştirme mekanizması içerir



Temel Bilgiler: Nedir?

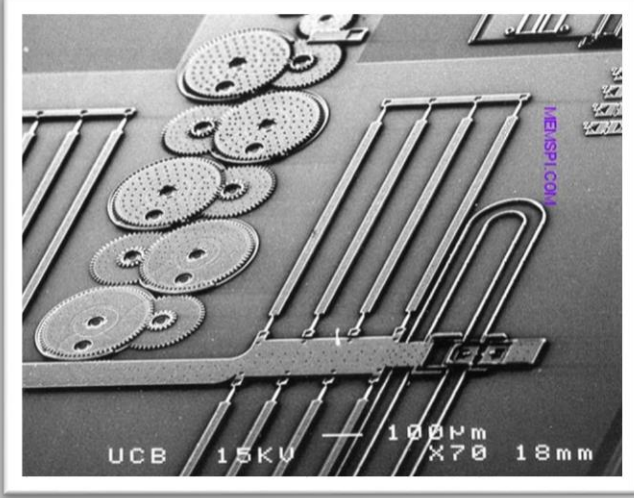


$$\varepsilon = a/R$$

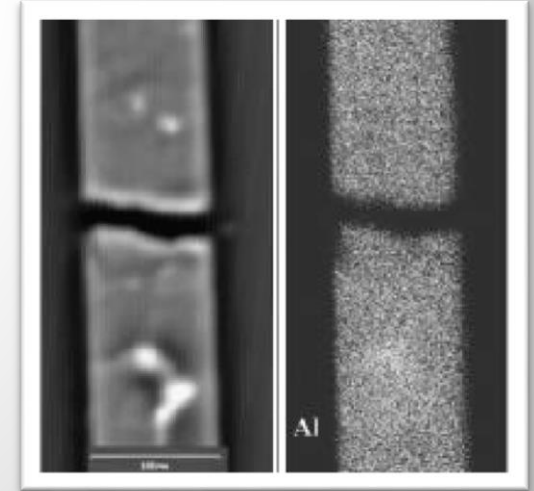
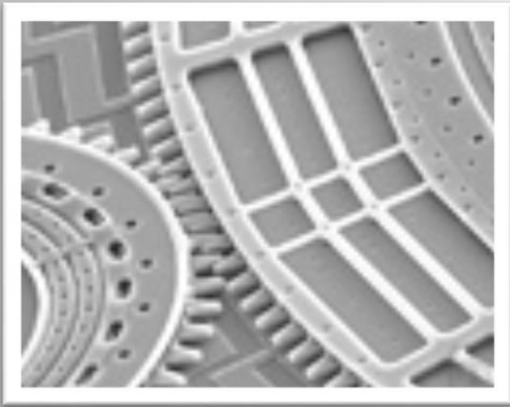
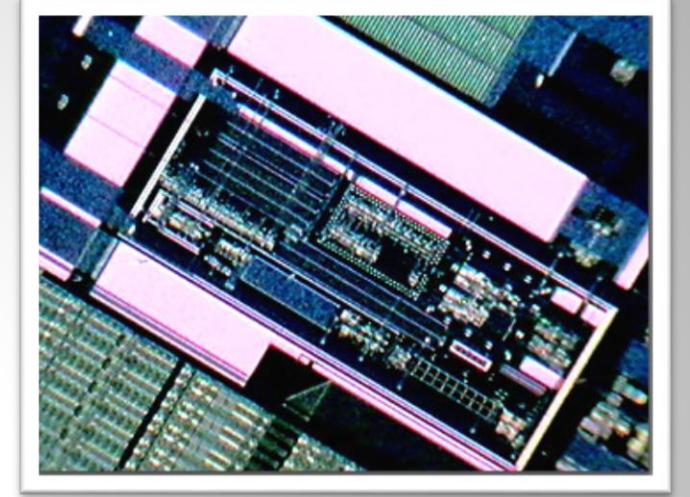
$$\sigma \propto P/\pi a^2$$

- Çok aksenel gerilme
- Tek ekseninde gerilme söz konusu değil
- İzi görebilmek için plastik şekil değiştirme gerekli

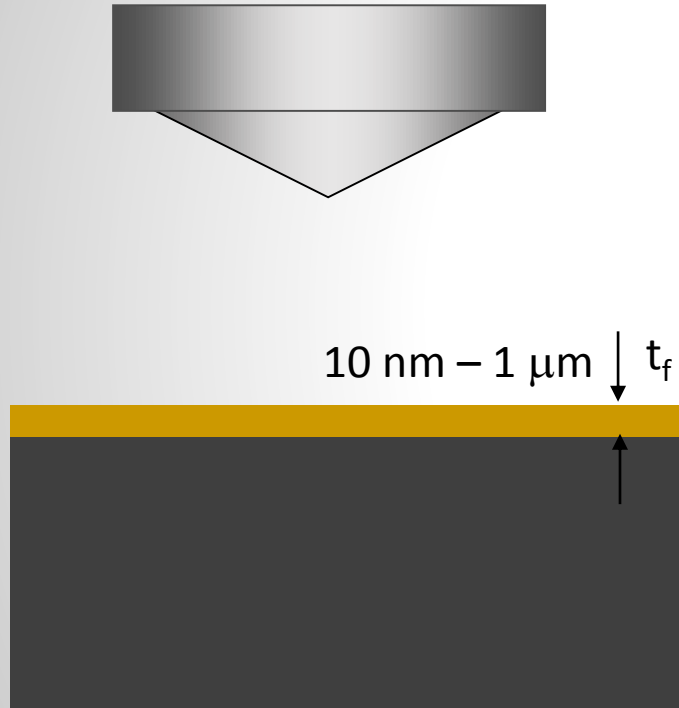
Temel Bilgiler: Neden?



- Mekanik özellikler boyutsal sınırlamalardan etkilenir mi?
- E , σ_y , n f boyut ilişkisi?
- Yapı boyutları atomik boyutlara yaklaştığı zaman mekanik özellikler nasıl nitelendirilebilir?



Temel Bilgiler: Neden?



- Microelektronik in filmler (1980 önceleri)
- İnce film kaplamalar

$$E \rightarrow t_f ?$$

$$\sigma_y \rightarrow t_f ?$$

$$n \rightarrow t_f ?$$

- Keskin kenarlı bir uç
- Nanoiz tekniği

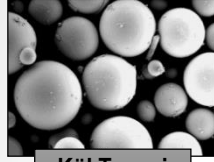
Ne kadar küçük?



Akar (mite)
200 μm



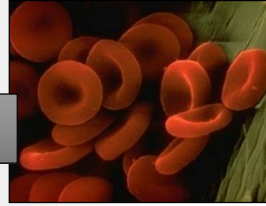
Karıncı
~ 5 mm



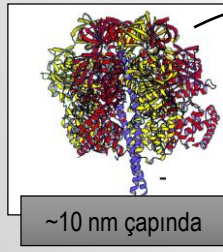
Kül Tanesi
~ 10-20 μm



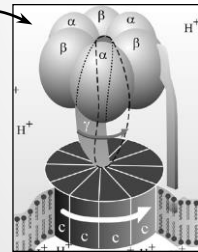
İnsan saçı
~ 60-120 μm wide



Kırmızı kan hücresi
(~7-8 μm)



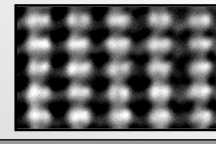
~10 nm çapında



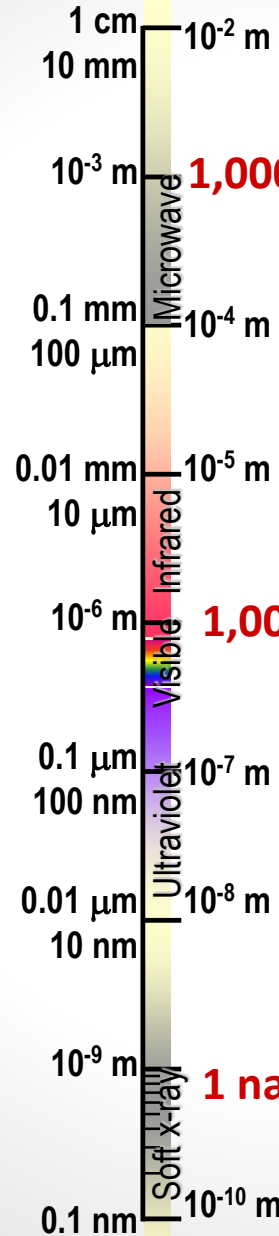
ATP sentezi



DNA
~2-1/2 nm çapında



Silikon atomları
arası mesafe
0.078 nm



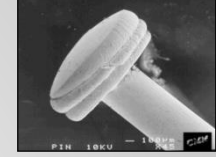
1,000,000 nanometre = 1 milimetre (mm)

Mikro Teknoloji
“Mikro Dünya”

1,000 nanometre = 1 micrometre (mm)

Nanotechnology
“The Nano World”

1 nanometre (nm)



İğne başı
1-2 mm



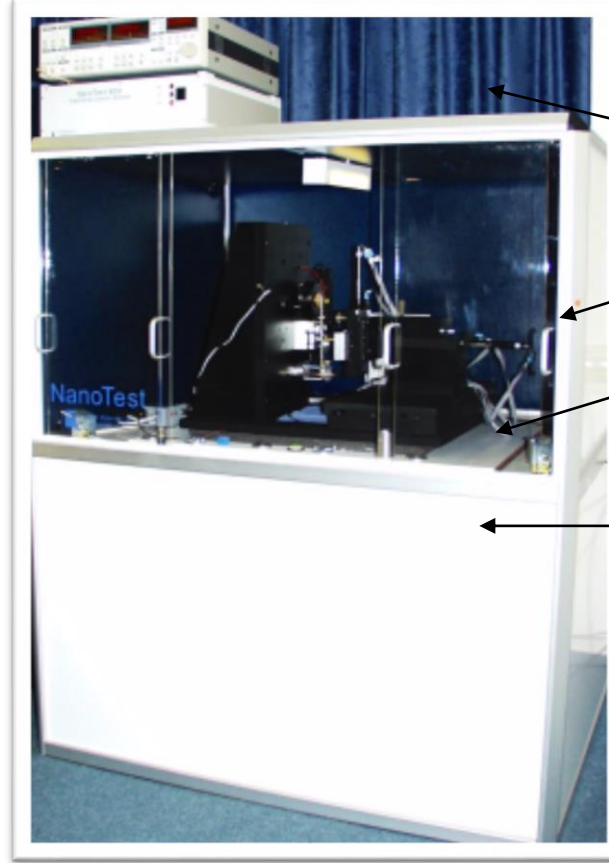
Kullanılan Cihazlar



Çalışma Şartları



Hysitron



MicroMaterials

Nem kontrolü (< 55%)

