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# Deformation Mechanisms of Geological Materials at the Nanoscale

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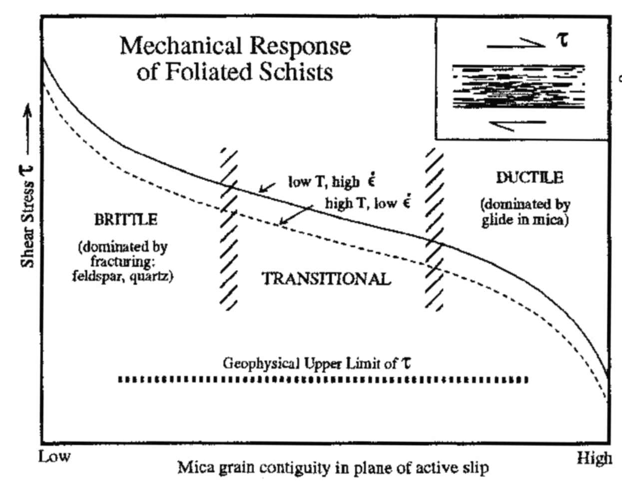
## Predicting Materials Behavior: Observations Across Length Scales

- A fundamental understanding of the deformation mechanisms of geological materials is critical when considering designs that require predictive mechanical behavior of geological materials
  - ex. Geological storage of $CO_2$, nuclear waste storage, geothermal heat pumps, and hydraulic fracturing
- Mica minerals
  - are abundant, comprising almost 15% of the upper continental crust[1]
  - have a layered sheet-like structure
  - shear along the basal plane with relative ease
  - greatly influence the mechanical properties of its host rock[2]
- Ex-situ[3] and qualitative in-situ[4] Transmission Electron Microscopy (TEM) straining experiments of mica have so far observed
  - strength is a function of shear stress and loading direction
  - dislocation glide is confined to the basal plane
  - Orowan dislocation bowing mechanism

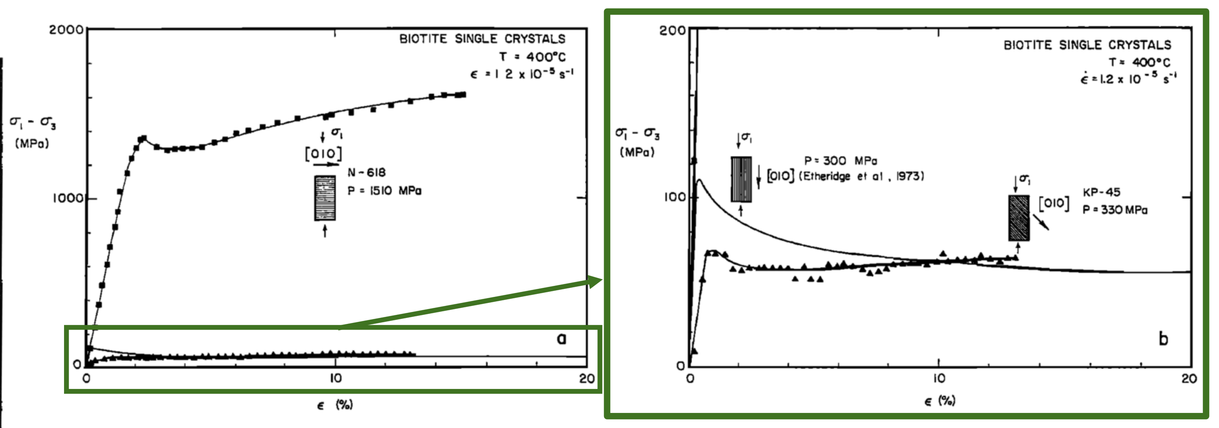
By quantitatively investigating deformation and fracture in mica minerals at the nanoscale, this research aims to generate a fundamental understanding of geological mechanical behavior by

- establishing deformation mechanisms in mica as a function of shear stress and loading direction
- quantitatively measuring activation and interaction energies of participating defects in mica

