Studying granular mechanics with neutron diffraction

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LUND UNIVERSITY

Granular media: What are they?

"**Granular materials are simple**:
they are large conglomerations of discrete macroscopic particles [...] Yet despite this **seeming simplicity**, a granular material **behaves differently** from any of the other familiar form of matter – solids, liquids, or gases – and should therefore be considered an **additional state of matter** in its own right."

«Granular solids, liquids and gases» – Jaeger et al. 1996

Inherently in a **non-equilibrium state**:

- Ordinary **temperature plays no role**
  - Lack of rearrangement under thermal fluctuations
- When in contact, the **interactions between grains** are **dissipative**
  - Loss of energy because of static friction and inelasticity of collisions

"… they phenomenologically reproduce equilibrium states of matter, exhibiting **characteristics of solids, liquids or gases, depending on the type and amount of driving** (state)."

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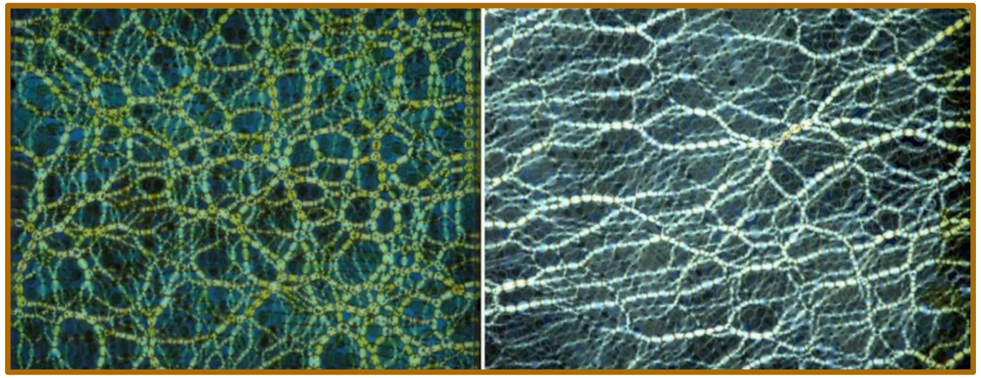
Granular solids under load

- When in **solid state**
  - ↳ Significantly **anisotropic** static configurations
- Under the effect of **applied stress**
  - ↳ Highly **heterogeneous** networks of **force chains** are **self-distributing the load** throughout the granular skeleton
- Material **failure**
  - ↳ Particle **interlocking** and **breakage**

Force chains are characterised by complex **spatio-temporal fluctuations**

- Varying and evolving **local stress-strain relationships**
- Localised **phenomena** and **mechanisms** of **different type** (e.g., shear/compaction bands) **across** the **spatial scales** (i.e., from the particle to the bulk)



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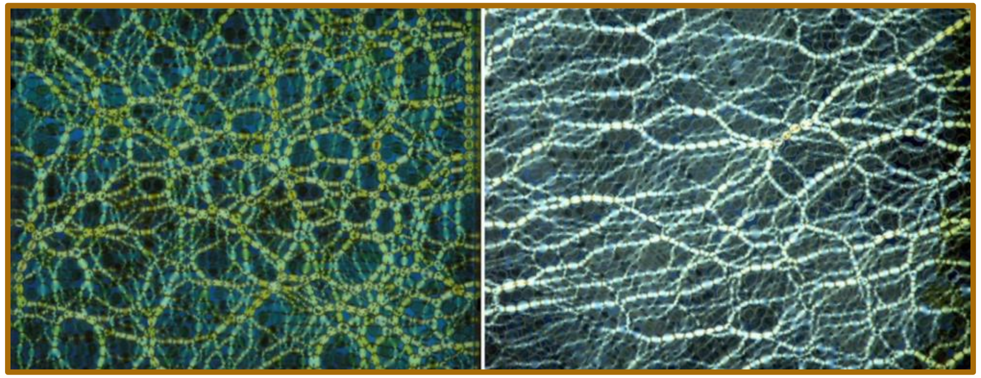

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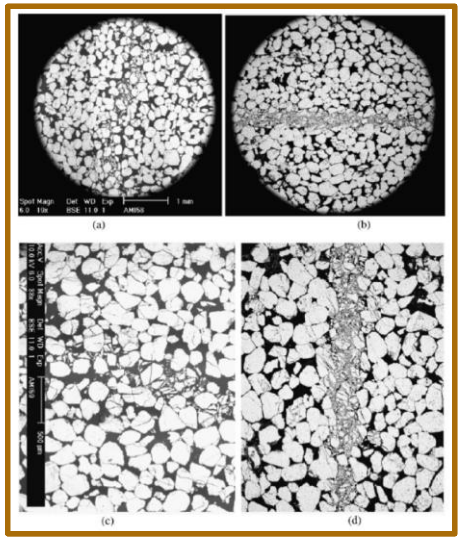
Stress: The key missing piece

A paradox: Granular media **still** often treated as **continuous materials!!**

- **Accurate models** must be built that take into consideration **grain scale phenomena**

- Traditional **macroscopic boundary measurements** provide a mere **approximation** of **(local) stress-strain relationships** and the **(micro)mechanisms** leading to material failure

  ↳ Solution: **Full-field measurements**



El Bied et al. 2002

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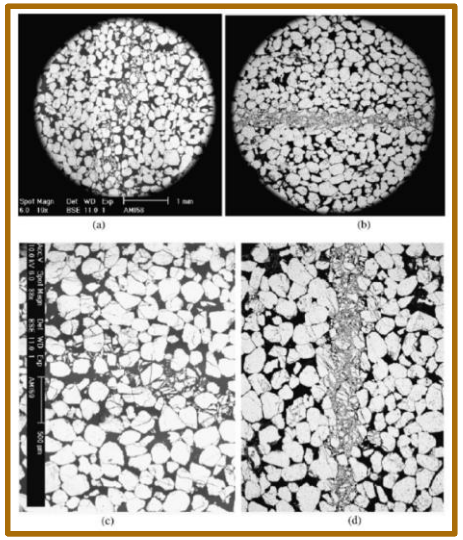

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Scattering based methods

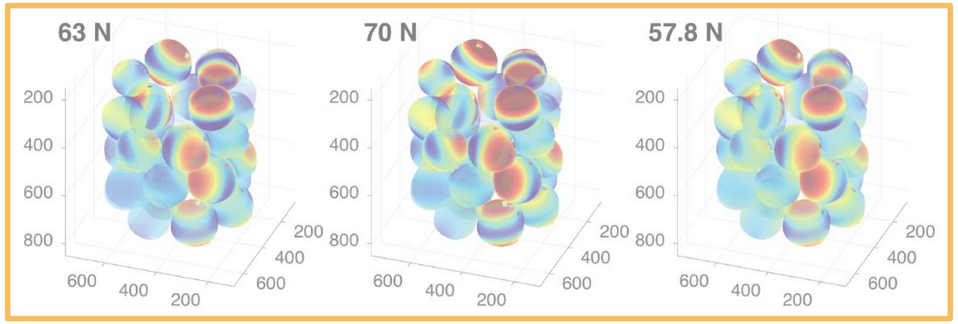
Principle to deduce stresses relies on the fact that the **constituent grains** of a granular specimen under load may **serve as intrinsic strain gauges**

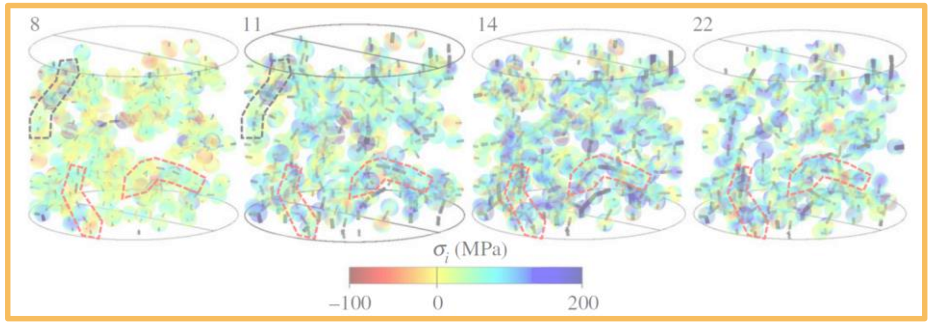
- Through **Bragg's law**, the **elastic** component of the **crystallographic – or grain – strains** can be derived
- Consequently, **grain-scale stresses** can be deduced directly by making use of **Hooke's law**

3D X-ray diffraction (3DXRD)

Individual grain **measurements** ⇨ **Discrete granular mechanics** for assemblies of a few hundreds of grains

Hall & Wright 2015





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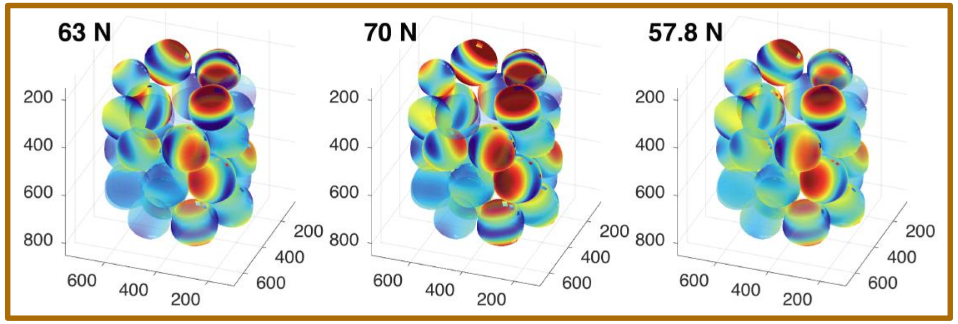

