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Universidad del País Vasco | *Euskal Herriko Unibertsitatea*

Internal Friction at Nano-scale and Size-effects on Damping in Shape Memory Alloys

J. San Juan, J.F. Gómez-Cortés & M.L. Nó

e-mail: jose.sanjuan@ehu.es

Dept. of Physics, Faculty of Science and Technology,
University of the Basque Country, UPV/EHU, Bilbao, Spain

Objective: Applying SMA at small scale

The SMA are more competitive as sensors and actuators when decrease de size of the device

Maximum workout per unit of volume ~ 10^7 J/m^3

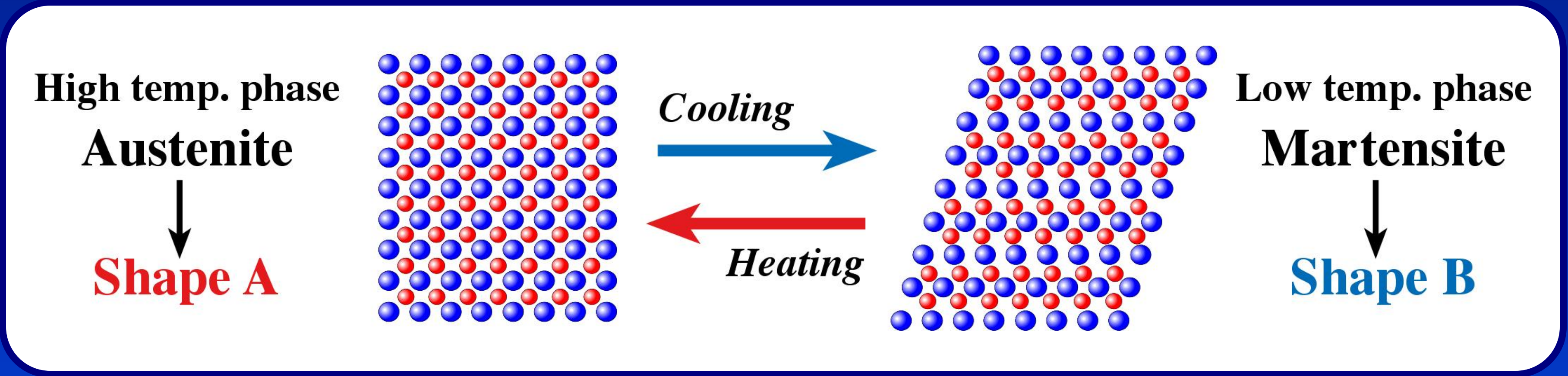
The Idea !!

Shape Memory Alloys for Micro Electro-Mechanical Systems (SMA for Smart MEMS called SMEMS)

Projects: MAT2017-84069-P from the Spanish Ministry MINECO. & ELKARTEK-CEMAP-2020 from Basque Government.

Shape Memory Alloy at small-scale

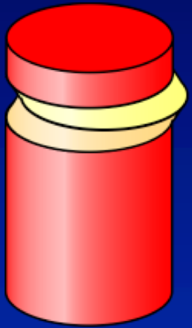
Thermo – elastic Martensitic Transformation



Thermally or stress-induced transformation

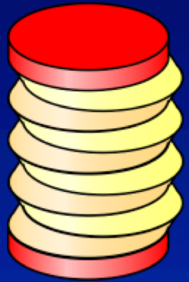
Stress-induced ⟹ Superelastic effect

Superelastic effect

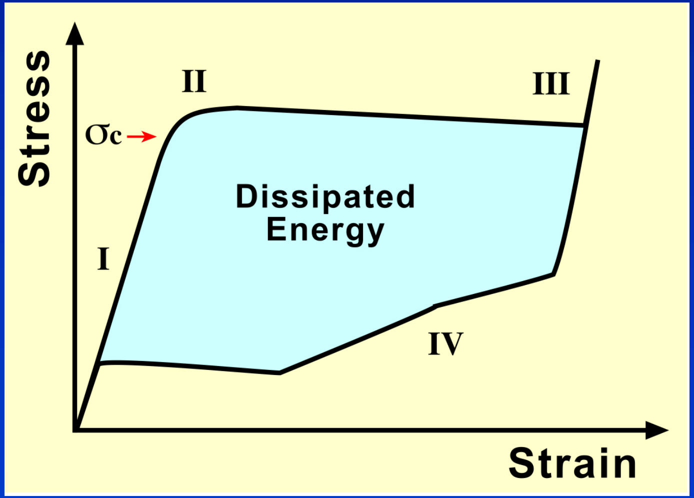


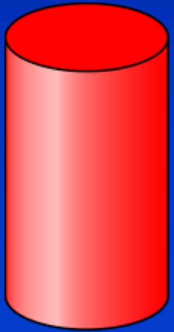
II. At a critical stress σ_c Martensite is induced

II to III Martensite variants compatible with the applied stress are induced



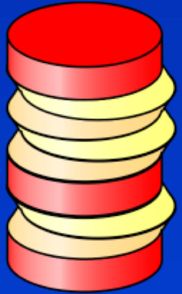
III. All the Austenite could be transformed to Martensite





I. Elastic deformation of the Austenite Pillar

Recovery when the stress is withdrawn



IV. The transformation is reversible during unloading

Focus Ion Beam (FIB) Micro / Nano Pillars

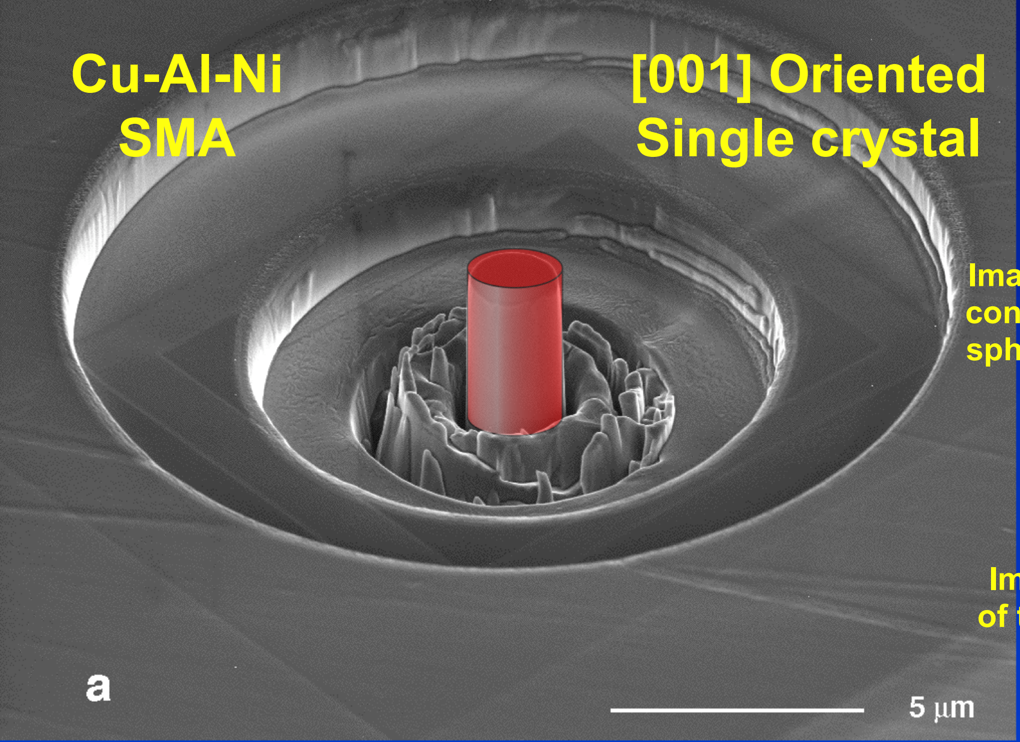


Image at constant contact force 2 µN sphero-conical tip

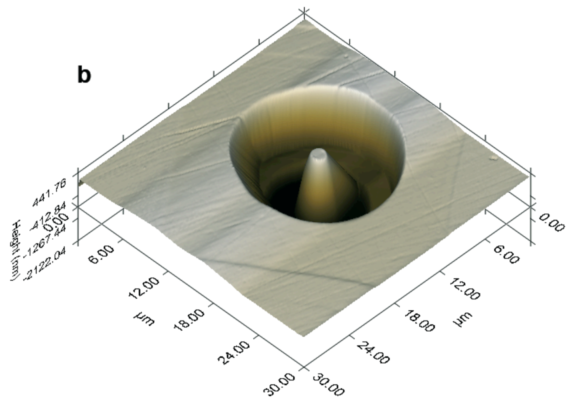
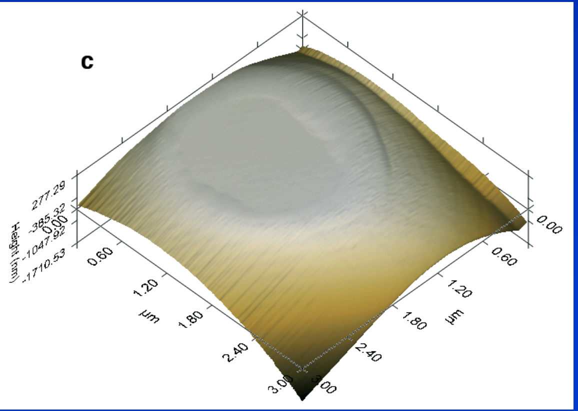


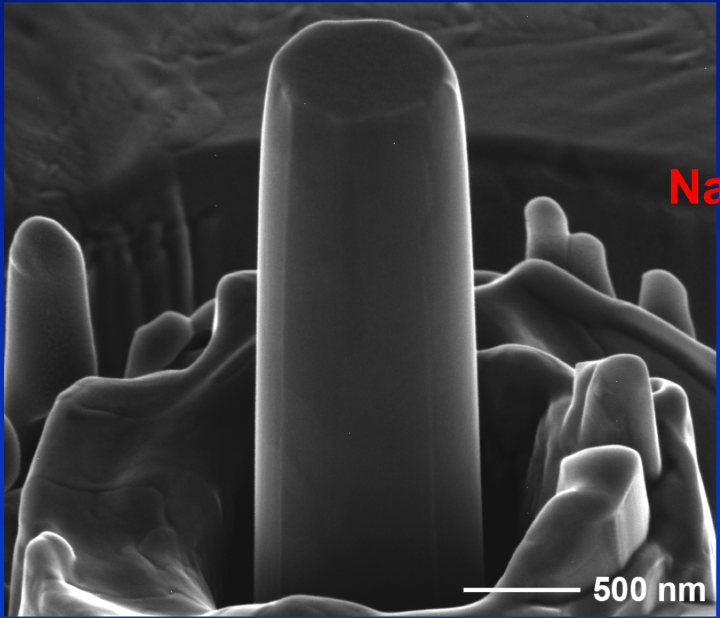
Image of the top of the micro-pillar



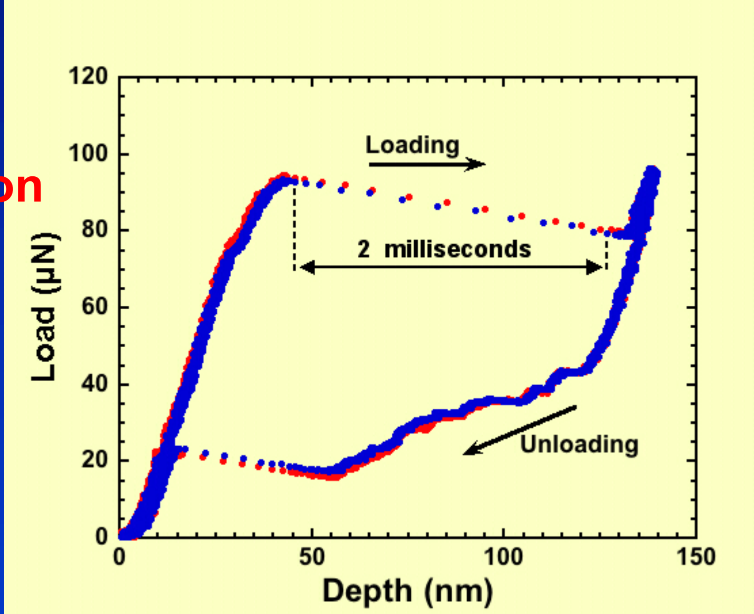
J. San Juan, M.L. Nó & C. Schuh,   Advanced Materials 20 (2008) 272

J. San Juan, M.L. Nó & C. Schuh,   J. Materials Research 26 (2011) 2461

Superelastic Micro & Nano - Pillars



Nanocompression behaviour



Superelastic behaviour of a sub-Micrometer Pillar

J. San Juan, M.L. Nó & C. Schuh, Nature Nanotechnology 4 (2009) 415-419

Are there size-effects on superelasticity ?

The beginning of the size-effects

All began in 2004 & 2005

When the pioneers on the size effects in confined crystal plasticity

Uchic, M.D., Dimiduk, D.M., Florando, J.N. & Nix, W.D. , Sample dimensions influence strength and crystal plasticity. *Science* 305, 986-989 (2004).

Gall, K., Diao, J. & Dunn, M.L. , The strength of gold nanowires. *Nano Letters* 4, 2431-2436 (2004).

Greer, J.R., Oliver, W.C. & Nix W.D. , Size dependence of mechanical properties of gold at the micron scale in the absence of strain gradients. *Acta Materialia* 53, 1821-1830 (2005).

Smaller is stronger !!

The beginning of the size-effects in SMA

A couple of years after, began the tests at micro-nano scale in SMA

1st Fully closed and reproducible superelastic test at nanoscale

San Juan, J., Nó, M.L. & Schuh, C.A. , Superelasticity and shape memory in micro- and nanometer-scale pillars. *Advanced Materials* 20, 272-278 (2008).

1st Size effect reported on superelasticity at nanoscale

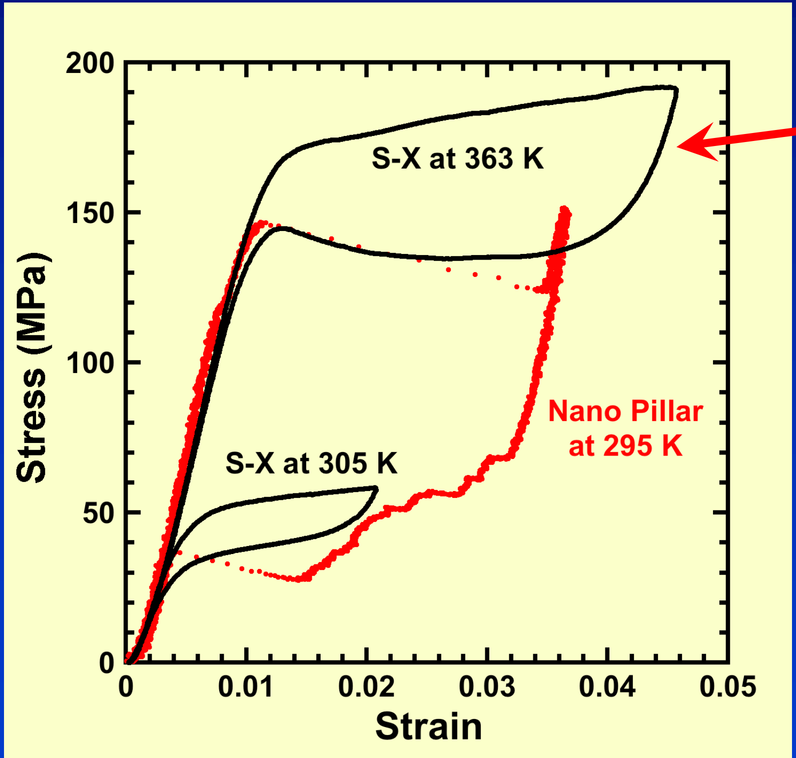
San Juan, J., Nó, M.L. & Schuh, C.A. , Nanoscale shape-memory alloys for ultrahigh mechanical damping. *Nature Nanotechnology* 4, 415-419 (2009).

