MEMS-based lab-on-chip electro-mechanical testing

A micro- or nano-mechanical on-chip test platform has been developed to deform under a variety of loading conditions freestanding thin films, ribbons and nanowires involving submicron dimensions. The lab-on-chip involves thousands of elementary test structures from which the elastic modulus, strength, strain hardening, fracture, creep properties can be extracted. The technique is amenable to in situ transmission electron microscopy (TEM) investigations to unravel the fundamental underlying deformation and fracture mechanisms that often lead to size-dependent effects in small-scale samples. The method allows addressing electrical, magnetic and thermal couplings as well in order to evaluate the impact of large mechanical stress levels on different solid-state physics phenomena.

This new concept of micro- or nano-mechanical test addresses the problem of measuring the mechanical and coupled properties of sub-micrometer films from a perspective that differs from most of the published techniques in the literature through essentially two aspects [1, 2].
(i) The first idea is to take benefit of one of the greatest advantages of microfabrication technology which is the capacity for easily reproducing large numbers of elementary patterns, and thus multiply elementary testing stages rather than build a complex multipurpose stage. Owing to this concept, thousands of machines can be processed on a single silicon wafer with the potential to replicate suites of tests structures at multiple locations on the wafer;
(ii) The second idea is to use internal stress presents in one material, referred as the “actuator,” to impose the load to another film (specimen) while completely avoiding any external loading and or electrical signal.

These micro- or nano-mechanical on-chip test platforms are produced in the following way starting with a silicon substrate: (i) a sacrificial layer, intended to be etched away, is deposited first (e.g. SiO_2, Cu, polysilicon, etc.); (ii) second, a thin film of a material characterized with a large internal stress in tension (e.g. Si_3N_4, Ni, Pt, etc.) is deposited and patterned by photolithography in the form of a very long beam named the actuator beam; (iii) third, the specimen layer (the material to be tested) is deposited and patterned with a dog bone shape. An overlap between the specimen beam and the actuator beam ensures a sufficiently wide contact zone between the two beams. The final geometry of the elementary test configuration is shown in Figure 1. Small cursors are also introduced in the overlap region.
When the sacrificial layer is removed, the internal stress in the actuator relaxes leading to the contraction of the actuator and to the deformation of the specimen beam until force equilibrium is attained, see Figure 2a. The displacement, after reaching the equilibrium position, of the overlap between the specimen beam and the actuator is measured at high magnification using a Field Emission Gun (FEG) Scanning Electron Microscope (SEM). The deformation (ε) and the applied stress (σ) of the specimen can be extracted based on the measured displacement (u) and mismatch strain in the actuator prior to release (ε_a^{mis}) with the following relationship:

$$\begin{cases} \varepsilon = \dfrac{u}{L} \\ \sigma = \dfrac{S_a}{S} E_a\left(-\dfrac{u}{L_a} - \varepsilon_a^{mis}\right) \end{cases}$$

where L and S are the specimen beam length and cross-section area, respectively, L_a and S_a the actuator beam length and cross-section area, respectively, E_a the actuator Young’s modulus.
In other words, the actuator acts also as a stress sensor. As a consequence, one structure gives one point in the specimen stress-strain response. The longer is the actuator beam, the larger is the deformation imposed to the specimen. Hence, by designing several test structures with various actuator lengths, several equilibrium points can be obtained and therefore several points of the stress-strain curve, see Figure 2b. Hundreds to thousands of tests are performed at the same time “on a chip”.

This characterization method has been successfully applied to a variety of materials over the last decade by our research group involving among others Si nanowires [3], polysilicon [2], Al, Pd, Cu, metallic glass (ZrNi) [4], SiN [5], SiO_2 films as well as graphene. The strain-stress curve up to fracture of the materials has been extracted but also the piezoresistance behavior of ultra-thin silicon [6], the relaxation/creep response of Al [7] and Pd [8] by simply measuring the evolution of displacement with time after release, as well as the impact of the environmental conditions (temperature, moisture, gas, irradiation, etc.) on the material mechanical properties [9].

Moreover, the loading configuration and geometry of the actuator can be varied in order to impose crack opening (Fig. 3a) [10], shear (Fig. 3b), equibiaxial, or multiaxial type loadings.