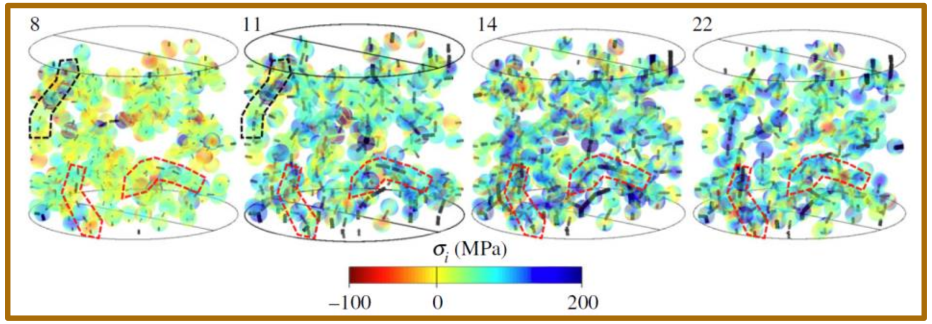

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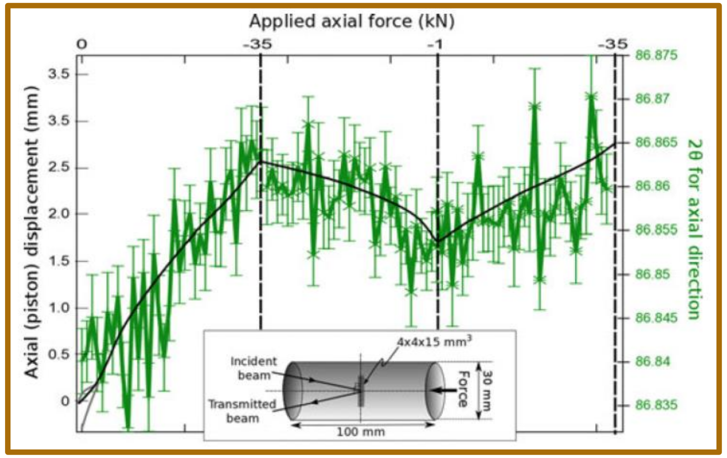
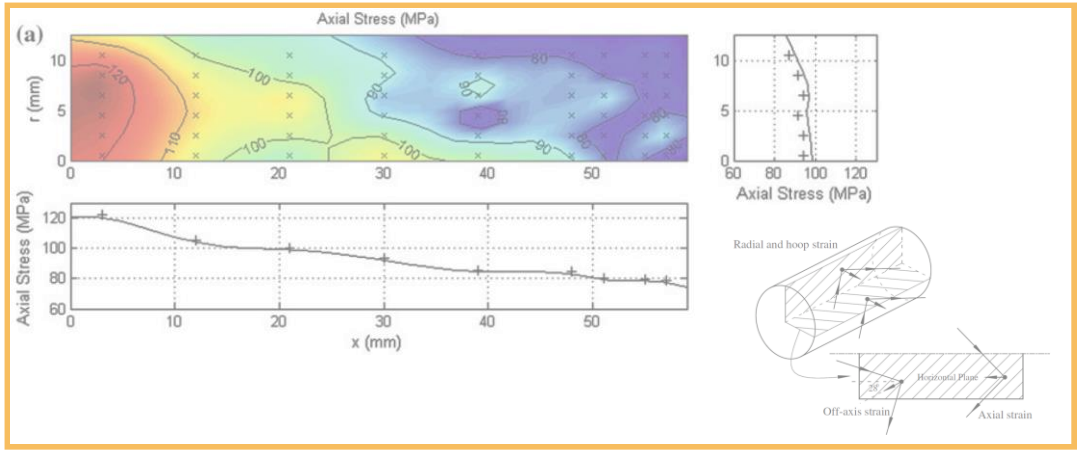
Scattering based methods

Neutron Diffraction (ND)

Averaged values over small sub-volumes ⇨ A continuum view of granular behaviour of larger, more representative sized specimens





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Provided insight into the evolution of elastic grain strains, however no information on spatial variations was achieved

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Reported the spatial distribution of granular stresses throughout a preloaded powder specimen, but no in-situ loading was involved

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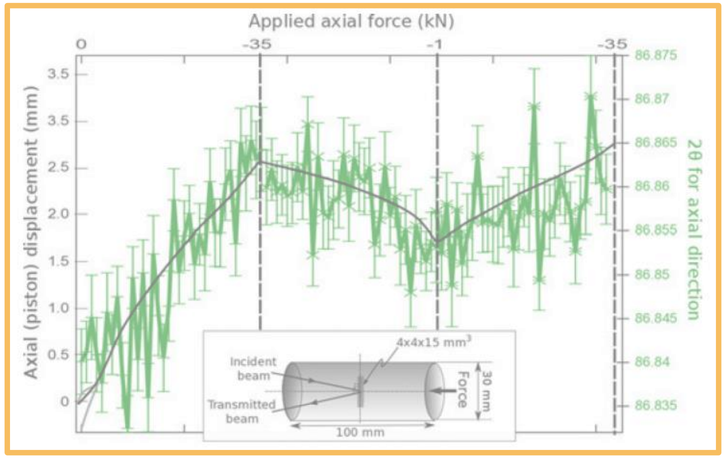

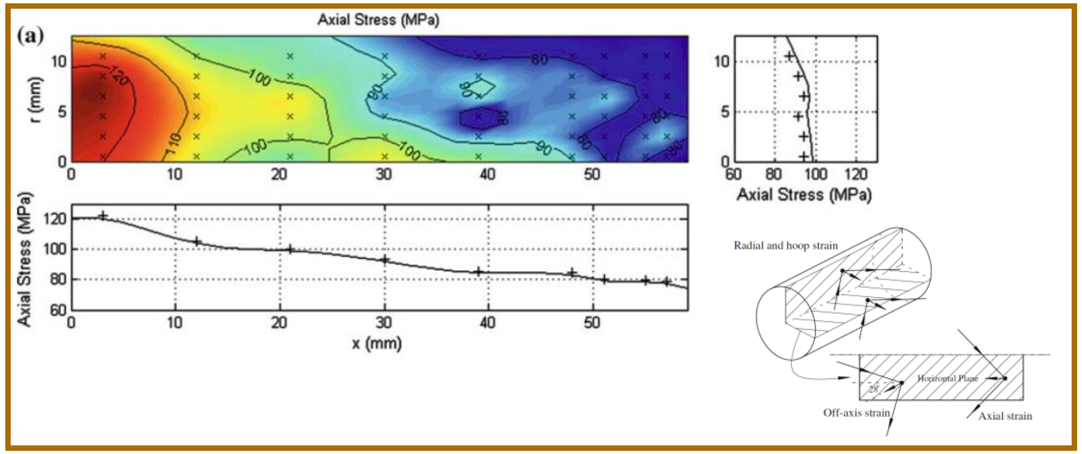

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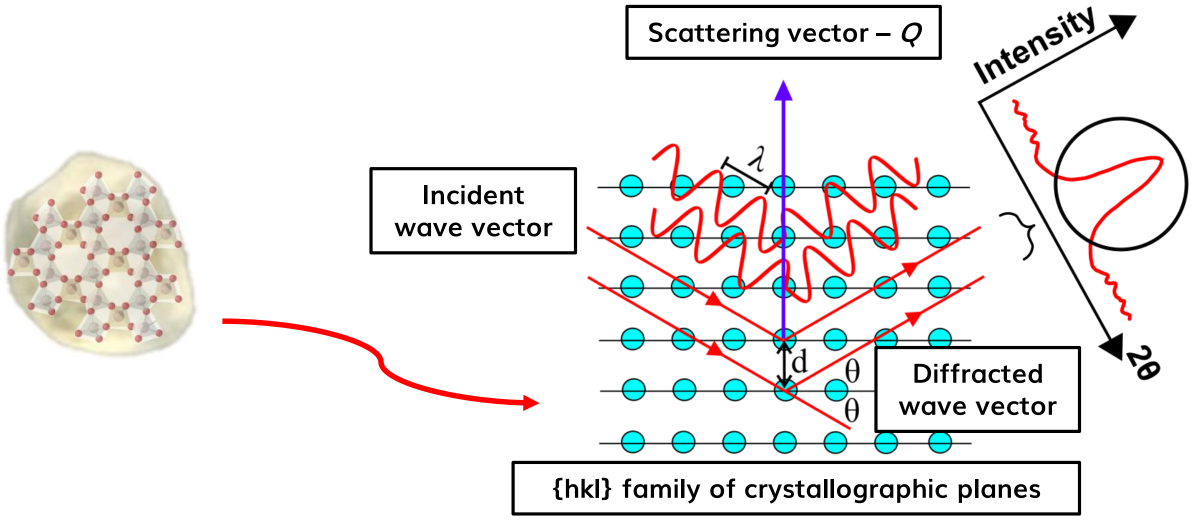
Bragg's law



Bragg's law

$$n\lambda = 2d \sin\theta$$

n: the order of the reflection
λ: the wavelength
d: the lattice spacing
θ: the diffraction angle