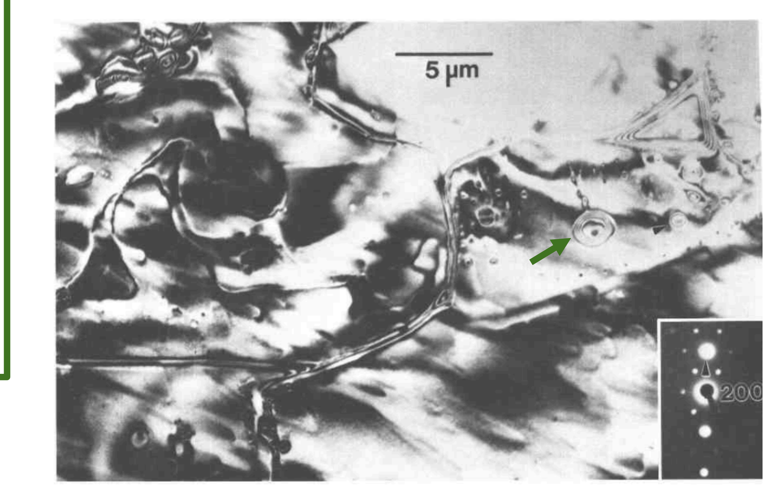
Constituent materials control the overall strength of rock[2]




Strength of mica is function of shear stress and loading direction[3]




Dislocation glide confined to mica basal plane, Orowan mechanism[4]

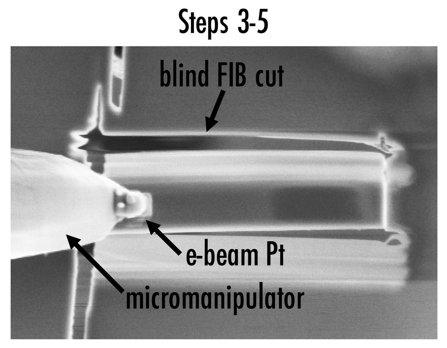




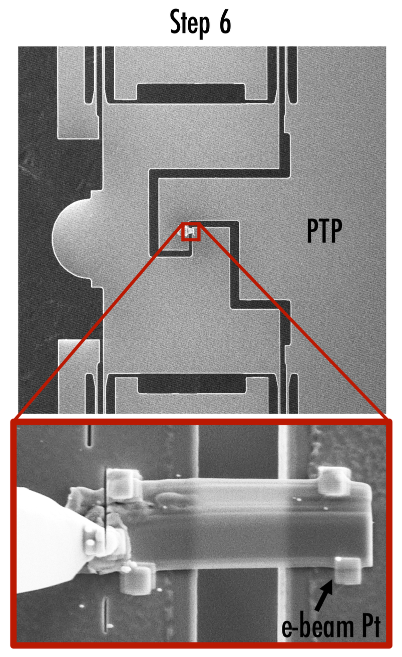
## Nanoscale Sample Preparation

1. Exfoliated or ultramicrotomed biotite mica sheet (obtained from Ward's Natural Science Establishment, Bancroft, Ontario, Canada) is floated in DI $H_2O$ onto Cu TEM grid
2. Crystallographic orientation determined using TEM diffraction
3. In a dual-beam SEM/FIB, 3 blind focused ion beam (FIB) cuts made with $Ga^+$ at 30 keV (blind = without imaging with the ion-beam)
4. Free end of sample affixed to micromanipulator with e-beam deposited Pt
5. Blind FIB cut made to free sample from sheet
6. Transferred and affixed to Push-To-Pull (PTP) device with e-beam Pt
7. Blind FIB cut made to free sample from micromanipulator
8. Blind FIB cuts made to shape tensile sample

