

Tensile properties of bone extracellular matrix at the microscale

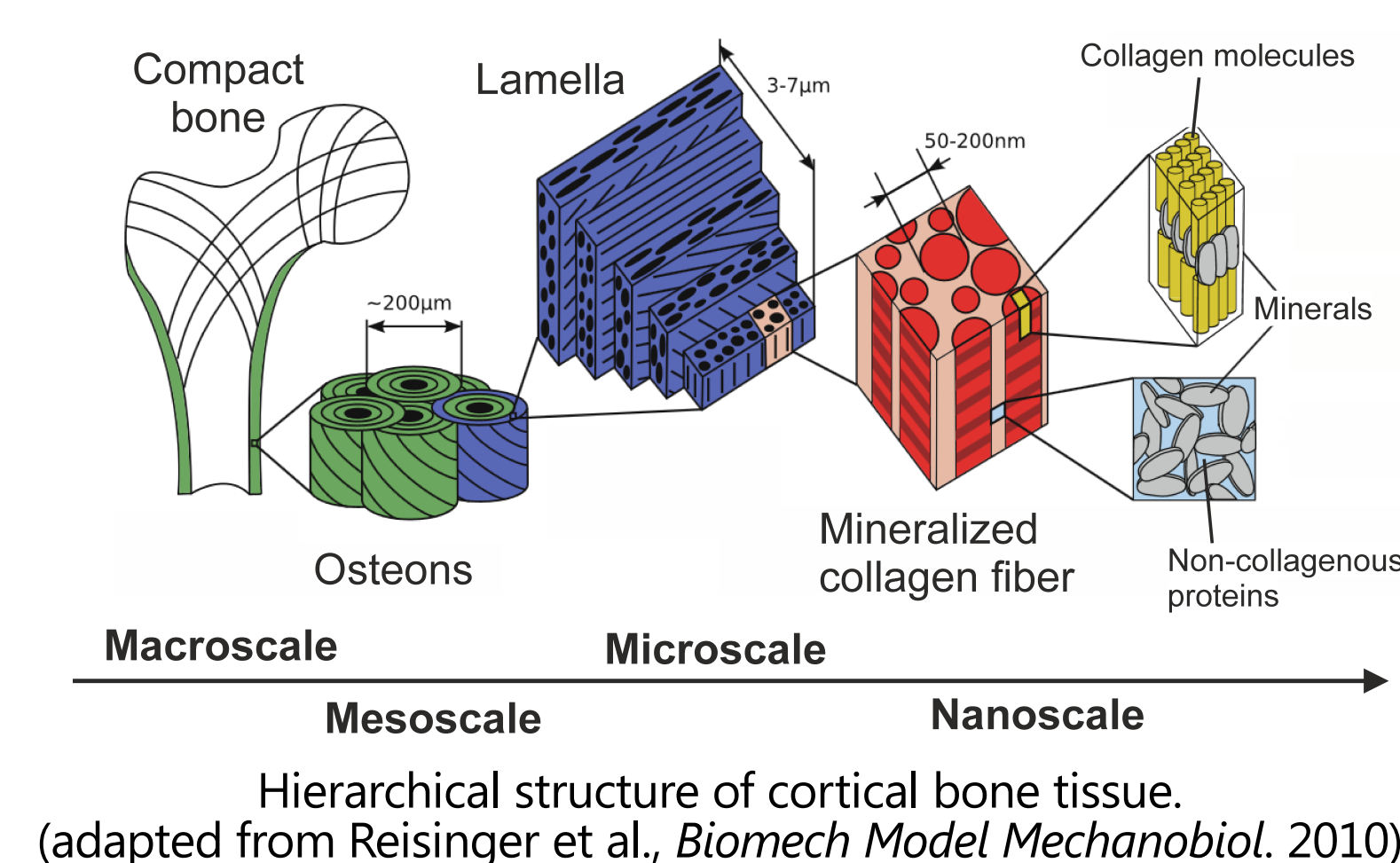
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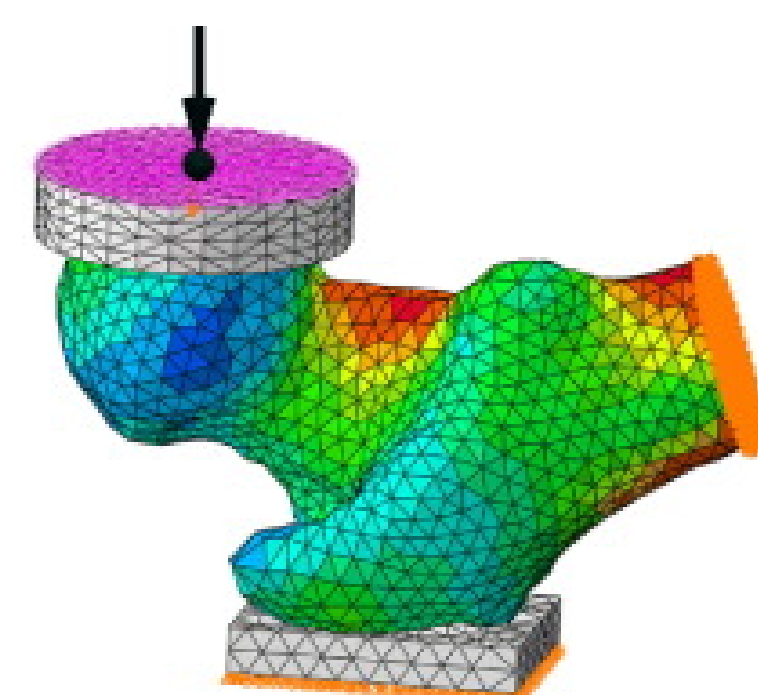
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Abstract: A micromechanical tensile setup was designed to probe focused ion beam (FIB) fabricated samples, while reducing measurement errors due to possible misalignment between the gripper and the sample. Ovine osteonal bone was tested in both axial and transverse orientations on the length scale of a single lamellae inside a scanning electron microscope in order to identify the elastic modulus, strength and strain at failure. Fracture surfaces were analysed in order to understand the dominant failure mechanisms.

