Journal of the Mechanical Behavior of Biomedical Materials 74 (2017) 371–382

Contents lists available at ScienceDirect

Journal of the Mechanical Behavior of Biomedical Materials

journal homepage: www.elsevier.com/locate/jmbbm

The micro-damage process zone during transverse cortical bone fracture: No ears at crack growth initiation

Thomas Willett[a,c,*], David Josey[b], Rick Xing Ze Lu[b], Gagan Minhas[b], John Montesano[c]

[a] Systems Design Engineering, Biomedical Engineering Program, University of Waterloo, Waterloo, Ontario, Canada
[b] Nanotechnology Engineering, University of Waterloo, Waterloo, Ontario, Canada
[c] Mechanical and Mechatronics Engineering, University of Waterloo, Ontario, Canada

ARTICLE INFO

Keywords:
Cortical bone
Fracture toughness
J-integral
Micro-damage
Micro computed tomography
Process zone

ABSTRACT

Objective: Apply high-resolution benchtop micro-computed tomography (micro-CT) to gain greater understanding and knowledge of the formation of the micro-damage process zone formed during traverse fracture of cortical bone.

Methods: Bovine cortical bone was cut into single edge notch (bending) fracture testing specimens with the crack on the transverse plane and oriented to grow in the circumferential direction. We used a multi-specimen technique and deformed the specimens to various individual secant modulus loss levels (P-values) up to and including maximum load (Pmax). Next, the specimens were infiltrated with a $BaSO_4$ precipitation stain and scanned at 3.57-μm isotropic voxel size using a benchtop high resolution-micro-CT. Measurements of the micro-damage process zone volume, width and height were made. These were compared with the simple Irwin's process zone model and with finite element models. Electron and confocal microscopy confirmed the formation of $BaSO_4$ precipitate in micro-cracks and other porosity, and an interesting novel mechanism similar to tunneling.

Results: Measurable micro-damage was detected at low P values and the volume of the process zone increased according to a second order polynomial trend. Both width and height grew linearly up to Pmax, at which point the process zone cross-section (perpendicular to the plane of the crack) was almost circular on average with a radius of approximately 550 μm (approximately one quarter of the unbroken ligament thickness) and corresponding to the shape expected for a biological composite under plane stress conditions.

Conclusion: This study reports details of the micro-damage fracture process zone previously unreported for cortical bone. High-resolution micro-CT enables 3D visualization and measurement of the process zone and confirmation that the crack front edge and process zone are affected by microstructure. It is clear that the process zone for the specimens studied grows to be meaningfully large, confirming the need for the J-integral approach and it does not achieve steady state at Pmax in most specimens. With further development, this approach may become valuable towards better understanding the role of the process zone in cortical bone fracture and the effects of relevant modifications towards changes in fracture toughness in a cost effective way.

1. Introduction

The study of cortical bone fracture mechanics is widely understood to be critical towards better understanding skeletal fragility, fracture prediction and prevention, and the development of new therapeutics (Martin et al., 2015). Over the last couple of decades, significant work has been done to appropriately measure cortical bone fracture toughness and to understand the complex mechanisms that toughen bone and, conversely, lead to its fragility. In recent years, Robert Ritchie's group at UC Berkeley has highlighted the importance of using the non-linear fracture mechanics J-integral approach (Yang et al., 2006) and on including the R-curve behavior in experimental studies of cortical bone quality and the like (Nalla et al., 2005; Zimmermann et al., 2011). The J-integral approach is promoted for cortical bone because linear elastic fracture mechanic theory does not hold due to the fact that a non-negligible process zone of “plastic” deformation occurs around the crack tip and meaningful non-linear behavior is also noted during testing, particularly for transverse fracture (Martin et al., 2015; Woodside and Willett, 2016; Yan et al., 2007; Yang et al., 2006).