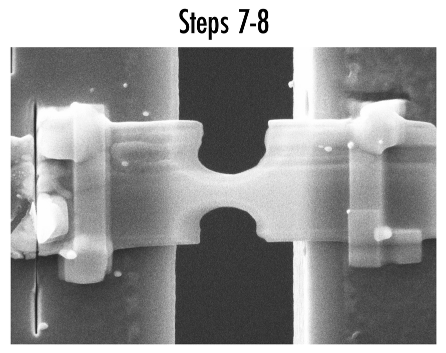
Steps 3-5




Step 6

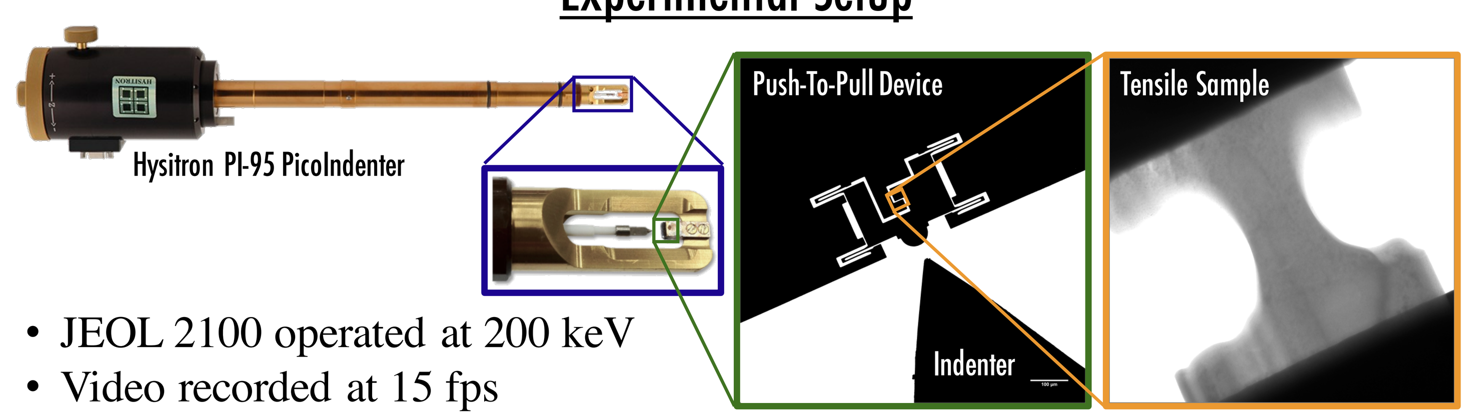



Steps 7-8



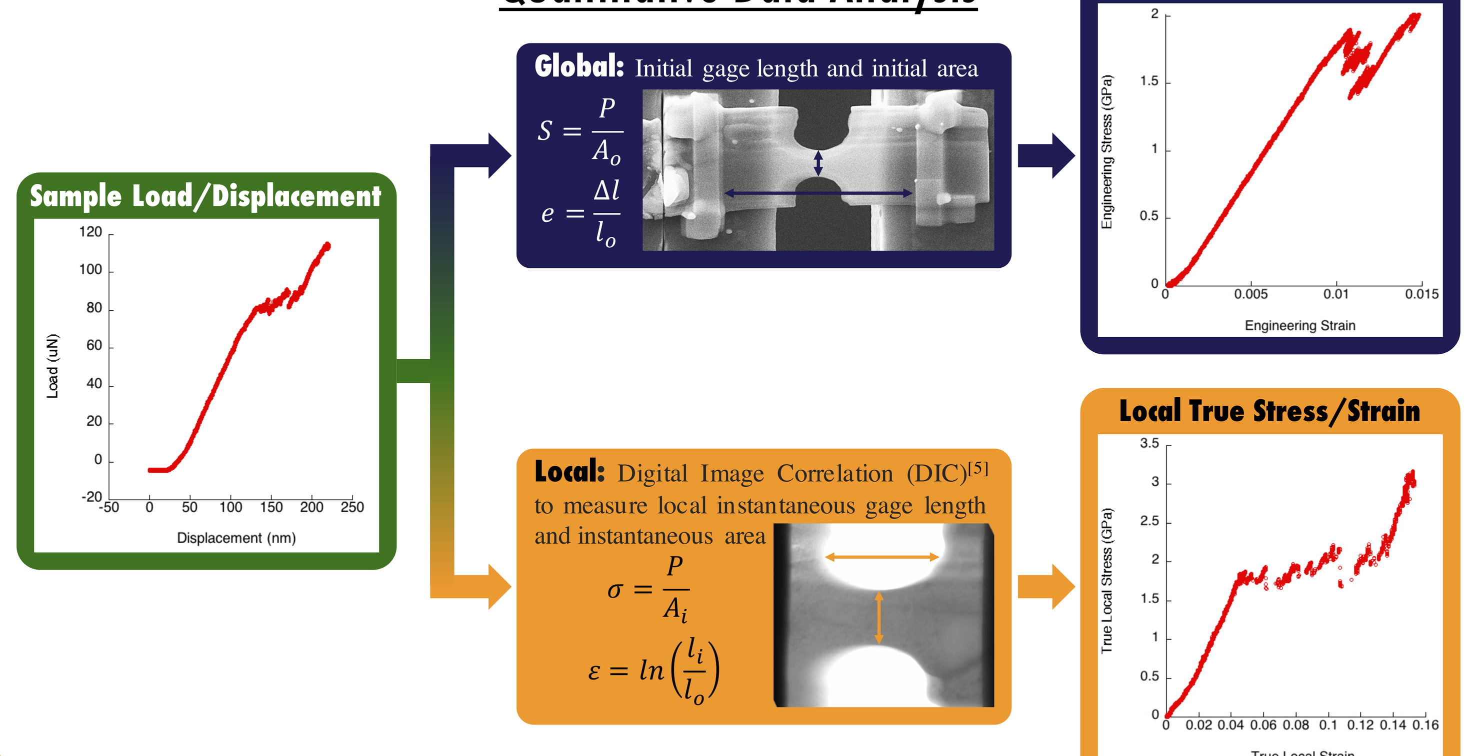
## Quantitative Mechanical Data Acquisition and Analysis