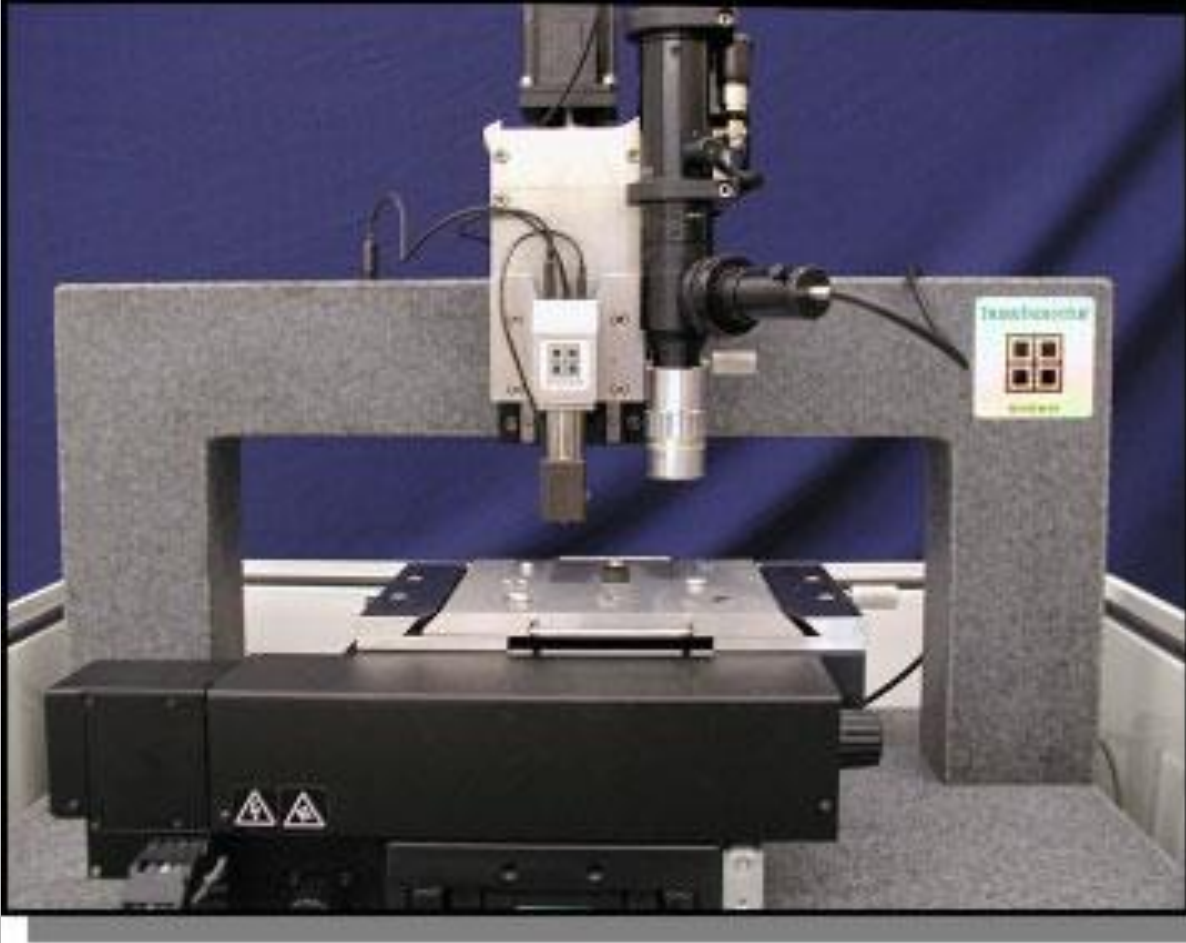
Ses yalıtımı (< 1 Hz)

Sıcaklık kontrolü (< 0.5 °C)

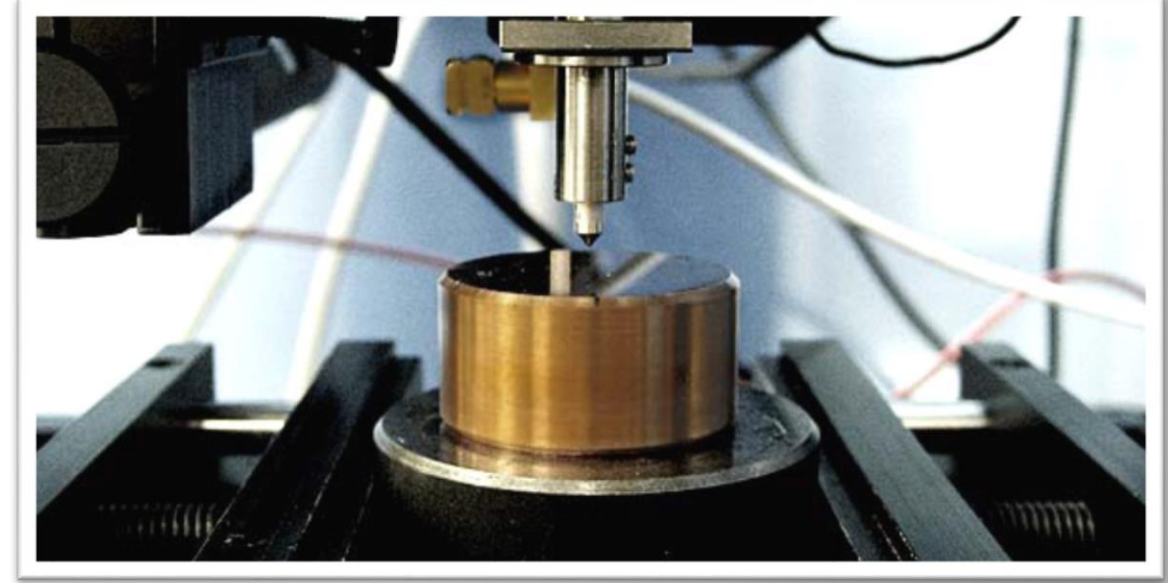
Titreşim kontrolü (< 1 Hz)



Nanoindenter (Nanoiz Oluřturucu)



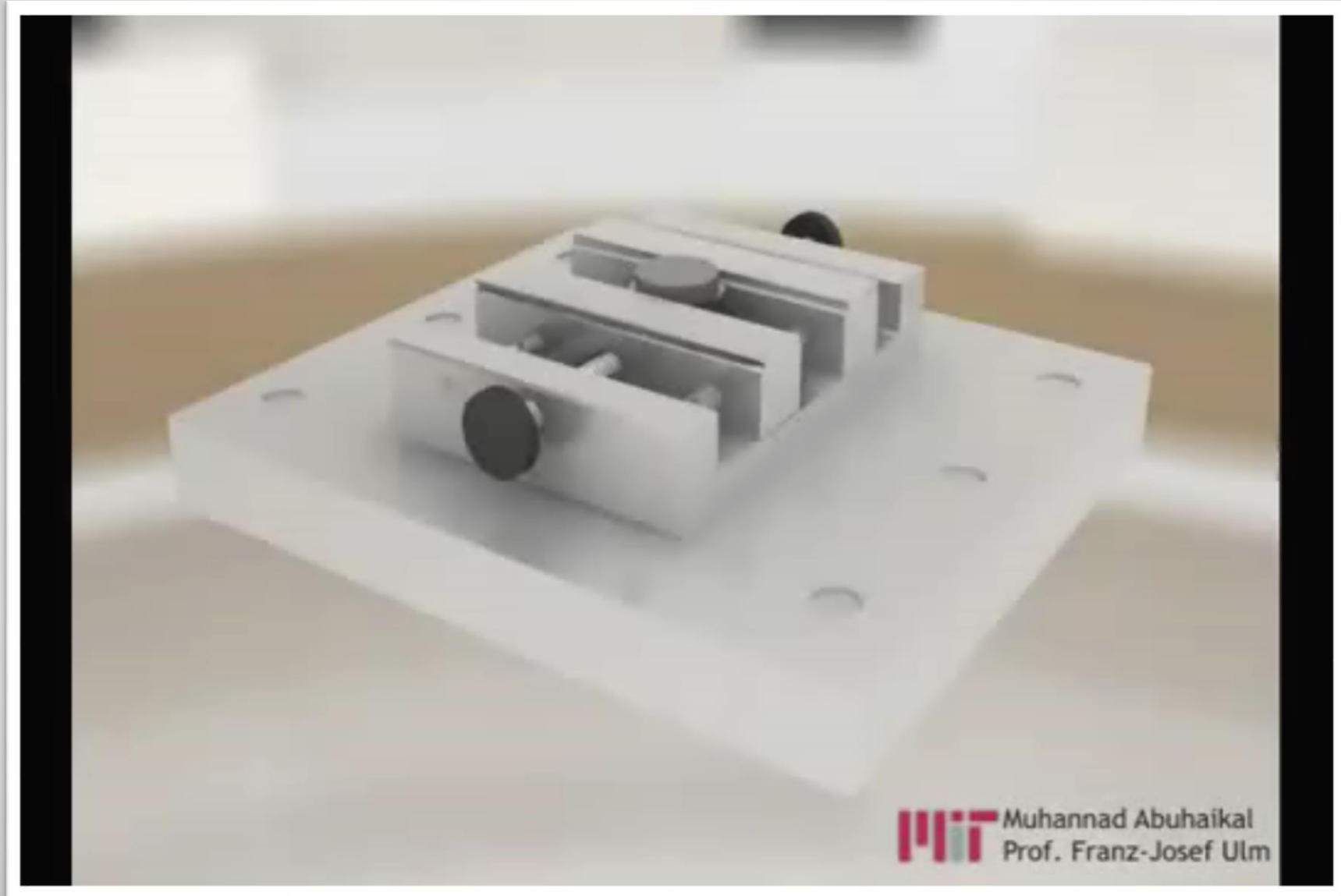
Nanoiz oluřturucu (Hysitron)

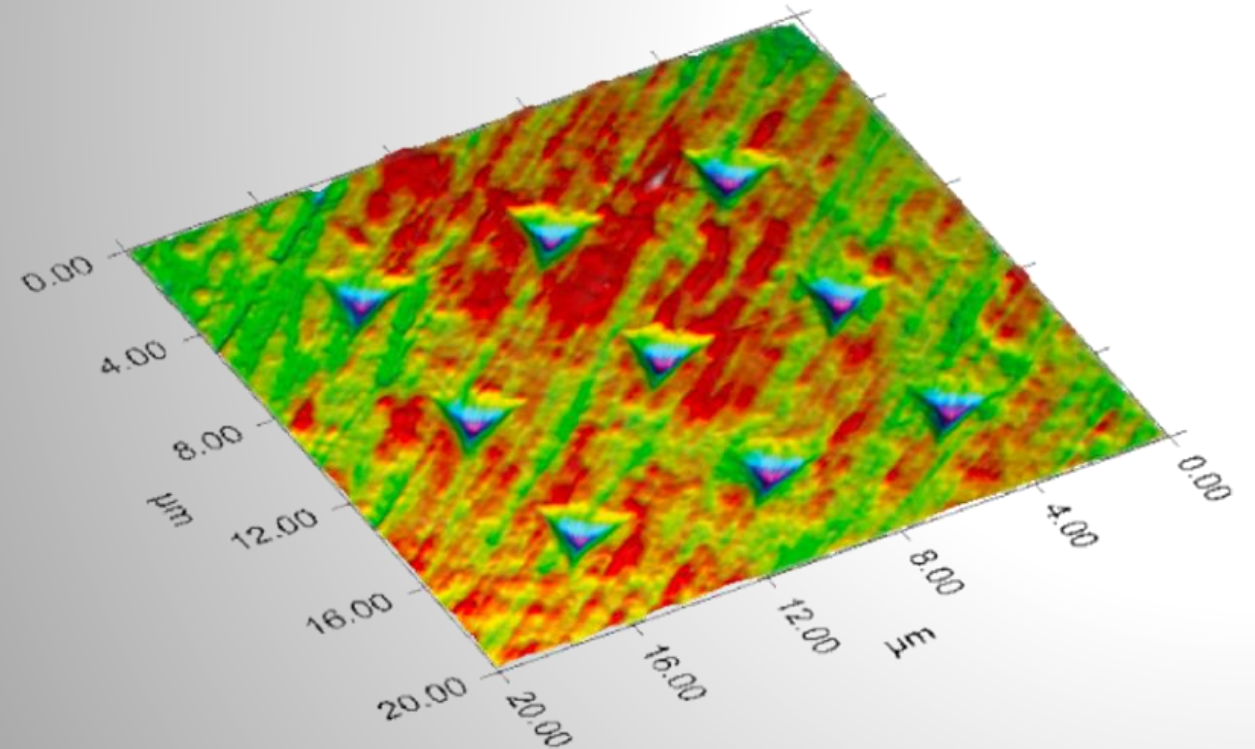
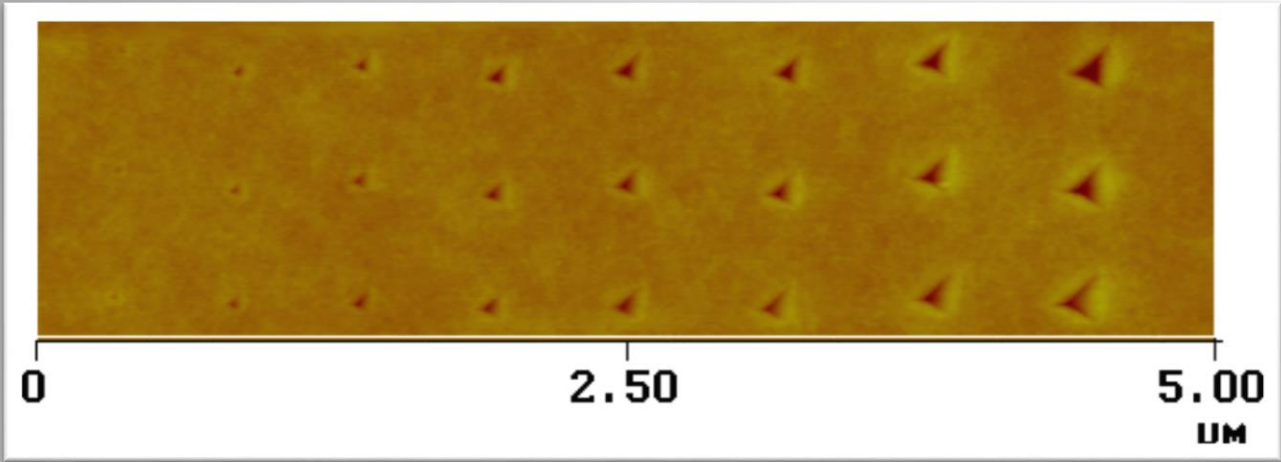


