Design and Validation of a Testing Setup to Measure Tensile Properties of Materials at the Microscale

Daniele Casari[a], Laszlo Pethö[a], Patrik Schürch[a], Laetitia Philippe[a], Johann Michler[a], Philippe Zysset[b], Jakob Schwiedrzik[a]

[a] Empa, Swiss Federal Laboratories for Materials Science and Technology, Thun, Switzerland
[b] Institute for Surgical Technology and Biomechanics, University of Bern, Bern, Switzerland

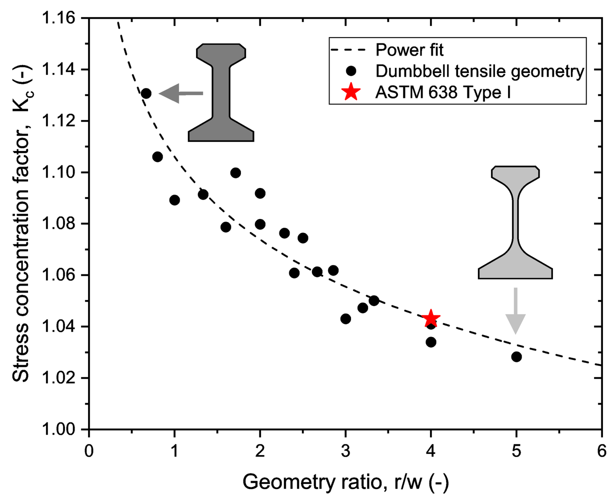
Abstract: Focused ion beam (FIB) technology was employed to to produce microtensile specimens through a time-saving manufacturing protocol while minimizing FIB artefacts. The sample geometry was optimized via finite element (FE) to mimic ASTM 638 tensile specimens stress conditions. A combination of reactive ion etching and FIB milling was used to manufacture self-aligning grippers which are able to ensure less than 5% error for in-plane misalignments up to 2° and up to 1/3 of the sample width. Single crystal Si and GaAs specimens were tested to validate the tensile setup.