1. Introduction



The mechanical properties of bone are defined by the individual elements that compose its hierarchical structure and the interaction between these across different length scales. To understand how bone can combine antagonistic properties like toughness and strength, it is important to characterize the mechanical properties of bone at different length scales.



FE calculations for fracture analysis. (Zysset et al., Bone, 2015)

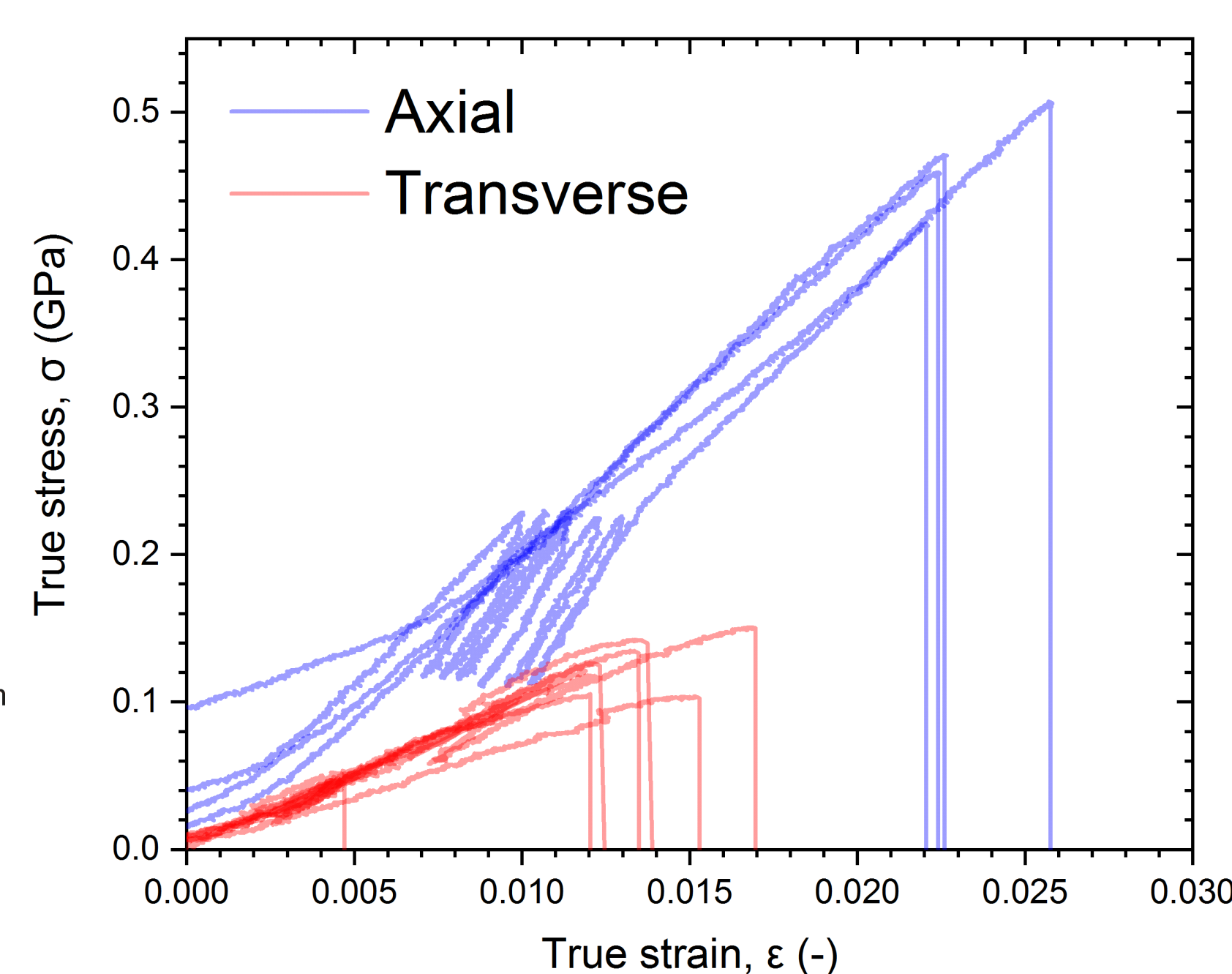
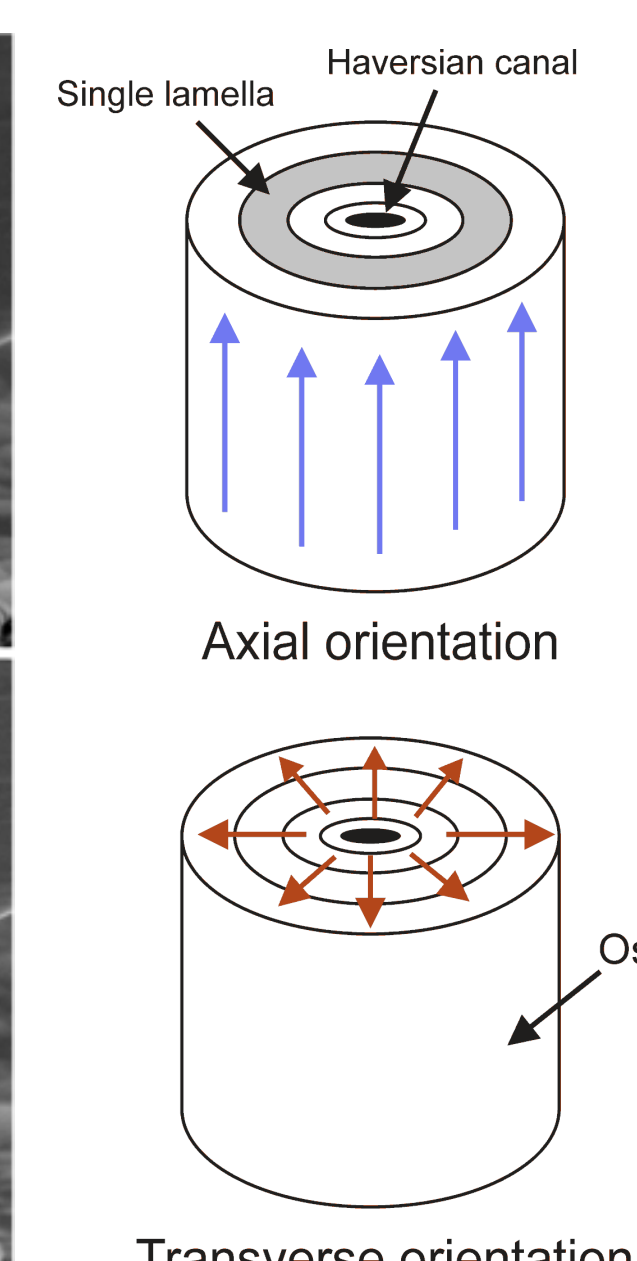
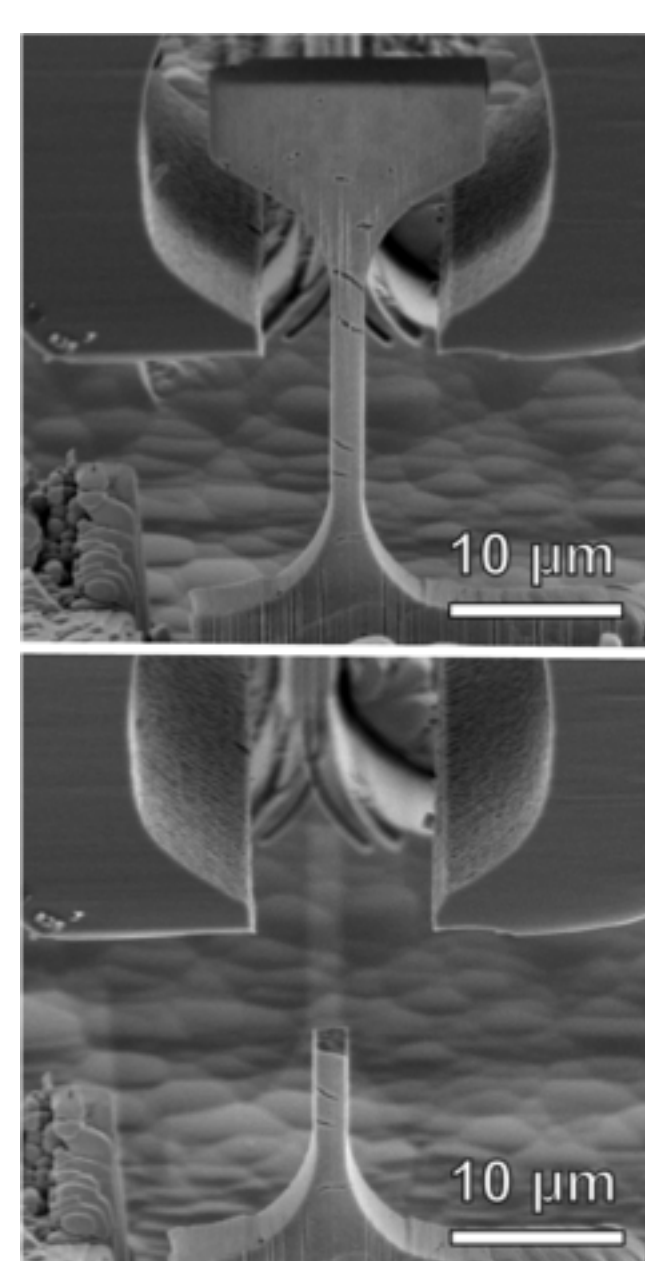
Multiscale biomechanics is an emerging field aiming at increasing the precision of mechanical models on the organ level by considering bone's hierarchical architecture. The study of bone micromechanics might allow to enrich existing finite element (FE) models for improved fracture risk prediction.