Bragg's law fulfilment

Constructive interference of radiation diffracted by the atoms of **adjacent planes in a *hkl* direction** of the crystal lattice gives **peaks** at well defined **θ^{hkl} angles**

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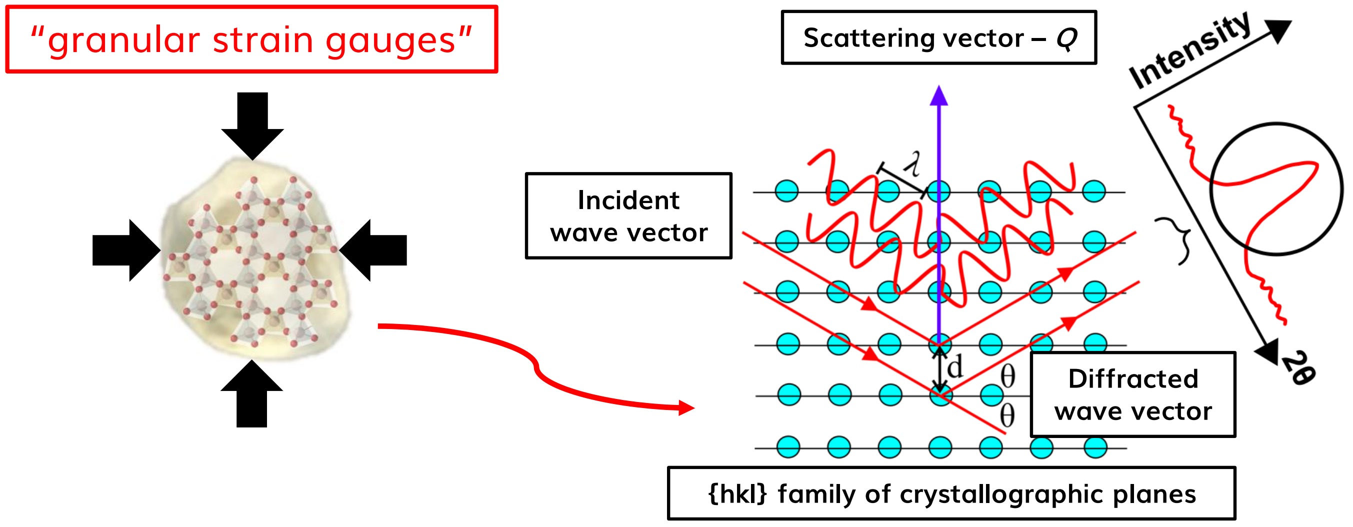

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Variations in θ^{hkl} angles indicate **d-spacing changes** of the crystal lattice

⇩

The **elastic crystallographic – or grain – strains** (e.g., due to loading) can be calculated by the use of **peak shifts**

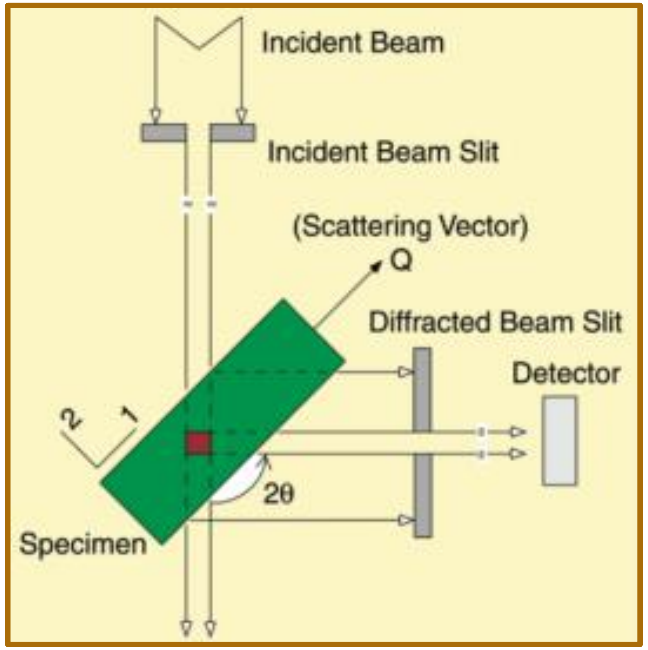
$$\varepsilon^{hkl} = \frac{d^{hkl} - d^{hkl}_{\text{ref}}}{d^{hkl}_{\text{ref}}}$$

Granular stresses may be inferred from grain strains, by direct use of **Hooke's law**

Neutron diffraction for granular media

Grain strains measured over **mm^3-sized** gauge volumes (GV) consisting of **thousands of grains** (i.e., a **"powder average"**)

Each GV acts as an **intrinsic strain gauge** embedded in a specimen



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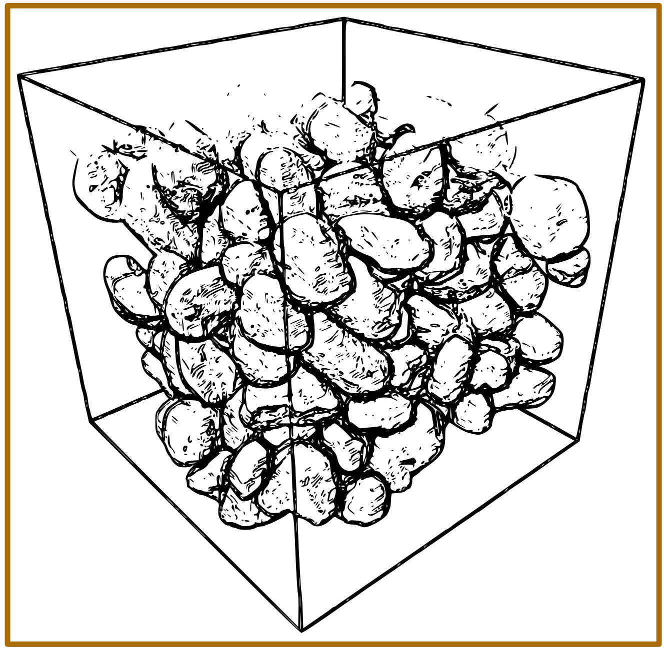

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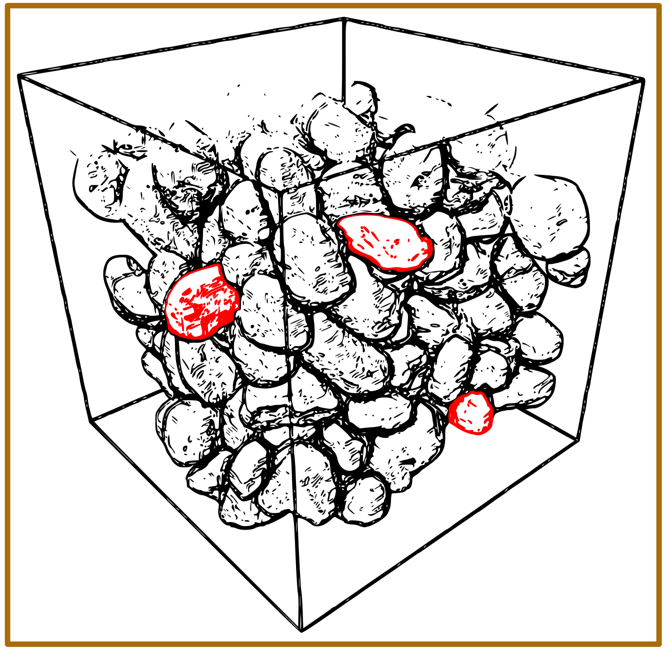

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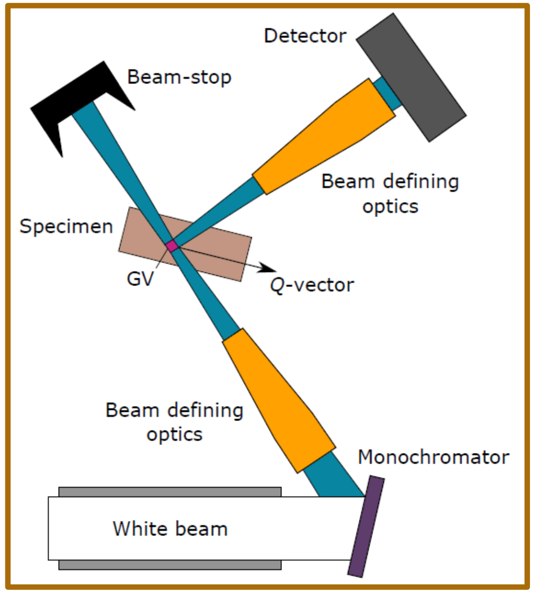
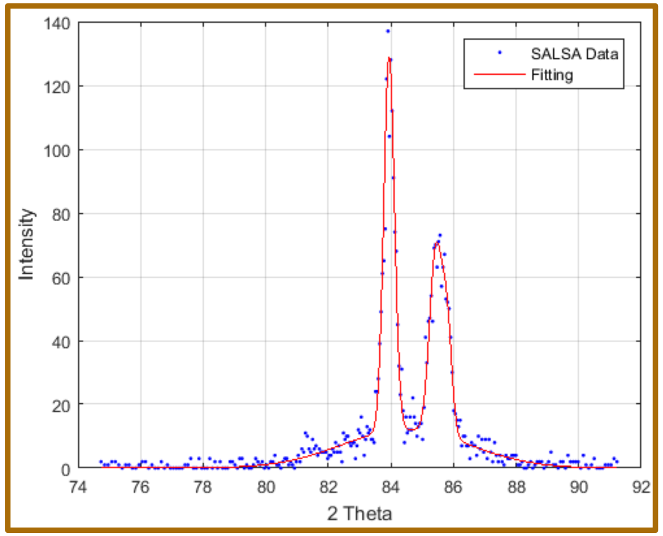
Single wavelength