Uç ve numune

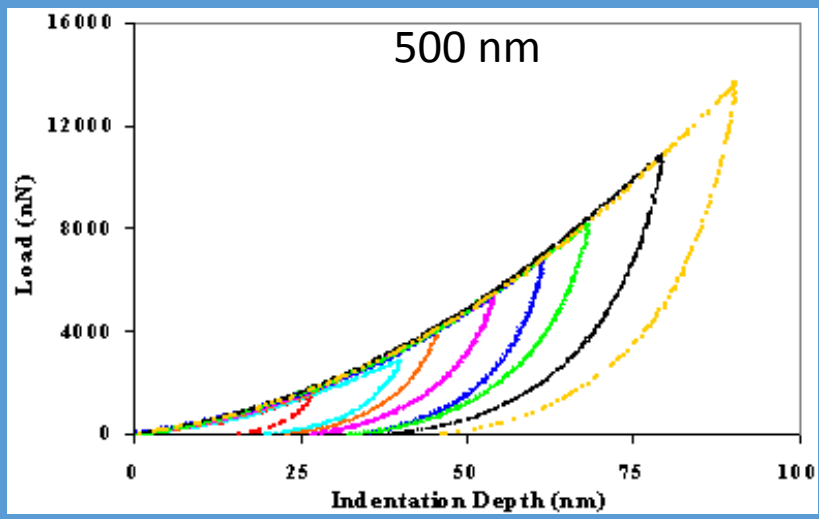
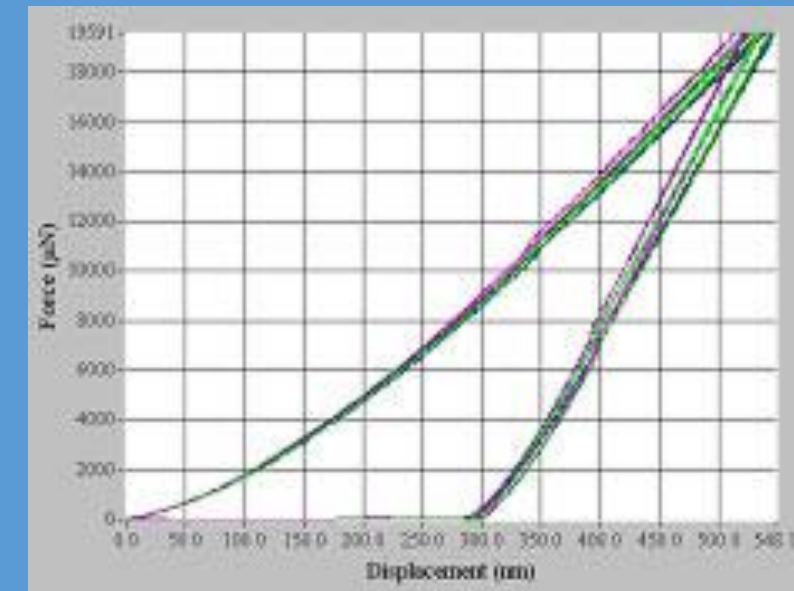


Nanoiz: Video



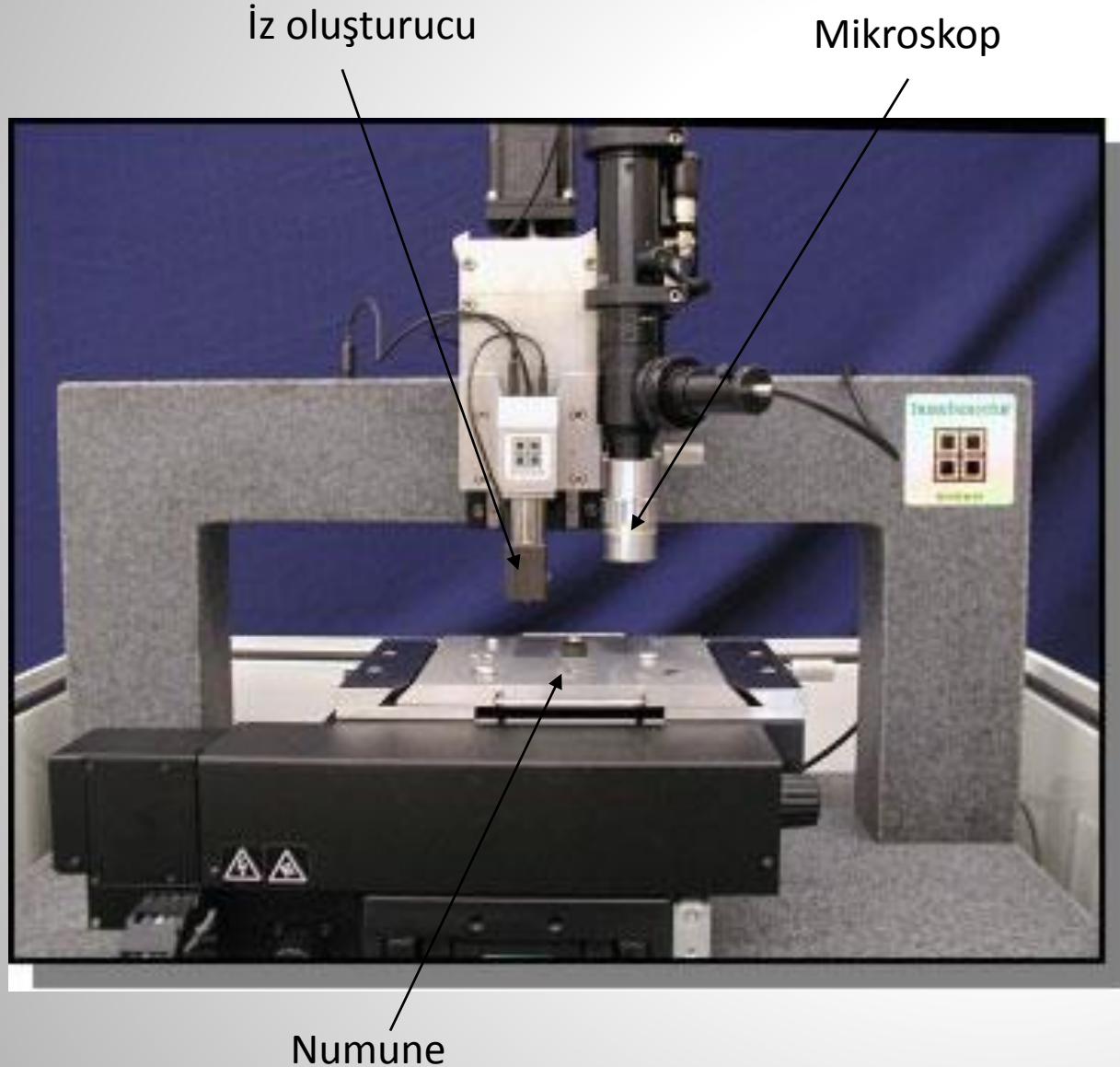


Yükleme



Şekil değiştirme (derinlik)

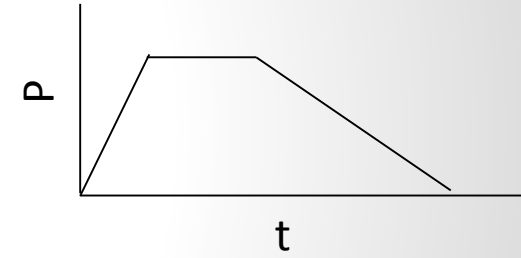
Mekanik Özellikler



1. Numune yüzeyini hazırla:
Düz, Pürüzsüz v.s

2. Numune üzerinde uygun yüzeyi bul

3. Program yükleme profili



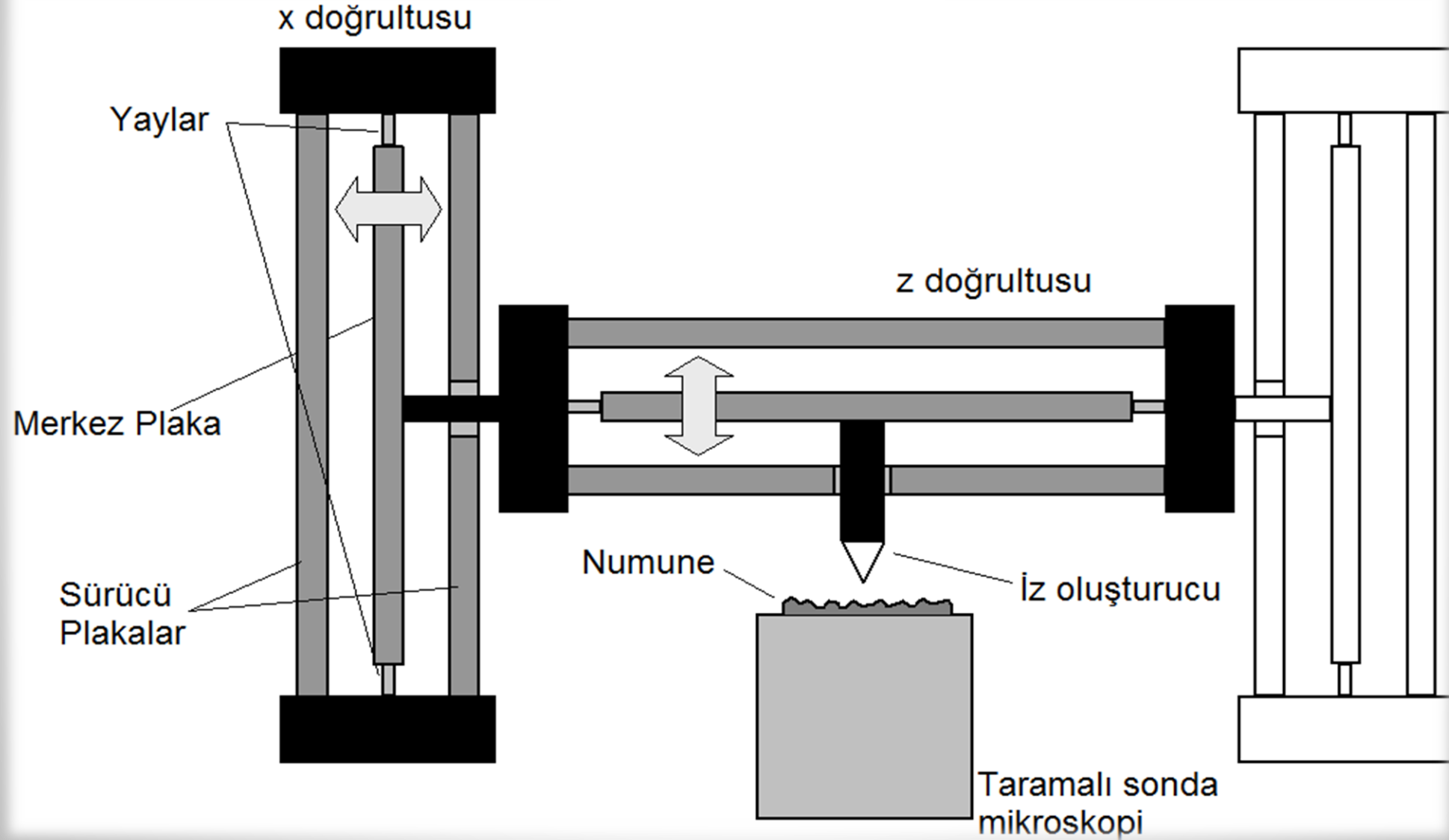
4. İzlerin sıklık-konum ve yapısını belirle