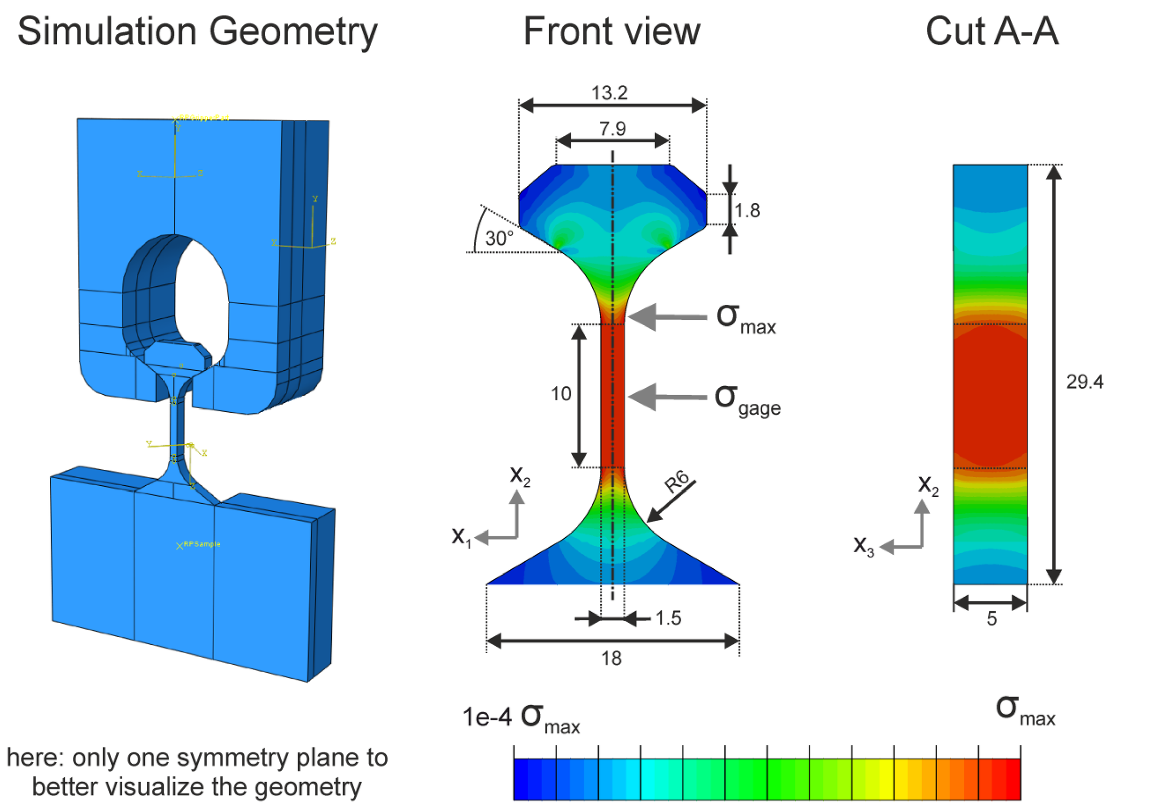
1. Sample Geometry

Self-standing sample geometry with stresses profile comparable to the one found in the ASTM 638 standard geometries: the FE optimized sample geometry ensures uniaxial and homogeneous stress in the gage section while minimizing stress concentrations.

Geometry Factor:

$$K_c = \frac{\sigma_{max}}{\sigma_{gage}}$$





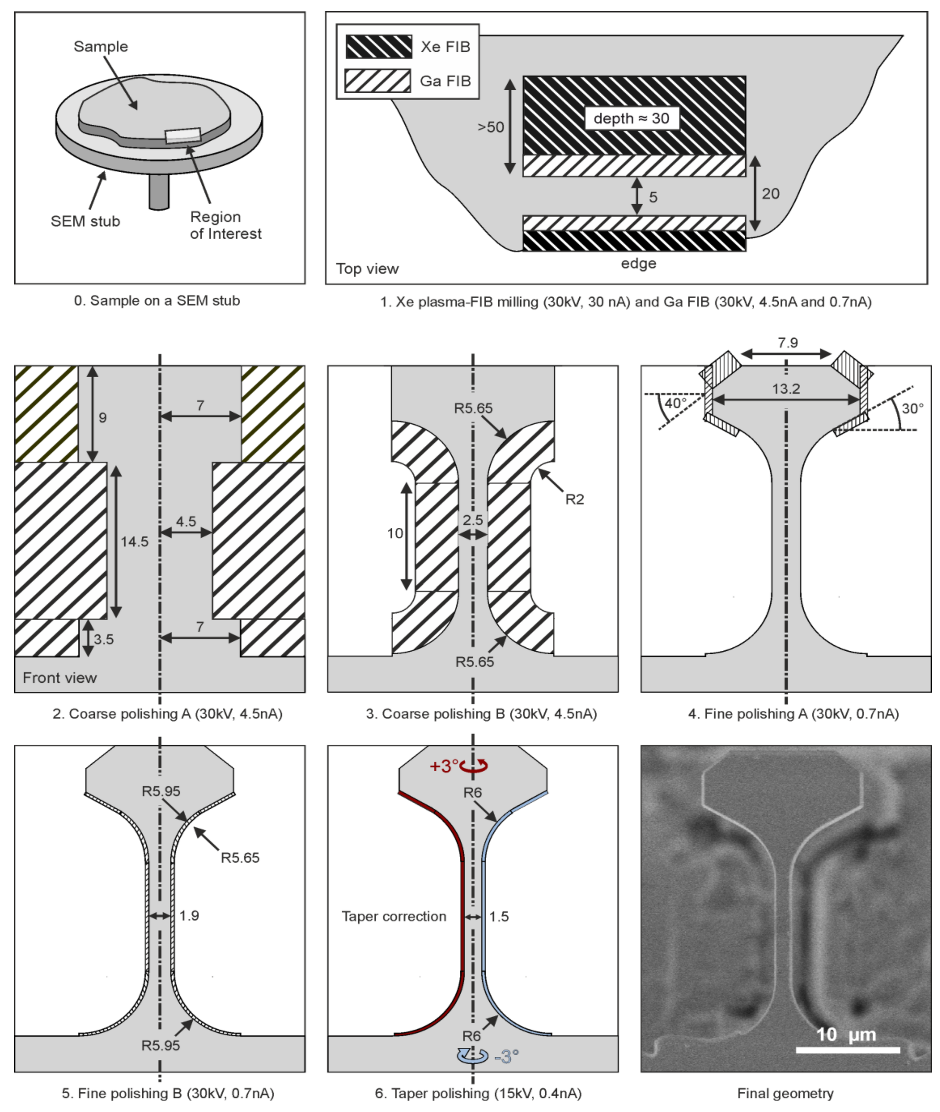
- Attached to its original substrate to facilitate handling and avoid pre-test damage.
- Stress deviations <5% with ratio junction radius/width sample ≥4.
- Size is in the order of few µm.
- Sample head designed to allow gripper guiding during self-alignment.