References

[1] D. Fabrègue, M. Coulombier, N. André, J.-P. Raskin and T. Pardoen, "Multipurpose nanomechanical testing machines revealing the size-dependent strength and high ductility of pure aluminium submicron films", *Micro & Nano Letters,* vol. 2, no. 1, pp. 13-16, March 2007, DOI: 10.1049/mnl:20065068.

[2] S. Gravier, M. Coulombier, A. Safi, N. André, J.-P. Raskin and T. Pardoen, "New on-chip nanomechanical testing laboratory - Applications to aluminum and polysilicon thin films", *IEEE Journal of MicroElectroMechanical Systems,* vol. 18, no. 3, pp. 555-569, March 2009, doi: 10.1109/JMEMS.2009.2020380.

[3] V. Passi, U. K. Bhaskar, T. Pardoen, U. Sodervall, B. Nilsson, G. Petersson, M. Hagberg, J.-P. Raskin, "High throughput on-chip large-strained Si nanowires", *IEEE Journal of ElectroMechanical Systems*, vol. 21, no. 4, pp. 822-829, August 2012, doi: 10.1109/JMEMS.2012.2190711.

[4] M. Ghidelli, S. Gravier, J.-J. Blandin, H. Idrissi, N. Schryvers, J.-P. Raskin, T. Pardoen, "Homogeneous flow and size dependent mechanical behaviour in highly ductile $Zr_{65}Ni_{35}$ metallic glass film", *Acta Materialia*, vol. 131, pp. 246-259, 2017, doi.org/10.1016/j.actamat.2017.03.072.

[5] A. Boé, A. Safi, M. Coulombier, T. Pardoen and J.-P. Raskin**,** "Internal stress relaxation based method for elastic stiffness characterization of very thin films", *Solid Thin Films*, vol. 518, pp. 260–264, 2009, doi.org/10.1016/j.tsf.2009.06.062.

[6] U. K. Bhaskar, T. Pardoen, V. Passi, J.-P. Raskin, "Piezoresistance of nano-scale silicon beams under tension up to 2 GPa", *Applied Physics Letters*, 102, 031911, 2013, DOI: 10.1063/1.4788919.

[7] M. Coulombier, A. Boé, C. Brugger, J.-P. Raskin and T. Pardoen, "Imperfection sensitive ductility of aluminium thin films", *Scripta Materialia*, vol. 62, pp. 742–745, 2010, doi.org/10.1016/j.scriptamat.2010.01.048.

[8] M. S. Colla, H. Idrissi, B. Amin-Ahmadi, L. Malet, S. Godet, J.-P. Raskin, D. Schryvers, T. Pardoen, "Dislocation mediated hardening and creep in nanocrystalline palladium thin films revealed by on-chip HRTEM in-situ testing", *Nature Communications*, 5 January 2015, doi: 10.1038/ncomms6922.

[9] P. Lapouge, F. Onimus, R. Vayrette, J.-P. Raskin, T. Pardoen and Y. Bréchet, "A novel on chip test method to characterize the creep behavior of metallic layers under heavy ion irradiation", *Journal of Nuclear Materials*, vol. 476, pp. 20-29, 2016, doi.org/10.1016/j.jnucmat.2016.04.014.

[10] S. Jaddi, M. Coulombier, J.-P. Raskin, T. Pardoen, "Crack on a chip test method for thin freestanding films", (**invited article**), *Journal of the Mechanics and Physics of Solids*, vol. 123, pp. 267-291, February 2019, https://doi.org/10.1016/j.jmps.2018.10.005.