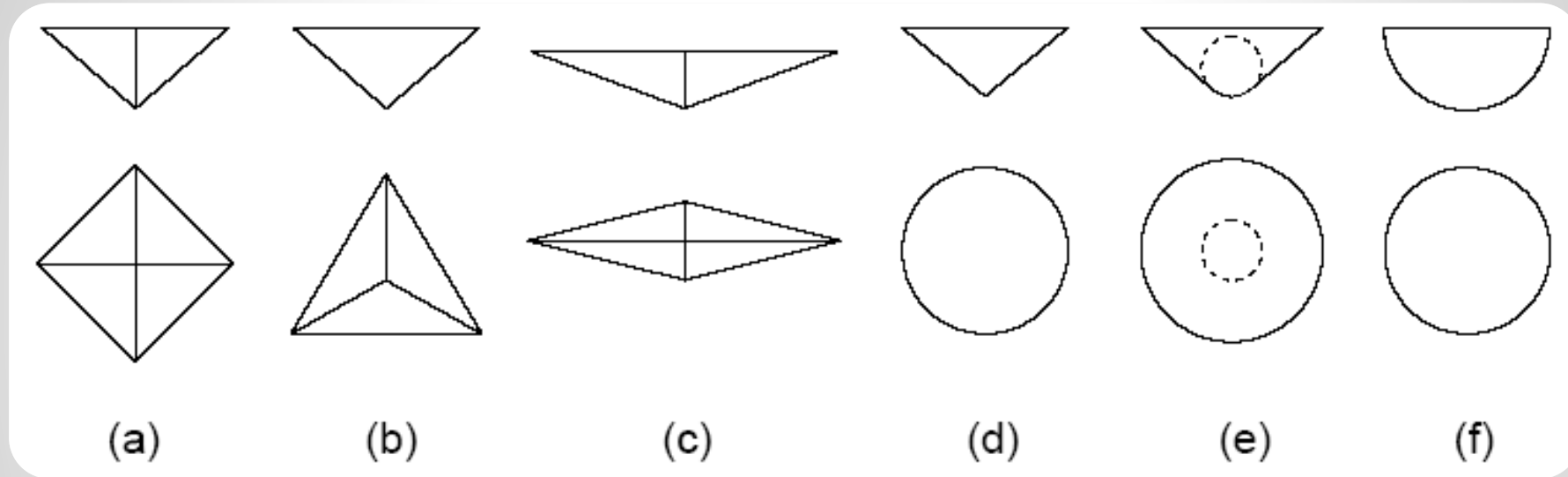
5. Deneyi başlat.

6. Elde edilen verileri değerlendir.

İz oluřturucu (Nanoindenter)



Uç Çeşitleri



(a)

(b)

(c)

(d)

(e)

(f)

(s)

(p)

(c)

(q)

(e)

(t)

Vickers

Berkovich

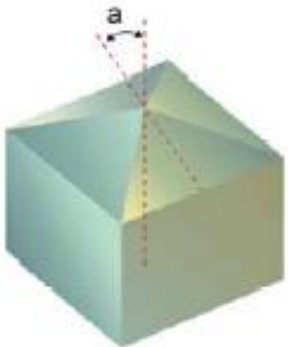
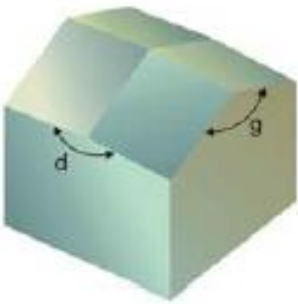
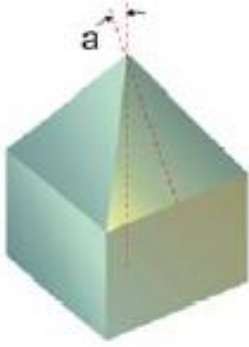
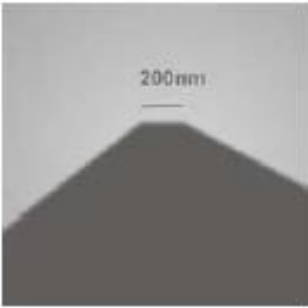
Knoop

Conical

Rockwell

Spherical

4 Kenarlı Uçlar

<p>VICKERS FV</p> 	<p>KNOOP INDENTER FK</p> 	<p>4-SIDED CUSTOM FD</p> 	<p>END LINE TEM micrograph</p> 
<p>Standard Vickers indenter: $a = 68.00^\circ$ Available as Traceable Standard</p>	<p>Standard Knoop indenter defined by 2 angles: $d = 172.50^\circ$, $g = 130.00^\circ$</p>	<p>Custom 4-sided indenters: $80^\circ > a > 20^\circ$</p>	<p>Micro Star indenters maximum line of conjunction: 400nm.</p>

3 Kenarlı Uçlar

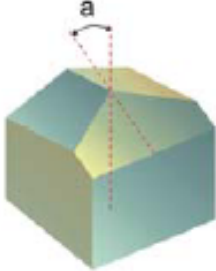
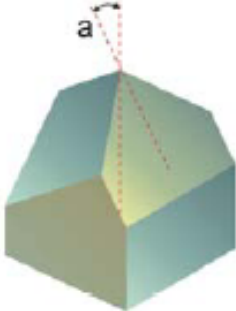


BERKOVICH TB 	CUBE CORNER TC 	3-SIDED CUSTOM TD 	SHARPNESS TEM micrograph 
Berkovich: $a = 65.03^\circ$ Mod. Berkovich: $a = 65.27^\circ$ Available as Traceable Standard	Cube corner: $a = 35.26^\circ$ Available as Traceable Standard	Custom 3-sided indenters: $80^\circ > a > 20^\circ$	Micro Star 3-sided sharp indenters tip radius $< 50\text{nm}$.

Figure 2 SHARP 3-SIDED INDENTERS

Konik Uçlar

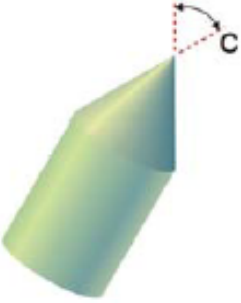
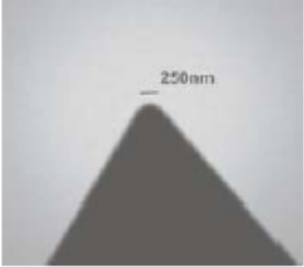


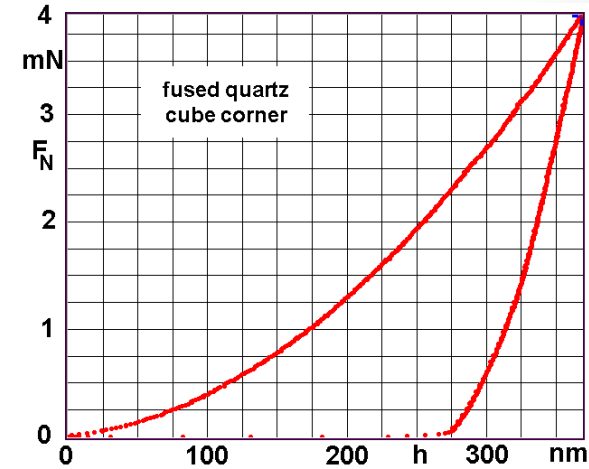
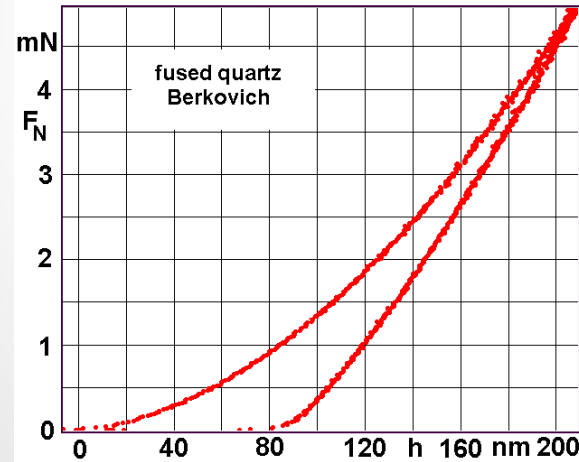
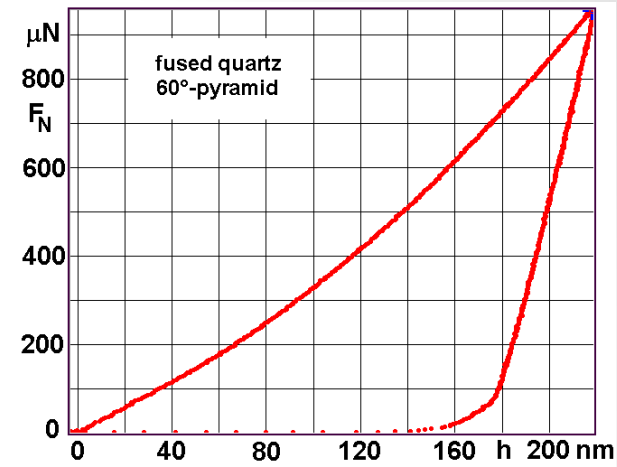
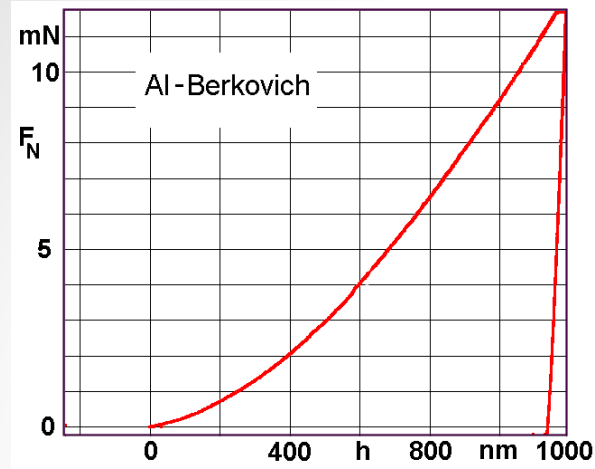
<p>CONE TIP VS</p> 	<p>POINT SHARPNESS TEM micrograph</p> 	<p>FLAT END CONE VP</p> 	<p>ROUND END CONE VR</p> 
<p>Included conical angle: $20^\circ > c > 140^\circ$</p>	<p>Micro Star sharp cone radius less than 300nm.</p>	<p>Flat from 500nm diameter to larger compatible sizes.</p>	<p>Spherical end radius 500nm to larger compatible sizes.</p>

Figure 6 CONE INDENTERS

Uç Geometrileri

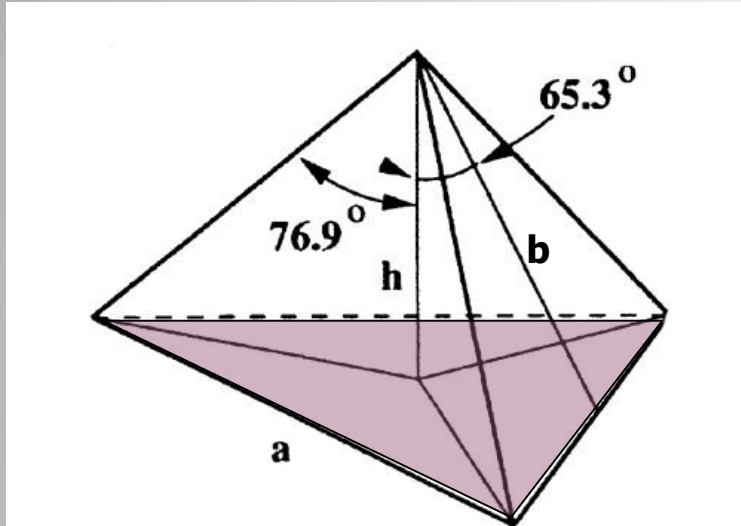
Uç Türü	İzdüşümü Alan	Semi angle (θ)	Etkin koniklik açısı (α)	Kesme Faktörü	Geometrik Düzeltme (β)
Sphere	$A \approx \pi 2Rh_p$	N/A	N/A	0.75	1
Berkovich	$A = 3h_p^2 \tan^2 \theta$	65.3 °	70.2996 °	0.75	1.034
Vickers	$A = 4h_p^2 \tan^2 \theta$	68 °	70.32 °	0.75	1.012
Knoop	$A = 2h_p^2 \tan \theta_1 \tan \theta_2$	$\theta_1 = 86.25^\circ$ $\theta_2 = 65^\circ$	77.64 °	0.75	1.012
Cube Corner	$A = 3h_p^2 \tan^2 \theta$	35.26 °	42.28 °	0.75	1.034
Cone	$A = \pi h_p^2 \tan^2 \alpha$	α	α	0.72	1