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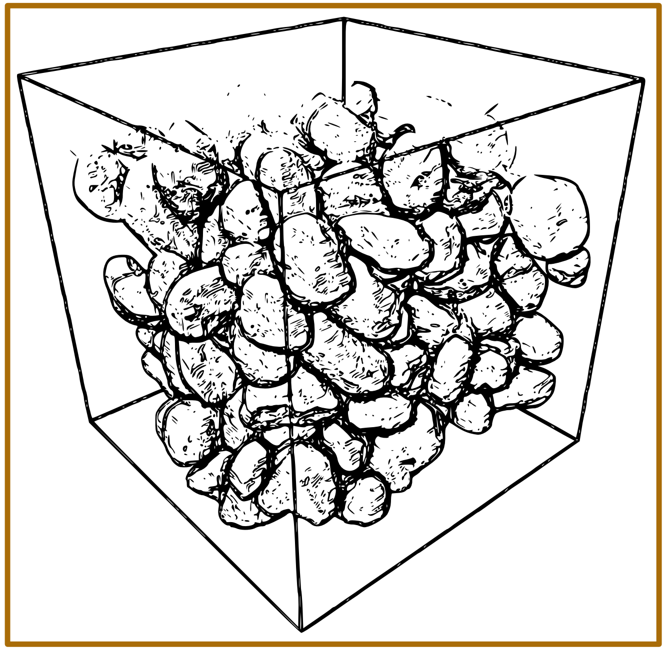

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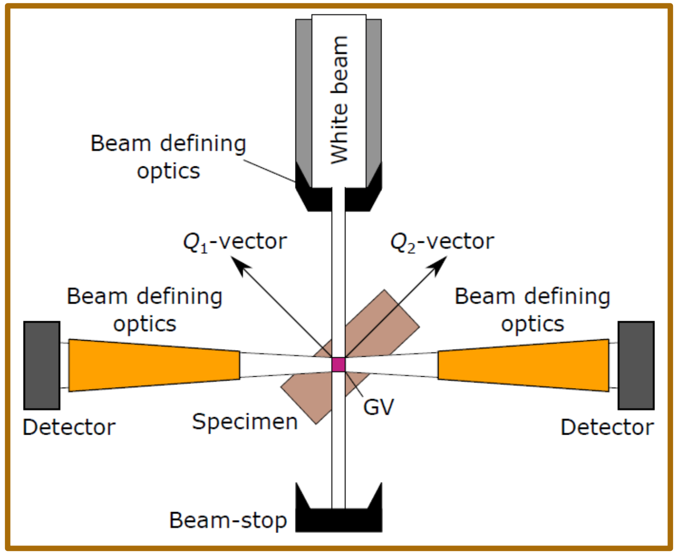
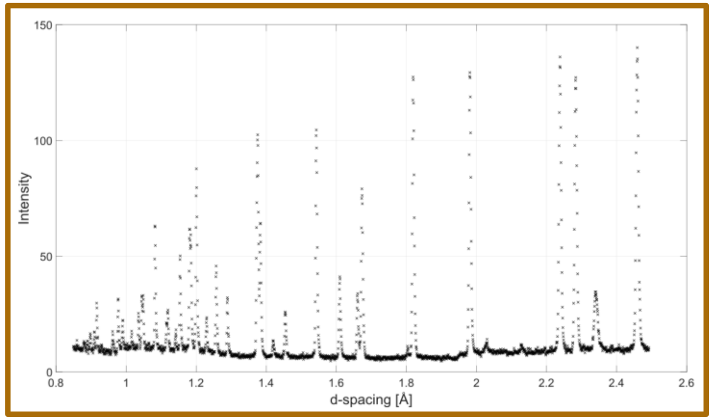
Multiple wavelengths





Neutron diffraction for granular media

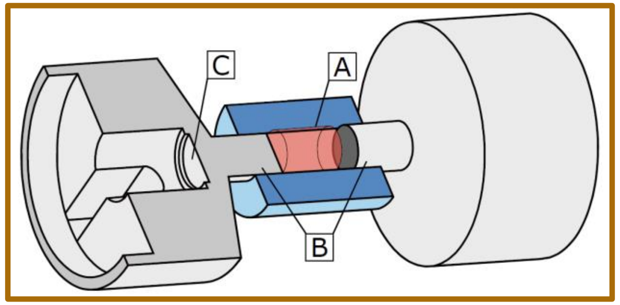
Newly defined formulas for multiple grain orientation dependent granular stress

$$\sigma_{micro,i}(l) = \frac{V_b}{V(l)} \sum_{hkl} \frac{w^{hkl}(l)}{\sum_{hkl} w^{hkl}(l)} C_{ij}^{hkl} \varepsilon_j^{hkl}(l) \qquad w^{hkl}(l) = \frac{I^{hkl}(l)/I_{Ref}^{hkl}}{\sum_{hkl} I^{hkl}(l)/I_{Ref}^{hkl}} \frac{m^{hkl}}{\sum_{hkl} m^{hkl}}$$

- l ⇨ Load step
- V_b & V ⇨ Bulk and total volume of specimen
- C_{ij}^{hkl} ⇨ *hkl*-associated stiffness matrix
- w^{hkl} ⇨ *hkl*-associated weighting factors
- m^{hkl} ⇨ Multiplicity
- I^{hkl} & I_{Ref}^{hkl} ⇨ Measured and reference Bragg peak heights

Representative results

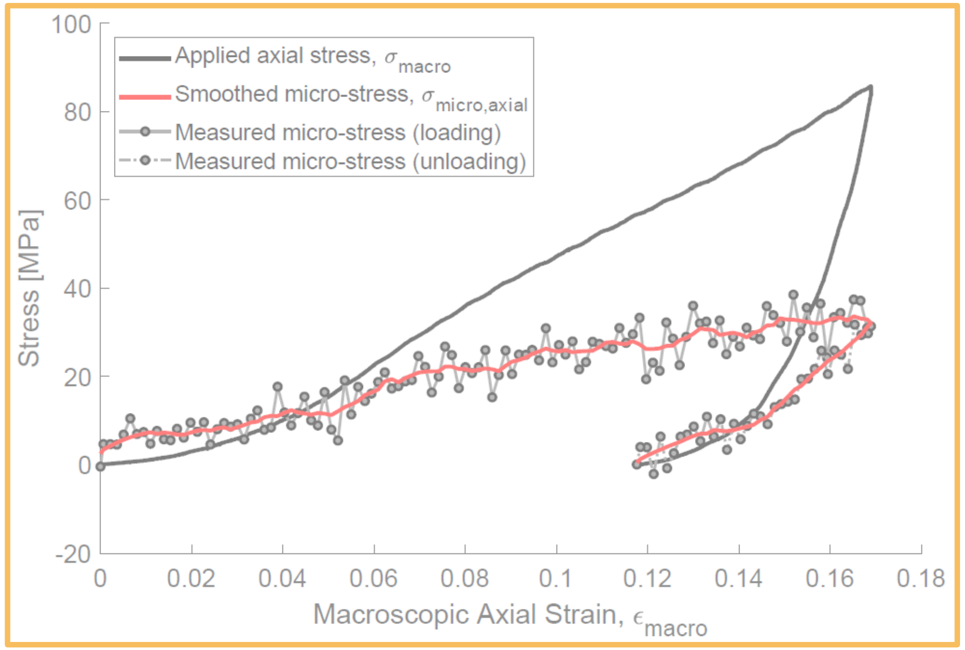
Uniaxial (oedometric) loading conditions



A: Specimen (Ø: 10 mm – H: ~10 mm)

B: Pistons

C: Ultrasonic transducer chamber



- **~20%** specimen coverage (single GV: **4x4x10 mm^3**)
- **153** measurements (continuous acquisition) – **3.5 min/GV**
- **Granular stress** calculated from **7 *hkl* subsets** of grains

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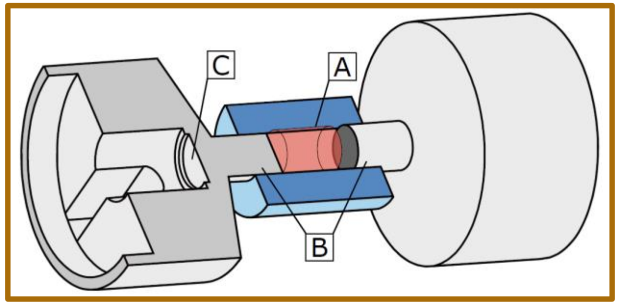