2. FIB-SEM Sample Fabrication Protocol

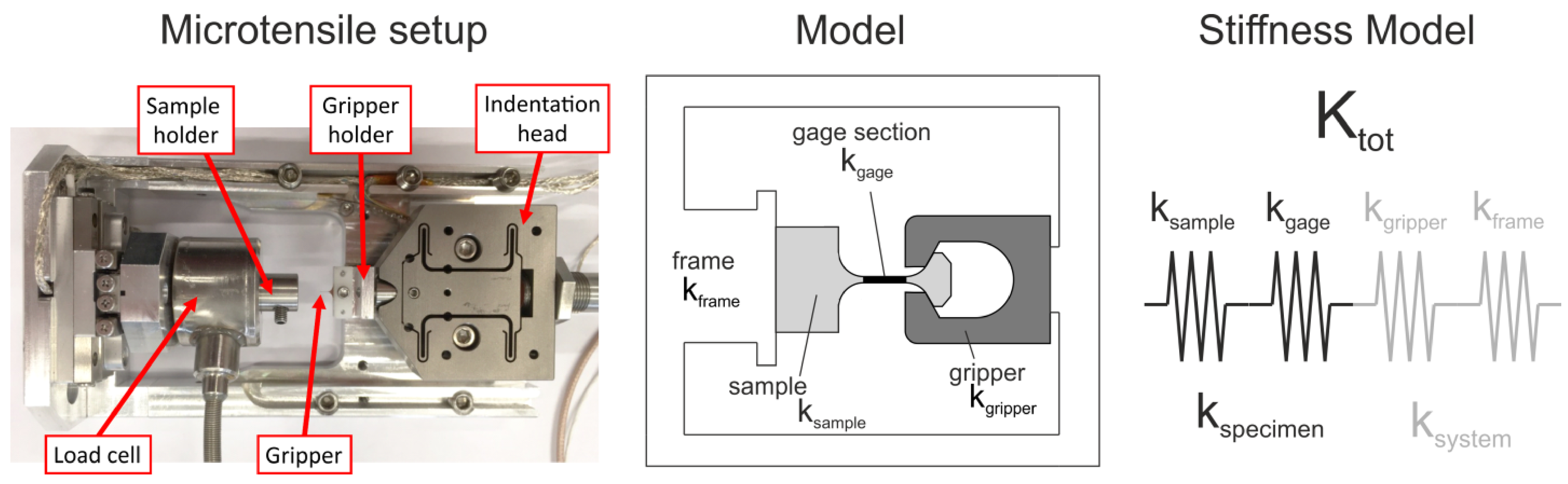
FIB milling allow the fabrication of microsized specimens of any material stable in vacuum and under the influence of electron beam. Nevertheless this method produces artefacts that could affect the mechanical test if not controlled.

Minimizing FIB Induced Artefacts:

- Curtaining is reduced by depositing a Pt layer cap (1-2 µm) on the sample head prior FIB milling.
- Damage layer is reduced by decreasing voltage and beam current. Estimated damage layer of less than 25 nm.
- Re-deposition minimized by parallel milling of the specimens.
- Taper to tolerances of <0.5° by overtilting the sample between 1 to 4° in the last step, depending on the material milled and the beam settings.