Farklı uçlar

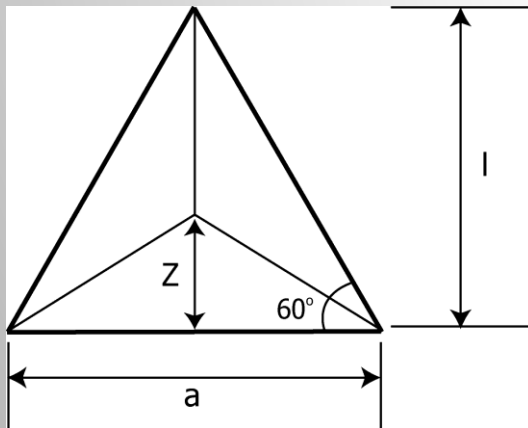


Farklı uçların farklı yüzeyler üzerine etkileri

Berkovich Uç



İz düşüm alanı



$$\tan 60^\circ = \frac{l}{a/2}$$

$$l = \frac{\sqrt{3}}{2} a$$

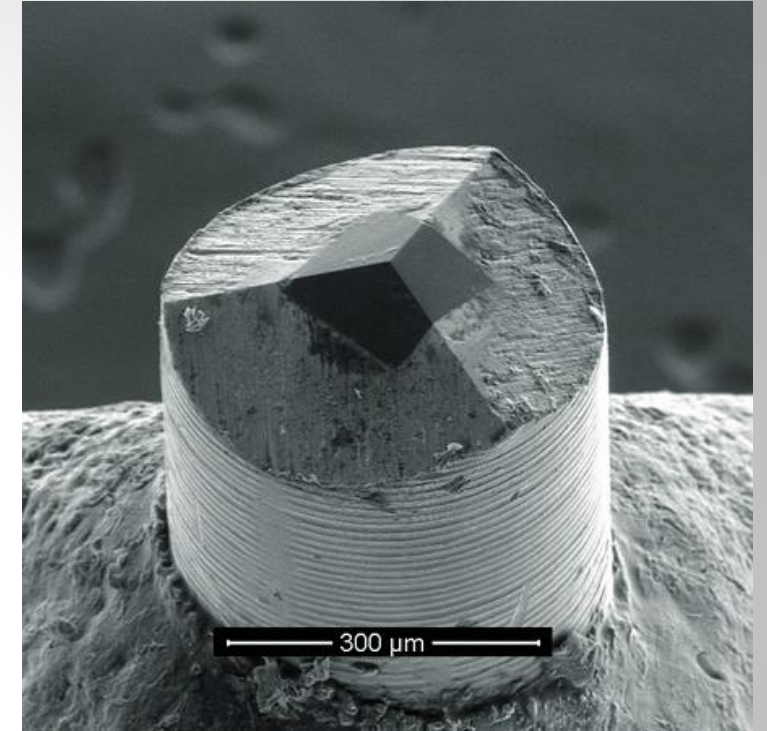
$$A_{proj} = \frac{al}{2} = \frac{\sqrt{3}}{4} a^2$$

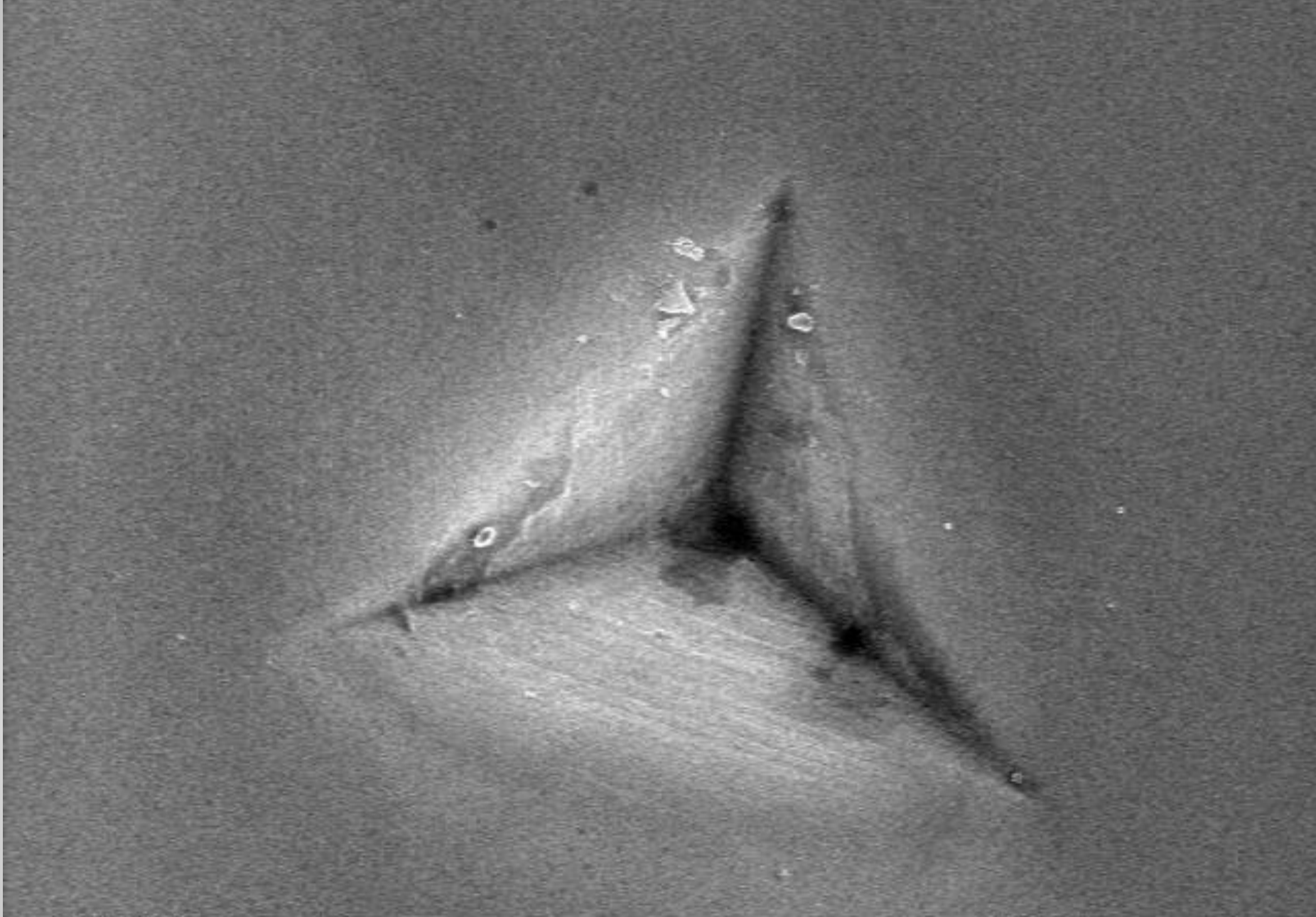
$$\cos 65.27^\circ = \frac{h}{b}$$

$$h = \frac{a \cos 65.3^\circ}{2\sqrt{3} \sin 65.3^\circ} = \frac{a}{2\sqrt{3} \tan 65.3^\circ}$$

$$a = 2\sqrt{3}h \tan 65.3^\circ$$

$$A_{proj} = 3\sqrt{3}h^2 \tan^2 65.3^\circ = 24.56h^2$$





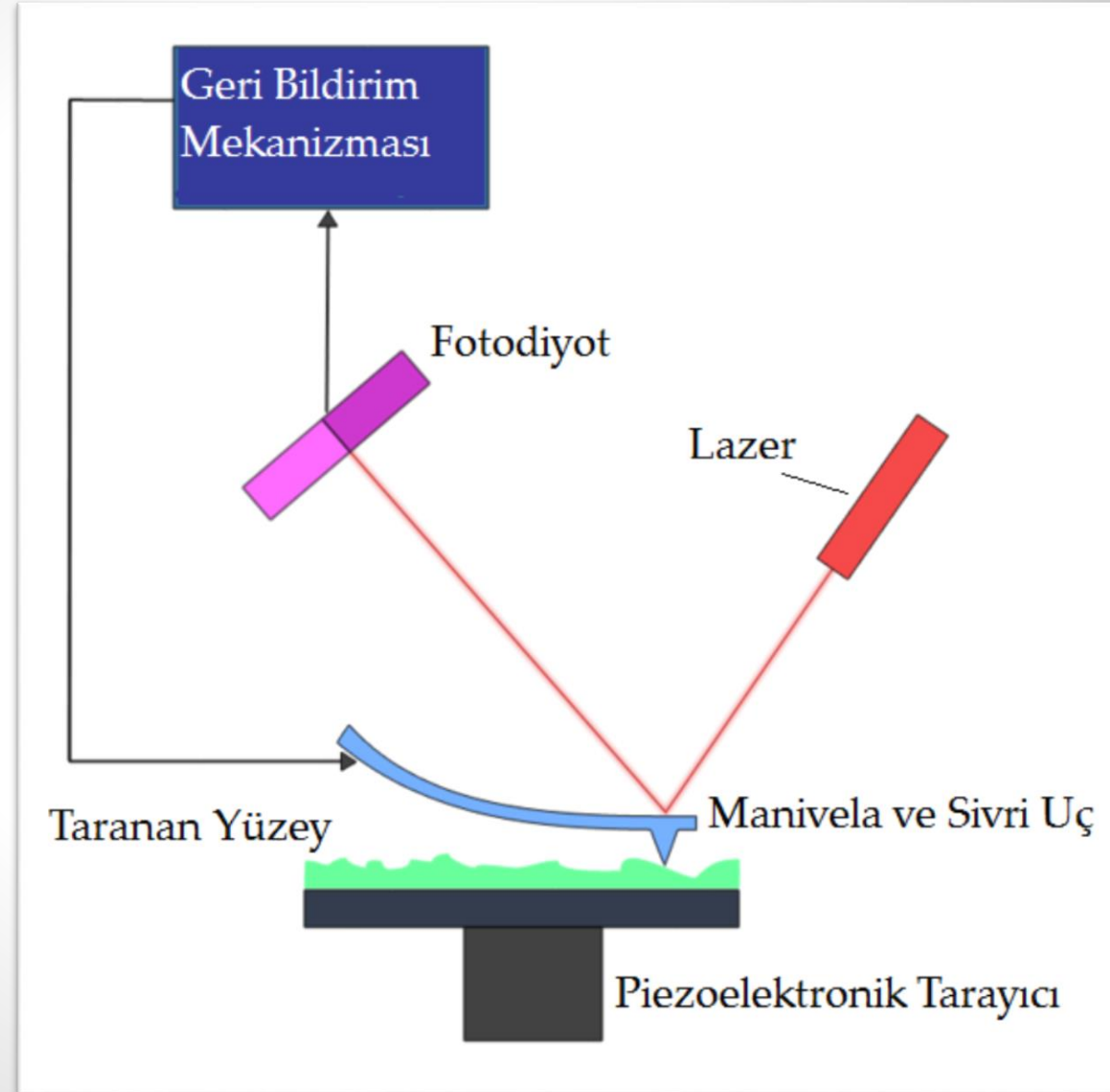
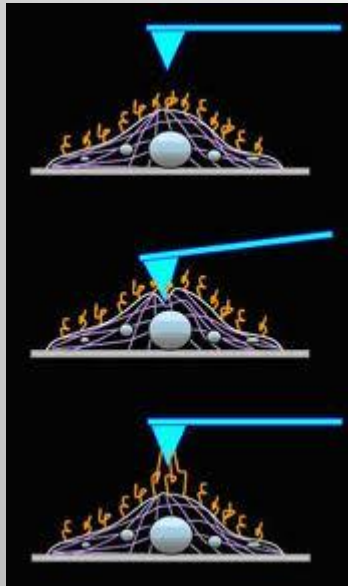
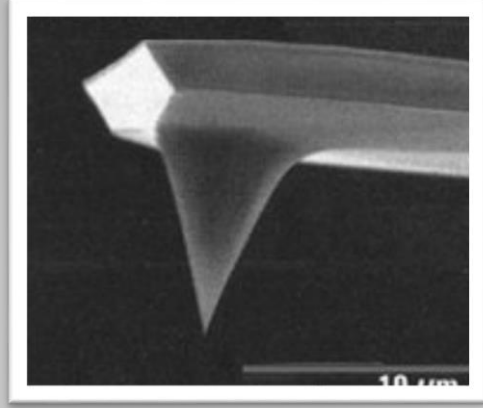
x20000
x20000
#0