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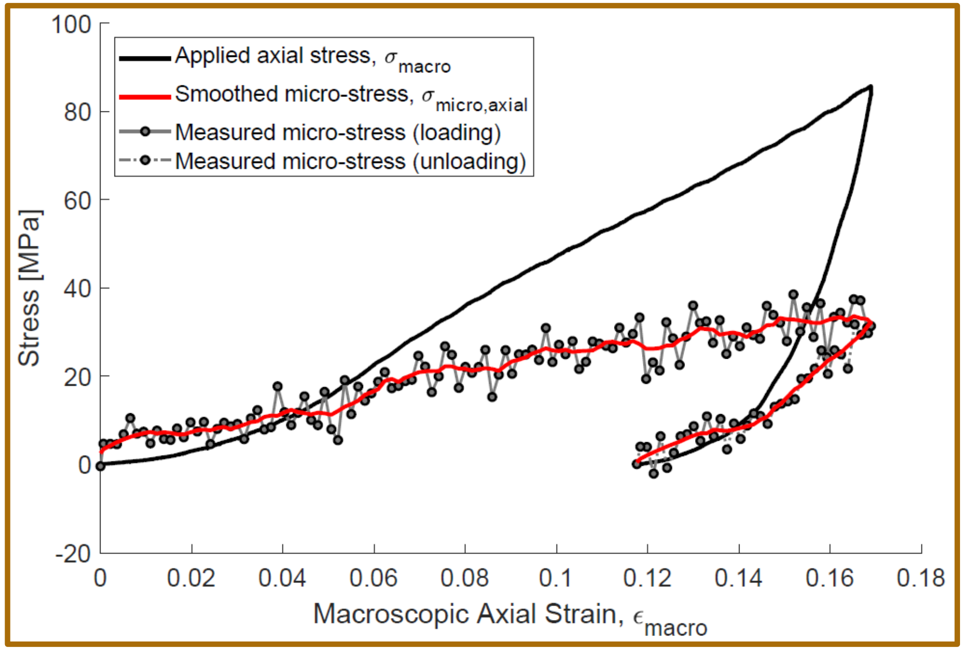

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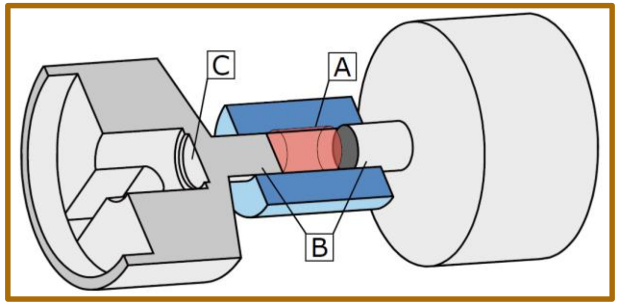

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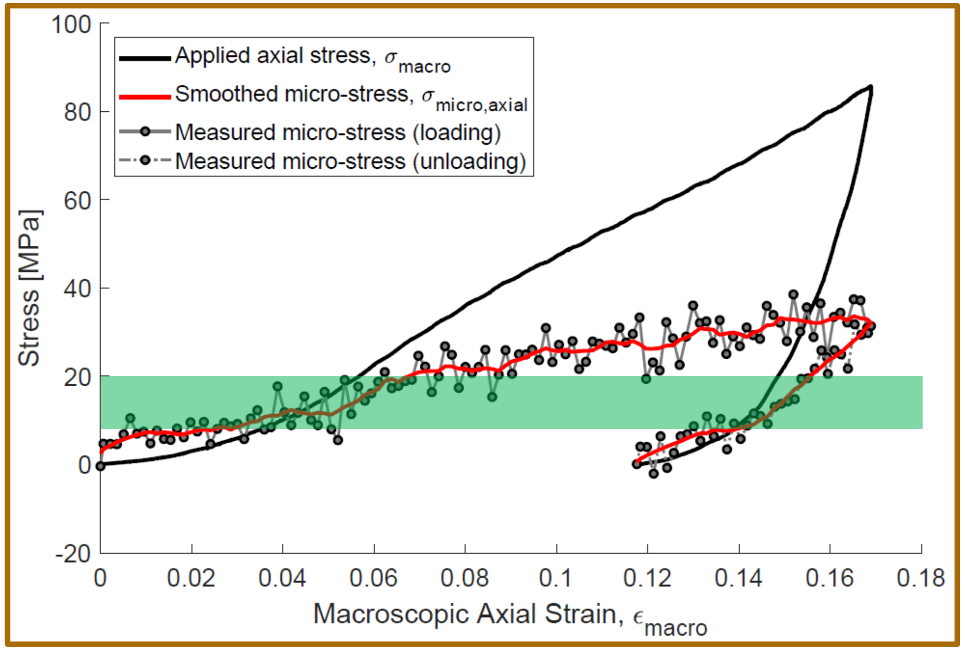

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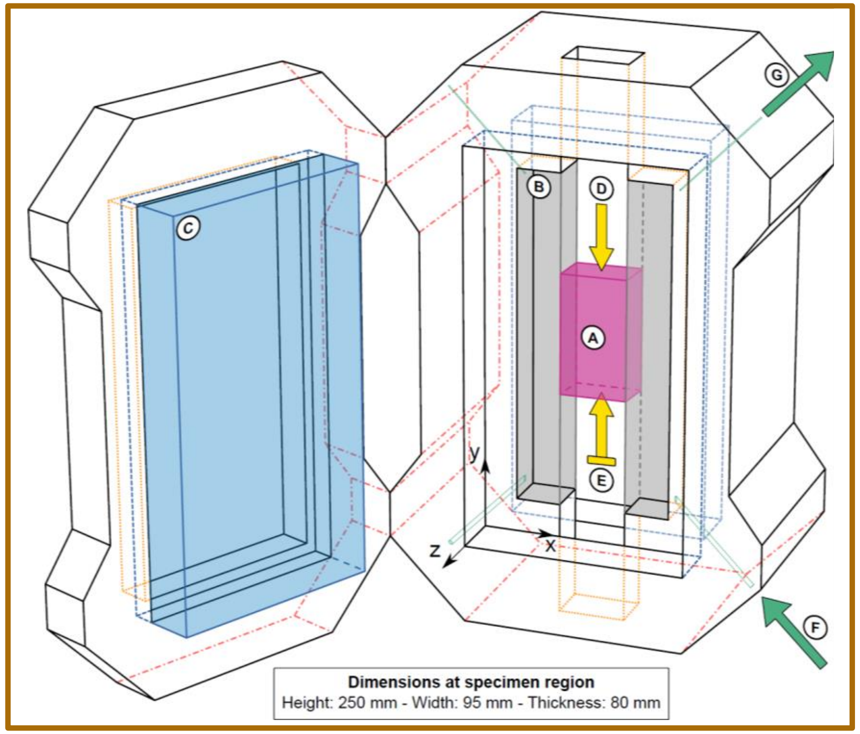

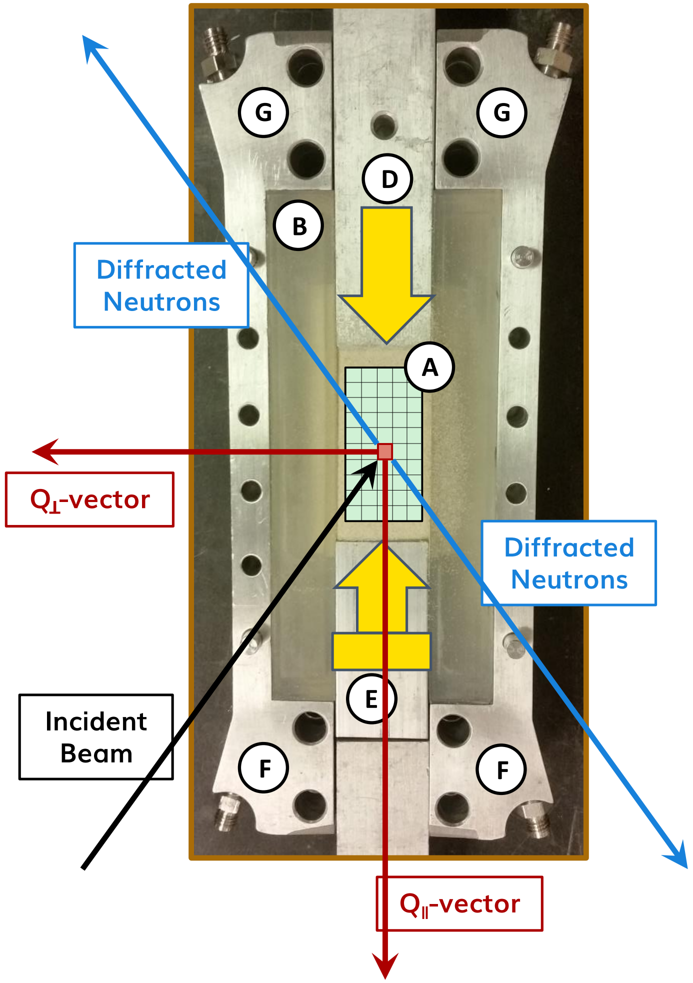

A: Specimen (60x30x20 mm^3)
B: Pressure-controlled cushions
C: Sapphire platens
D: Moving piston
E: Fixed piston
F: Pressure liquid supply
G: Air escape

Representative results

A novel multiscale neutron diffraction-based experimental approach for granular media
Géotechnique Letters 9(4), pp. 284-289