1st In-situ TEM showing the size effect on selection rules of martensite at nanoscale

Nó, M.L., Ibarra, A., Caillard, D. & San Juan, J. , Quantitative analysis of stress-induced martensites by in situ transmission electron microscopy superelastic tests in Cu-Al-Ni shape memory alloys. *Acta Materialia* 58, 6181-6193 (2010).

Smaller is … different !!

Size - Effects at Nano - Scale



Compression test in Bulk Material

A.Ibarra, J. San Juan, M.L. Nó
Acta Materialia 55 (2007) 4789

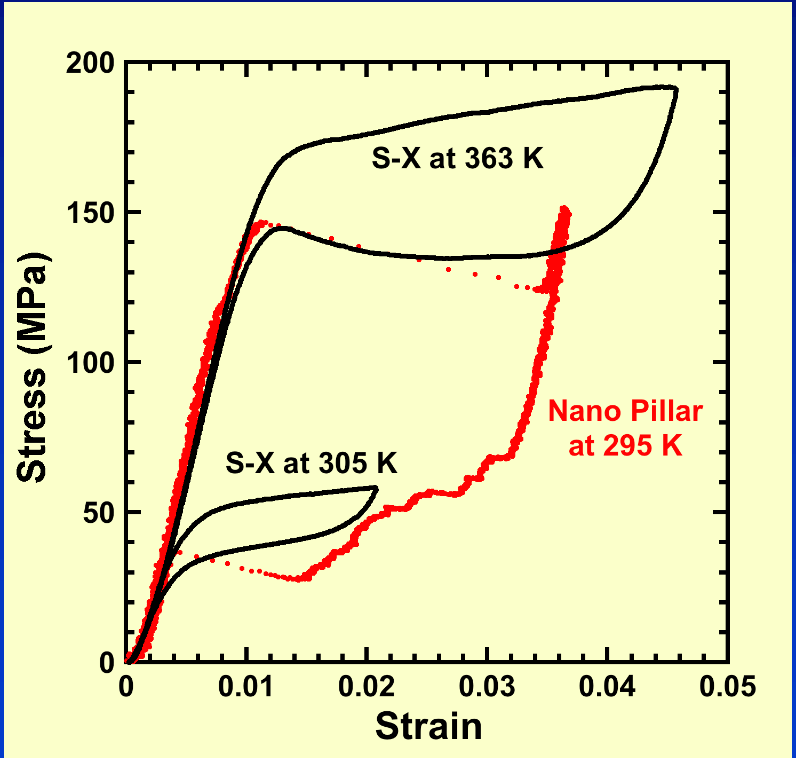
Size – effects
Superelastic behaviour
at Nano - scale

1st Size effect:

Increase of σ_c

J. San Juan, M.L. Nó & C. Schuh, Nature Nanotechnology 4 (2009) 415-419

Size - Effects at Nano - Scale



1^{st} Size effect:

Increase of σ_c

Paucity of dislocations for heterogeneous nucleation

2^{nd} Size effect:

Change of selection rules for martensite

Nucleation of Martensites with best oriented basal planes

3^{rd} Size effect:

Decrease of the σ for recovery

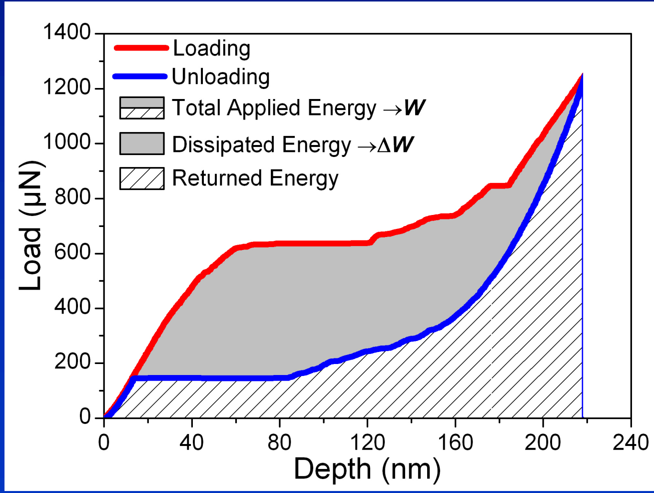
Martensite relieves the elastic stored energy at the free surface

M.L. Nó, A. Ibarra, D. Caillard, J. San Juan. Acta Mater 58 (2010) 6181

J. San Juan, M.L. Nó , J. Alloys & Compounds 577S (2013) S25

Size Effects: Ultra-high Damping

For a positive stress - strain cycle
Loss Factor or Internal Friction



High Damping Materials for Structural Applications
High Stiffness & High Damping

Columns loaded in compression
or Beams loaded in bending

$$Merit\ \ Index\ = E^{1/2}\cdot\eta$$

$$\eta = \tan(\phi) = \frac{\Delta W}{\pi\cdot W_{max}}$$

Size - Effects at Nano - Scale

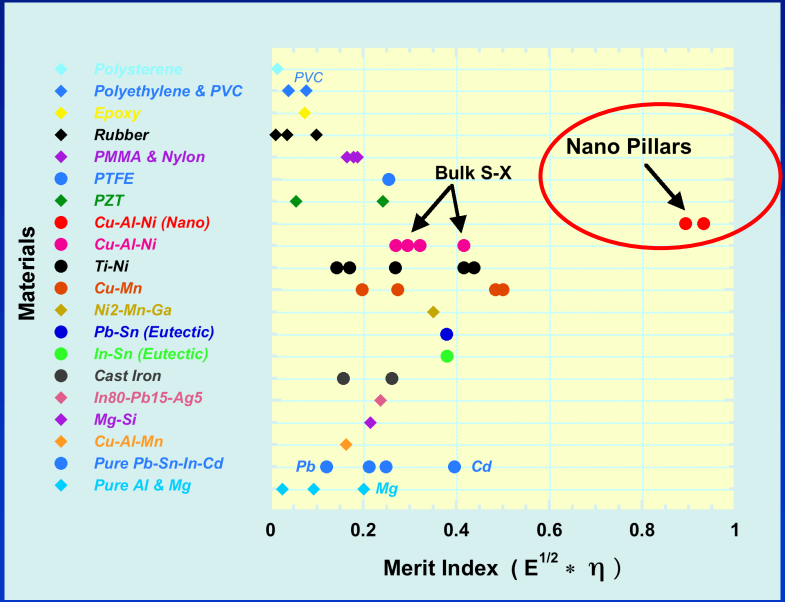
Increase of the critical stress for superelasticity

+

Decrease of the stress for recovery

↓

Increase of the cycle enclosed area



J. San Juan, et al, Nature Nanotechnology 4 (2009) 415-419

4^{th} Size effect: Ultra-High Damping at Nanoscale

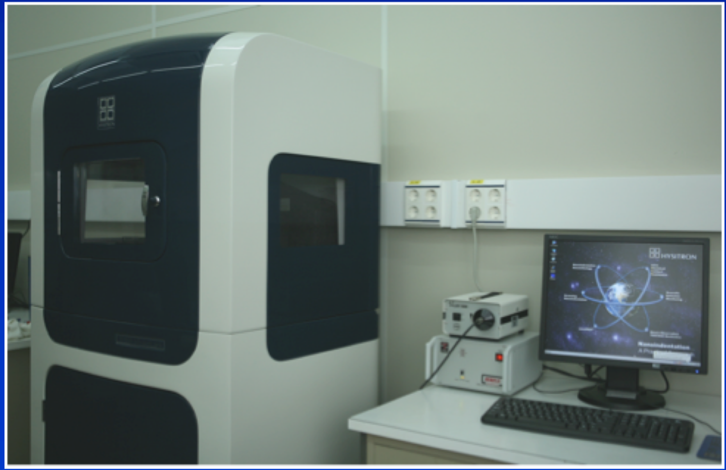
Objective: Size effect on σ_c for superelasticity

- Cu-Al-Ni & Cu-Al-Be Shape Memory Alloys. *Single crystals [001]*

- Micro & nano pillars milled by FIB
  (FEI Helios Nanolab 650)

- Nano – compression tests
  (Hysitron TI-950) &
  (Jeol 7000F + Hysitron PI-85)
  (Sphero-Conical indenter 2 μm radio)

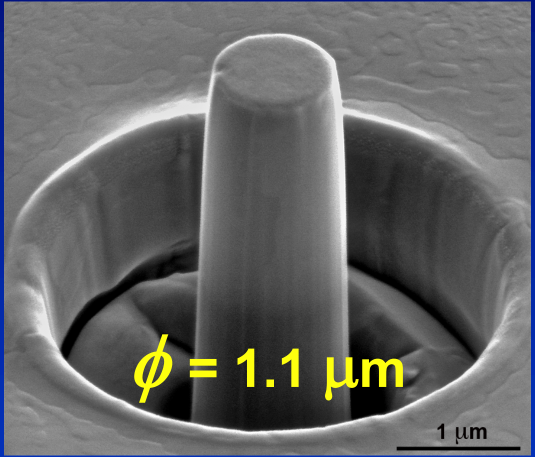
- A series of micro-nano pillars
  (from 2 μm to 260 nm ϕ)