2 μm 
2 μm 
INDENT 3000 MN

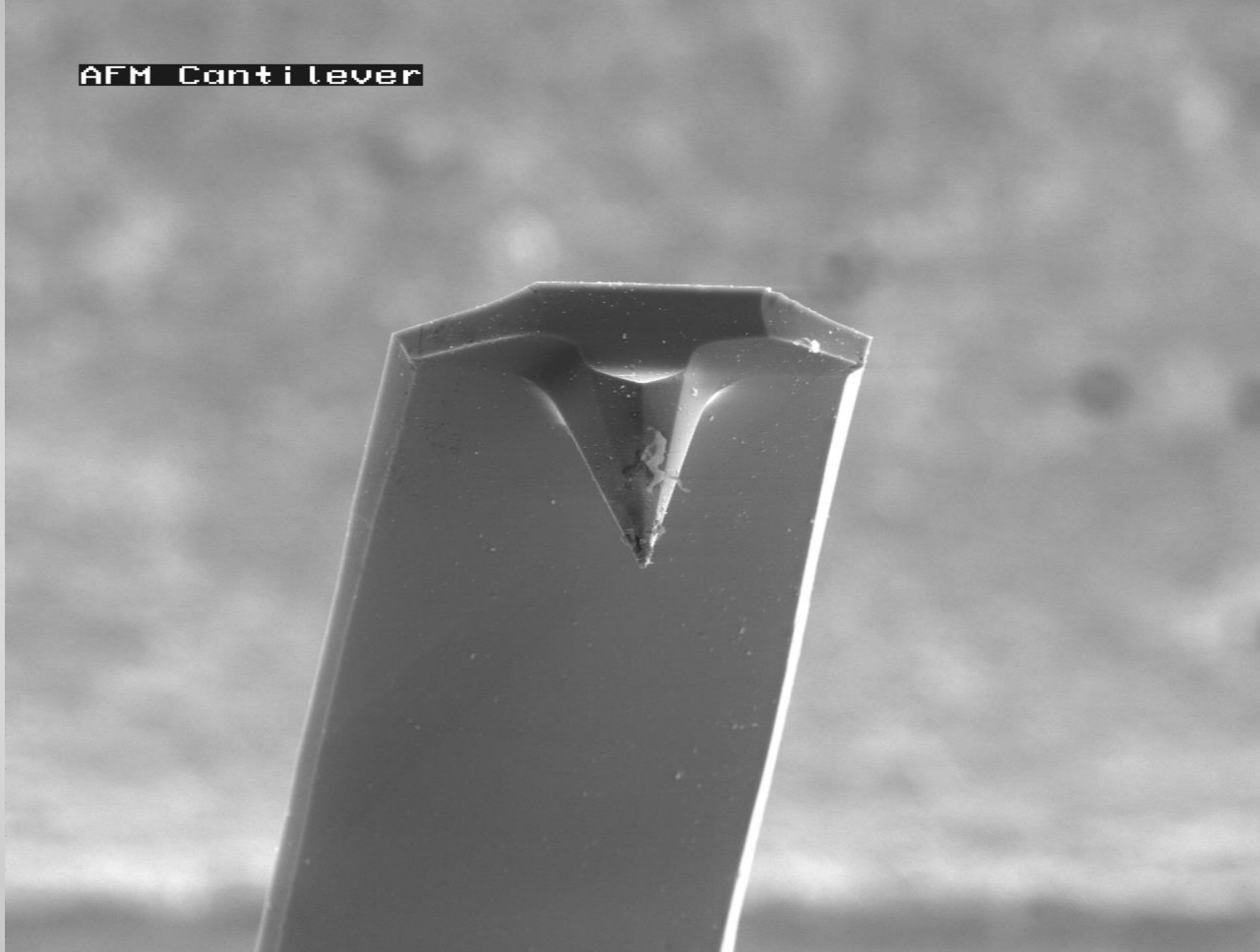
20kV
20kV

2mm
2mm

Atomik Kuvvet Mikroskobu (AFM)



AFM Cantilever



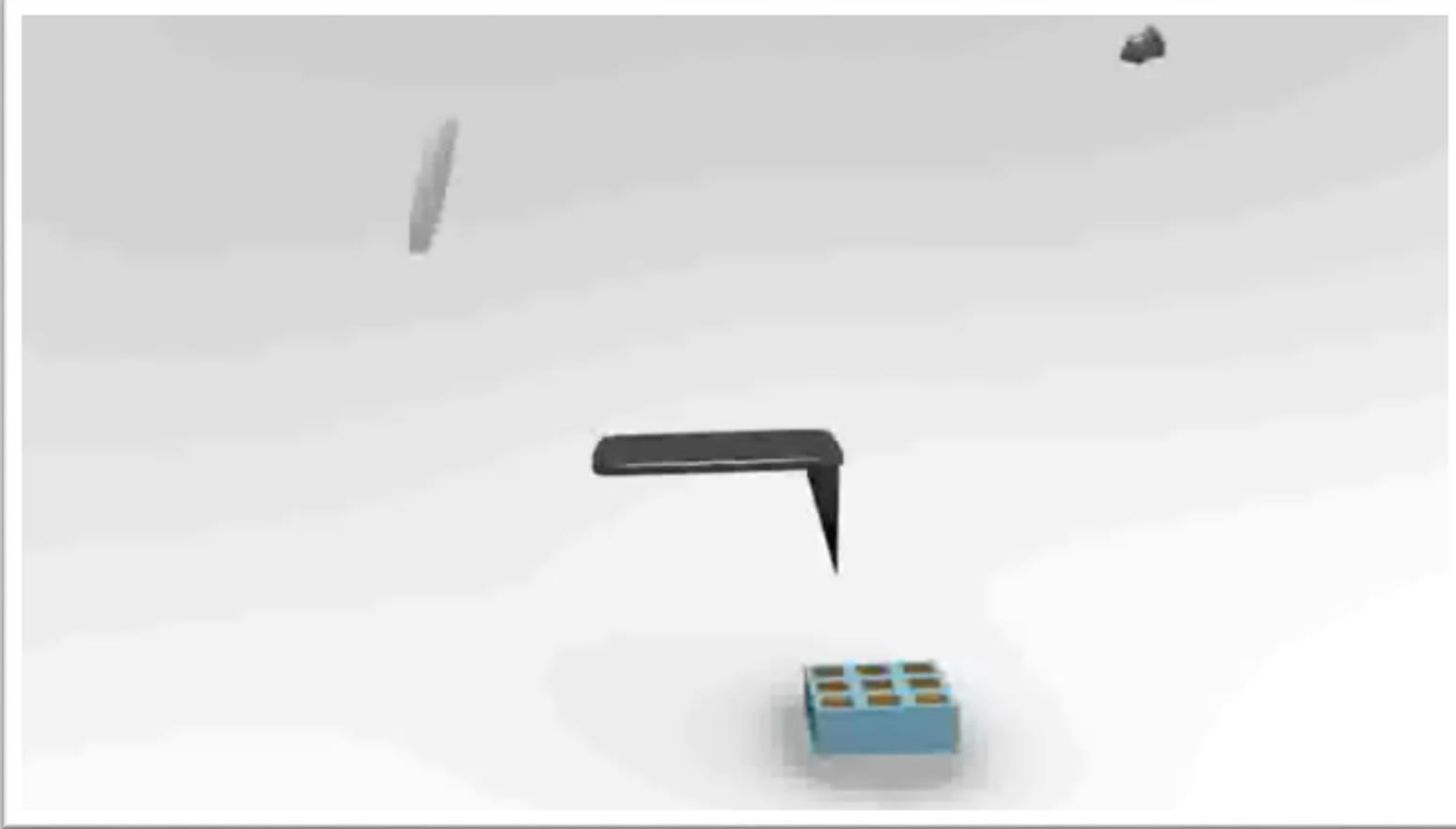
x1000

20 μ m

8kV

22mm

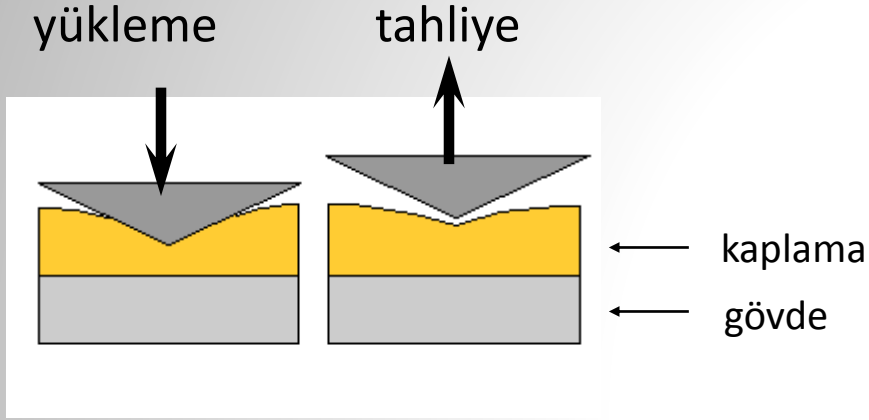
Atomik Kuvvet Mikroskobu (AFM)



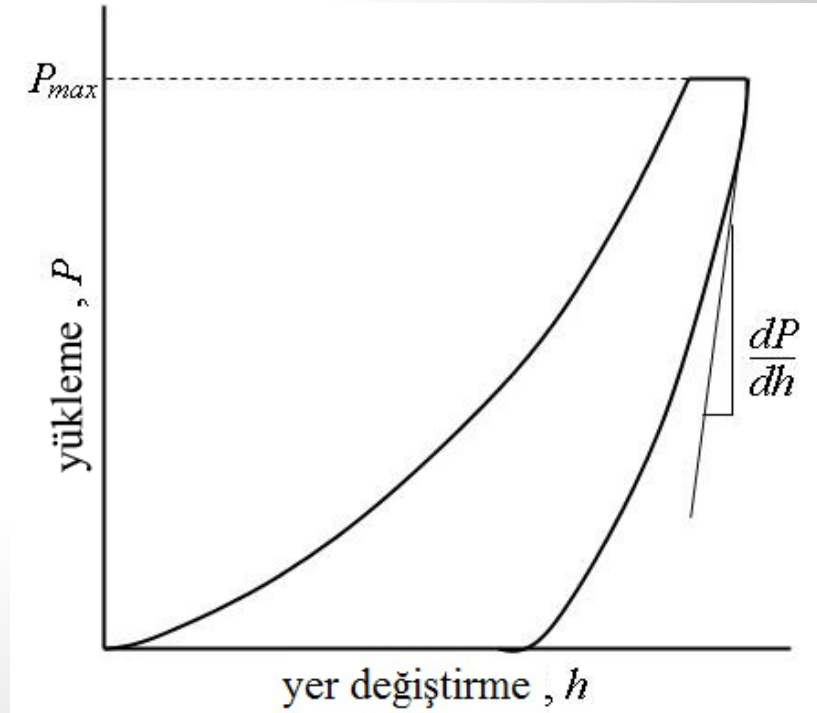
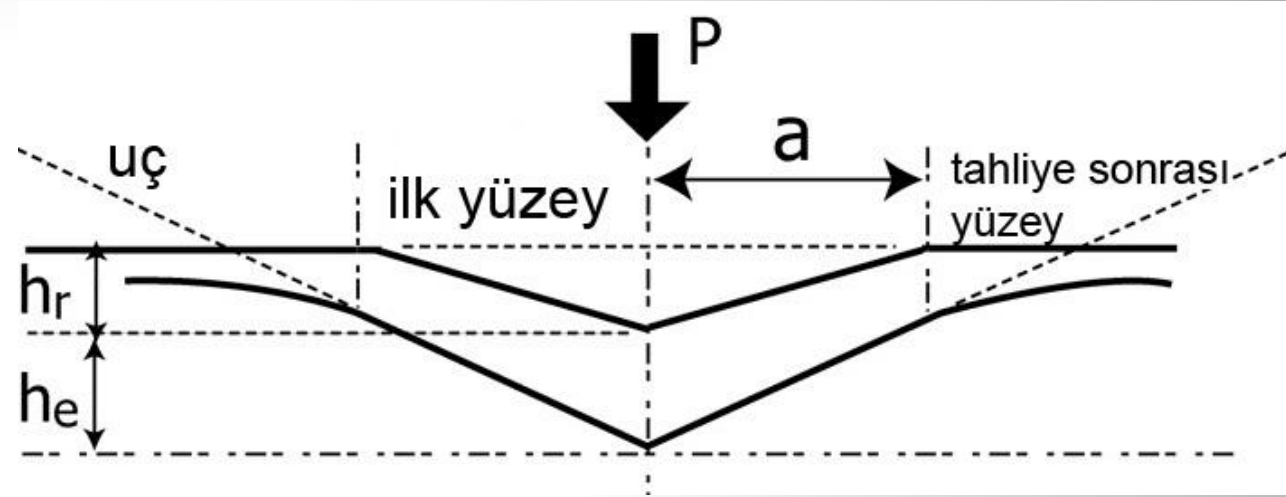
Atomik Kuvvet Mikroskobu çalışma animasyonu