3. Microtensile Setup



Stress strain data can be obtained with a correction for the system compliance. The compliance of the tested specimen is found by knowing the compliance of the system (which is fixed and can be found with calibration samples). With the help of simulations two geometry factor describing the variation of stiffness in function of specimen width and thickness are defined:

$$f_{gage}(\Delta w, \Delta t) = \frac{k_{gage}}{k^0_{gage}} = \left(1 + \frac{\Delta w}{w_0}\right)\left(1 + \frac{\Delta t}{t_0}\right) \qquad f_{specimen}(\Delta w, \Delta t) = \frac{k_{specimen}}{k^0_{specimen}} = \left(1 + \frac{\Delta w}{w_0}\right)\left(1 + \frac{\Delta t}{t_0}\right)^{0.783}$$

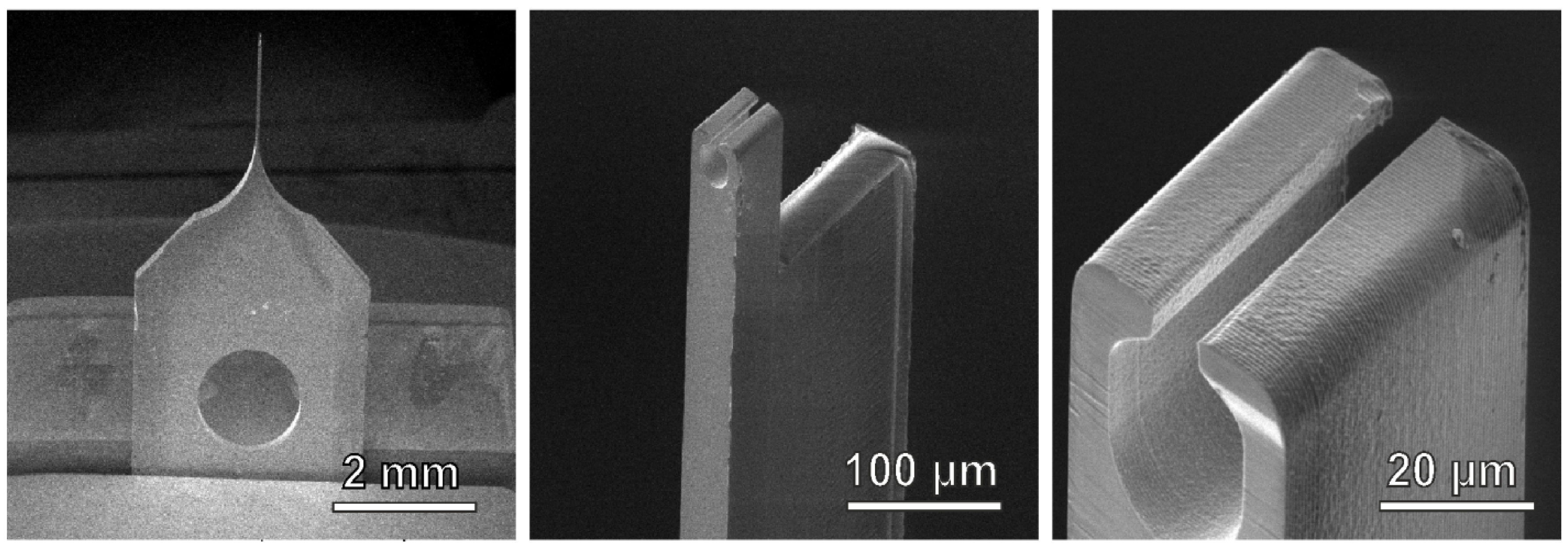
Hence, even when FIB milling does not produce the exact specimen shape defined above, one can still extrapolate both modulus and displacement in the gage section:

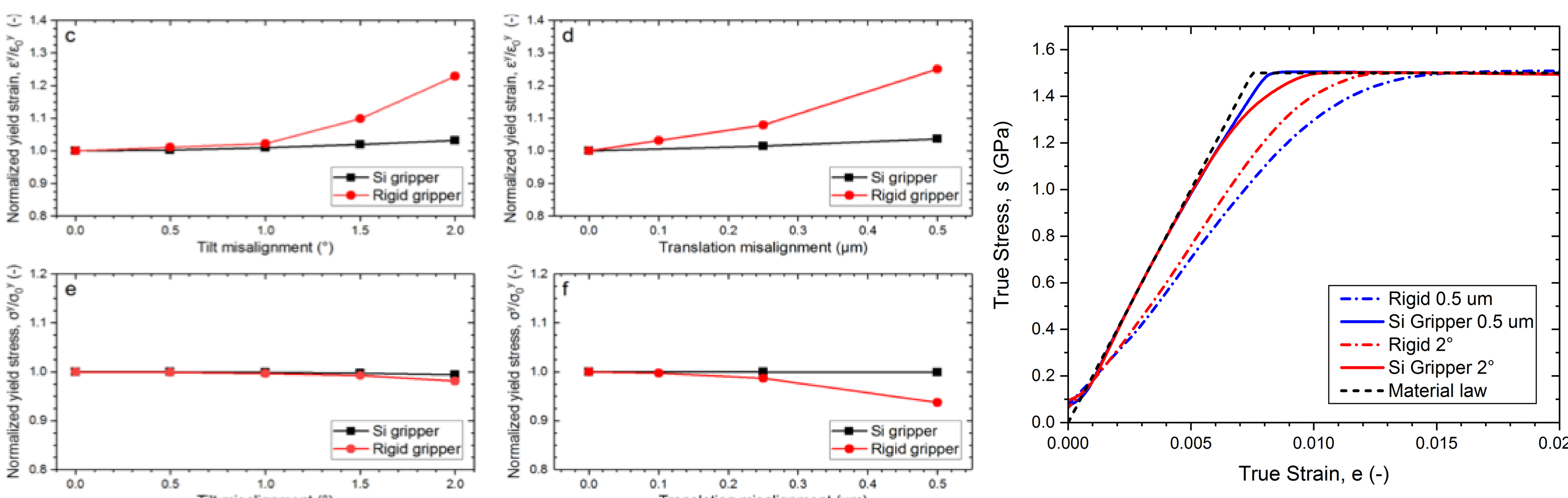
$$E = \frac{f_{specimen}}{0.398}\left(\frac{1}{k_{tot}} - \frac{1}{k_{system}}\right)^{-1} \qquad d_{gage} = d_{tot} - F\left(\frac{1}{k_{tot}} - \frac{1}{k_{gage}}\right) = d_{tot} - F\left(\frac{1}{k_{tot}} - \frac{1}{0.746\, f_{gage}\, E}\right)$$

Error Propagation (setup accuracy):

Assuming an error of 100 nm for all the geometric dimensions necessary to calculate the stress strain data, a peak-peak noise of ΔF =10 µN for the load cell and a misalignment 0.5 µm, this study revealed an uncertainty in the measured elastic modulus of only 7.2%.

4. Silicon Grippers



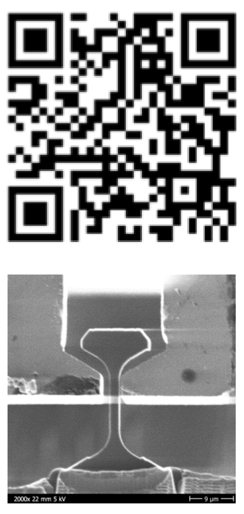


Laterally compliant silicon (Si) grippers are fabricated by deep reactive ion etching (Bosch etch). This process allows the manufacturing of 96 gripper of 200 µm thickness in one single Si wafer. The grippers have high reproducibility and the process allow resolution up to 0.5 µm. To ease the manoeuvring of the tensile gripper on the sample, a part of the gripper end can be removed by Xe plasma-FIB milling. FE analysis showed that by using laterally compliant grippers instead of a rigid gripper is advantageous in terms of measurement accuracy of both stress and strain.