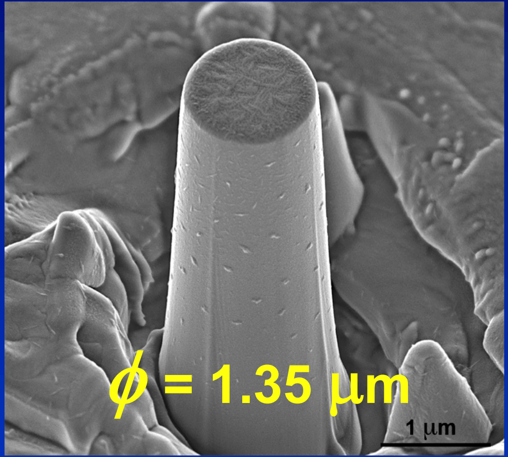


Size Effects on Superelasticity

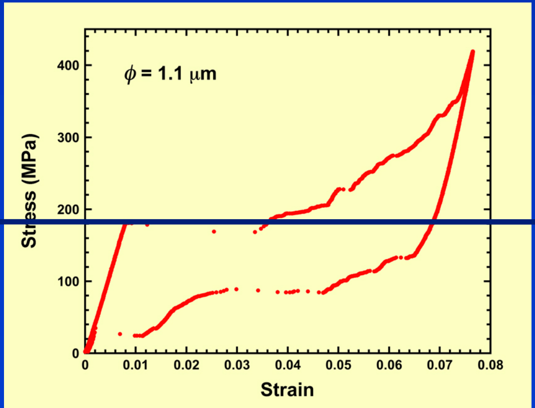
Cu-Al-Ni

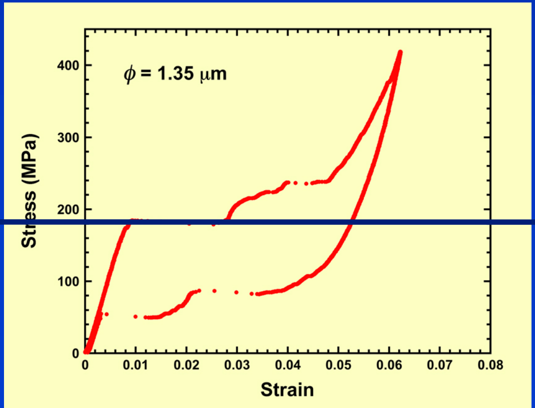
Pillars > 1μm





Nano-compression Stress – Strain curves



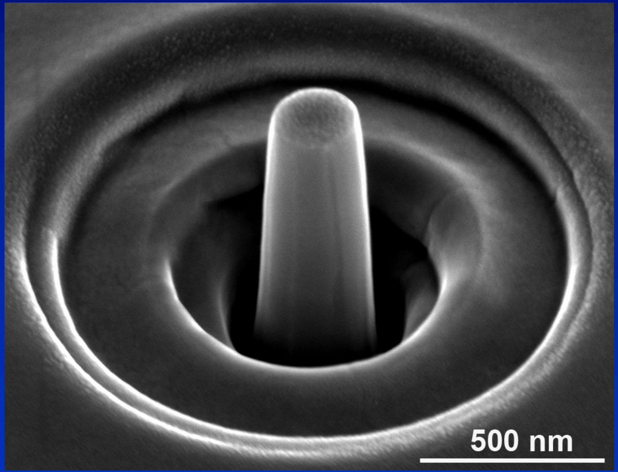


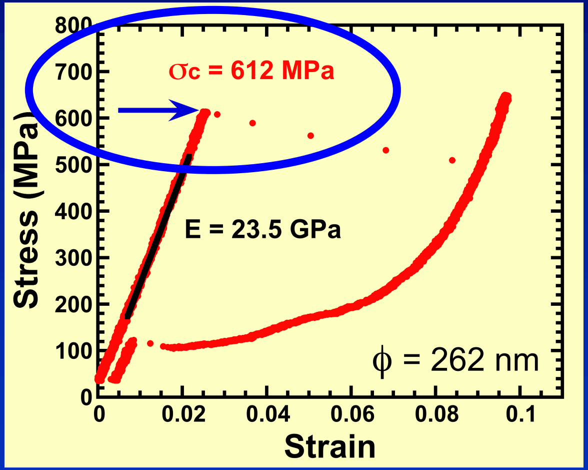
Superelastic testing at Nano-scale

Cu-Al-Ni

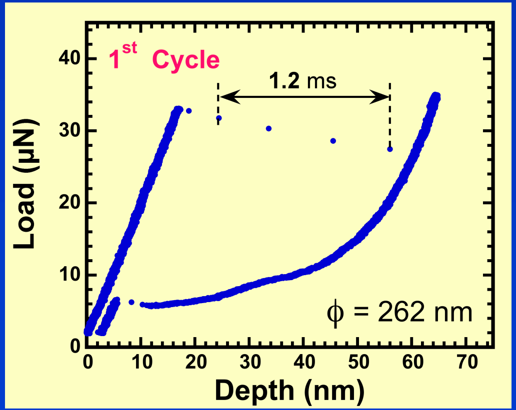
Small Pillar

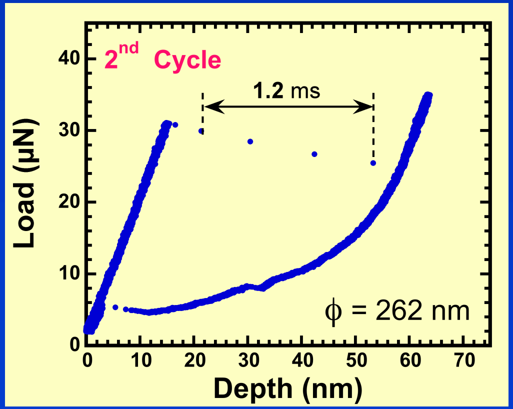
260 nm ϕ





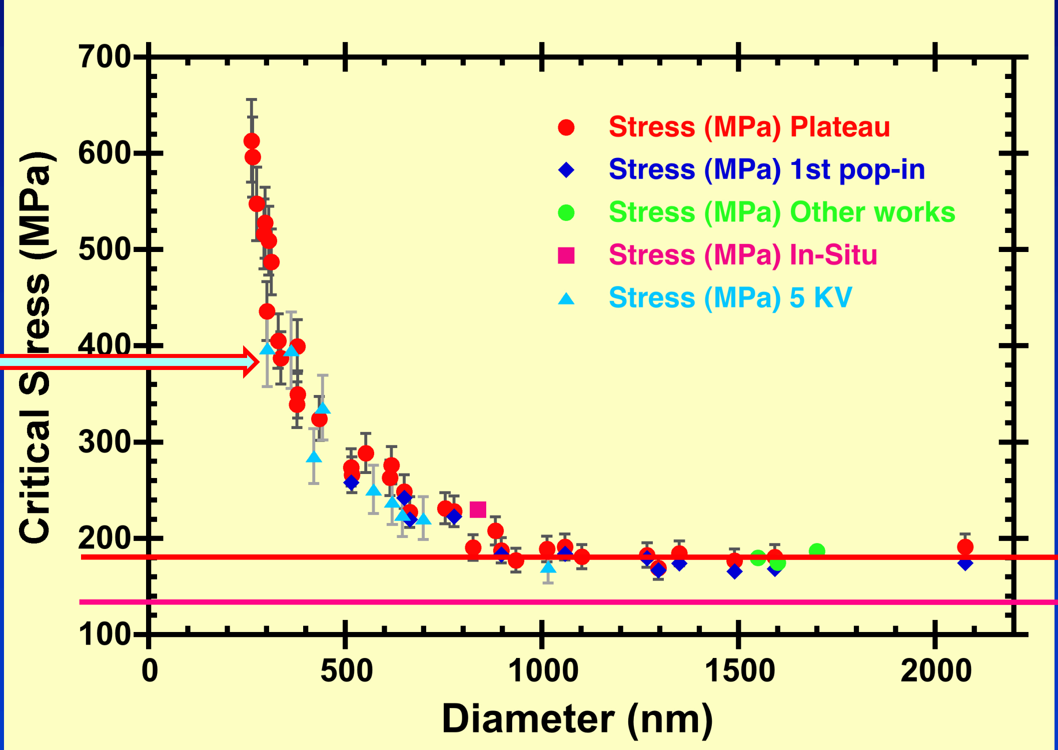
Comparison of the two first cycles





Size effect in Superelasticity

Strong increase on the critical stress for Superelasticity



Many Experimental conditions

Non-dependent Stress value σ_0

Bulk S-X

These results demonstrated that there is a size effect in Superelasticity

J.F. Gómez-Cortés et al., Nature Nanotechnology 12 (2017) 790 - 796

What is the scaling - law

for

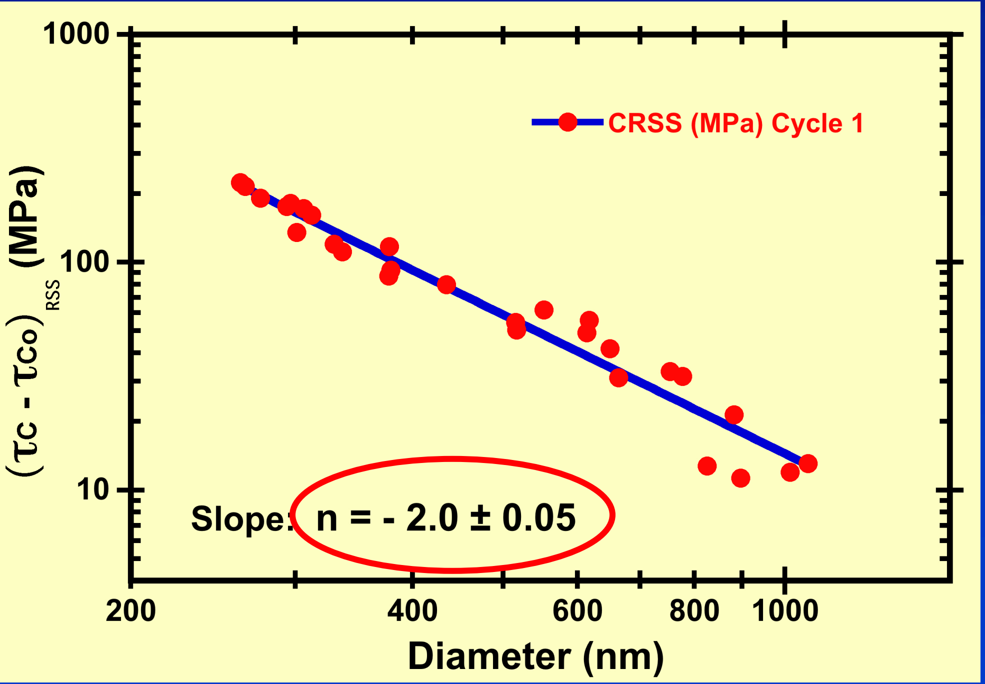
this size-effect ?

Scaling-Law at Nano-scale

First approach: $\sigma_C = \sigma_{C0} + A \times d^{-n}$

As proposed for the size – effect in confined plasticity

Cu-Al-Ni [001] single crystals



J.F. Gómez-Cortés et al., Nature Nanotechnology 12 (2017) 790 - 796

Universal Scaling-Law for superelasticity

Similar behavior for Cu-Al-Ni and Cu-Al-Be

Strong increase of the critical stress σ_C for superelasticity when decreasing the size of the pillar below 1 μm

Scaling-law for size-effect on the critical stress in superelasticity

n takes a value - 2

Universal Scaling-Law for superelasticity in Cu-based SMA

Universal Scaling-Law for superelasticity

Scaling power-Law : $\sigma_C = \sigma_{C0} + A \times d^{-n}$

Scaling-law for size-effect in plasticity: n vary from - 0.6 to - 0.8

Scaling-law for size-effect on the critical stress σ_C in superelasticity

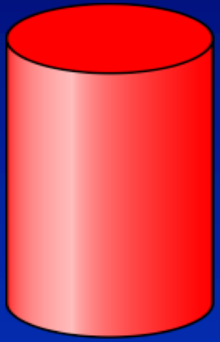
n takes a value - 2

Proposed model to explain this exponent 2

Due to starvation of dislocations and clean surfaces, there are no specific points for heterogeneous nucleation of martensite

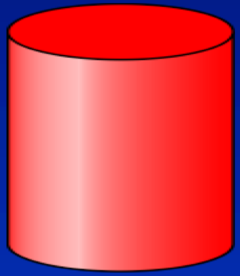
Homogeneous Nucleation of Martensite by shearing the atomic lattice of austenite parallel to {110} planes

Model for homogeneous nucleation



Elastic Compresion deformation
of the Austenite Pillar

$$\varepsilon_{rr} = - \nu \, \varepsilon_{zz}$$



Elastic Longitudinal contraction
Lateral expansion

Important effect because of the high value of the Poisson ratio

$$\nu = 0.47$$

(In-situ experiment)