Mekanik Özellikler



- P : Uygulanan yüklemeye
- h : Şekil değiştirme
- h_r : Yüklemeden sonra meydana gelen plastik şekil değiştirme
- h_e : Yüzey-temas bölgesi şekil değiştirmesi



Mekanik Özellikler

•İndirgenmiş Modül $\frac{1}{E^*} = \frac{1-\nu^2}{E} + \frac{1-\nu'^2}{E'}$

E: Numunenin elastik modülü
E': İzi oluşturunun elastik modülü

•Rijitlik $\frac{dP}{dh} = 2E^* \frac{\sqrt{A}}{\sqrt{p}}$

•Temas Alanı $A = 3\sqrt{3}h_p^2 \tan^2 65.3 = 24.5h_p^2$

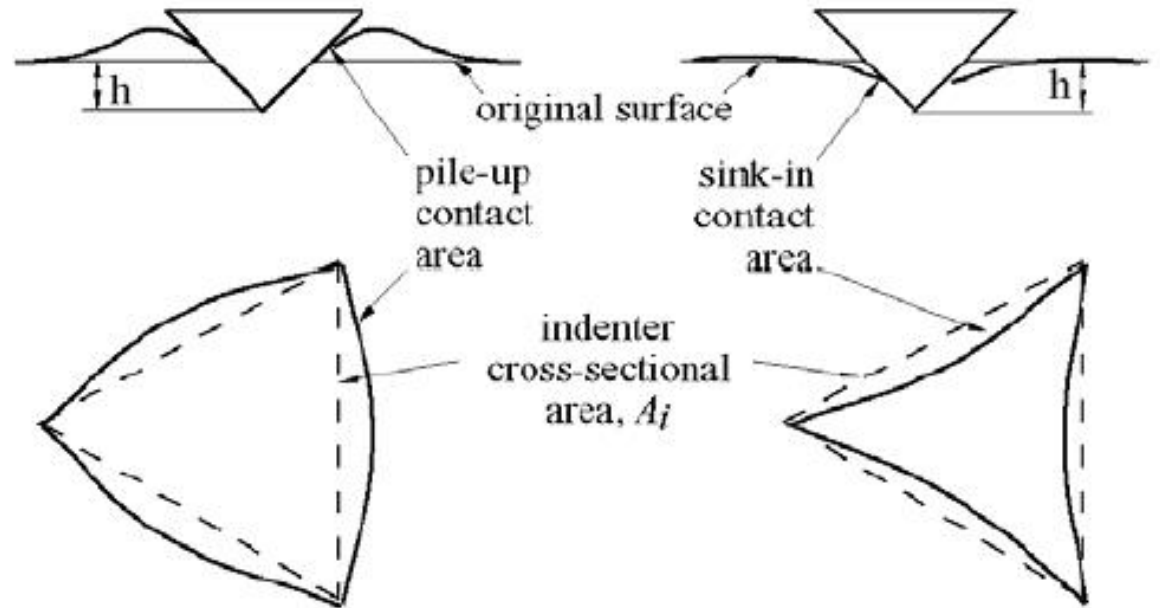
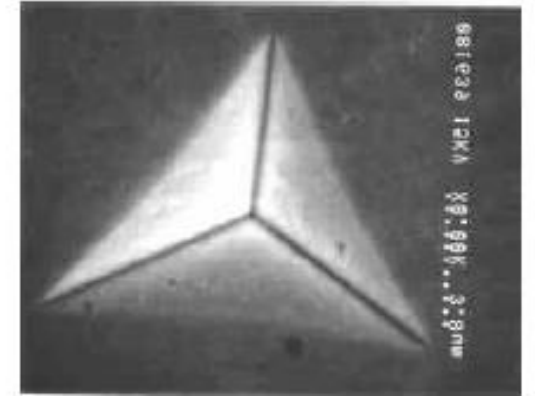
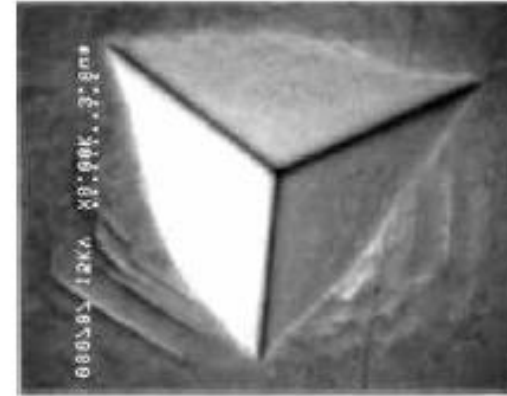
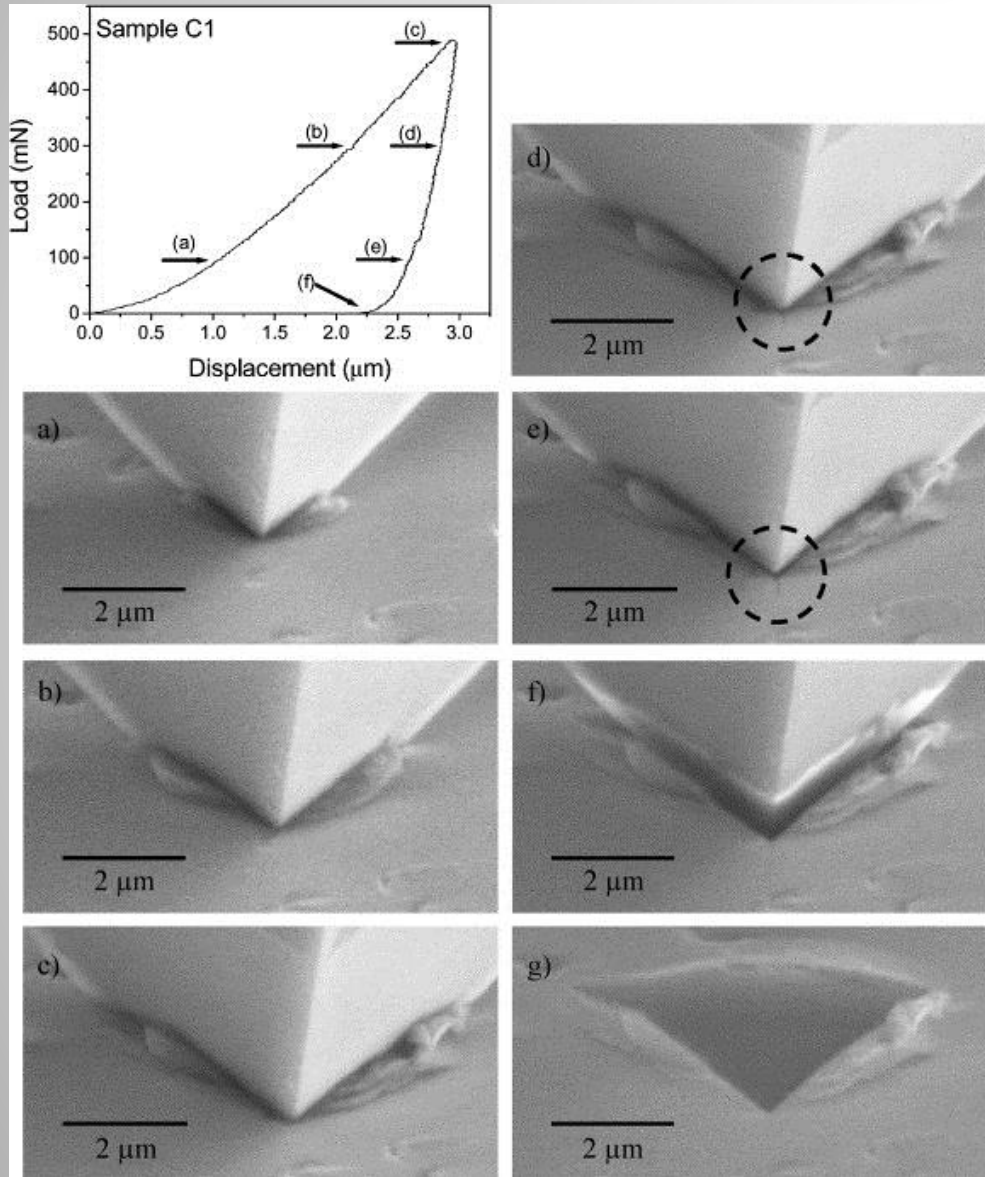
Berkovich tipi uçlar için

•Sertlik $H = \frac{P}{24.5h_p^2}$

•Elastik Modül $E^* = \frac{dP}{dh} \frac{1}{2h_p} \frac{1}{\beta} \sqrt{\frac{\pi}{24.5}}$