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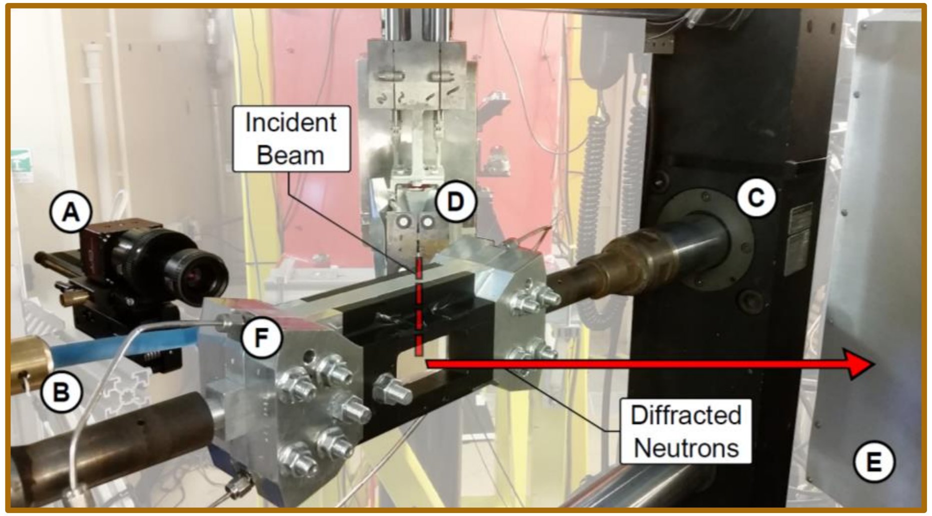
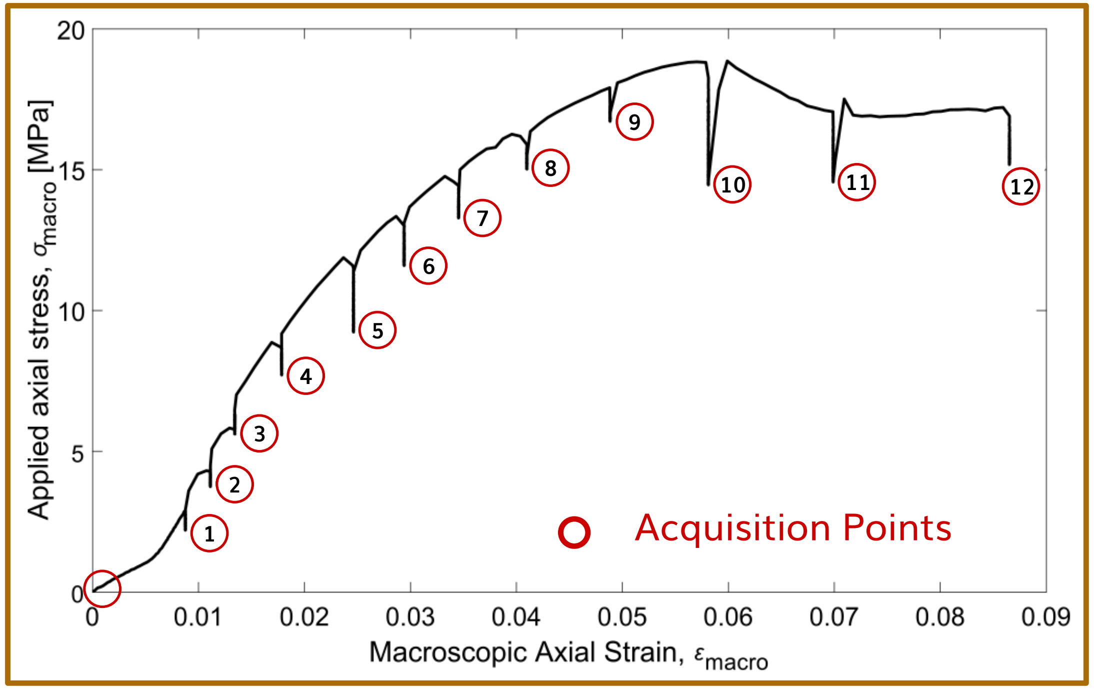


A: High resolution camera (28.8 MP)
B: Custom-made LED lighting system
C: Stress rig
D: Beam defining optics system
E: Detector
F: Connection to pressure controller



- **~56%** specimen coverage (grid of **6x10 GVs** – GV: **3x3x4 mm^3**)
- **13** mappings (acquisition in load steps) – **4 min/GV**

Representative results

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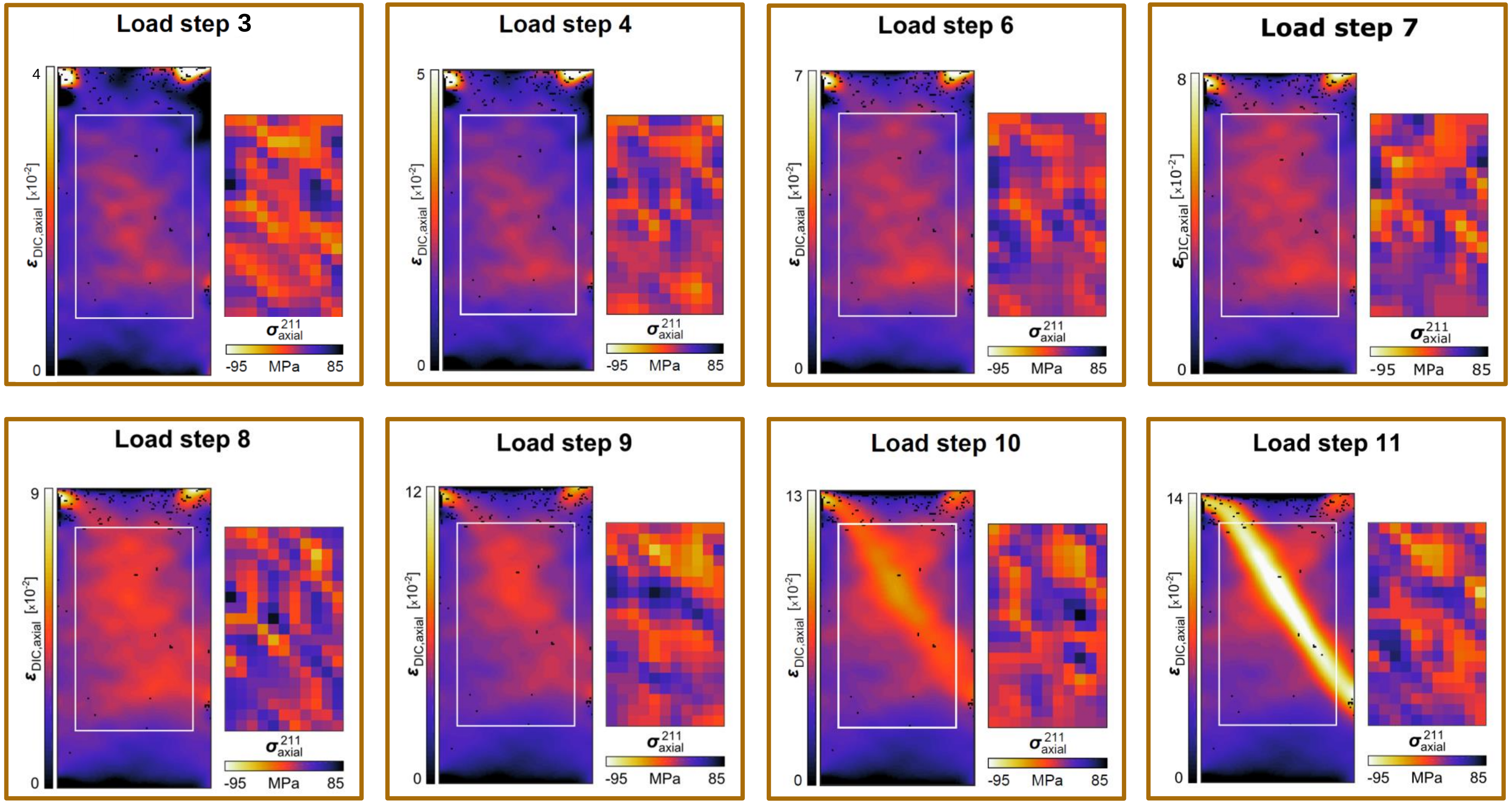

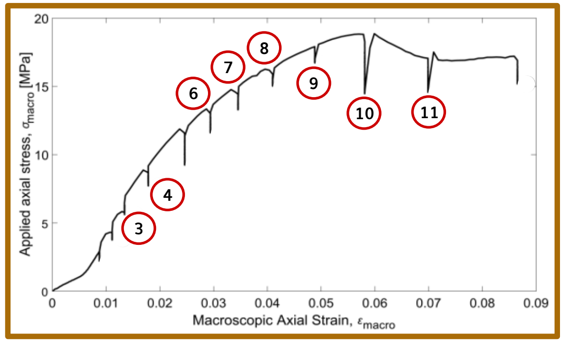

- **Stress mappings** produced only from ND measurements in the **axial direction**
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OBS! Darker colours in the **stress mappings** indicate **compression**