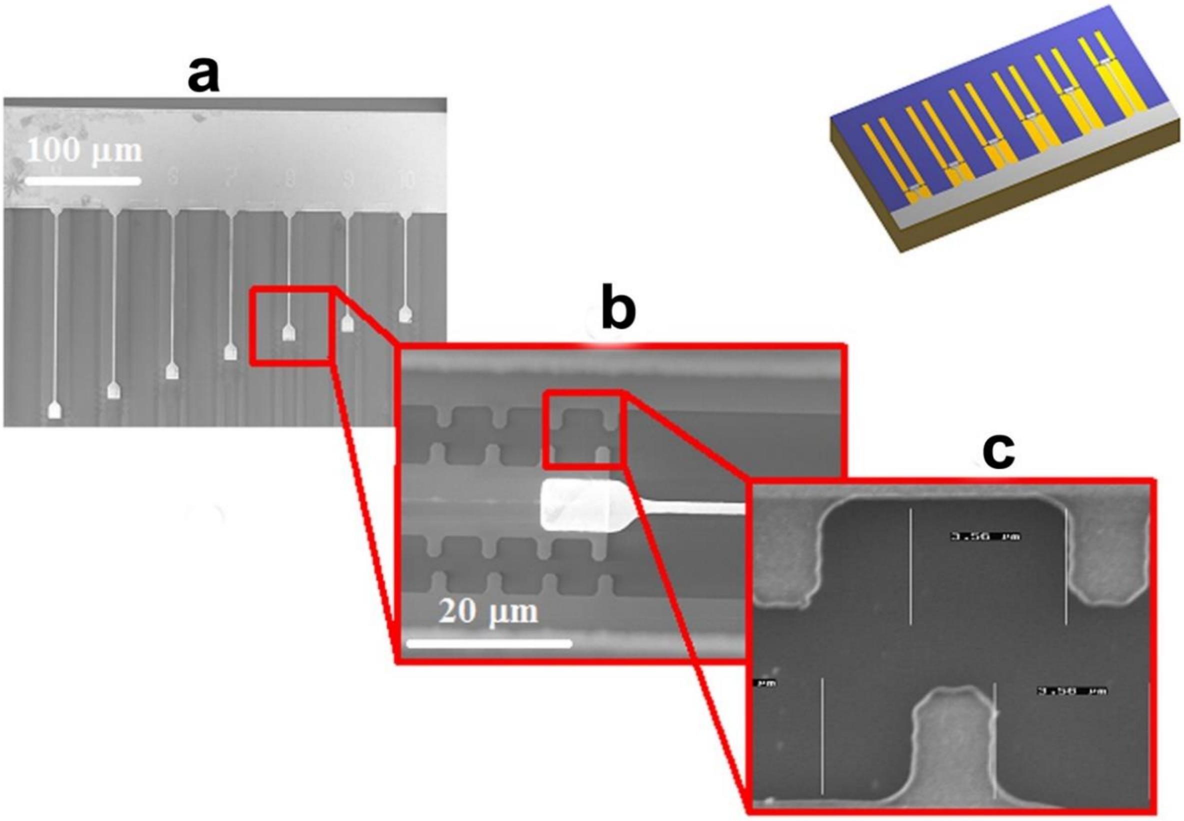
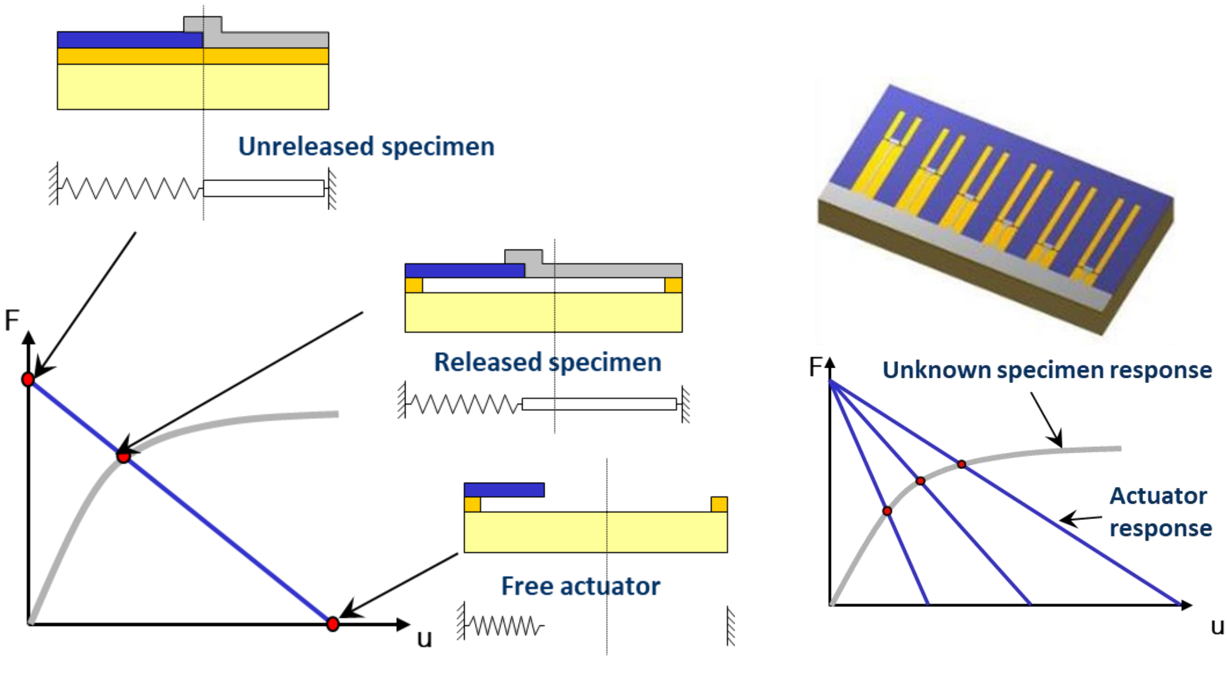
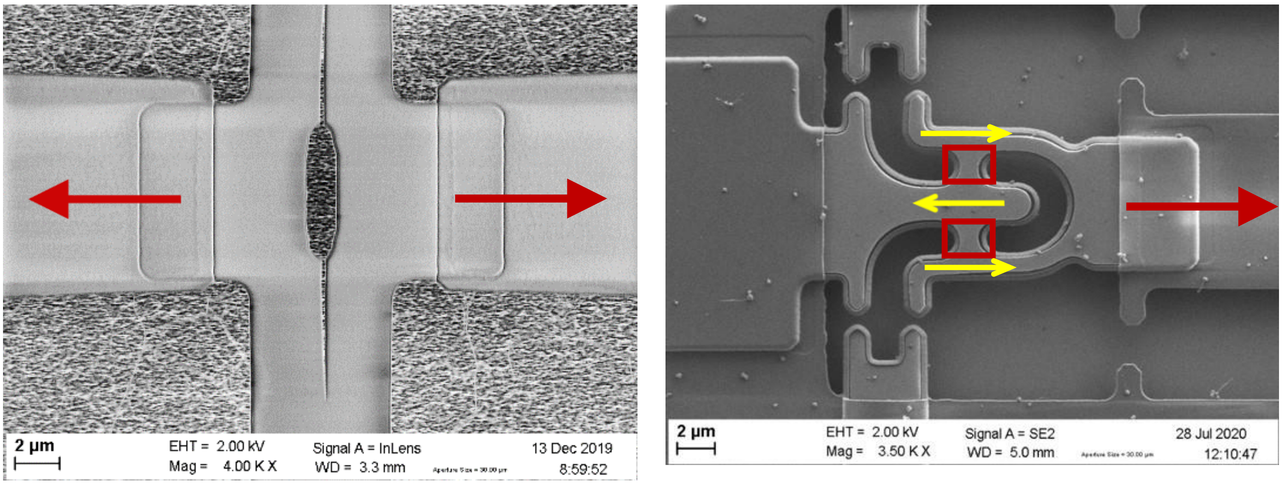