Instrumented Pico - Indenter at SEM

SEM - FEG
JEOL – 7000F

+

Hysitron Pico-indenter
PI - 85

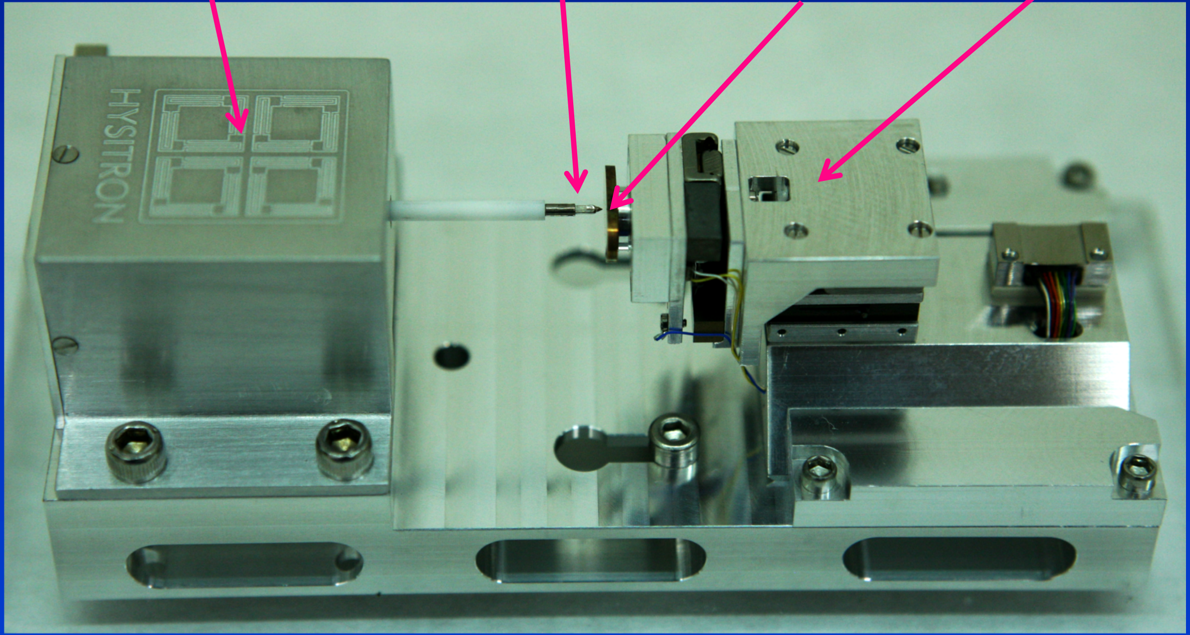
Instrumented Pico - Indenter at SEM

Hysitron electrostatic transducer

Conductor diamond Tip

Sample

Piezoelectric X-Y-Z Table



SEM - FEG
JEOL – 7000F

+

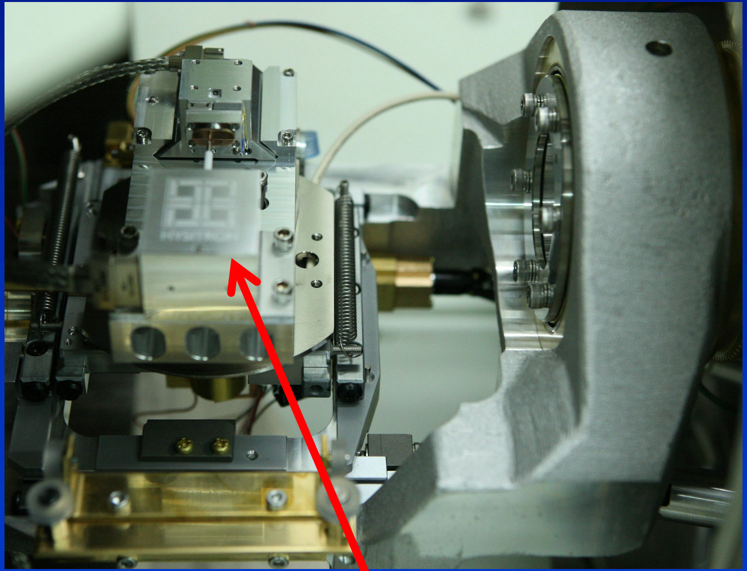
Hysitron Pico-indenter PI - 85

In-Situ Superelastic testing at SEM

Open the main door

SEM - FEG
JEOL – 7000F

+

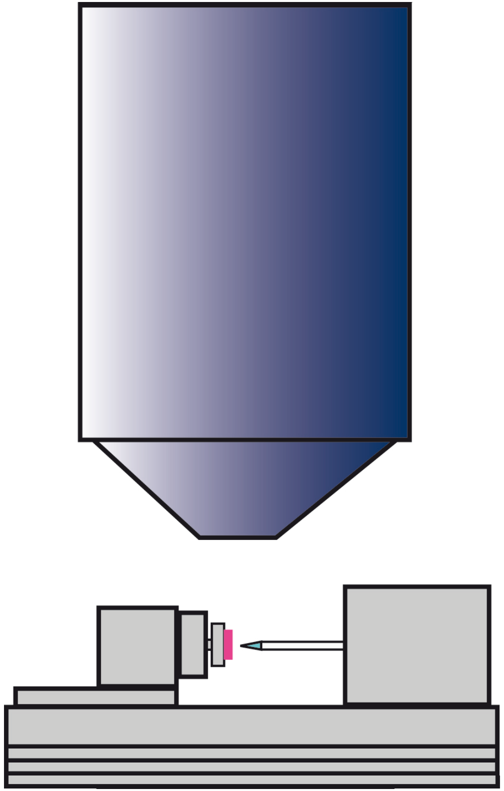


Hysitron Pico-indenter
PI - 85

In-Situ Superelastic testing at SEM

Pole piece of the SEM

Picoindenter

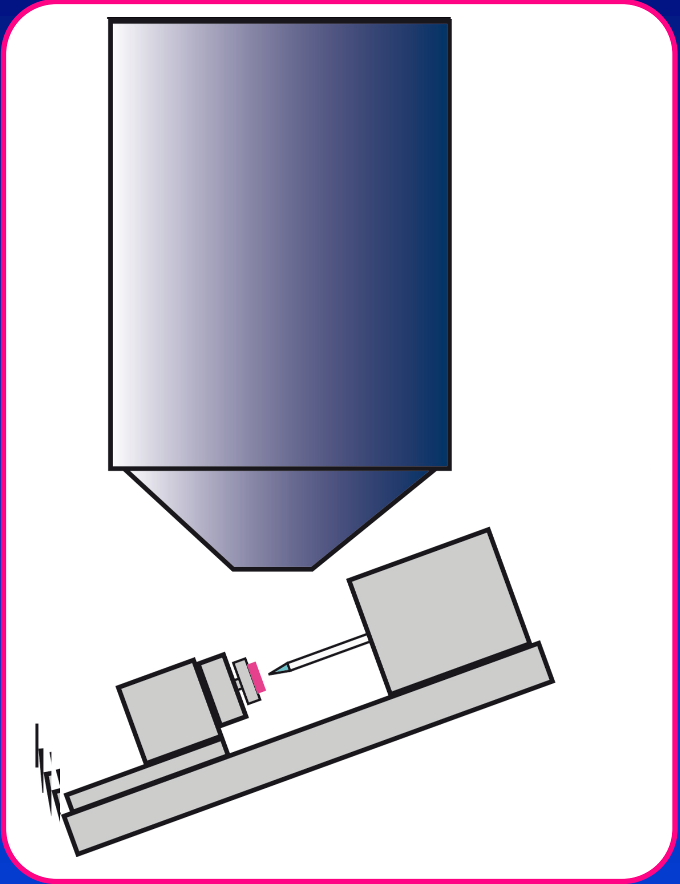


Rotation of the Picoindenter

Approach the Picoindenter to the WD

In-Situ Superelastic testing at SEM

Pole piece of the SEM

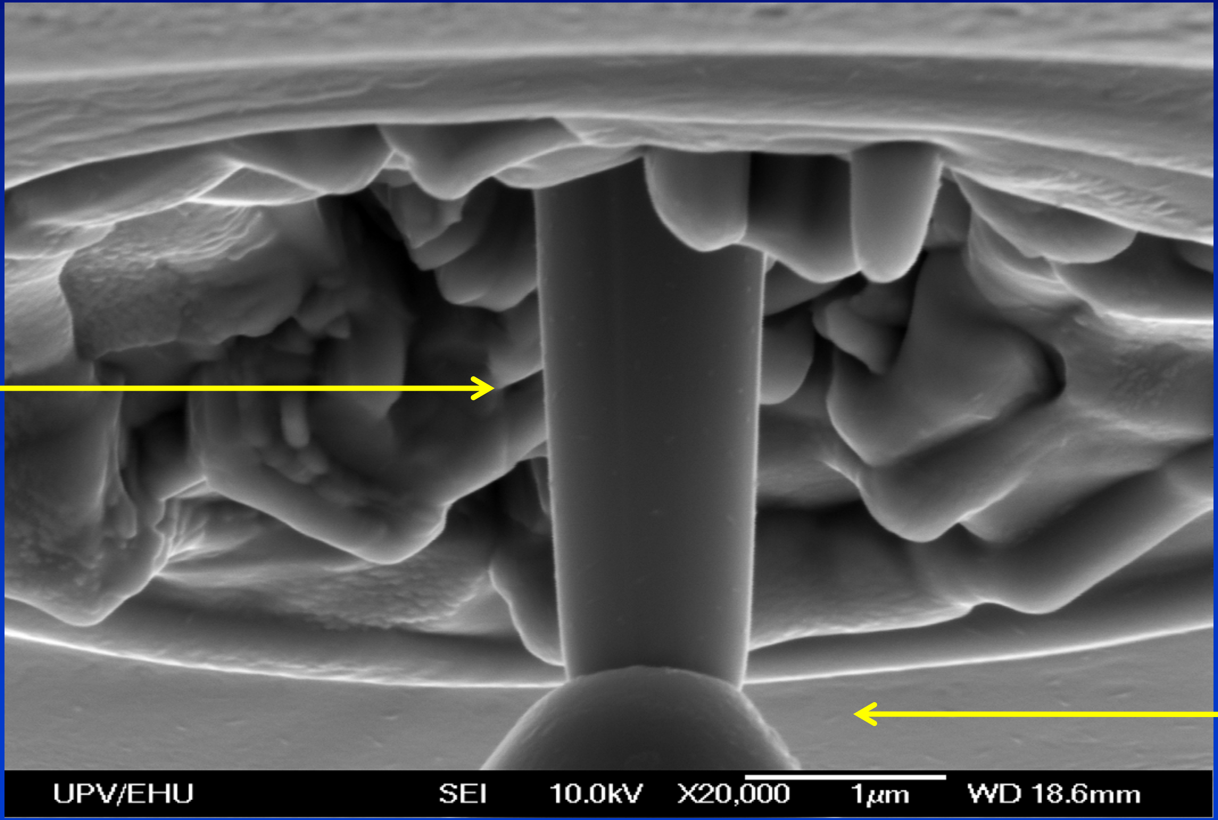


Picoindenter

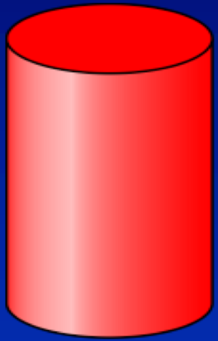
Tilt of the Picoindenter

Approach the sample to Picoindenter

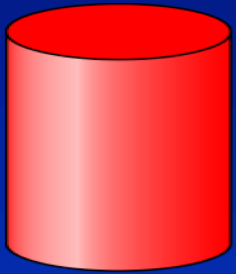
In-Situ Superelastic testing at SEM



Model for homogeneous nucleation

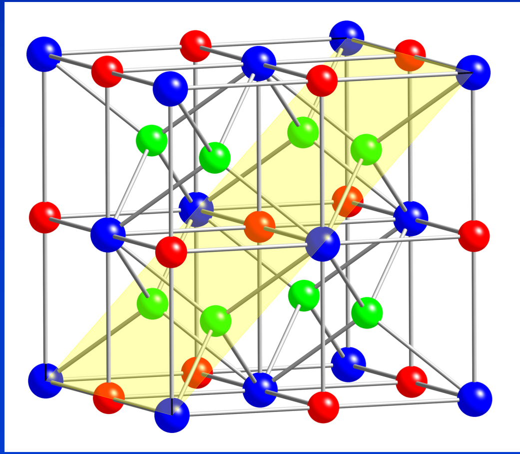


$$\varepsilon_{rr} = -\nu\, \varepsilon_{zz}$$



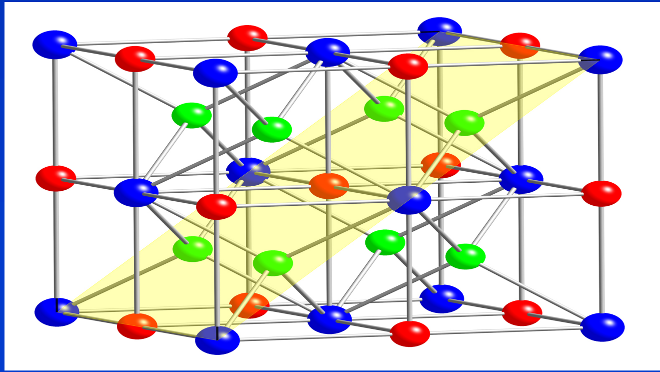
Elastic Compresion deformation of the Austenite Pillar

Elastic Longitudinal contraction Lateral expansion



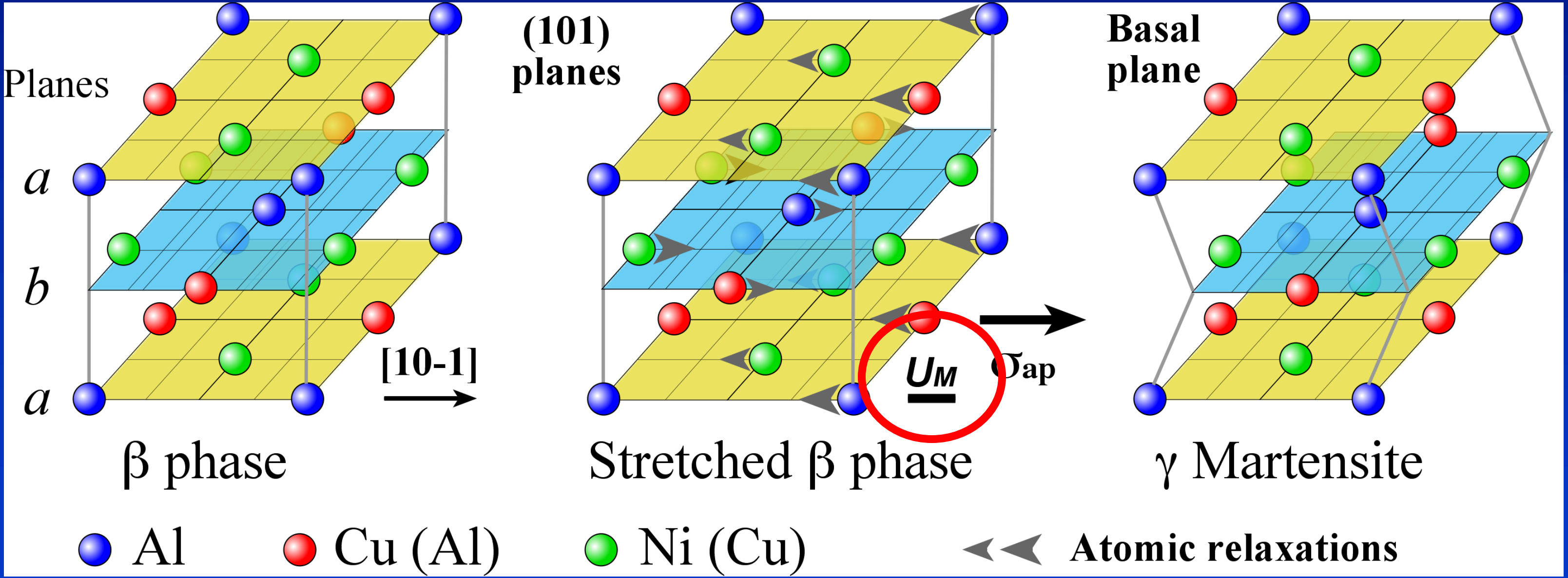
Compressed $L2_1$ Lattice

(101) planes expanded



Model for homogeneous nucleation

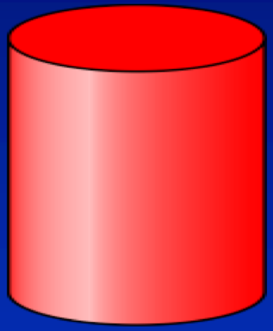
Atomic model for Homogeneous Nucleation of Martensite



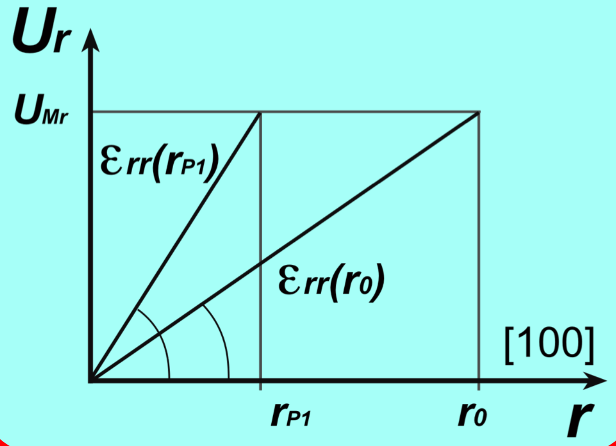
When the elastic displacement on (110) planes reach U_M the homogeneous transformation takes place.

Where is coming the size-effect from ??

Model for homogeneous nucleation



$\varepsilon_{rr} = -\nu\,(\sigma_{ap}/E)$



Evaluate

$$\frac{\partial \sigma_{apc}}{\partial r_P}$$

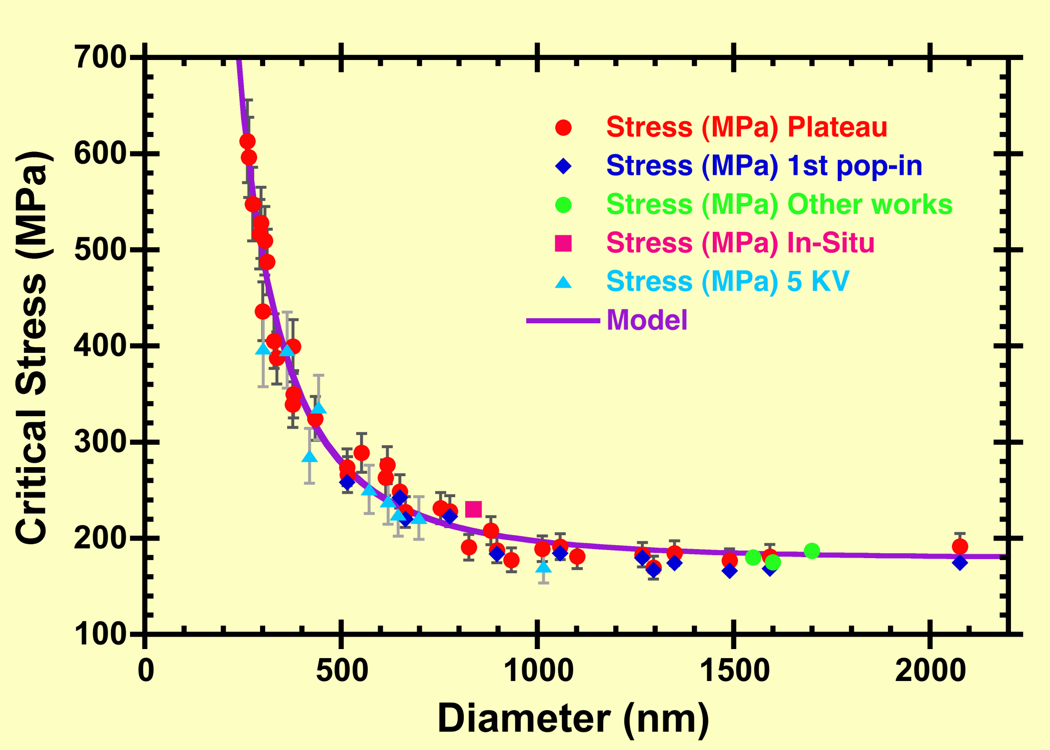
To reach U_M

$$\sigma_{apc}(d_P) = \sigma_{apc}(d_0) + \frac{2\sqrt{2}\cdot E\cdot U_M}{m_b\cdot \nu}\cdot (d_0 - d_P)\cdot \frac{1}{d_P^2}$$

Dependence of the Critical Stress on the pillar Diameter

Atomic model & Scaling-Law

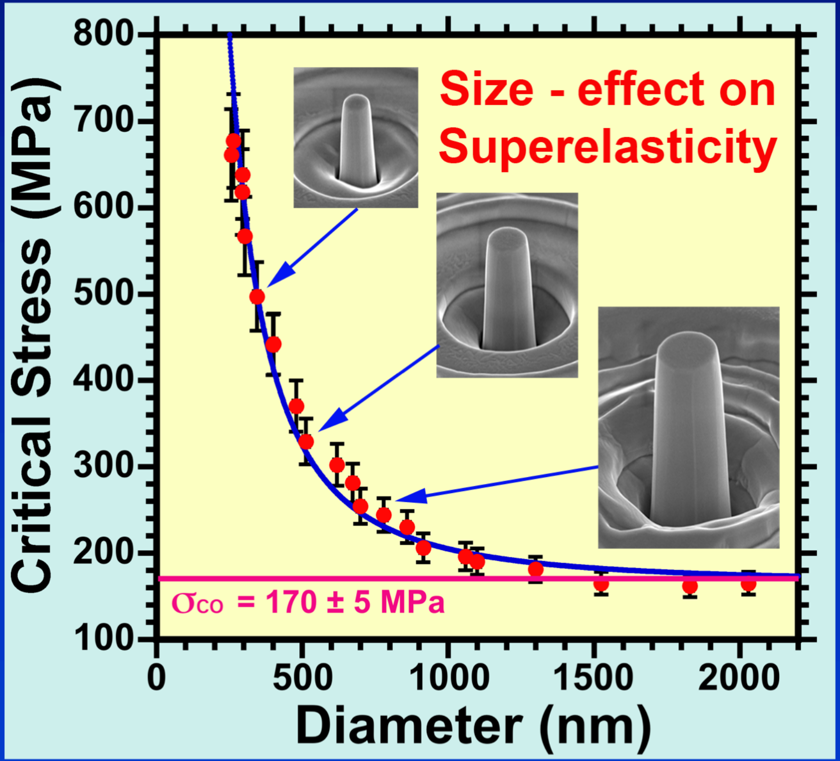
Cu-Al-Ni data



The Scaling-Law Model for Superelasticity is consistent with the experimentally observed Size-effect

Is this a particular behaviour or it is a general one ?

Universal Scaling-Law for the Size effect



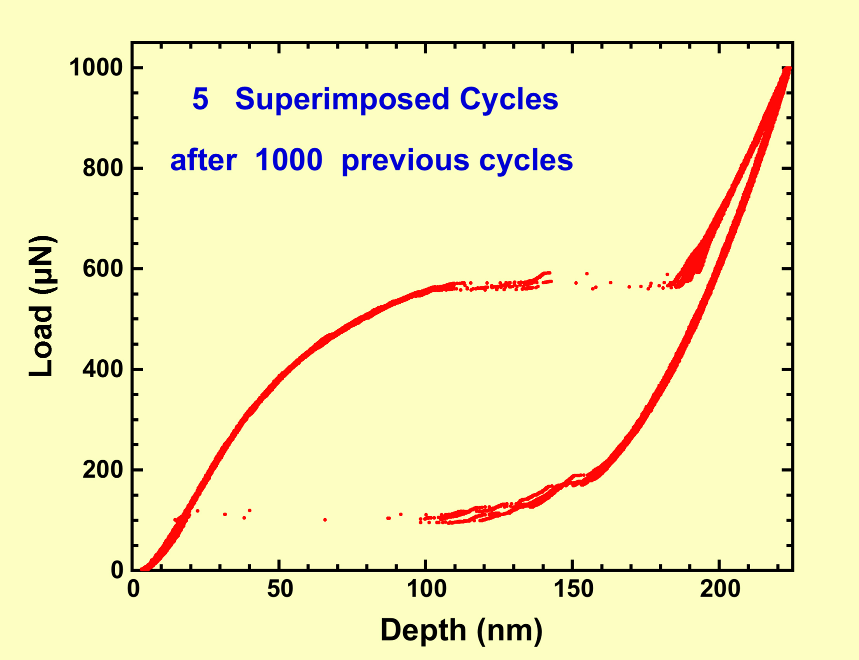
Similar results for Cu-Al-Be

Universal Scaling–Law for Superelasticity in Cu-based SMA

V. Fuster et al., Adv. Electr. Materials 6 (2020) 1900741

What about Damping at the nano-scale ?