Representative results

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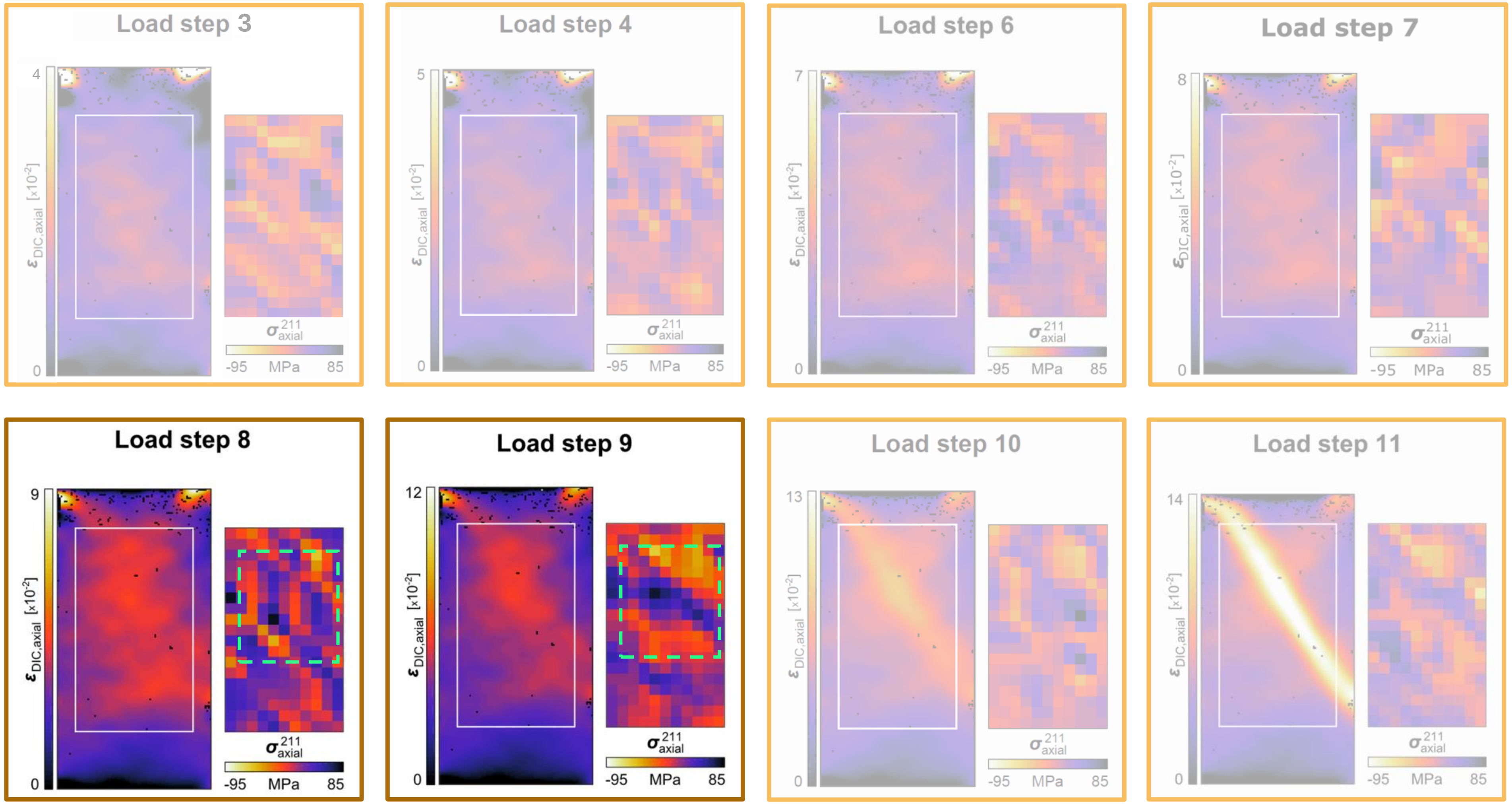

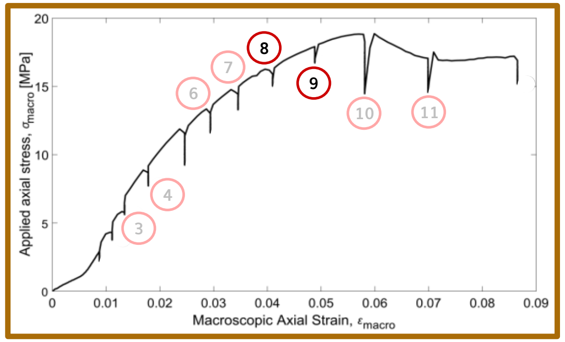

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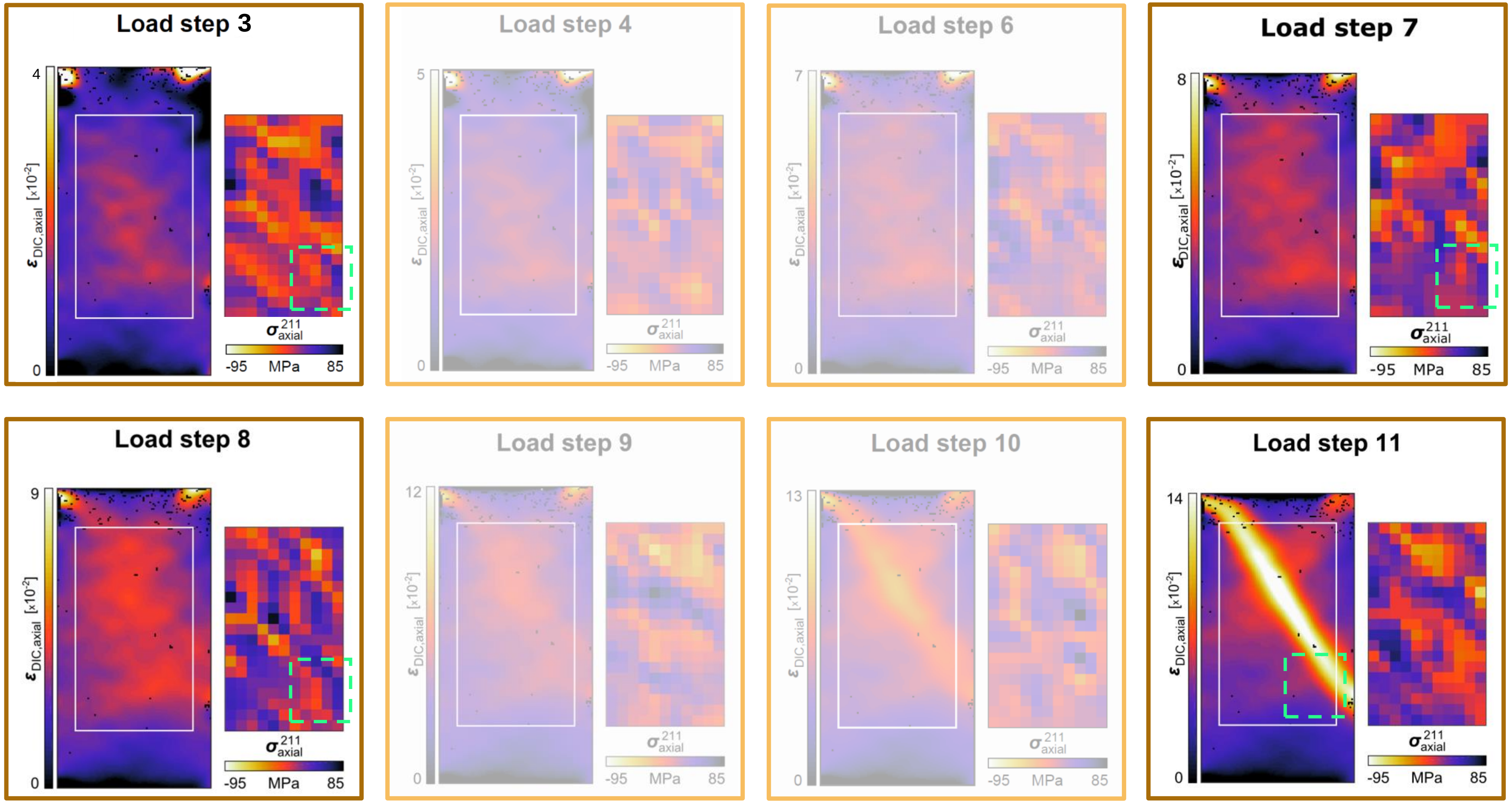

