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Nanomechanical mapping of the osteochondral interface with contact resonance force microscopy and nanoindentation ☆

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ABSTRACT

The bone–cartilage, or osteochondral, interface resists remarkably high shear stresses and rarely fails, yet its mechanical characteristics are largely unknown. A complete understanding of this hierarchical system requires mechanical-property information at the length scales of both the interface and the connecting tissues. Here, we combined nanoindentation and atomic force microscopy (AFM) methods to investigate the multiscale mechanical properties across the osteochondral region. The nanoindentation modulus M ranged from that of the subchondral bone (M = 22.8 ± 1.8 GPa) to that of hyaline articular cartilage embedded in PMMA (M = 5.7 ± 1.0 GPa) across a narrow transition region <5 μm wide. Contact resonance force microscopy (CR-FM), which measures the frequency and quality factor of the AFM cantilever's vibrational resonance in contact mode, was used to determine the relative storage modulus and loss tangent of the osteochondral interface. With better spatial resolution than nanoindentation, CR-FM measurements indicated an even narrower interface width of 2.3 ± 1.2 μm. Furthermore, CR-FM revealed a 24% increase in the viscoelastic loss tangent from the articular calcified cartilage into the PMMA-embedded hyaline articular cartilage. Quantitative backscattered electron imaging provided complementary measurement of mineral content. Our results provide insight into the multiscale functionality of the osteochondral interface that will advance understanding of disease states such as osteoarthritis and aid in the development of biomimetic interfaces.

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1. Introduction

Within the articular joint, forces are transmitted across the mechanically dissimilar layers of tissue that comprise the osteochondral region: rigid subchondral bone (SCB), a thin (~50 μm to 100s μm) layer of articular calcified cartilage (ACC), and compliant hyaline articular cartilage (HAC). In vivo loading of the osteochondral region generates high stresses [1,2]. In particular, a stress concentration exists at the leading edge of mineralization, or the tidemark interface, between the mineralized ACC and the unmineralized HAC. Collagen fibers that traverse this interface perpendicularly are thought to dissipate and resist shear stresses [3]. The mineral within the ACC has long been thought to vary in mineral volume fraction to functionally grade properties from the SCB to the HAC [4]. Mineralization of the SCB and ACC often increases with age and altered loading conditions, and likely plays a key role in the development of disease states such as osteoarthritis [5–7]. However, our limited understanding of load transmission and mechanical properties across the osteochondral region restricts our ability both to understand disease progression and to engineer replacement materials [8].

Past investigations of the osteochondral region have focused primarily on bulk techniques [9,10] that test the combined mechanical response of several tissues (i.e. SCB, ACC and HAC). More recently, nanoindentation studies have probed the mechanical properties of these individual tissues [6,11–13]. However, the leading tidemark interface between ACC and HAC has been studied much less [11], primarily due to the micrometer-scale spatial resolution constraints of nanoindentation. Furthermore, to our knowledge, no studies have examined the spatial distribution of viscoelastic properties within the osteochondral region.

Atomic force microscopy (AFM) methods can provide nanometer-scale mechanical property measurements on a wide array of materials [14–16]. Here, we demonstrate contact resonance force microscopy (CR-FM) [17,18], an AFM method for quantitative mapping of viscoelastic properties across the tidemark interface. The results are compared to complementary information about microscale mechanical properties obtained by nanoindentation and to mineral content obtained by quantitative backscatter electron microscopy (qBSE) imaging. Our results provide new insight into multiscale mechanical properties of the osteochondral region that

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