Damping at Nano-scale



J. San Juan, et al., Appl. Phys. Lett. 104 (2014) 011901

Preliminary works on Cu-Al-Ni pillars

Very good reproducibility

Complete reversible recovery

Develop micro-dampers to protect MEMS

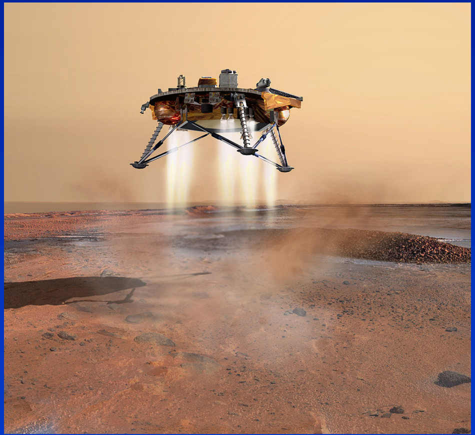
Micro-Nano Dampers of SMA

Vibration Damping in MEMS for aero-space applications

Spatial travel between the Earth and Mars

Take off: Mechanical vibrations. It is important to damp them

210 days of travel without relevant vibrations



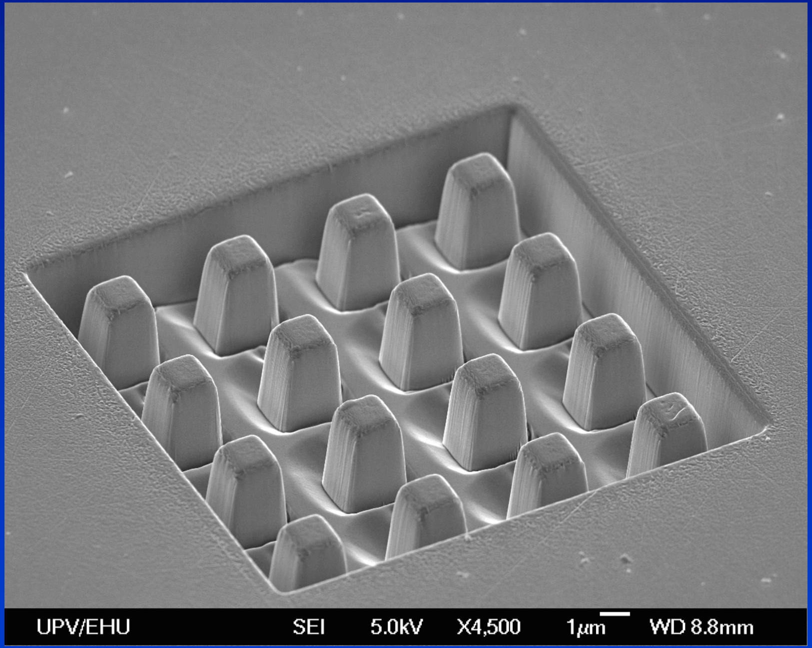
Landing: Mechanical vibrations.
Again, important to damp them