### Experimental Setup




- JEOL 2100 operated at 200 keV
- Video recorded at 15 fps
- Displacement control at 0.5 nm/s ($\dot{\varepsilon} \sim 1\times10^{-4}\ s^{-1}$)
- PTP has 4 laterally stiff springs (k = 450 N/m) that translate the compressive motion of the indenter into tensile motion at the sample gage section

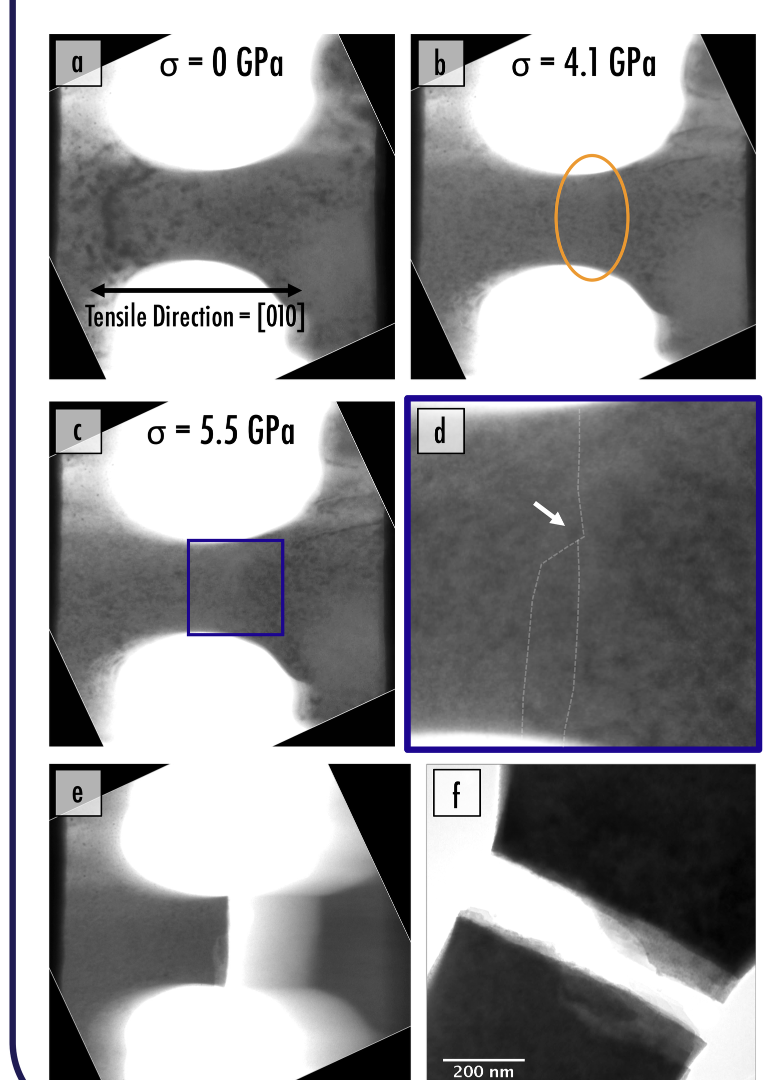
### Quantitative Data Analysis

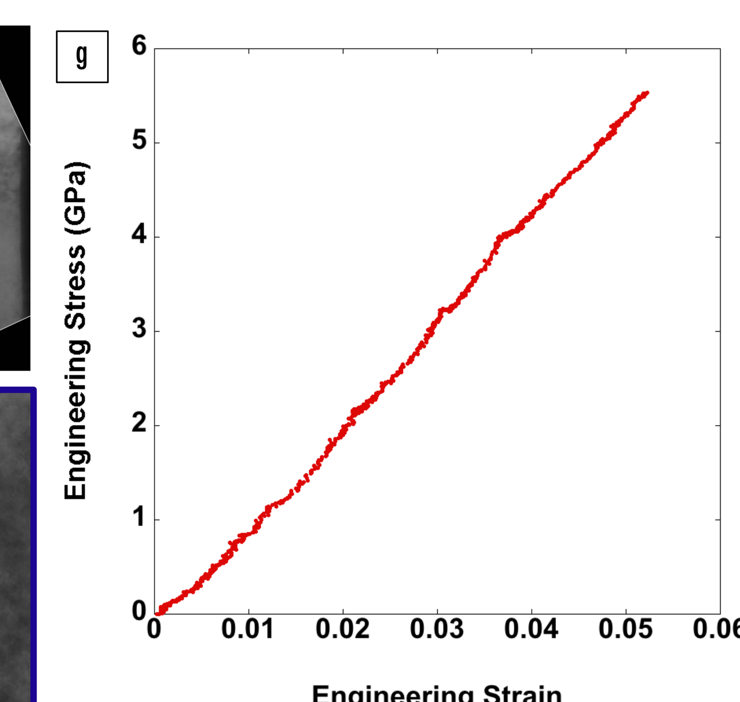



*Data are representative

## Quantitative In-Situ TEM Tensile Testing of Mica

- Sample
  - 115 nm initial thickness
  - 675 nm initial gage width
  - 1.2 μm initial gage length
  - Tensile loading // to [010](001)
  - Experienced drift during blind FIB cuts that exposed top ~¼ thickness of gage section to unknown dose of 30 keV $Ga^+$ ions
- $E_{[010]meas}$ = 110 GPa ($E_{[010]calc}$ = 170 GPa[6])
- $\sigma_{max\text{-}meas}$ = 5.5 GPa ($\sigma_{max\text{-}ideal}$ ~ 4-9 GPa[7])




In-situ TEM tensile test of biotite mica. (a-e) Still frame images from experiment video. (a) Initial sample. (b) FIB damage that has begun to clear at failure site at 3.2 GPa, is now obvious at 4.1 GPa (yellow circle). (c) Right before failure, defect seen to grow, indicating failure initiated in the middle of the gage width, likely in the FIB-damaged top layer. (d) Magnified area from blue inset in (c), arrow indicates defect location, dashed lines indicate relation to fracture surface. (e) Frame after failure. (f) Bright field TEM micrograph showing fracture surface. (g) Engineering stress-strain curve. Correlated in-situ video indicates plasticity at 0.8, 1.1, 2.1, 2.5, 3.2, and 4.0 GPa due to cooperative motion of FIB-induced defects.

## Conclusions

- A methodology to quantitatively measure the deformation of constituent geological materials at the nanoscale has been developed.
- A nanoscale biotite mica sample loaded in tension parallel to the basal plane along a multiple-slip direction showed nominally elastic behavior until brittle failure, after reaching near-ideal strength.
- Role of FIB damage unclear and sample drift mitigation techniques will be employed during future sample preparation.
- Slip-oriented (τ≠0) samples are expected to yield observable/measurable dislocation activity.

## References

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