5. Setup Validation

Si

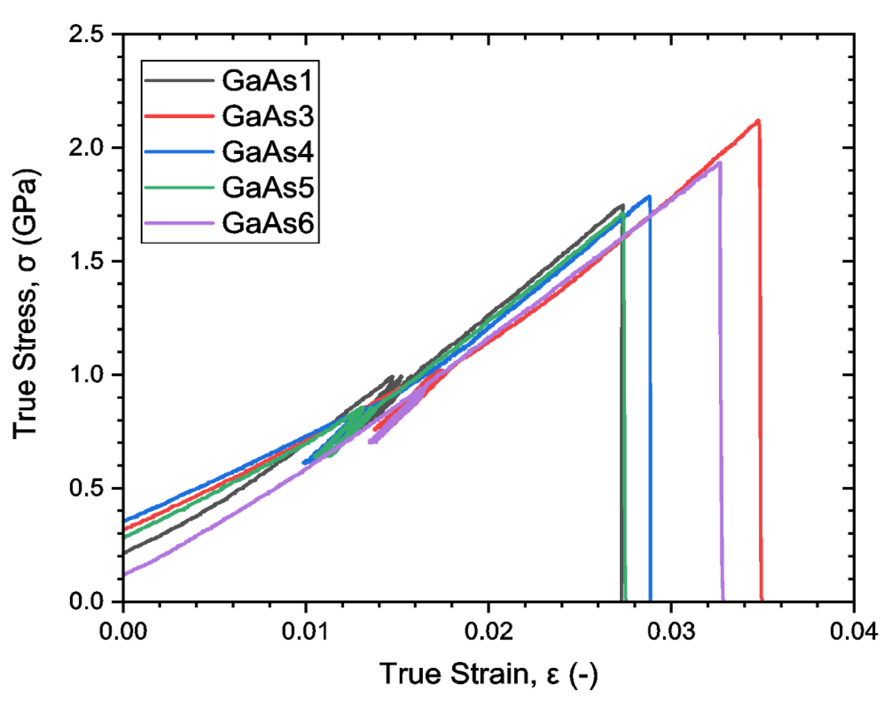
Video



(Self-alignment)

GaAs

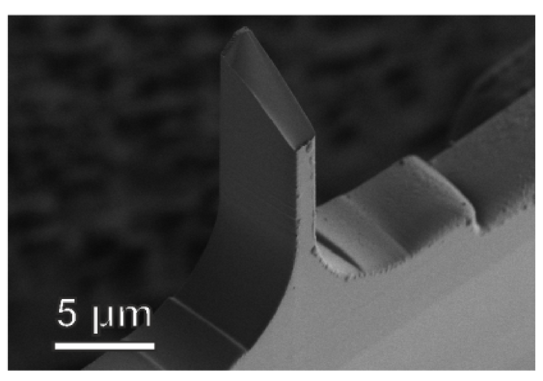
Stress strain data



Accuracy setup:

Measured modulus: 81.4 ± 4.6 GPa
Theoretical modulus: 85.5 GPa

Sample Geometry:



Gage section failure in more than 90% of the samples.

6. Conclusions

- The versatility of FIB manufacturing techniques is highlighted as it allows applications such as the study of micromechanical properties of materials.
- The testing setup was design to be insensitive to in plane misalignments and it might be used to test all class of material that can be milled by FIB methods.
- Sample geometry was optimized through FE analysis to avoid stress concentrations while a self-aligning tensile setup was developed using a compliant gripper.
- The methodology was demonstrated experimentally on single crystal GaAs and Si specimens using an Alemnis nanoindenter.

Empa Materials Science and Technology
u b UNIVERSITÄT BERN
alemnis Mechanical testing at the micro- and nano-scale
ScopeM

FNSNF
FONDS NATIONAL SUISSE
SCHWEIZERISCHER NATIONALFONDS
FONDO NAZIONALE SVIZZERO
SWISS NATIONAL SCIENCE FOUNDATION
Grants: # 165510 # 174192

Corresponding author:
Daniele Casari
Swiss Federal Laboratories for Materials Science and Technology
Feuerwerkerstrasse 39
3602 Thun
Switzerland
daniele.casari@empa.ch
+41 58 765 62 31