496 waiting days in Mars

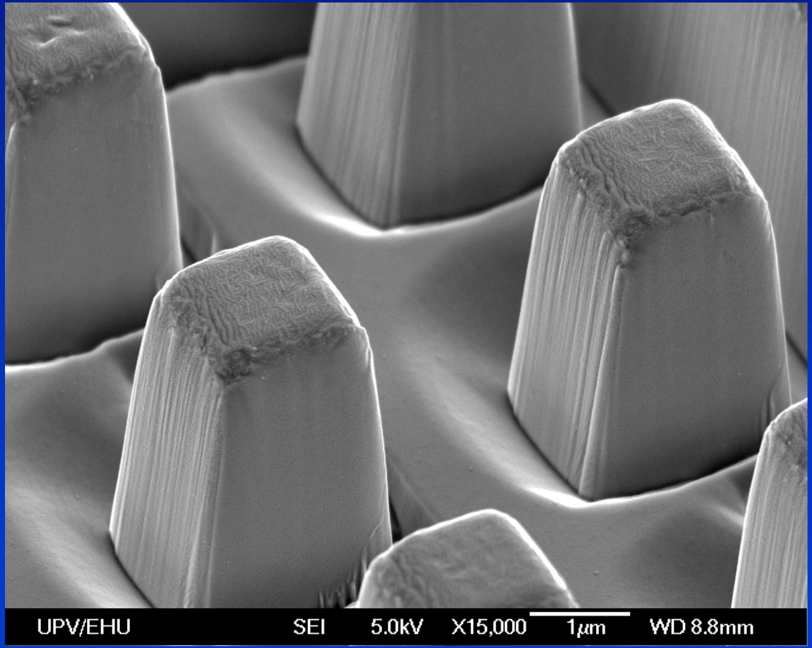
Landing at Earth

Damping at Nano-scale

To test the reproducibility of Damping on cycling and on time



Array of 16 micropillars : 20 μm^2



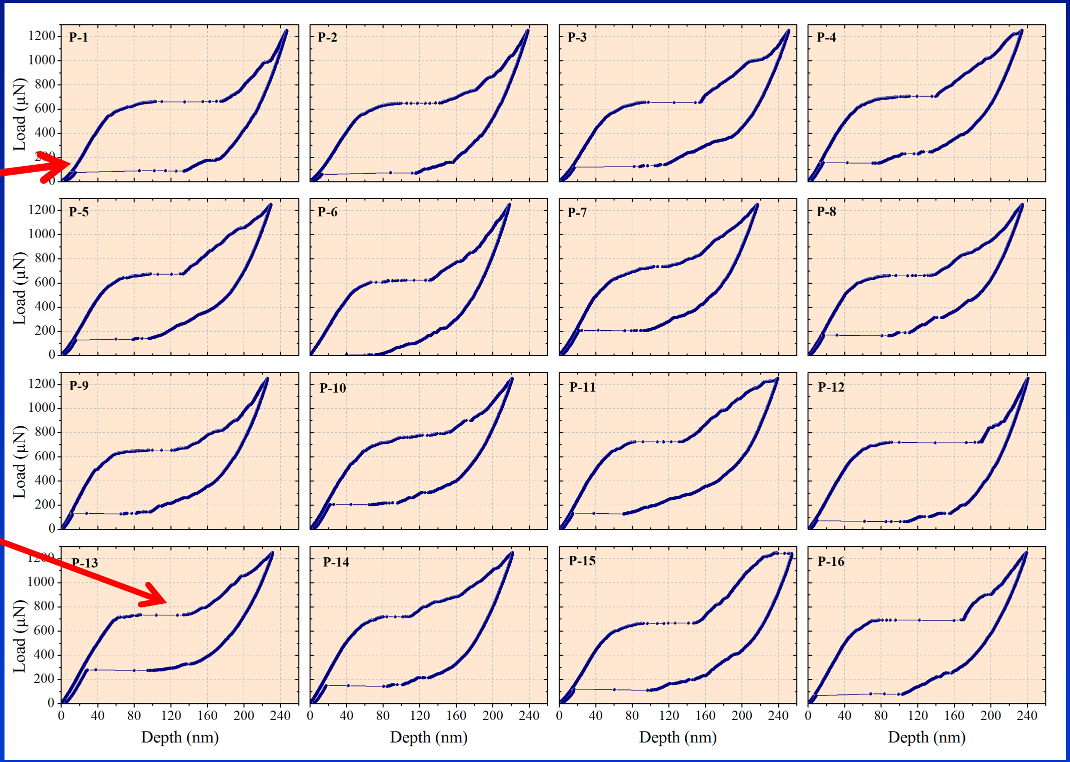
Pillars size = 1,7 x 1,7 x 3,5 μm^3

Damping at Nano-scale

β_3

Superelastic tests
Nano-compression

martensite

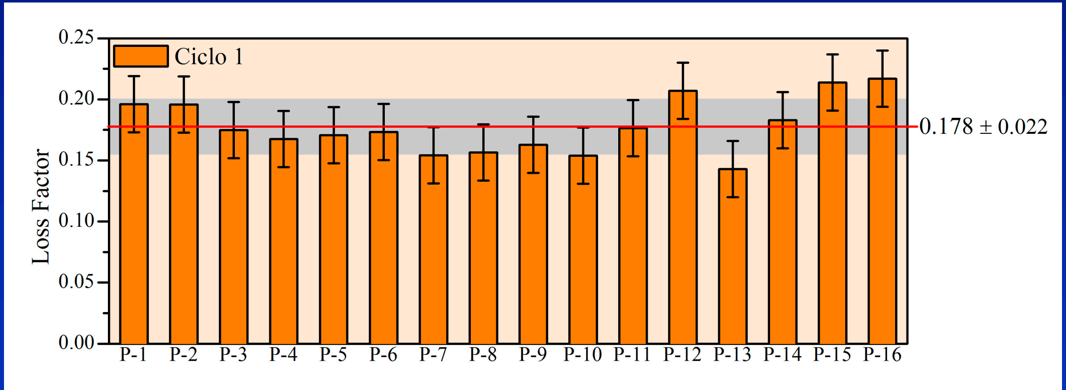


Pillars of the array

Similar behaviour for all pillars

J.F. Gómez-Cortés et al., Acta Mat. 166 (2019) 346-356

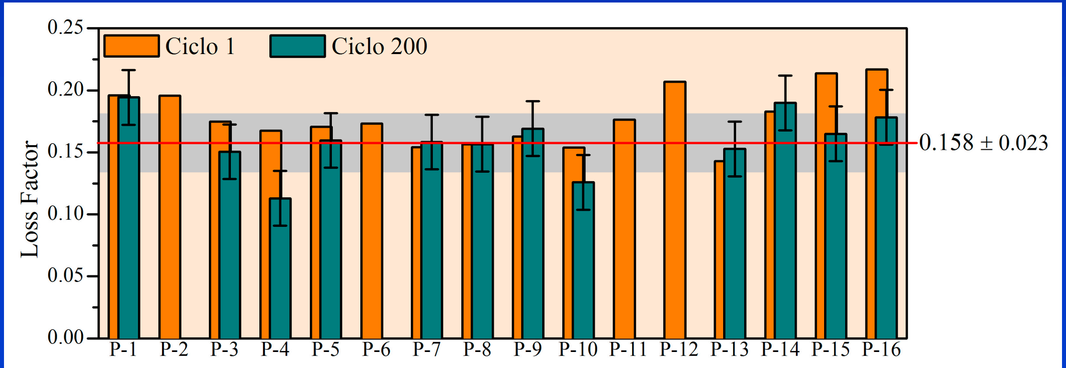
Damping at Nano-scale



Loss factor for the 16 pillars

Mean value

$\eta = 0.178$



Damping after 200 cycles

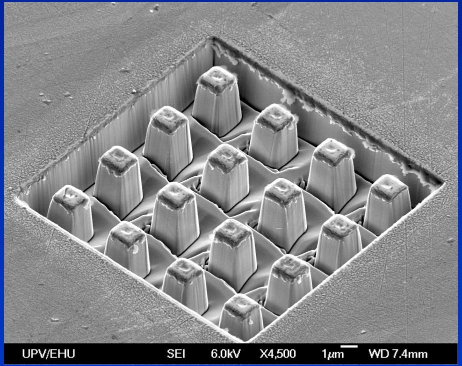
Mean value

$\eta = 0.158$

Slightly lower but still very high

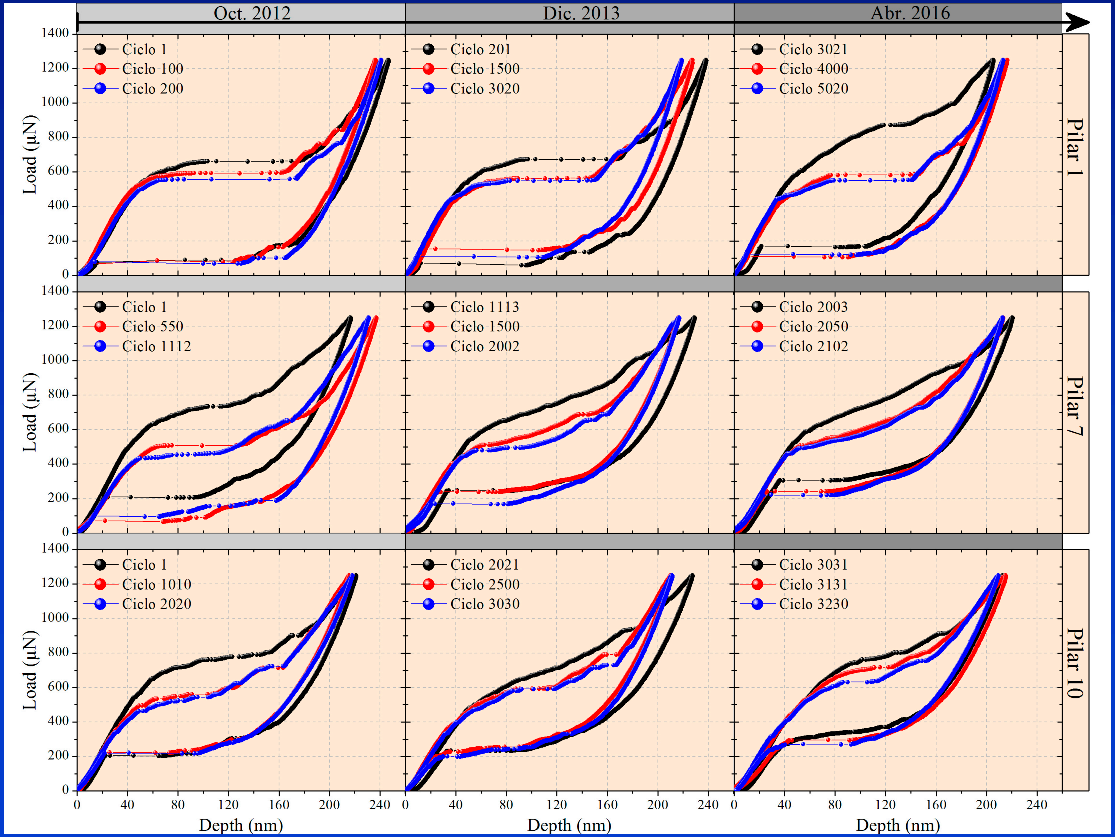
Damping at Nano-scale

All pillars were tested for 200 cycles



Some pillars were tested for more than 2000 cycles

First campaign | 1 Year later | After 4 years



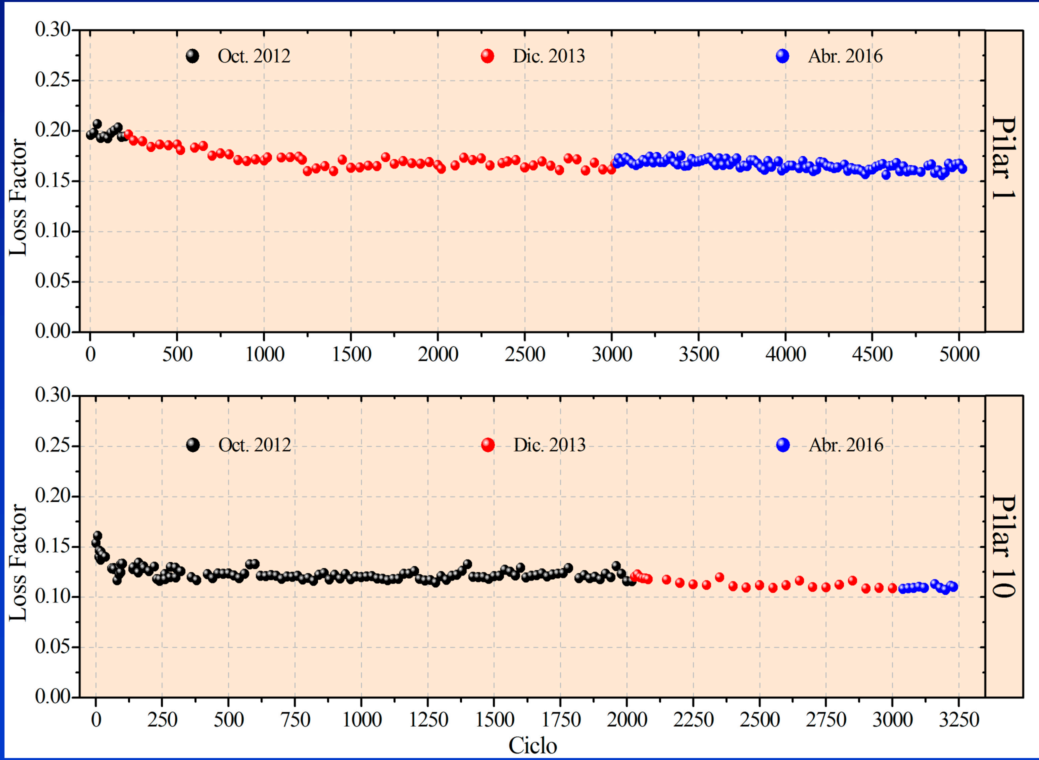
Pillar 1
5020 cycles

Pillar 7
2102 cycles

Pillar 10
3230 cycles

Damping at Nano-scale

Loss Factor



Very good reliability as a function of time

P1: η = 0.17

P10: η = 0.12

Ultra-high Damping along cycling & on time

J.F. Gómez-Cortés et al., Acta Mat. 166 (2019) 346-356

Is this damping behaviour only present in compression?

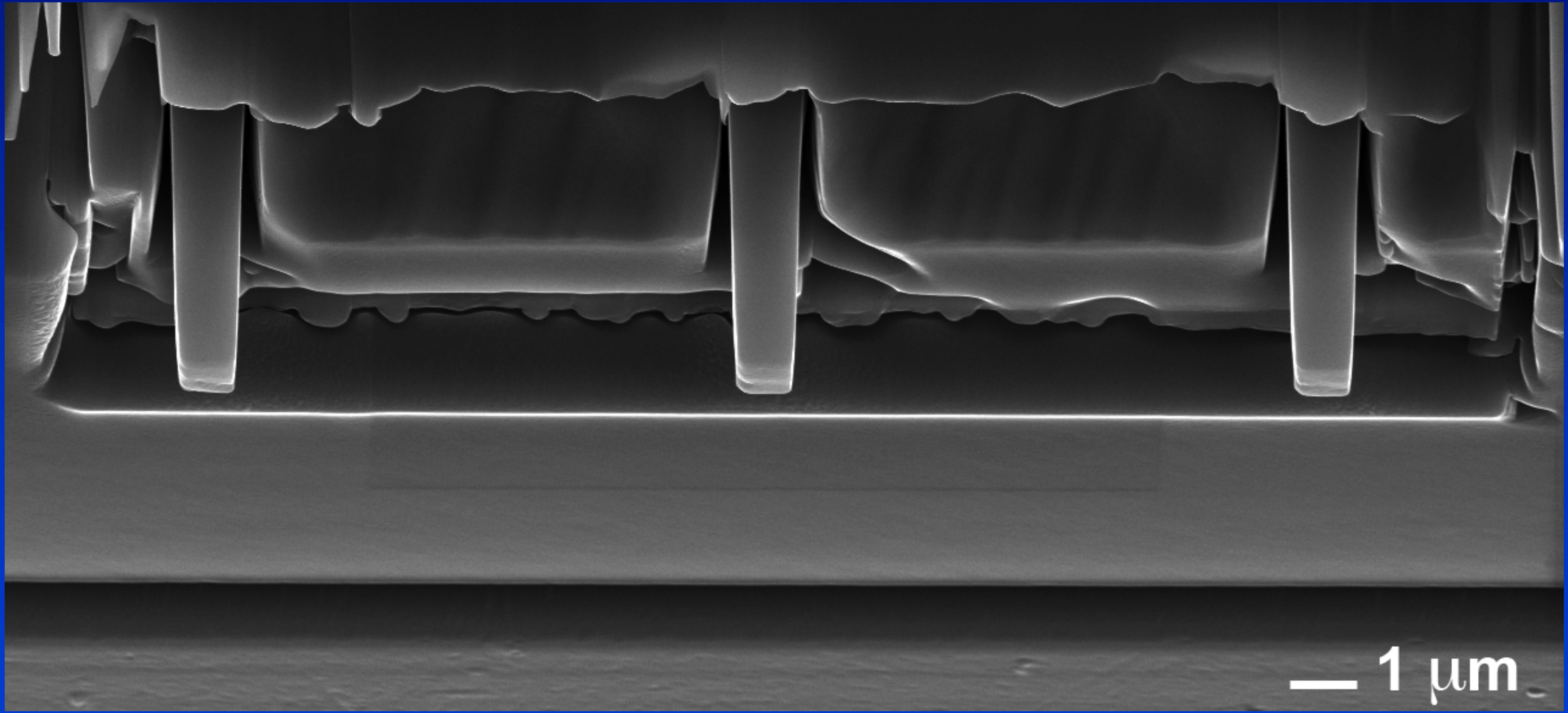
Damping at Nano-scale

To answer the above question we start by doing some preliminary in-situ off-axis tests in bending mode.

Then, several micro-beams were produced by FIB, to be tested in bending at the nanoindenter

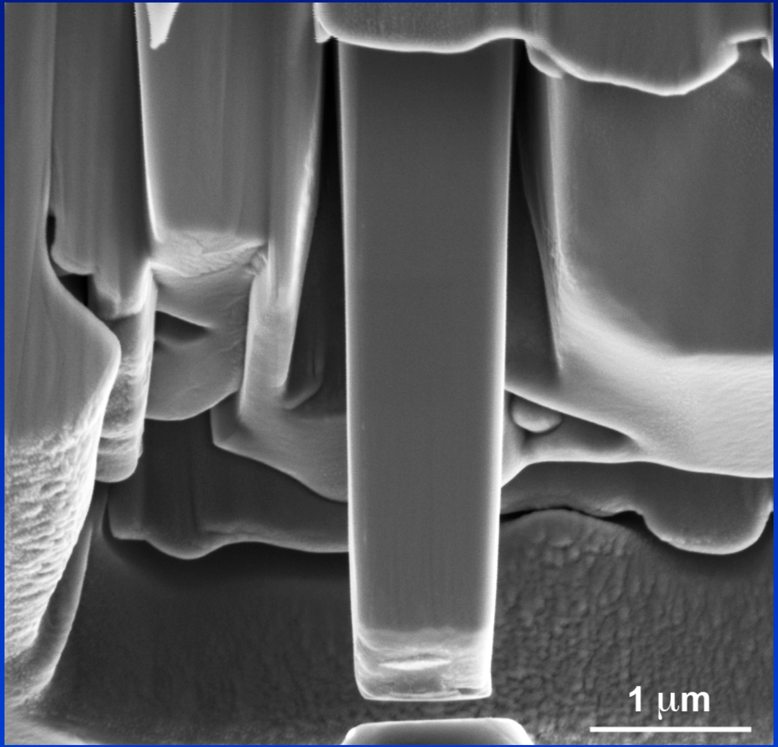
The results from these tests evidence …

Damping at Nano-scale: In-situ tests

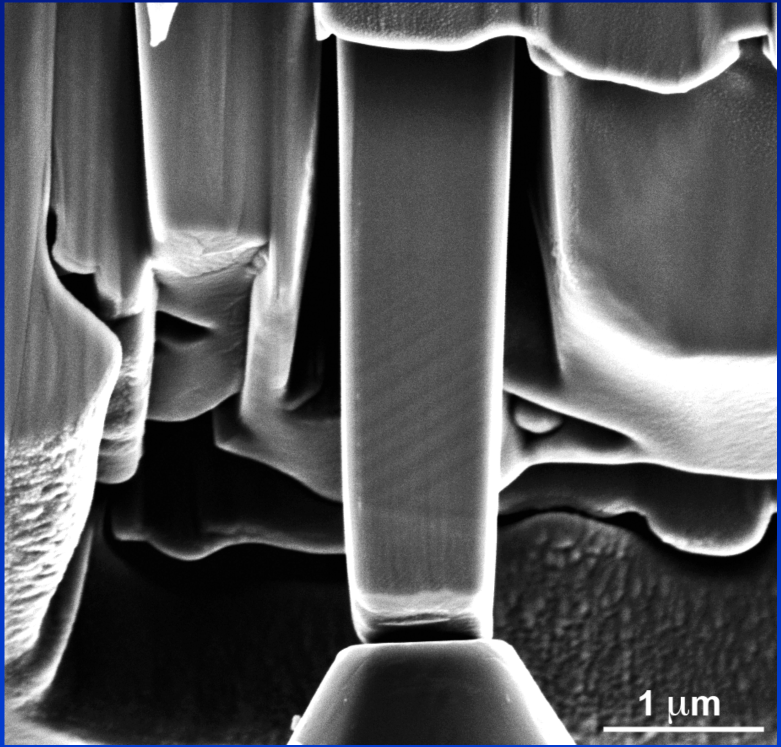


Array of Cu-Al-Ni micropillars milled by FIB on [001] single crystal

Damping at Nano-scale: In-situ tests

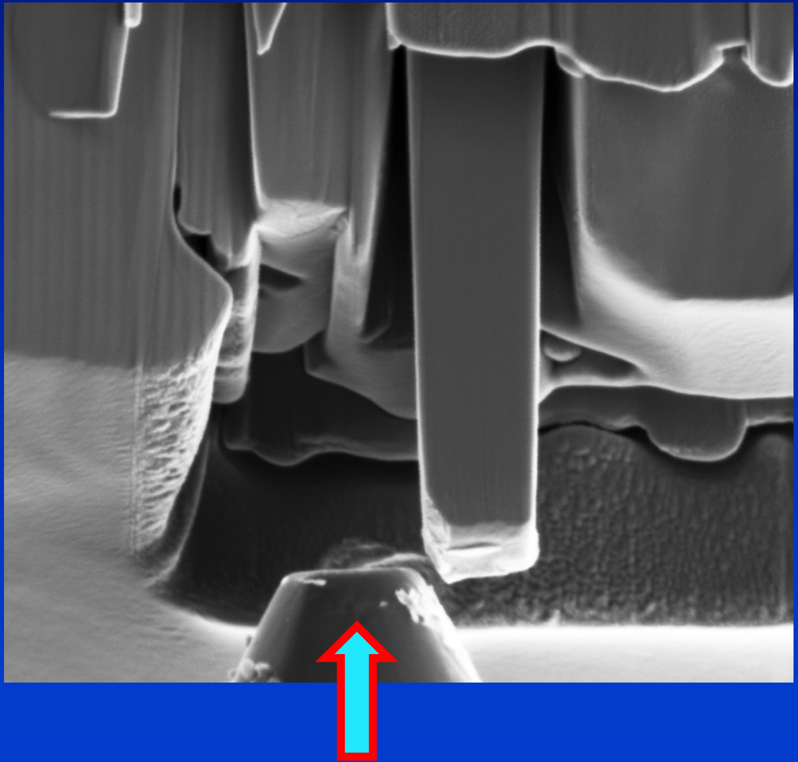


Before the test



Under applied stress

Damping at Nano-scale: In-situ tests



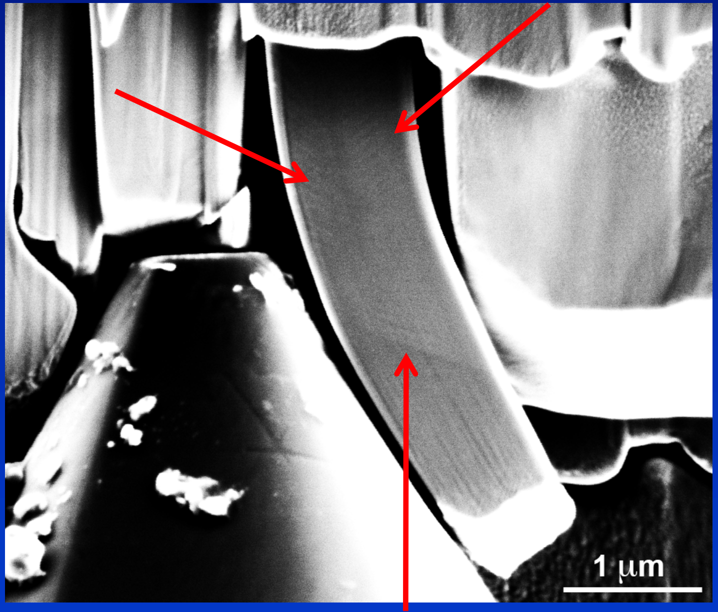
Diamond indenter

Experiment off-axis

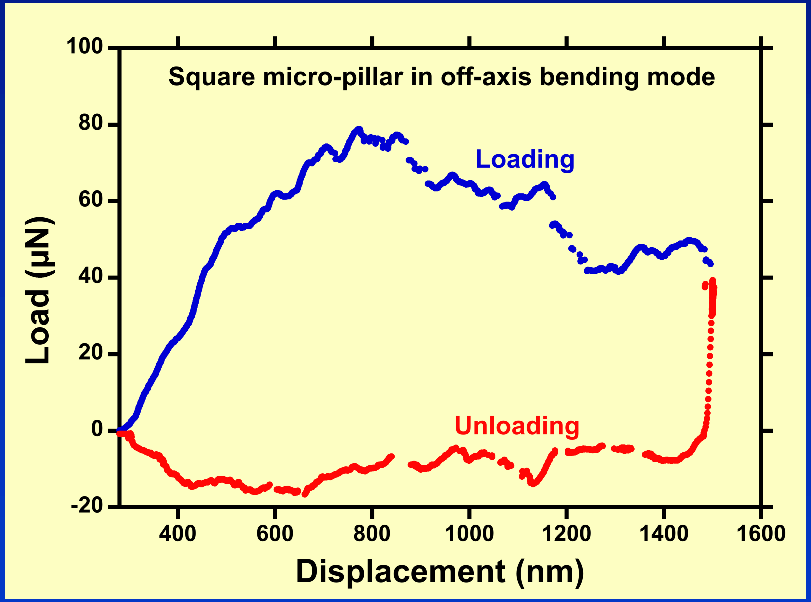
The stress is applied with the lateral side of the diamond indenter

