MICROCRACK EVOLUTION IN MICROINDENTATION OF OVINE CORTCAL BONE INVESTIGATED WITH TIME LAPSED SRμCT

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Introduction

Skeletal diseases such as osteoporosis constitute a serious socio-economic burden in aging societies. Patient specific tissue strength estimation could be helpful for personalised treatment strategies. Reference point indentation (RPI) [1] that derives from instrumented indentation [2] has been proposed. With imprints of several 100 µm at indentation depths of ~80 µm several structural units are affected. It is necessary to better understand the processes under the indenter tip but studies investigating the impact of larger indenters are currently missing. Combining high resolution imaging with *in situ* microindentation could help to investigate the zone underneath the indenter and observe emerging cracks. We aimed at (i) combining microindentations with synchrotron radiation micro-computed tomography (SRµCT) to investigate (ii) crack systems under two different indenters (iii) in axial or transverse testing direction.

Materials and Methods

Parallelepipeds of ~4x4x35 mm (n = 41) were cut from three ovine femoral diaphysis and dried at ambient temperature for 24 h. Four mm long axial and transverse subsections these were glued on 8x12 mm aluminium cylinders. The free end was lathed (Promac) to a 3 mm diameter and flattened using an ultramiller (Reichard-Jung). A custom made microindenter was implemented in beamline ID19 at ESRF that was equipped with a 30 µm high Berkovich or a 20 µm diameter spherical indenter. Samples were manually loaded with three ~10 µm deformation steps through a µm-screw followed by 300 s relaxation. A bespoke phase contrast scanning protocol (31 keV, 1499 projections, 40 mm sample-detector distance, 0.2 s shutter time) was developed to image the samples at 670 nm spatial resolution prior to loading, after each relaxation step, and after unloading. Force, time, and displacements were recorded. Image reconstruction was performed using ESRF software (pyHST) including Paganin phase retrieval. 3D images were reduced to 900x900x950 voxels. Bone tissue, indenter, and air were separated by a custom made software (Python, Simple ITK) that also delivered contact depth and area. Microcracks and pores were segmented with a previously developed segmentation algorithm [3]. Beam damage was investigated with post-test Raman analyses of the v_1PO_4, v_2PO_4, amid I and III bands and a control sample. Data was evaluated with Gnu R or python. Nonparametric tests to a significance level $p = 0.05$ were chosen due to the sample number.

Results

Twenty-eight specimens survived mounting, loading, and could be scanned yielding seven specimens in four groups (axial, transverse, Berkovich, Spherical). Cellular porosity under the tip decreased up to 26% except for transverse spherical indentations. Significantly more microcracks were produced in axial than in transverse direction. The Berkovich indenter produced significantly more cracks than the spherical indenter in axial but less in transverse direction. The X-ray beam significantly influenced the relaxation behaviour independent of the tip (Fig. 1). Raman analyses did not show significant changes in specimen composition after irradiation compared to non-irradiated tissue.

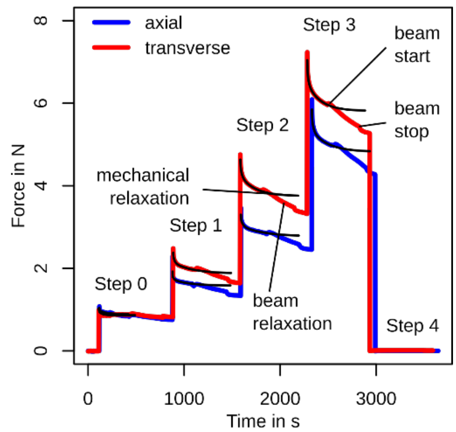

Figure 1: Force-time plots of two specimens illustrate the stepwise loading protocol. Mechanical relaxation is extrapolated to illustrate the behaviour without beam.

Conclusions

Time-lapsed microindentation tests were successfully combined with SRµCT imaging to study microcrack systems under the indenter tip. The X-ray irradiation relaxation suggests that the used SRµCT protocol was too harmful. Less detrimental protocols such as post-test scans of different microcrack states could be more beneficial. The data may help to improve our understanding of such indentation tests and their results.

References

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Acknowledgements

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