Nevertheless there are major experimental challenges encountered when testing miniaturized samples that need to be solved to obtain reliable data:

- Sample handling
- Sample fabrication
- Misalignment

Different testing methodology from macroscale is required!

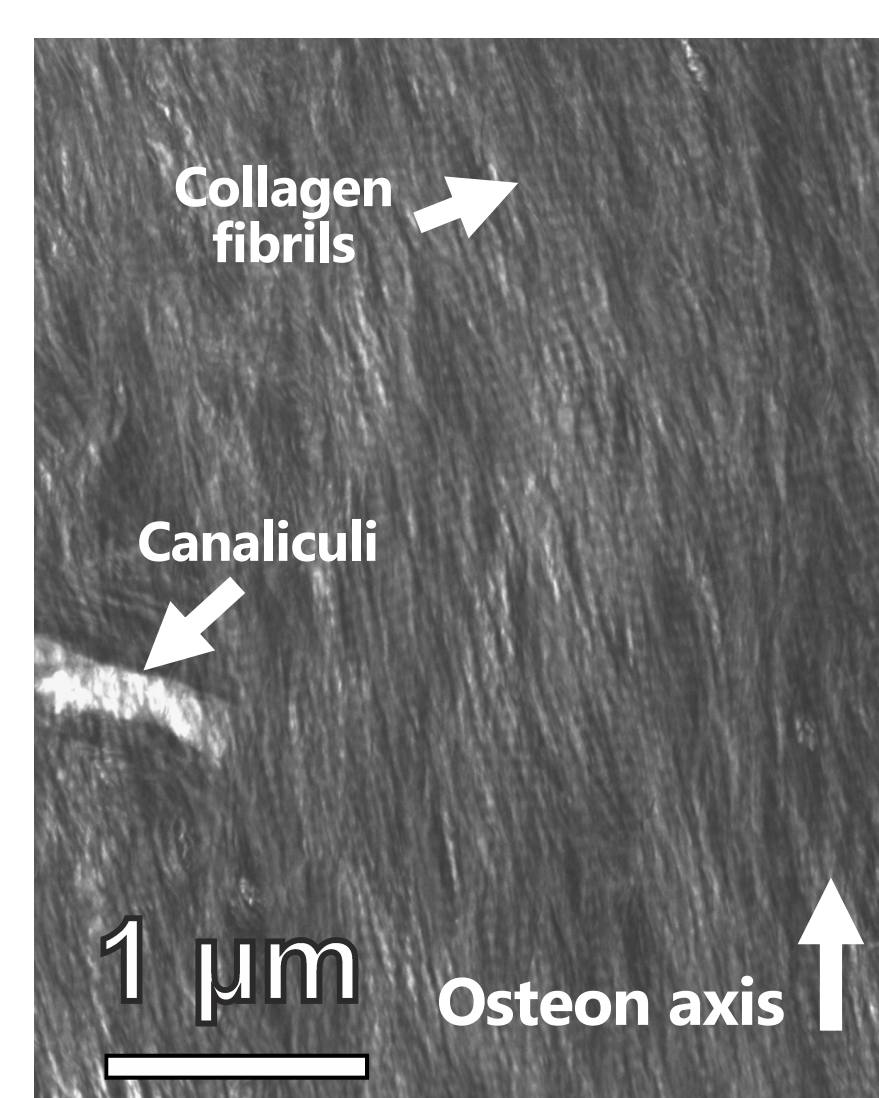
3. Microtensile properties



| Property | Axial Tension | Axial Compression [1] | Transverse Tension | Transverse Compression [1] |
|--------------------------------------|-------------------|-----------------------|--------------------|----------------------------|
| Strength, σ_{max} (GPa) | 0.47 ± 0.03 | 0.75 ± 0.06 | 0.12 ± 0.03 | 0.59 ± 0.04 |
| Strain, $\epsilon(\sigma_{max})$ (-) | 0.023 ± 0.002 | 0.054 ± 0.017 | 0.013 ± 0.004 | 0.121 ± 0.025 |
| Elastic modulus, E (GPa) | 33.6 ± 0.7 | 31.1 ± 6.5 | 13.9 ± 1.2 | 16.5 ± 1.5 |

[1] J. Schwiedrzik et al. *Nature Materials*, 13, 740–747 (2014)