Video of the In-situ test

Damping at Nano-scale: In-situ tests



Stress-induced Martensite during superelastic bending



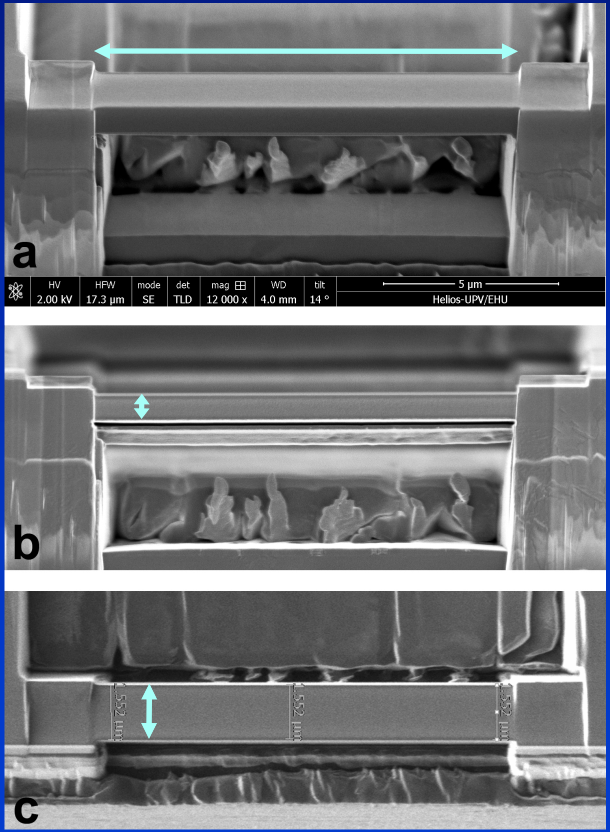
Apparently, the mechanical energy is fully dissipated

Damping at Nano-scale

12 µm long

740 nm thick

1.5 µm width

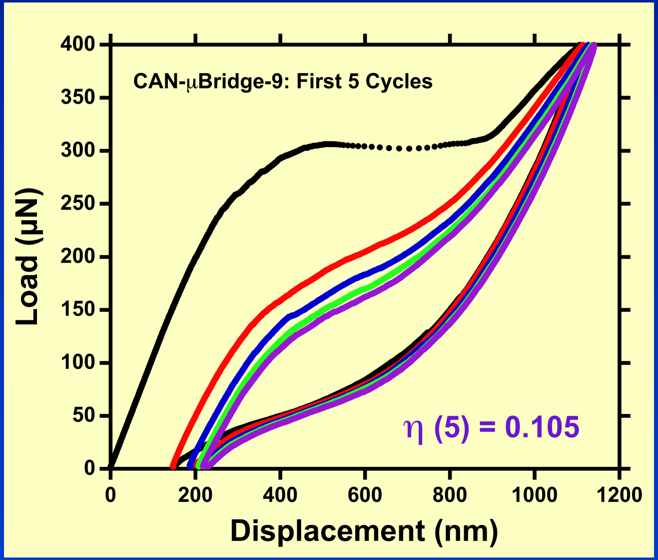


Micro - beam milled by FIB

Cu-Al-Ni

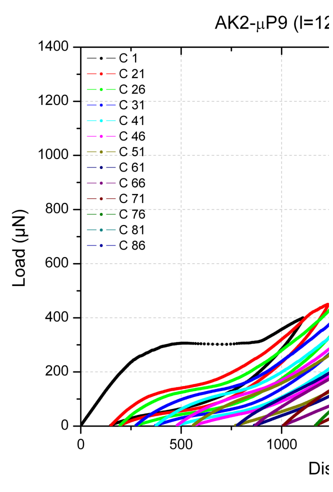
[001] oriented

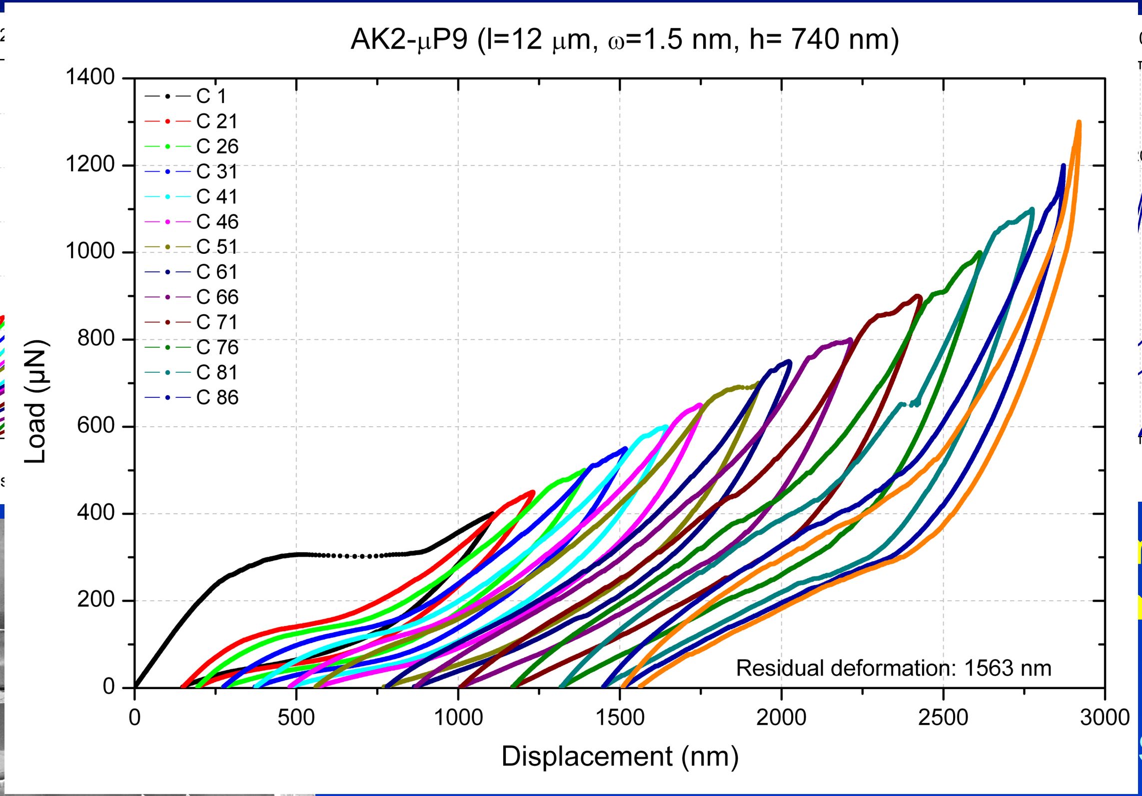
Single crystal

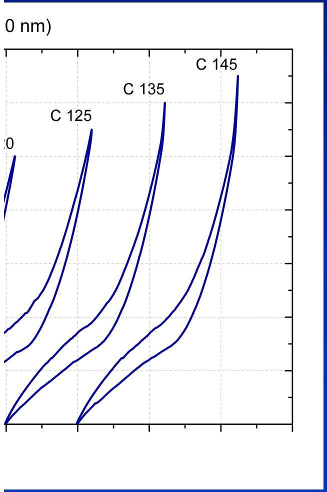


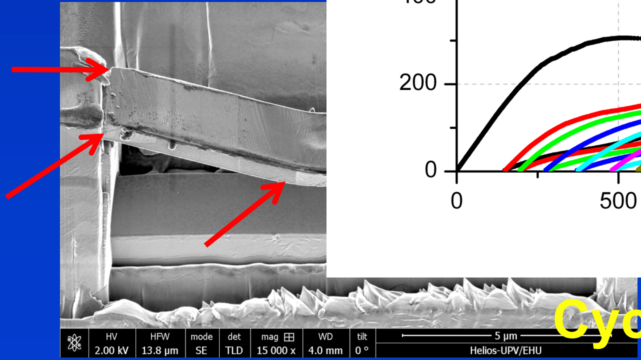
High damping in bending

Damping at Nano-scale









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nes closed

stant strain

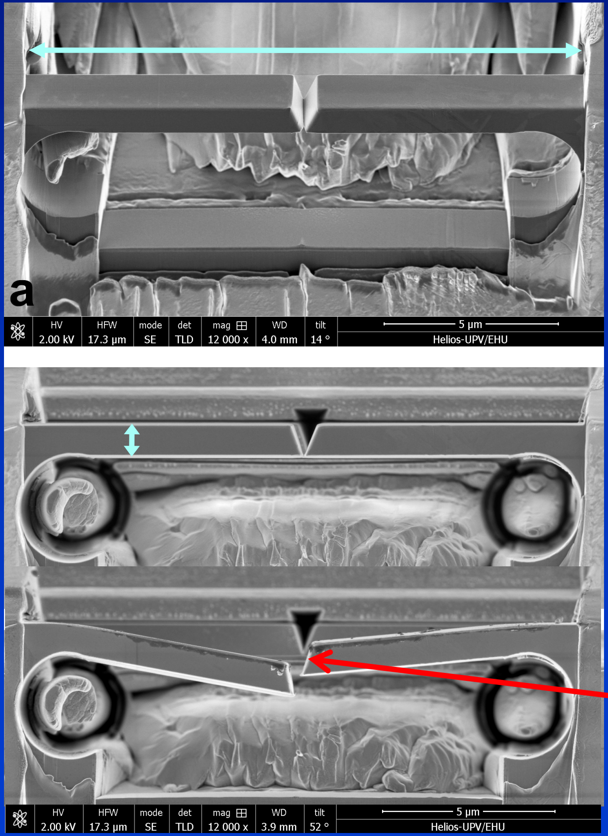
Cycling at increasing load

Damping at Nano-scale

16 μm long

820 nm thick

1.5 μm width

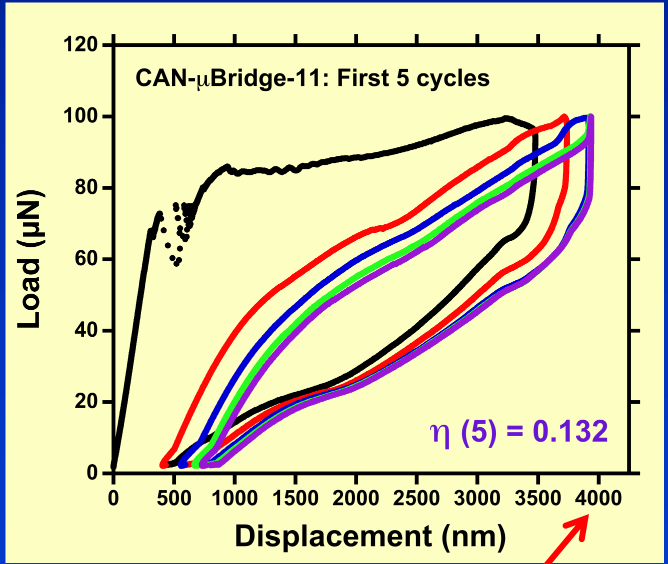


Micro - cantilevers milled by FIB

Cu-Al-Ni

[001] oriented

Single crystal



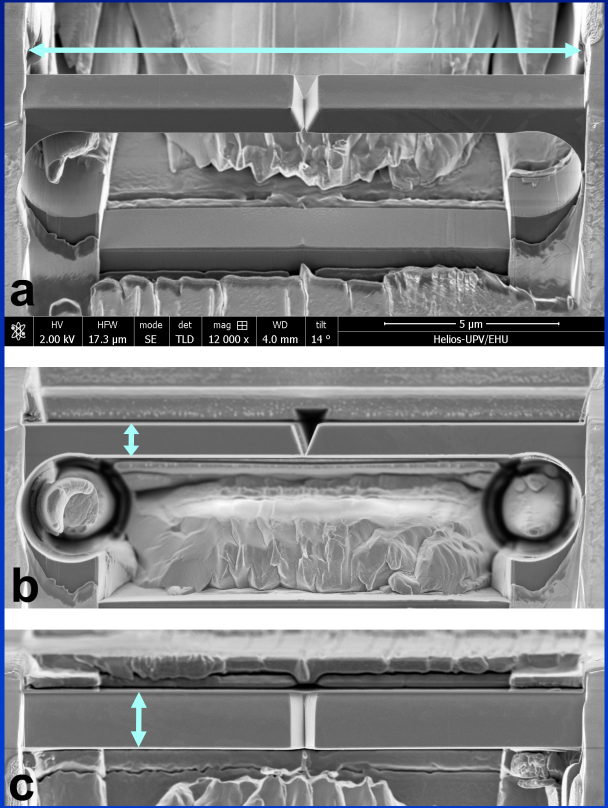
Some plastic deformation for large strains