Cortical bone recruits multiple mechanisms to inhibit crack growth

* Corresponding author at: Systems Design Engineering, University of Waterloo, Waterloo, Ontario, Canada.
E-mail address: thomas.willett@uwaterloo.ca (T. Willett).

http://dx.doi.org/10.1016/j.jmbbm.2017.06.029
Received 18 March 2017; Received in revised form 19 June 2017; Accepted 22 June 2017
Available online 24 June 2017

(Launey et al., 2010). The relative importance of each of the toughening mechanisms is somewhat debated (Launey et al., 2010; Nalla et al., 2005; Ritchie et al., 2005). Take micro-crack and micro-damage formation as a specific example (Launey et al., 2010; Nalla et al., 2004; Vashishth et al., 2003). During cortical bone fracture, the phenomenon termed "constrained micro-cracking" occurs in the process zone ahead of the crack tip both before crack propagation (i.e. it is an intrinsic toughening mechanism) and, as the crack grows, a field of "constrained micro-cracking" is left in the wake (Launey et al., 2010; Nalla et al., 2004; Vashishth et al., 2003). This form of micro-cracking has a finer, more diffuse morphology (Poundarik et al., 2012; Poundarik and Vashishth, 2015; Sun et al., 2010) than the classic Frost-Burr micro-cracking, which has been intensely studied over the last several decades (Donahue and Galley, 2006; Taylor and Lee, 2003). This finer diffuse micro-cracking (herein furthermore referred to as 'micro-damage') is much smaller in scale, with lengths on the order of microns rather than the hundreds of μm reported for the Frost-Burr type (Poundarik et al., 2012). Both forms of micro-cracking can be formed in vivo and in vitro, but micro-damage may be the precursor to the larger micro-cracks and indeed fracture. It is associated with the apparent "yielding" of bone under tension in the longitudinal direction (Poundarik et al., 2012; Poundarik and Vashishth, 2015). It is thought to occur as mineralized collagen fibrils are stretched and small-scale fractures nucleate in the mineral phase (Poundarik et al., 2012; Poundarik and Vashishth, 2015). Many suspect, and some evidence supports, the idea that the collagen and other organics and their interface with the mineral phase contribute to controlling the micro-damage (Burton et al., 2014). While cracking of the hydroxyapatite-like mineral phase alone may not absorb much energy (Nalla et al., 2004), it is currently widely thought, though not entirely proven, that the accompanying deformation of the bone collagen (Gupta et al., 2006), and perhaps other organics such as the non-collagenous proteins (Poundarik et al., 2012), and even their debonding from the mineral (Gupta et al., 2007) absorb a significant amount of strain energy (Nalla et al., 2004).

The contribution of micro-damage formation in the process zone to fracture toughness depends on the size and shape of the process zone and the mechanisms that engage upon formation of the diffuse micro-damage within the process zone (Anderson, 2005; Launey et al., 2010). We know very little about these mechanisms, and, to date, the size and shape have been largely assumed based on general predictions from linear elastic fracture mechanics and simpler materials (Martin et al., 2015). A recent textbook portrays the zone as consisting of symmetrical "ears" that form on either side of the crack propagation direction (Martin et al., 2015).

This manuscript reports the results from our first attempts to study the micro-damage process zone in bovine cortical bone specimens undergoing transverse-circumferential fracture in Mode-I. The single edge notch bending configuration and high resolution micro-computed x-ray tomography (HR-micro-CT) combined with a $BaSO_4$ contrast agent were used. The aim was to increase our understanding of this important process in cortical bone fracture in order to improve the mechanistic study of skeletal fragility and the fidelity of computational modeling of cortical bone fracture.

2. Material and methods

Conveniently, the formation of diffuse micro-damage within the process zone enables the visualization and quantification of its dimensions. In the past decade or so, a few studies have reported using micro-CT with heavy metal stain contrast enhancement to study the accumulation of micro-cracking and micro-damage resulting from mechanical loading in bone specimens. (Tang and Vashishth, 2010) used lead uranyl acetate to study micro-cracks in trabecular bone. More recently, a more benign barium sulfate precipitation stain has found application in studies of cortical bone under quasi-static and fatigue loading in compression, tensile and even fatigue of notched bending specimens (Landrigan et al., 2011, 2010; Leng et al., 2008; Turnbull et al., 2011). Recently, (Choudhari et al., 2016) used the barium sulfate approach to study micro-damage formation in the vertebrae of tumorous spines from a rat model exposed to in vitro loading.