Figure 1. Implementation of the internal stress-based test structures; (a) set of tensile test structures with various actuator and specimen lengths used to generate a full stress-strain curve; (b)-(c) zoom on the cursors used to measure the displacement applied to the test specimen.



(a) (b)

Figure 2. (a) Configurations that actuator-specimen beams deposited on top of a sacrificial layer can take: (i) actuator-specimen beams are not yet released, (ii) actuator-specimen beams are released from the substrate and thus free to move and reach an equilibrium, (iii) actuator beam is released and free to fully contract. (b) Series of actuator-specimen structures of various dimensions to build up the complete stress-strain curve of the specimen knowing the mechanical properties of the actuator beam.



(a) (b)

Figure 3. (a) Crack arrest test in 140 nm-thick SiO_2 film, (b) shear test in 200 nm-thick Si film.

Involved researchers:

- Prof. Thomas Pardoen
- Prof. Hosni Idrissi
- Dr. Paul Baral
- Dr. Marie-Stéphane Colla
- Dr. Michaël Coulombier
- Dr. Audrey Favache
- Mr. Alex Pip
- Mrs. Farzaneh Bahrami
- Mr. Luis Angel Velosa Moncada (Universidad Veracruzana, Mexico)
- Mr. Mohammad Wasil Malik
- Mrs. Nargisse Khiara (CEA-Saclay and INP-G)
- Mr. Nicolas Roisin
- Mrs. Sahar Jaddi