Damping at Nano-scale

16 μm long

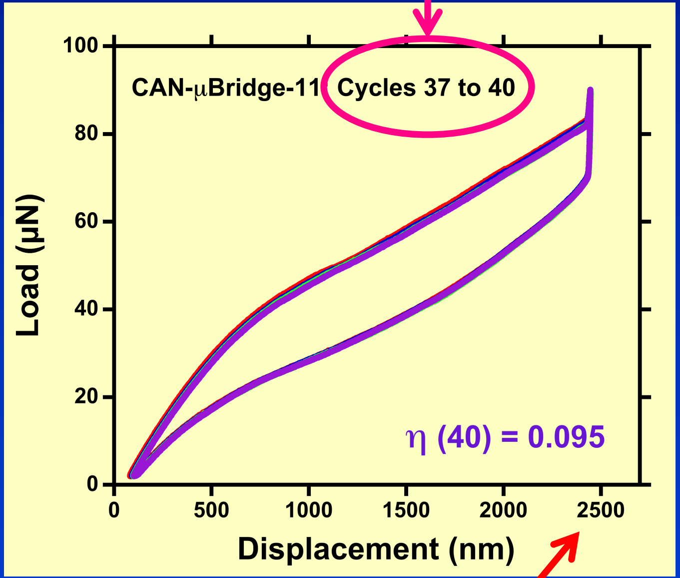
820 nm thick

1.5 μm width



Micro - cantilevers milled by FIB

Good cycling behavior



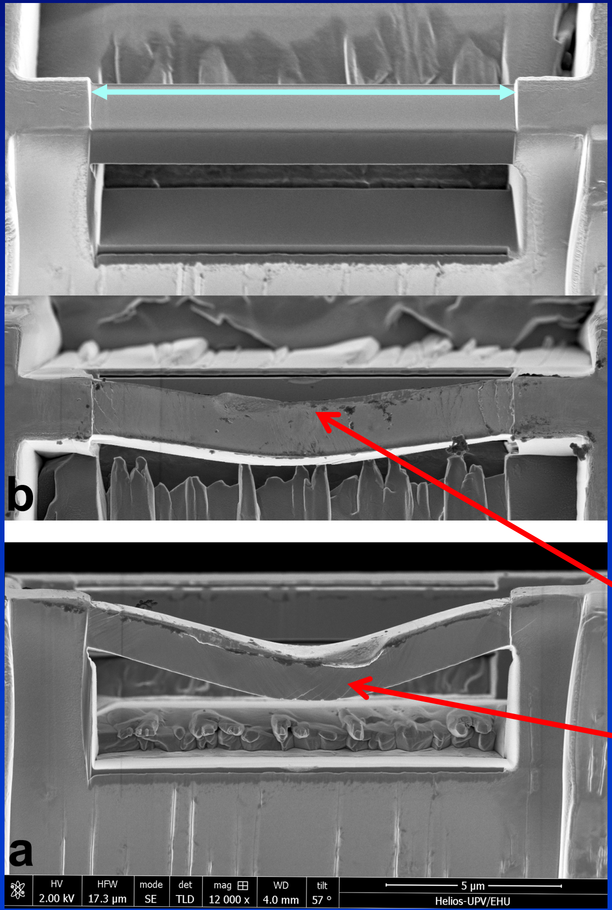
High damping with a large strain

Damping at Nano-scale

12 μm long

1.2 μm thick

1.5 μm width



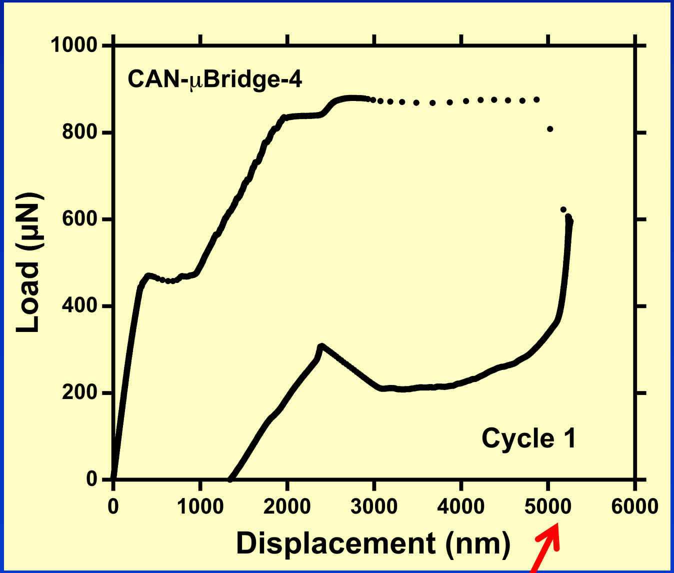
Micro - beam milled by FIB

Tested in bending + torsion

Cu-Al-Ni

[001] oriented

Single crystal



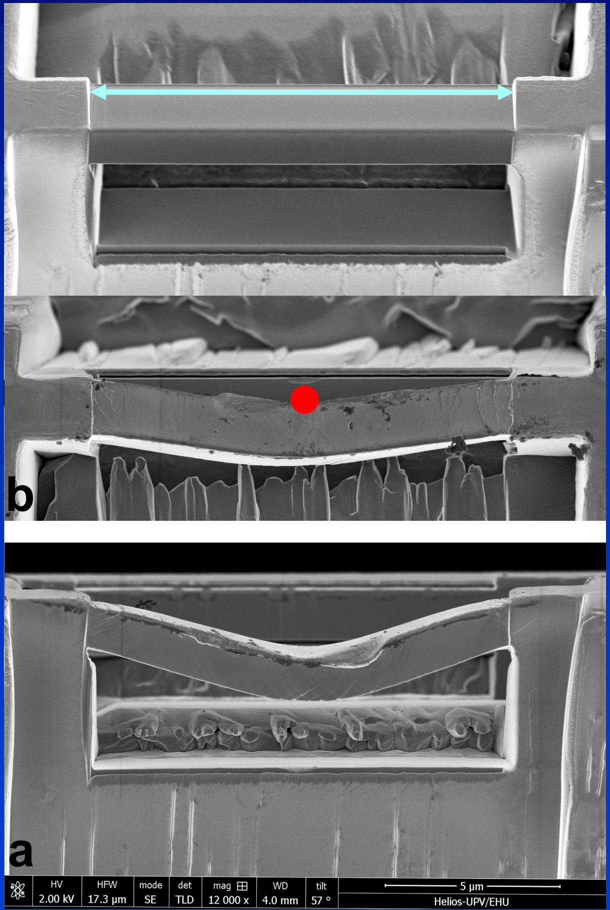
Some plastic deformation for large strains

Damping at Nano-scale

12 μm long

1.2 μm thick

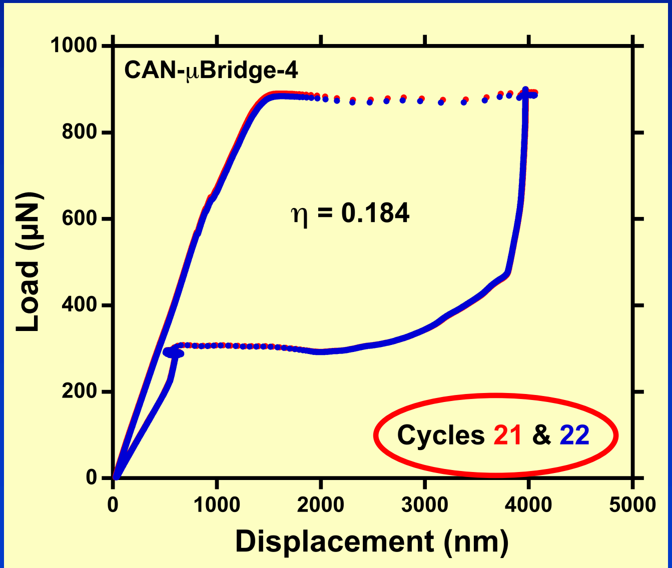
1.5 μm width



Micro - beam milled by FIB

Tested in bending + torsion

Reproducible Bending+torsion cycling



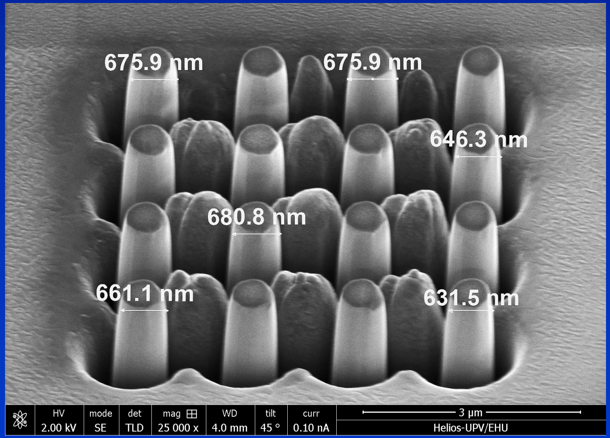
Ultra-high damping at high strains

Then …

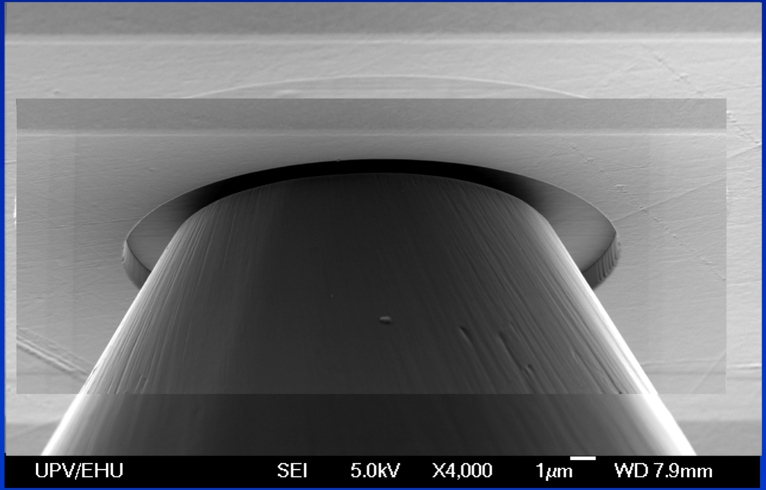
What is the next step ?

Applying Damping at Nano-scale

Is damping scalable for applications ?

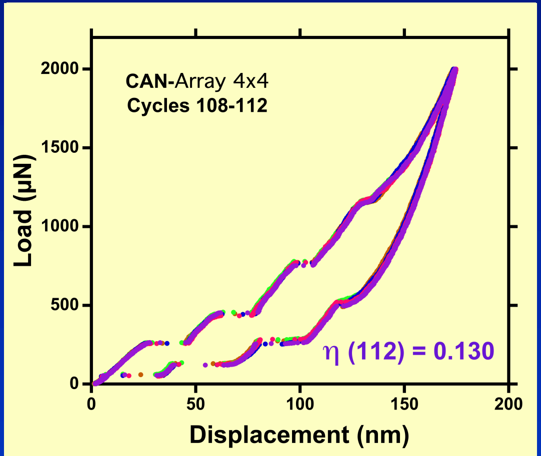


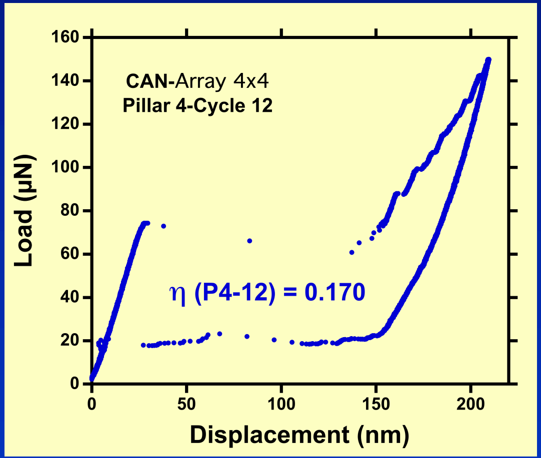
Array of 4 x 4 Cu-Al-Ni pillars of about 660 nm in diameter

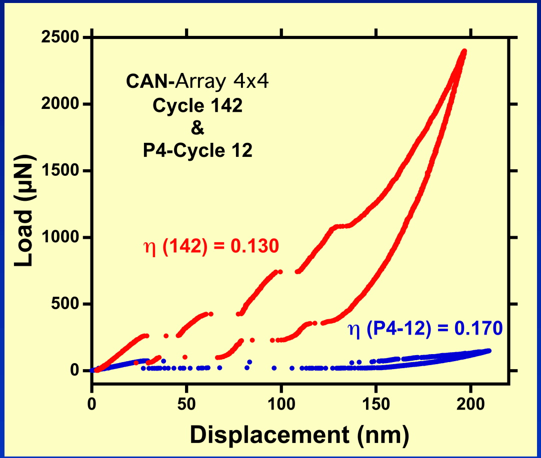


Testing simultaneously all the array

Applying Damping at Nano-scale







Stars on load due to lack of parallelism

Good reproducibility on cycling

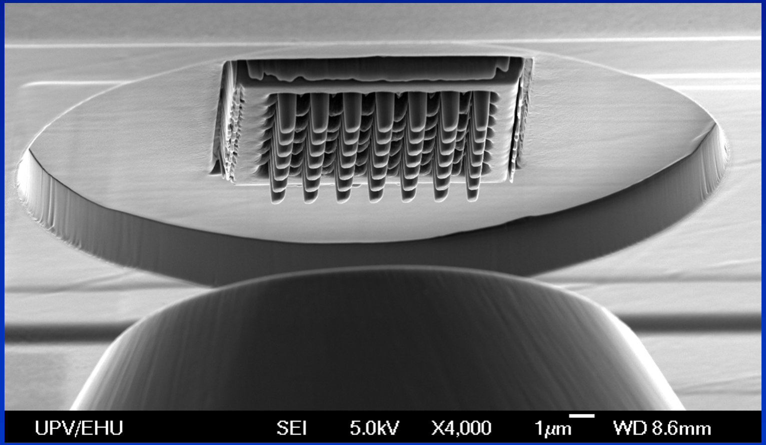
Individual micro pillar

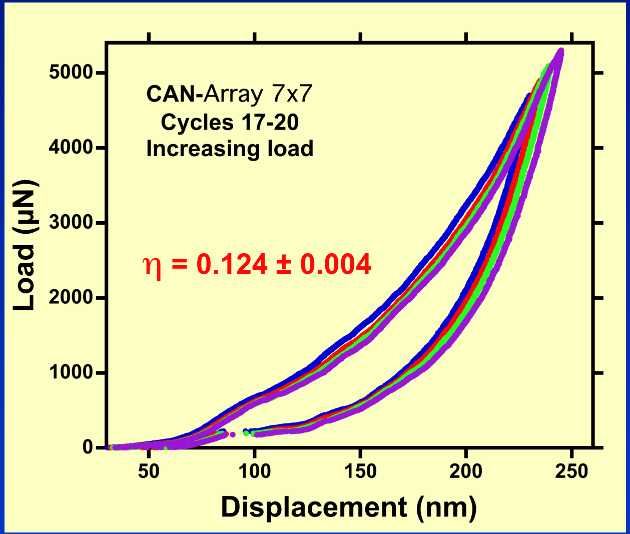
Comparison with the array

Ultra-high damping when increasing the number of pillars and consequently the load

Applying Damping at Nano-scale

Array of 7 x 7 nano pillars

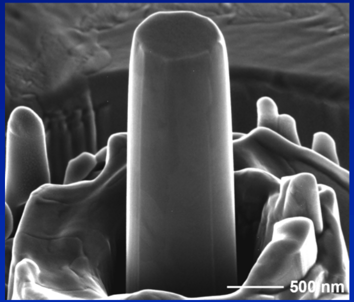


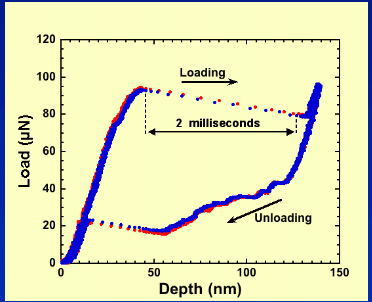


Ultra-high damping is scalable when increasing the number of pillars

Conclusions 1st

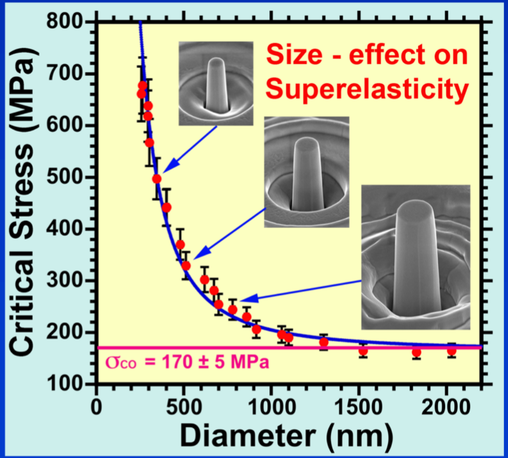
Micro & Nano pillars of Cu-Al-Ni exhibit completely reversible superelastic effect, above 8% even at Nano – Scale.





Superelastic cycling at nano-scale is very fast and perfectly reproducible above.

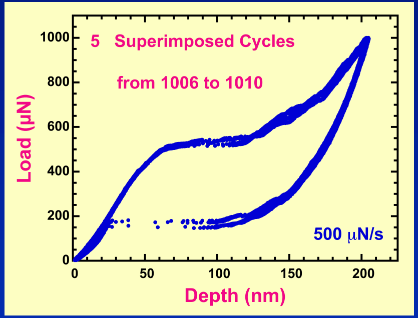
The Size-Effect on the critical stress is present in both Cu-Al-Ni & Cu-Al-Be SMA, with the same scaling-law.



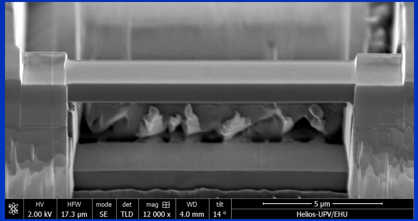
This scaling-law with n = - 2 for the critical stress on superelasticity, seems to be an Universal Scaling-Law for all Cu-based SMA.

Conclusions 2nd

Three different size-effects have been reported, being responsible for Ultra-High Damping at nano-scale, and reproducible above thousands cycles and along years.



Superelastic bending in micro pillars and micro beams of shape memory alloys, also exhibits Ultra-High Damping.



Micro & Nano devices of Cu-Al-Ni SMA exhibit Ultra-High Damping and could offer a novel solution to protect MEMS.

The road is paved for applications of Damping at nano - scale


Thank you
for your attention

Prof. Jose San Juan
jose.sanjuan @ ehu.es

Bilbao
B.S.L.

https://www.researchgate.net/profile/Jose_San_Juan2

Damping at Nano-scale

Damping at Nano-scale