EXPERIMENTAL DESIGN FOR UNIAXIAL TENSILE MEASUREMENTS AT THE MICROSCALE

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Bone's unique combination of mechanical properties like strength, stiffness, toughness and low weight are the result of its complex hierarchical structure spanning several length scales. Despite extensive research on bone, there is still a lack of understanding on how its micromechanical behavior relates to its macroscopic failure behavior. While recent research has mostly utilized microcompression and nanoindentation, pure tensile testing at the lamellar level has not yet been reported. Nevertheless, critical failure events in bone are often attributed to tensile stresses. In this study a tensile experiment is designed using an in-situ micromechanical testing platform (Alemnis AG, Switzerland). The setup consists of a translatable silicon gripper driven by a piezo motor and a movable sample stage mounted atop a load cell. The setup is compact and can be installed within an electron microscope for in-situ testing.

Three main topics were considered in order to ensure a correct tensile test: sample fabrication, dimensioning and alignment. As the manipulation and gripping of a free-standing specimen of only a few µm in size could easily introduce pre-test damage, we utilize a sample design for which the tensile specimen remains attached to its original substrate. Finite element methods were employed for the optimization of the sample geometry with the aim of reducing stress concentrations at the connecting regions. A maximum tolerance of 5% stress deviation was chosen to select acceptable sample geometries. This value was chosen to be comparable with stress deviations present in samples conforming to ASTM D638 for tensile testing of polymers. Reduced linear hexahedral elements (C3D8R) were used as they allow a good compromise between accuracy and calculation time. Mesh convergence was verified for a mesh size of 0.05 µm near stress concentrations. In a second study, the gripper geometry (Figure 1, top) was also optimized to reduce stress deviation induced by any translational or tilt misalignment. To do so, both beam theory and finite element methods were employed to analyze the sample stress distribution for several gripper geometries. Computational time was drastically reduced by idealizing the thin, deflecting gripper section (Figure 1, top, in black), as an Euler-Bernoulli beam, thereby preserving the bending properties of the original complex shape as calculated analytically. The beam was coupled with the gripper end to guarantee a realistic interface with the sample. Quadratic elements (C3D20R and B32) were selected as they can accurately describe bending in both the sample and the silicon gripper. The gripper's geometry results in a tradeoff between translational and tilting misalignment sensitivity. A compliant gripper is tolerant to translation misalignment, but produces a stress concentration when the sample is tilted. For a stiff gripper the opposite is true. The ideal compromise to minimize misalignment sensitivity with the selected sample geometry is achieved when the ratio between the sample and the gripper stiffness is equal to 0.3. In this case, stress deviations within the sample are kept below 10% for translational and tilt misalignments up to 0.5 µm and 1°, respectively.

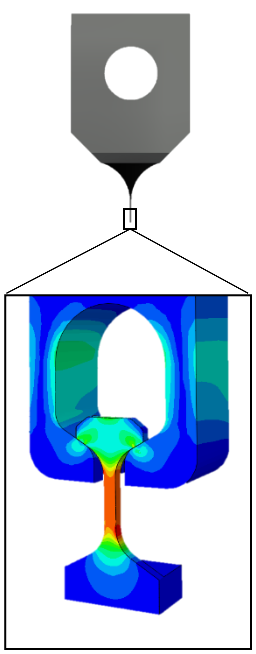

Figure 1 – Silicon gripper (top) and dumbbell bone sample (bottom)

A protocol for manufacturing the tensile specimens was developed. A bone cube with a side length of 3 mm is mechanically polished perpendicularly on two adjacent surfaces (front and top). Picosecond laser ablation is then used to remove most of the material behind the polished front surface, leaving a bone wall at the front of the cube. This wall is then thinned down and finally milled to the final desired dumbbell geometry using a focused ion beam. Tapering along the specimen thickness is avoided by milling with a 1° undercut on both the left and right sample edges. Preliminary tests on (100) silicon demonstrate the practicability of this fabrication step. The validation of the setup will be performed on nanocrystalline nickel, for which specimen size effects may be neglected due to the high number of grains present. Besides allowing the analysis of bone strength and yield stress at the microscale, the presented tensile device could be a useful tool for studying different types of ductile materials such as metals and polymers.