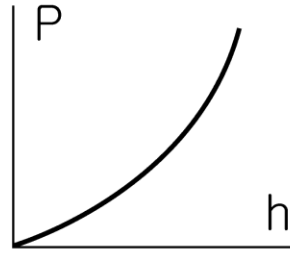
Berkovich uçlar için $\beta = 1.034$

Batma ve Taşmalar

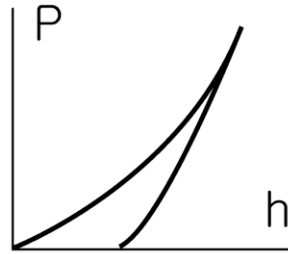


Farklı Mazemeler

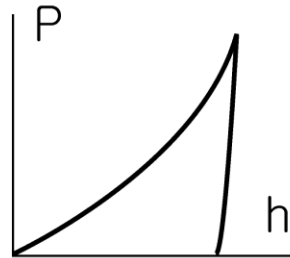
(a) Elastic solid



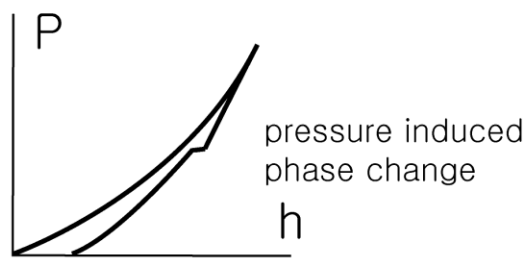
(b) Fused silica



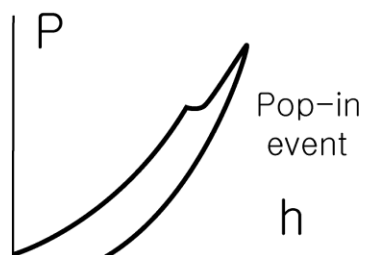
(c) Steel



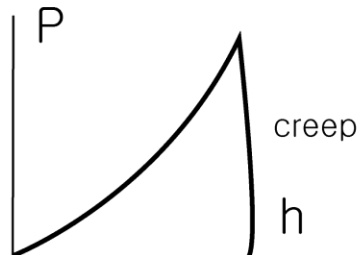
(d) Crystalline silicon



(e) Sapphire



(f) Polymer

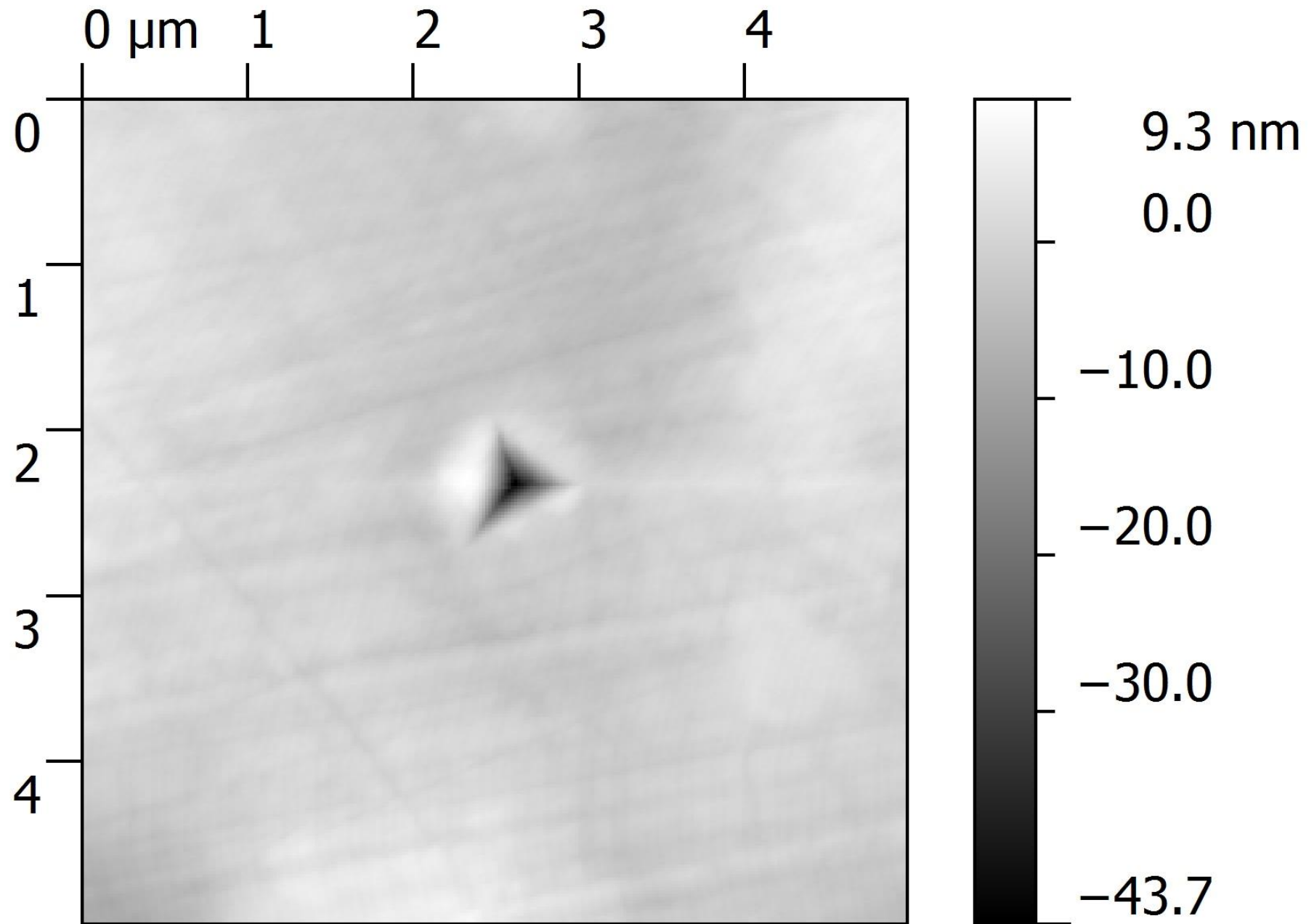


Farklı malzemelere ait yükleme eğrileri

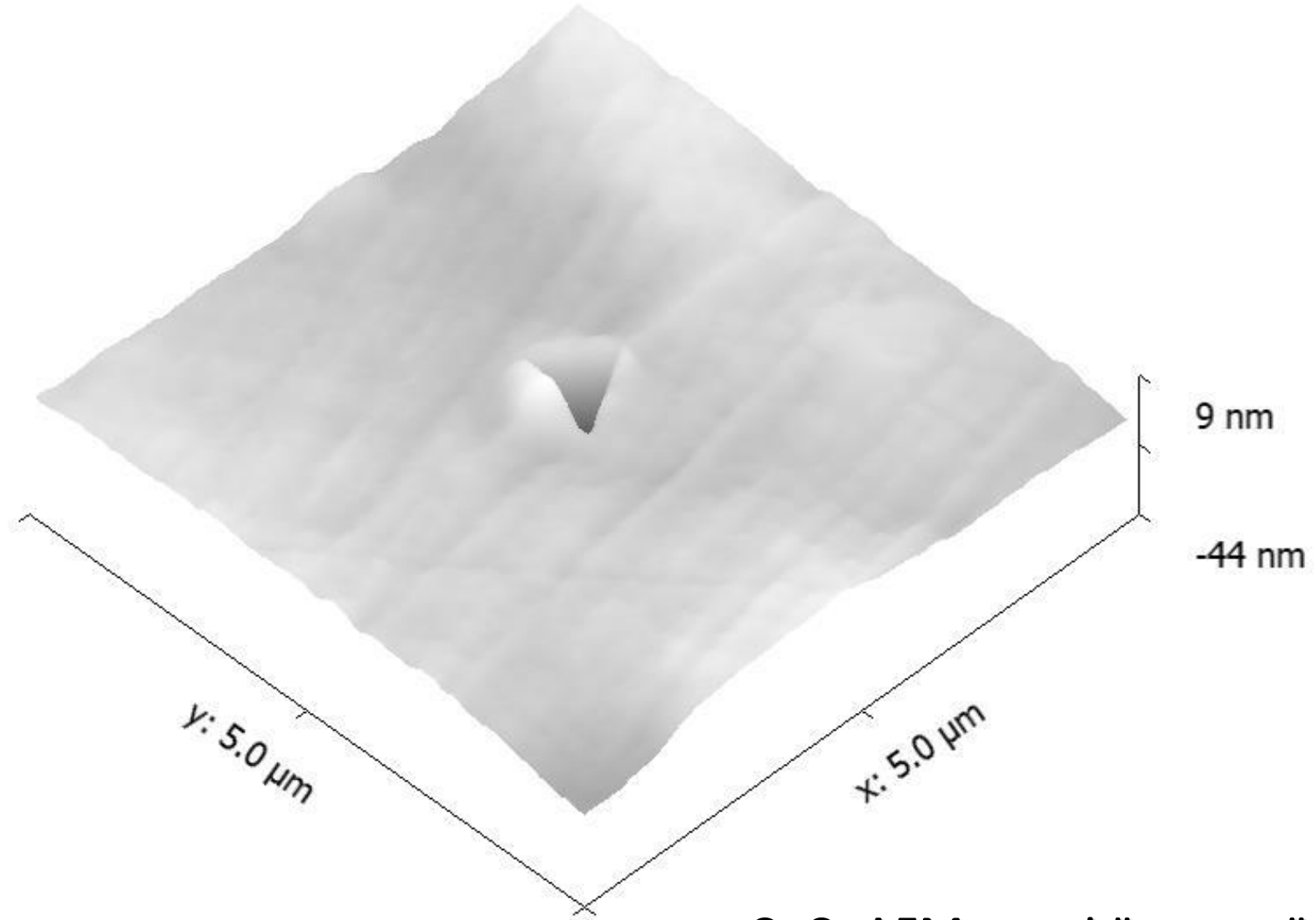
Nanoiz Uygulamaları:Video



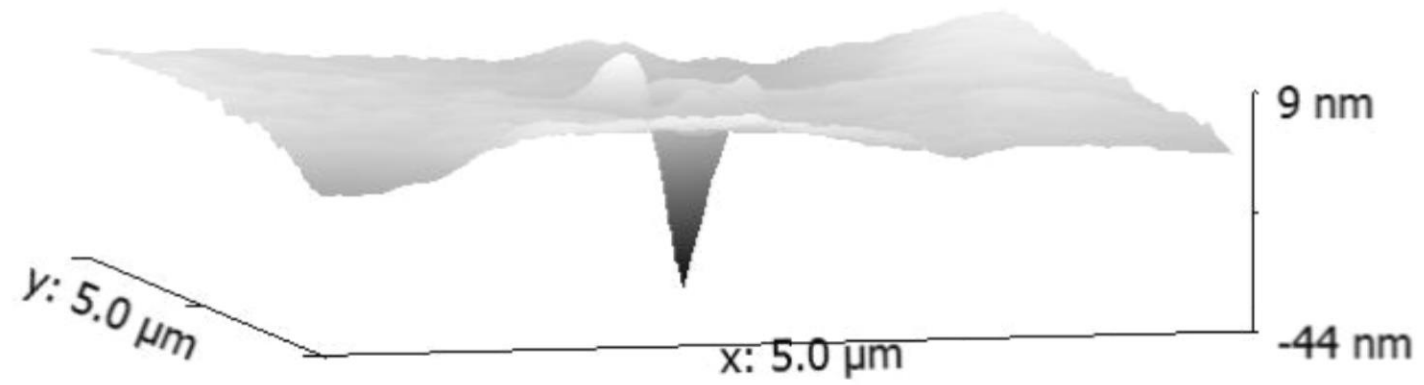
Atomik Kuvvet Mikroskobu ile Virüs üzerine Nanoiz uygulaması



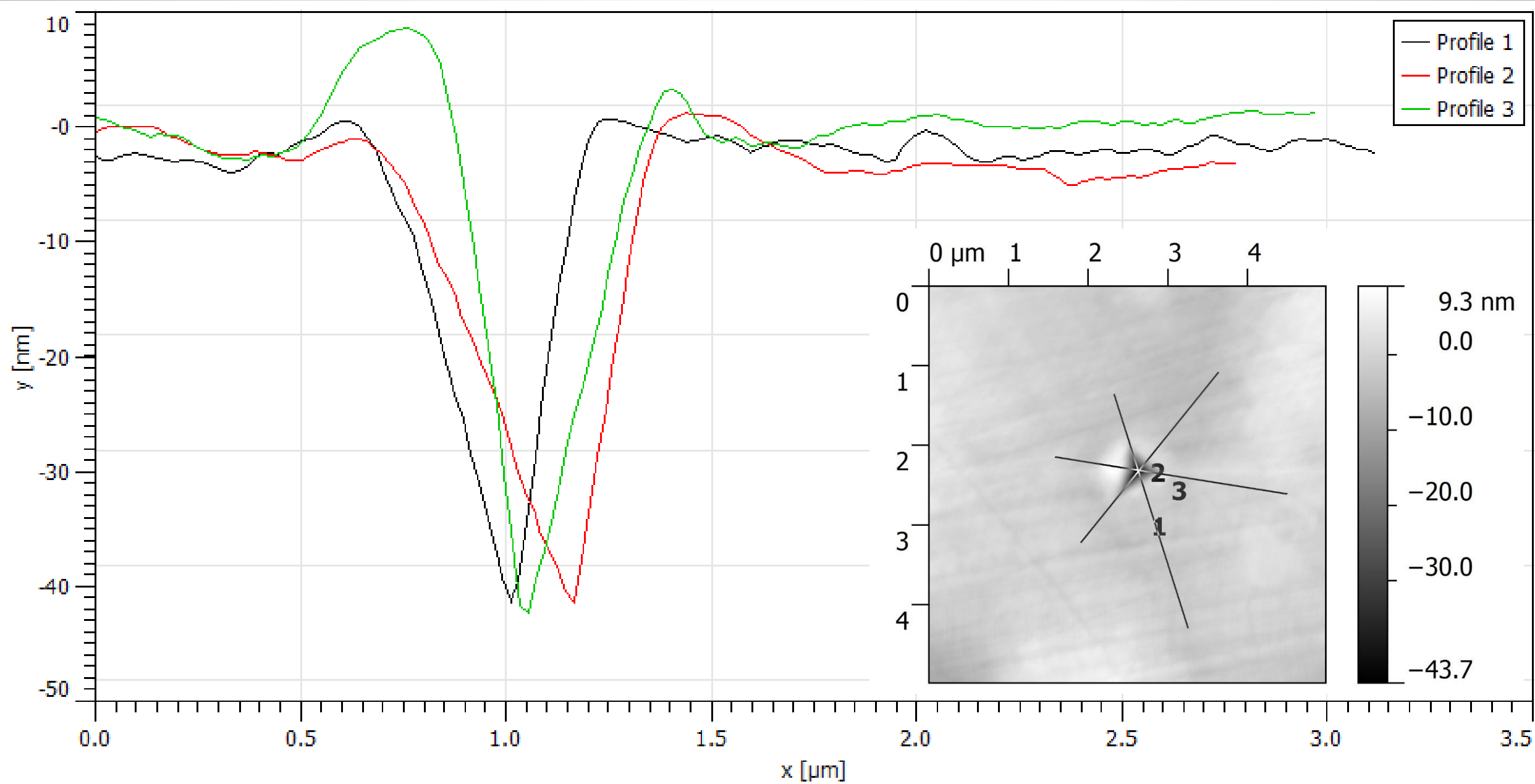
CoCr AFM resminin üstten görünüşü



CoCr AFM resmi üstten görünüş



CoCr AFM resmi üstten görünüş



Teşekkürler



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