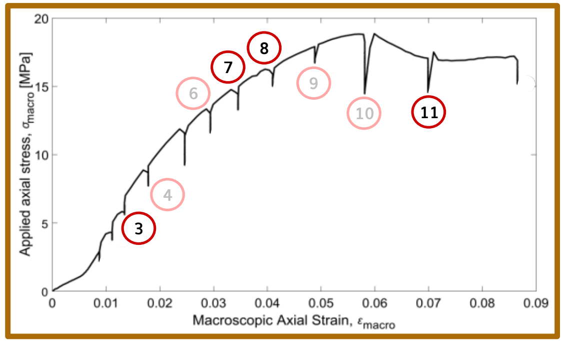

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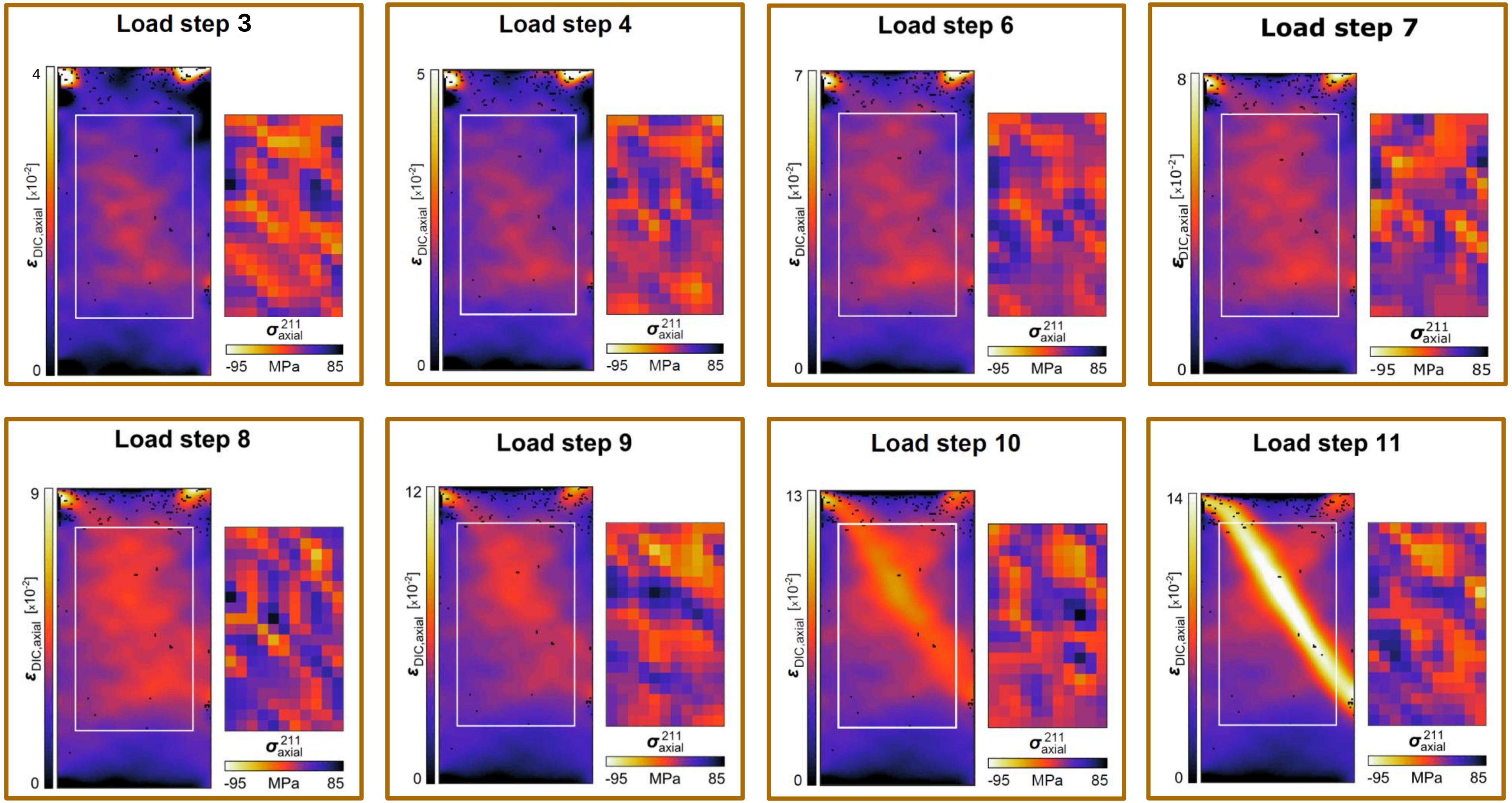

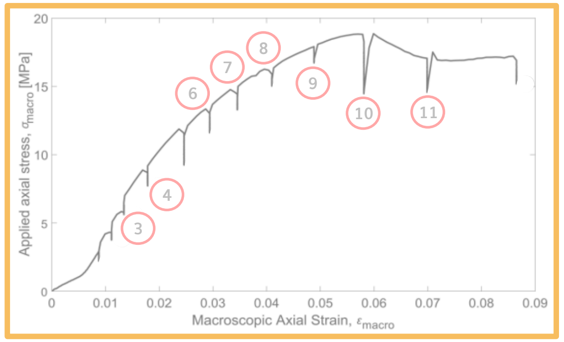

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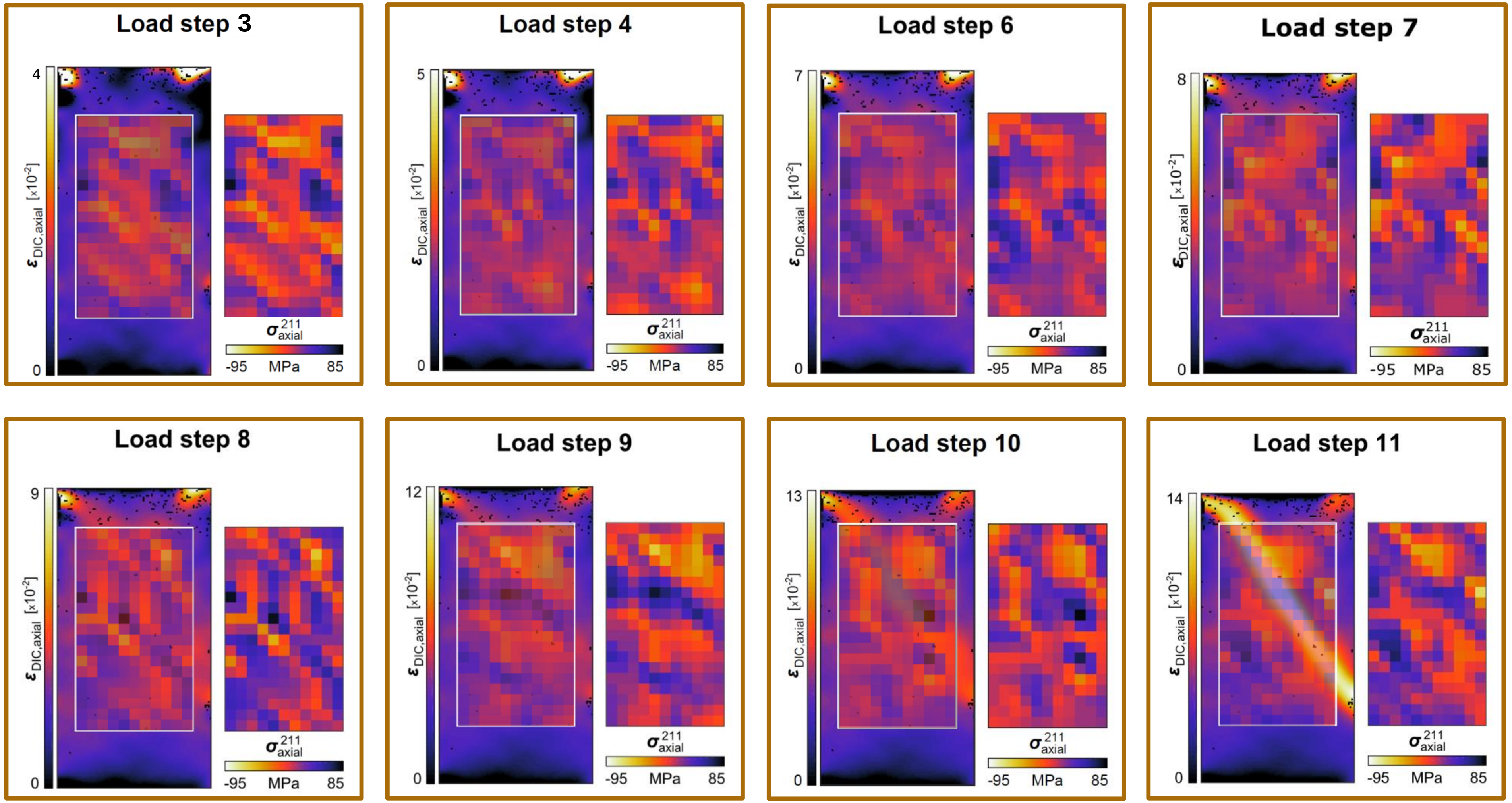

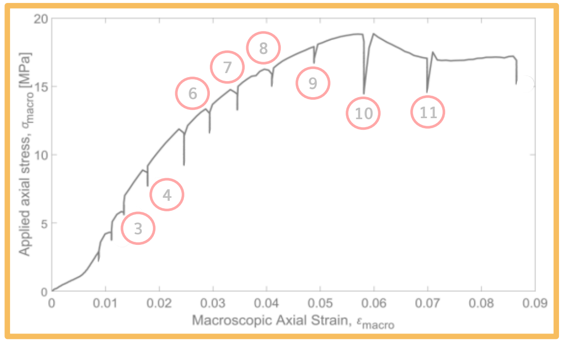

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Summary

- Association of **macro-/meso-** and **microscale** mechanical responses
  - Traditional macroscale **boundary measurements** – microscale **ND-inferred stress** (& mesoscale **DIC-derived strain** / **Ultrasonic-derived stiffness**)
  - Key component: **Specially-designed devices**
- Determination of **grain-orientation** dependent **granular stresses**
  - **Physically realistic** manner to account for grains in **multiple orientations**
- **Promising correlation** of structures in the **ND** and **DIC** results
  - Novel insight into the **coupled evolution of stress-strain distributions** throughout granular media
  - **Non-agreeing features** are likely to **reveal** more information on the mechanics **across different scales**
- **Ongoing** work …
  - Investigation of **stress-strain relationships** in a **localised manner**
  - Detailed investigation of the **sources of uncertainty/errors**

Acknowledgements

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Sergio Carcia Martinez
Dr Sandra Cabeza
Dr Alessandro Tegnattini

Prof. Gary D. Couples
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