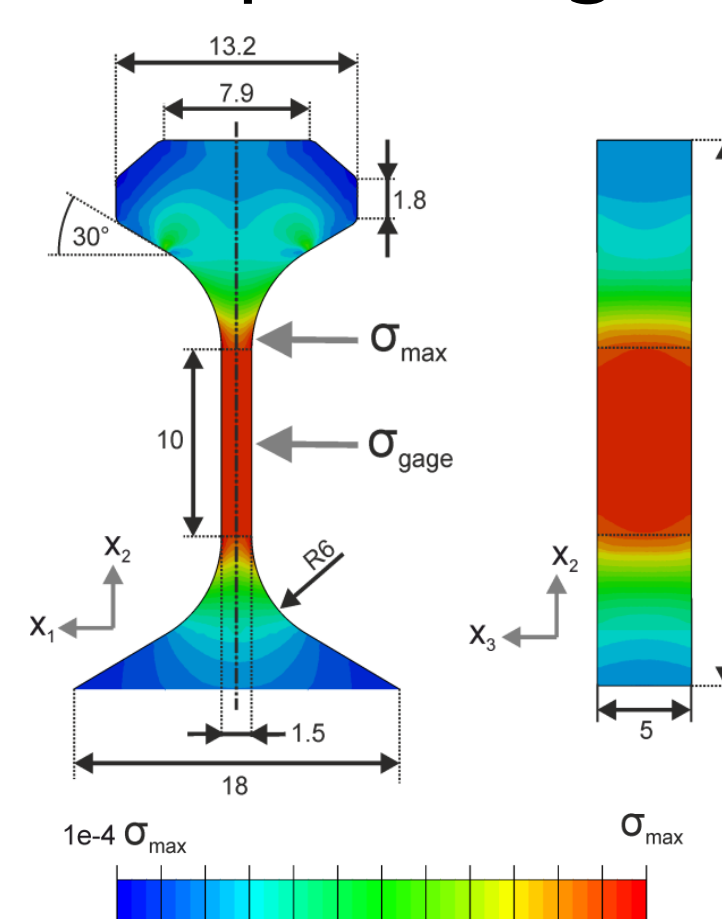
Similarly to micropillar compression [1], microtensile tests on the length scale of a single lamella showed a size effect and a clear anisotropy of the mechanical properties of lamellar bone. While elastic properties were comparable between compression and tension for both loading directions, failure strength and strains were different. Bone micromechanical properties showed an increase in value by a factor of 2.8 from macroscopic mechanical properties, highlighting the influence of the hierarchical structure. Generally, tensile properties exhibited lower values than compressive properties and axial samples were stronger than transverse samples. The anisotropy is more pronounced in tension compared to compression. This is in line with previous observations, that showed that in bovine bone the mineralized collagen fibrils (MCF) are mainly aligned with the longitudinal axis of the osteons.



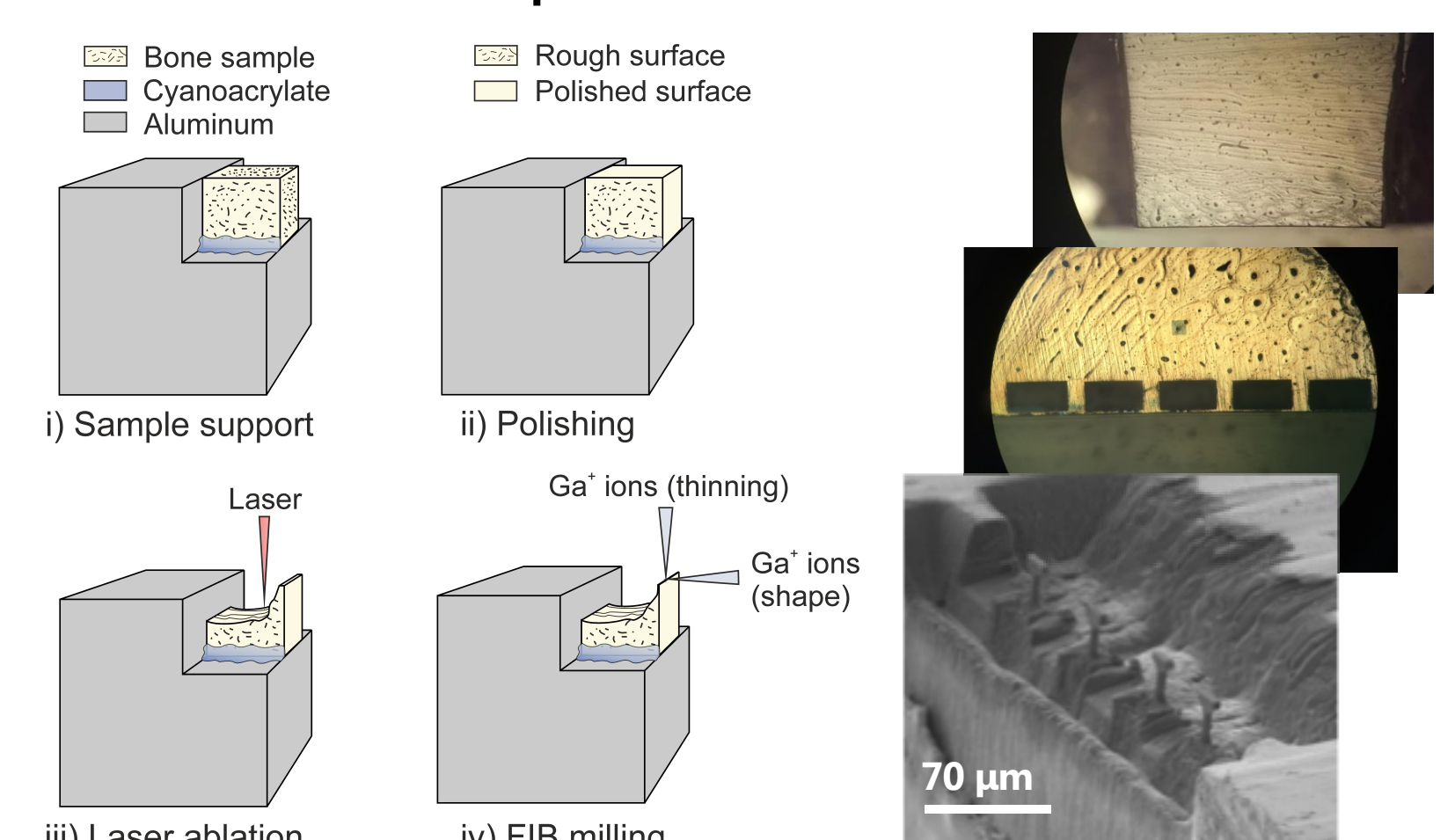
Scanning transmission microscopy on axial lamellar bone