The studies mentioned above inspired the development of our own barium sulfate-based, contrast-enhanced, high-resolution micro-CT technique. We have used the technique to study the diffuse micro-damage within the fracture process zone of elastic-plastic fracture mechanics (J_{Ic}) qualified single edge notched bending fracture of cortical bone specimens undergoing Mode-I transverse plane-circumferentially directed fracture. We sought 3-D qualitative characterization and quantification of the dimensions of the process zone as the process zone developed up to and including crack growth initiation.

2.1. Specimen production

Consistent with our previous works, cortical bone was sourced from five tibiae of bovine steers (aged 1.5 – 2 years old) obtained immediately after slaughter from a local abattoir and processed as follows (Burton et al., 2014; Willett et al., 2015; Woodside and Willett, 2016). Steer bone is a useful model when developing new approaches, as in this project, because the bone is relatively more homogeneous, denser, and less porous than typical human cadaveric specimens. The bone was kept frozen (−20 °C) for up to 10 days following sourcing. Bones were thawed and stripped of all soft tissue. Using a morgue band saw, each tibiae was cut into two blocks approximately 70 mm × 25 mm × 6 mm; one mid-diaphysis anterior block and one mid-diaphysis posterior block. Each block was cut into four rectangular beams using an Isomet 1000 diamond wafer saw (Buehler Canada, Whitby, ON, Canada). The length was oriented along the longitudinal direction and the width in the radial direction. Beams were 60 mm × 4 mm × 4 mm (l × w × t). The endosteal side of the beam was marked to track orientation. The bone beams were ground and polished by hand to a 1-μm finish. Beams were stored at −20 °C while wrapped in saline soaked gauze.

The beams were prepared for single-edge notched bend (SEN(B)) fracture testing in three-point bending complying as closely as possible with requirements drawn from ASTM E1820 and ASTM D6068 and consistent with previous studies (Burton et al., 2014; Willett et al., 2015; Woodside and Willett, 2016; Yan et al., 2007). The beams were thawed to room temperature and a starter notch was cut to a depth of 1.9 mm at mid-span into one face in the circumferential direction on the transverse plane using a 300-μm diameter diamond wire saw (Delaware Diamond Knives, Wilmington, DE, USA). Orienting the starter notch in this way meant that the crack would propagate across any osteons, which run longitudinally, if present in the tissue.

Each crack was propagated in the circumferential direction in this study because less severe crack deflections occur relative to the radial direction where the cracks interact with lamellar interfaces and can deflect up to 90°. Thus, avoiding crack deflections simplified later measurements of the micro-damaged process zone. To further avoid severe crack deflections, side grooves aided in maintaining crack propagation direction on the transverse plane, which corresponds to the peak drive force. In previous work, specimens without side-grooves demonstrated significant deflections and crack branching (Woodside and Willett, 2016). Furthermore, side grooves promote a tri-axial ("plane strain") stress state (Anderson, 2005). Rounded side grooves (300-μm wide and 400-μm deep) were cut with the same 300-μm diameter diamond wire saw.

The starter notch was sharpened using an ultra-fine razor blade (McMaster-Carr, Elmhurst, IL, USA) lubricated with 1-μm diamond slurry (Buehler) to produce a total starter notch length of approximately 2 mm and ~ 5-μm tip radius (Burton et al., 2014; Willett et al., 2015; Woodside and Willett, 2016; Yan et al., 2007). While consistent with ASTM D6068, sharpening with a razor blade was also necessary because we have found that starter cracks induced from a blunt notch using fatigue loading deflect almost immediately due to the anisotropy of the