## TENSION-COMPRESSION STRENGTH ASYMMETRY OF BONE EXTRACELLULAR MATRIX

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## Key Words: Lamellar bone, tension-compression asymmetry, deformation mechanisms

Bone features a hierarchical architecture, as a result of which antagonistic properties like toughness and strength are achieved. On the macroscale, bone exhibits a distinct anisotropy and loading mode dependence, with a considerably lower strength in tension compared to compression. To better understand the mechanisms leading to this behavior, anisotropic tensile yield and failure properties of ovine bone were characterized on the length scale of a single lamella (3-7 µm) and then compared to compression data for the same scale [1]. *In situ* microtensile experiments were carried out using an improved testing methodology, developed to overcome typical issues encountered during small scale testing related to sample fabrication, sample handling and misalignment [2]. The methodology is based on self-aligning silicon grippers prepared by means of reactive ion etching and an optimized microtensile sample geometry that can be fabricated via focused ion beam (FIB) milling. The measured elastic modulus, strength, yield stress and strain at maximum stress are summarized in table 1.

| Sample      | Loading         | E          | $\sigma^{max}$ | $\sigma^{y}$ | ε (σ <sup>max</sup> ) |
|-------------|-----------------|------------|----------------|--------------|-----------------------|
| Orientation | mode            | (GPa)      | (GPa)          | (GPa)        | (%)                   |
| Axial       | Compression     | 31.1 ± 6.5 | 0.75 ± 0.06    | 0.49 ±       | 5.4 ± 1.7             |
|             | (N=20)          |            |                | 0.10         |                       |
|             | Tension (N=10)  | 27.7 ± 3.4 | 0.35 ± 0.05    | -            | 1.8 ± 0.2             |
| Transverse  | Compression     | 16.5 ± 1.5 | 0.59 ± 0.04    | 0.30 ±       | 12.1 ± 2.5            |
|             | (N=19)          |            |                | 0.02         |                       |
|             | Tension (N =12) | 13.6 ± 1.1 | 0.13 ± 0.02    | -            | 1.3 ± 0.3             |
|             |                 |            |                |              |                       |

|  | Table 1 – | Micromechanical | data for | ovine | lamellar | bone. |
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Both compression and tensile experiments performed at quasistatic rates of 3x10<sup>-4</sup> s<sup>-1</sup> revealed a clear size effect with an increase in strength by a factor of 2.5 compared to macroscopic data. It was observed that strength anisotropy was more prominent in tension. Contrarily to compression, for which micropillars exhibited a plastic onset followed by shear failure at higher strains, microtensile specimens showed brittle failure associated with rough fracture surfaces. Fibril-matrix interface failure was found to be the dominant failure mechanism for both tension and compression. SEM imaging of micropillar cross sections suggest that failure during compression is related to the shear deformation of the extrafibrillar matrix for both sample orientations. As for tension, fracture surface analysis highlighted a change in failure mode: axial samples failed via interfacial shear (fibril pull-out), while the transverse sample showed fibril-matrix debonding. The change in failure mode observed in tension, however, not in compression, can explain the substantial difference in anisotropy seen when comparing strengths from the two loading modes. Finally, cross section analysis via scanning transmission electron microscopy (STEM) was used to develop a composite failure model based on fibril orientation. Interestingly, the highest tensile strength was found for fibrils oriented with an offset of 25° from the loading direction. The model fits well with the individual experiments and their respective fibril orientation measured by STEM. These results are the first reported measurements experimentally showing strength tensile-compression asymmetry of bone on the length scale of a single lamella. They highlight the importance of studying bone deformation mechanisms at several length scales to improve the understanding on bone mechanics. As the mechanical properties of bones strongly depend on hydration [3], a micromechanical setup for testing bone on the microscale in hydrated state is currently under development.

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- 2. Casari et al. J. Mater. Res. Accepted, 2019.
- 3. Schwiedrzik et al. *Acta Biomater.* 60: 302-314, 2017.