2. Experimental

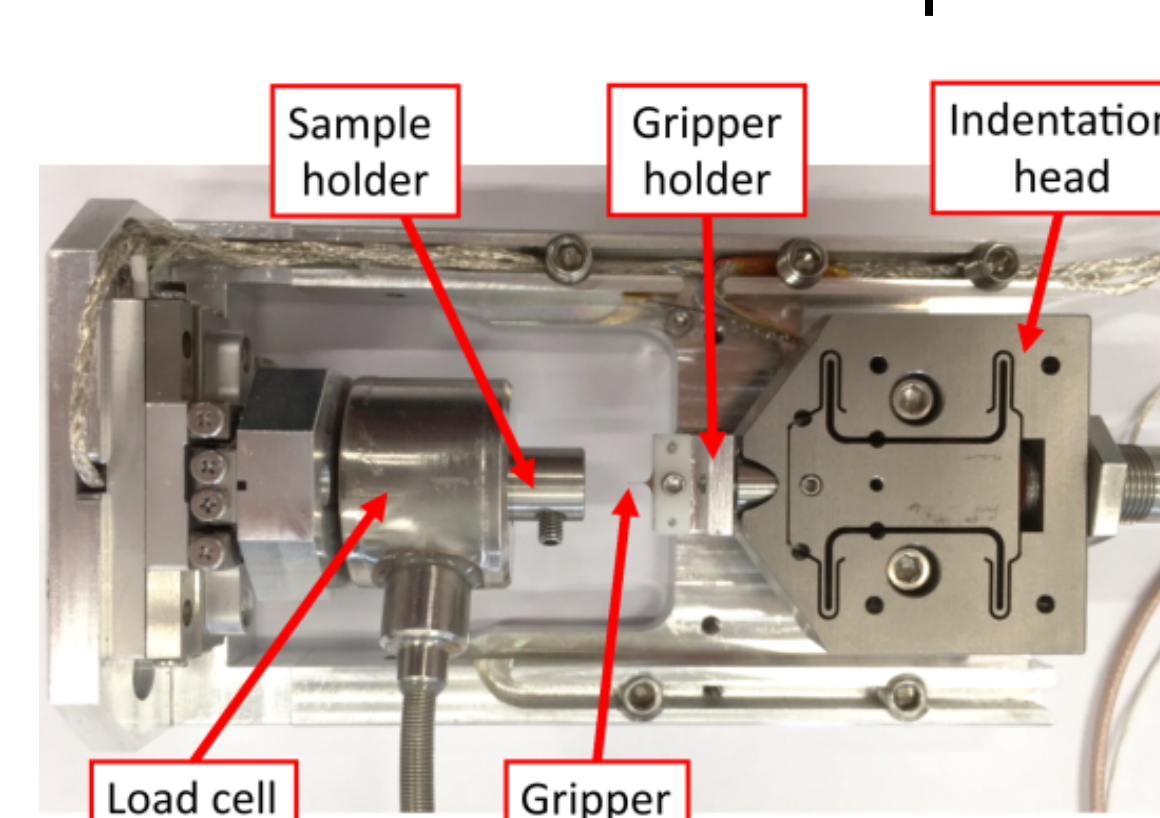
Sample design



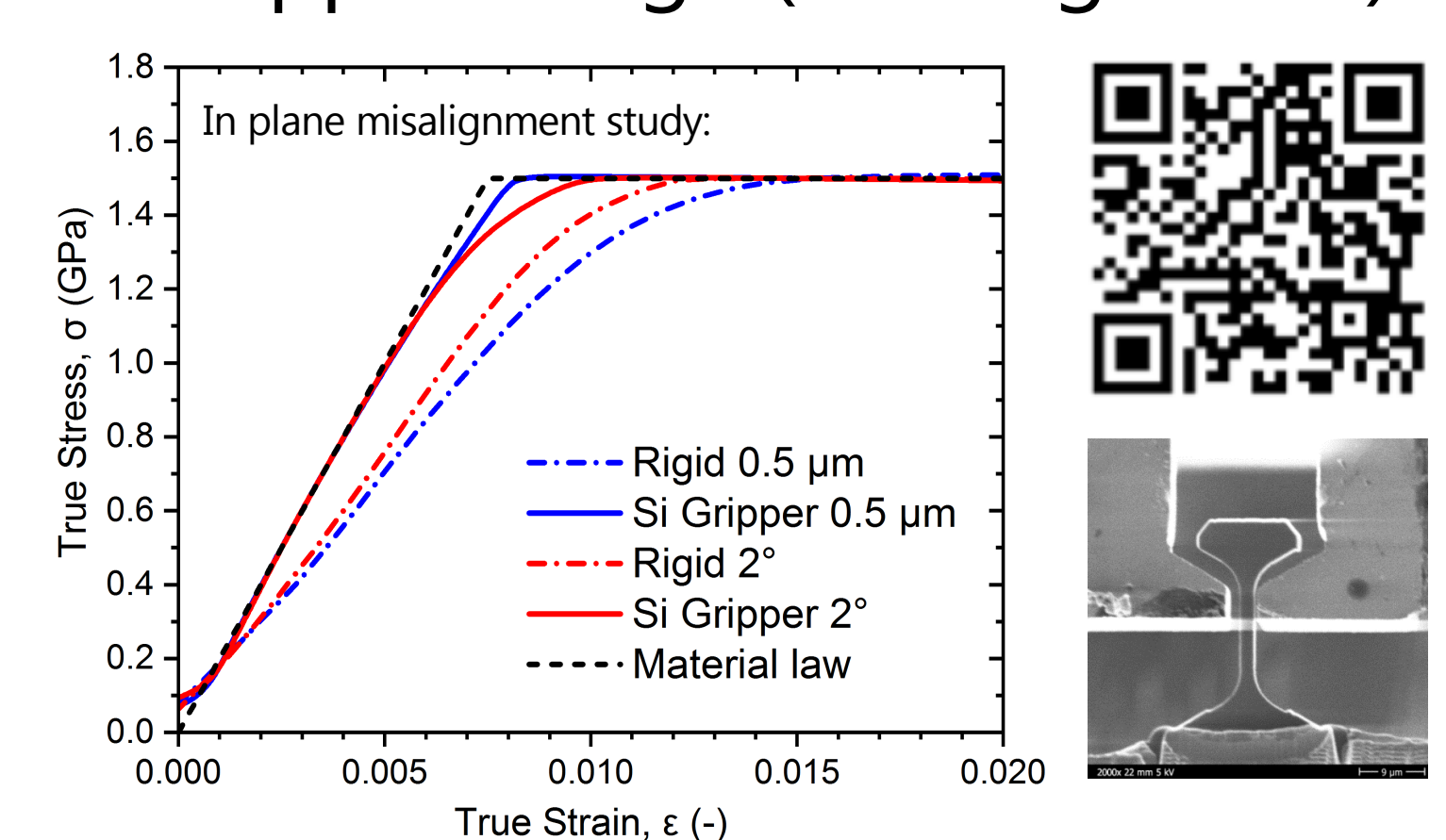
Sample fabrication



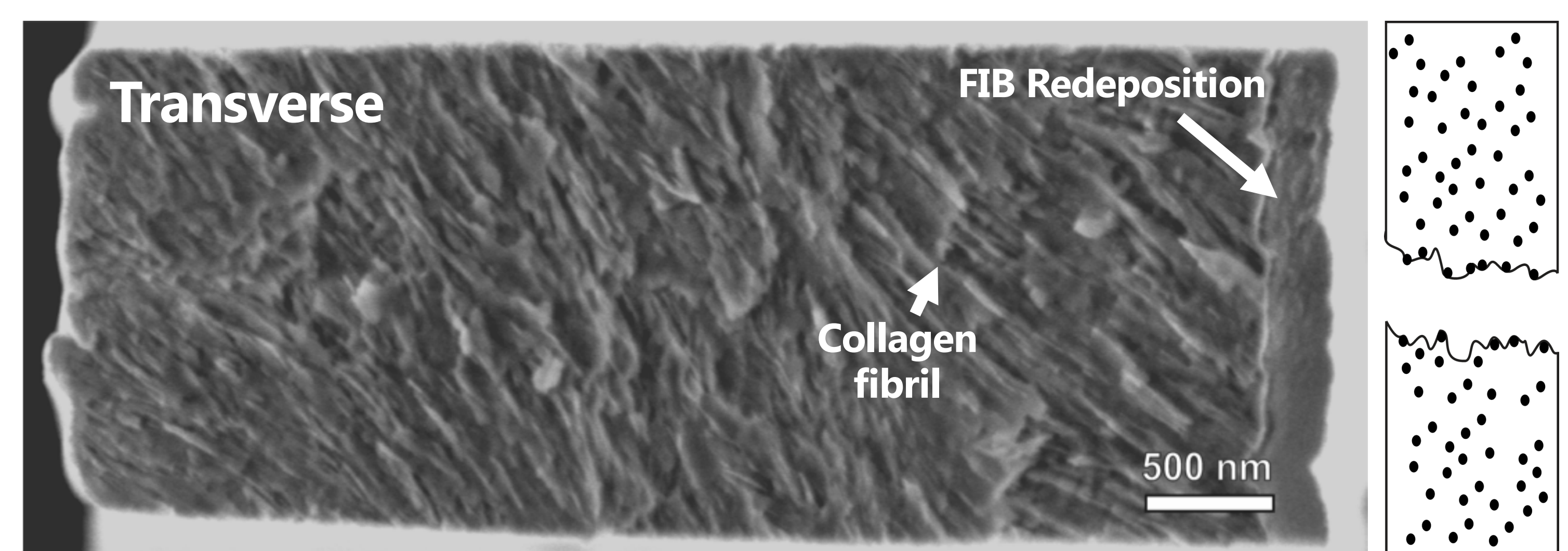
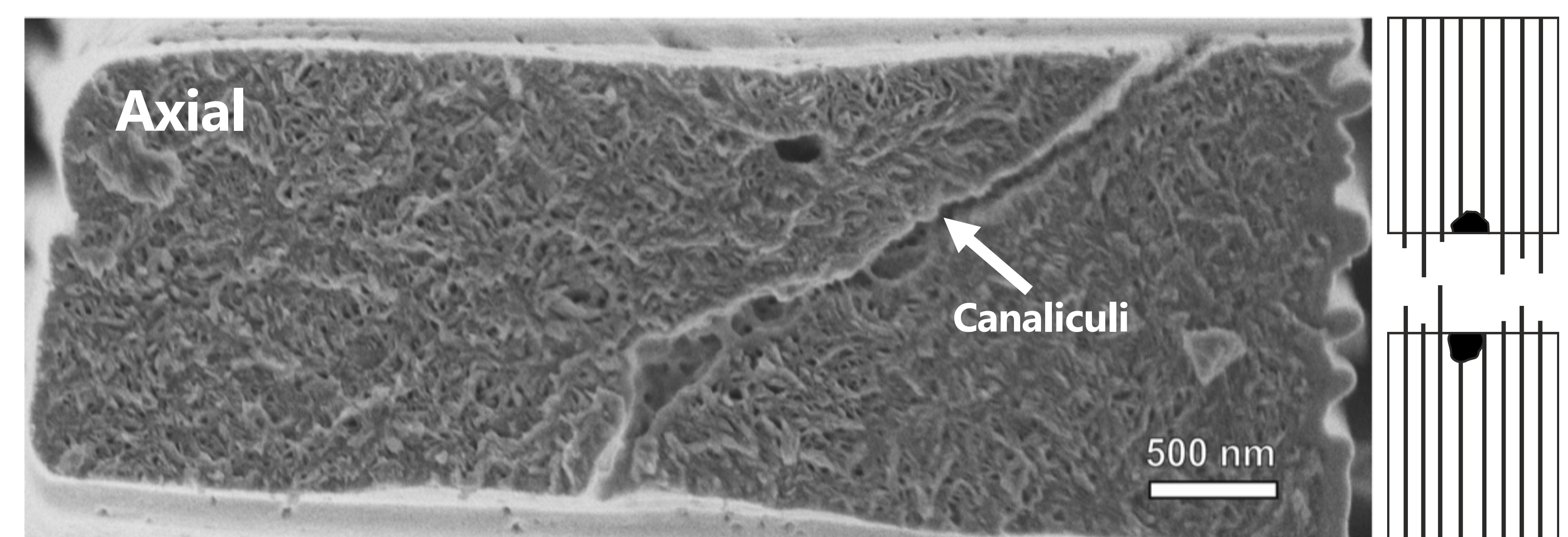
Microtensile setup



Gripper design (self-alignment)



4. Fracture surfaces



Two distinct failure modes were identified from the *in situ* observation and post-failure high resolution scanning electron micrographs. In the case of axial specimens, canaliculi were always found on the fracture surface, presumably acting as a stress concentration point and causing the failure. While observing brittle failure in dry conditions, the rough surfaces suggest MCF pull-out as a possible toughening mechanism. We expect to see this mechanism to be more pronounced in hydrated conditions since the MCF will be able to undergo larger deformations before failure [2]. Efforts are being undertaken to extend the testing methodology to probe lamellar bone in hydrated conditions. In transverse samples the MCF are oriented perpendicular to the applied load and extrafibrillar matrix (EFM)-fibrils debonding was observed as the principal failure behavior.

[2] J. Schwiedrzik et al. *Acta Biomaterialia*, 60, 302–314 (2017)

5. Conclusions

- Uniaxial microtensile properties of bone were characterized on the length scale of a single lamella using a new experimental setup.
- Tensile tests revealed size effects and anisotropy of the micromechanical properties of bone.
- Axial samples had consistent strength of 0.47 ± 0.03 GPa and their failure was associated to the presence of canaliculi.
- Transverse samples exhibited lower strength of 0.12 ± 0.03 GPa and showed interface failure between the MCF and the EFM.