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Research paper

Mechanistic modeling of a nanoscratch test for determination of *in situ* toughness of bone

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ABSTRACT

The objective of this study was to develop a nanoscratch technique that can be used to measure the *in situ* toughness of bone at micro/nanostructural levels. Among the currently possible techniques, the surface scratch test may be conducted on very small regions, thus exhibiting a potential in determining the *in situ* failure behavior of materials. To adapt such a technique for assessing bone toughness at the micro/nanostructural levels and for limited stocks in small animal bone models (e.g. zebra finish and mice), a simple but reasonably accurate mechanistic model for the nanoscratch test was developed in this study. This model was based on the assumption that the removal energy of the tissue required during the nanoscratch test is the manifestation of the *in situ* toughness and the shear flow stress during the removal process is a measure of the *in situ* strength of bone. In addition, the experimental methodologies were developed to determine the elastic recovery force and frictional coefficients between the scratch tip and bone specimens that are required by the model. Finally, the efficacy of the nanoscratch technique was verified by testing bone samples from control (wild type), mild, and severe osteogenesis imperfecta (OI) mice, which have a distinct degree of brittleness. The experimental results indicated that the nanoscratch test could sensitively detect the *in situ* brittleness and strength of bone from the animal models.

1. Introduction

Bone is a highly hierarchical material (Fritsch et al., 2009; Nikolov and Raabe, 2008; Ritchie et al., 2004; Rubin et al., 2004), which is quasi-brittle in nature while allowing for appreciable plastic deformation (Cointry et al., 2005; Dong et al., 2010a,b; Peterlik et al., 2006). It is well known that the bulk behavior of bone is dependent on the mechanical behavior at local regions that are representative of its hierarchical structures (Gao et al., 2004; Gupta et al., 2006; Nyman et al., 2009; Yuan et al., 2010). However, measuring the bulk behavior alone may not provide enough information to discriminate the local changes at different structural levels (Currey, 2004; Leng et al., 2009; Nyman et al., 2010, 2009). In addition, due to the limited bone stock for small animal models (*e.g.*, mice and zebra fish) in bone research (Dong et al., 2010a,b; Ge et al., 2006; Hawse et al., 2008; Pennypacker et al., 2009; Wang et al., 2002) it is very difficult to use conventional mechanical

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testing techniques in assessing mechanical properties of small bone specimens available in these models. Thus, advanced techniques that can measure local properties of bone at submicron and even nanoscopic levels have become essential for addressing these issues.

Facing the challenge, recent development in nanotechnology (*e.g.*, nanoindentation) has allowed for the estimation of the stiffness and hardness of bone tissues at submicron and nanometer length scales (Lewis and Nyman, 2008). For instance, nanoindentation techniques have been utilized to examine the elastic, viscous (strain rate), plastic deformation of bone matrix in the vicinity of *in vivo* microcracks (Wang et al., 2002), and to assess the stiffness loss at the damaged region of bone (Diab et al., 2005). Similarly, elastic anisotropy has also been estimated by performing nanoindentation measurements in different orientations (Pennypacker et al., 2009). Moreover, nanoindentation techniques also have been extended to investigating time-dependent properties of osteonal lamella in human cortical bone samples (Hawse et al., 2008). Combining with other biochemical and structural properties, local material properties determined by nanoindentation have helped investigators to perform a more comprehensive assessment of the interdependence among bone quality, quantity, and fracture risk (Kruzic et al., 2009). Nonetheless, some issues still remain to fill the gap between the present knowledge and the actual mechanical behavior of bone (Mullins et al., 2007; Wang et al., 2007). One of the issues is how to estimate the strength of bone at local regions. In addition, measurement of the fracture toughness of local bone tissues is another challenging issue in bone research. In the past, the indentation fracture toughness measurements have been performed on the dehydrated bone samples using the technique for brittle materials (Kruzic et al., 2009; Mullins et al., 2007). However, such indentation approaches are not suitable for testing wet bone samples because no measurable cracks would be observed at the edges of indentations. Indeed, there exist significant limitations for the current techniques in measuring the local strength and toughness of bone. Addressing this issue, a pilot study by this group has indicated that nanoscratch tests may have a great potential for assessing the *in situ* energy dissipation during the post-yield and failure deformation (or toughness) of bone tissues (Wang et al., 2007).

Hence, the objective of this study was to establish a simple but reasonably accurate mechanistic model for the nanoscratch approach and to verify its efficacy in the quantitative assessment of the local mechanical properties of bone. This approach is based on the assumption that the local failure strength and local toughness of bone could be estimated by measuring the shear flow stress and energy dissipation during the removal process of bone tissue by scratching.

2. Analytical treatment

2.1. *Mechanistic model of the nanoscratch test*

Generally speaking, scratching consists of two concurrent actions: indentation and cutting. The indentation action leads to the elastoplastic deformation of the test material, whereas the cutting action results in the removal of material by the scratch tip. The outcome of the two actions is the formation of a scratch groove (Fig. 1). The typical profiles of the scratch groove in the longitudinal and cross-sectional directions are shown in Fig. 2, which were obtained using a nanoindentation system (Agilent Technologies, Santa Clara, CA).

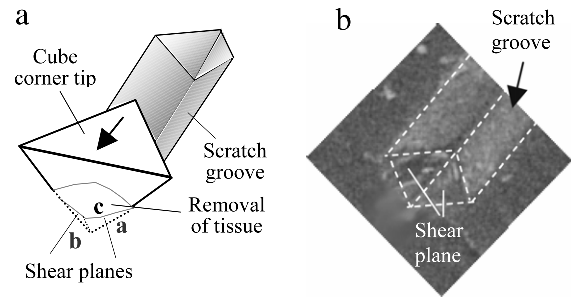

Fig. 1 – (a) Schematic representation of the nanoscratch test, showing the shear planes in front of the scratch tip; and (b) scanning electron microscopic image of the end of scratch groove, which shows the shape of the failure zone by the scratch process.

Based on the principle of cutting mechanics, the material in front of the scratch tip would be pushed out along a so-called shear plane (Fig. 1), where the shear stress reaches the maximum. For ductile materials, large plastic deformation would occur along the plane and the flow of the plastically deformed material eventually leads to the formation of a continuous cutting chip (plastic removal). In this case, the energy consumed during the chip formation is not equivalent to the toughness (*i.e.*, energy consumption till failure) because no failure is reached in this case. On the other hand, scratching on a brittle material would cause a random shattering of varying volume of the material in front of the tip, thus making it very difficult to quantify the energy dissipation during the random process. Different from ductile and brittle materials, bone is quasi-brittle and may sustain an appreciable plastic deformation (up to 10%) in compression (Leng et al., 2009; Nyman et al., 2009). Such a behavior would enable the formation of the shear plane, along which bone is deformed initially by plastic shear flow, but pushed out later by the scratch tip due to failure. In a previous pilot study by this group (Wang et al., 2007), the total work required to create a unit volume of the scratch groove (u_T) was used to estimate the toughness of bone:

$$u_T = \frac{W_T}{V_g}, \tag{1}$$

where, W_T is the work done during the scratch test and V_g is the volume of the scratch groove, which could be readily determined by the average cross sectional area and length of the scratch groove. However, the limitation of this approach is that it is only valid for relative comparisons of the *in situ* tissue toughness between samples.

To address this issue, this study proposed a mechanistic model to quantitatively measure the energy dissipation and the flow stress in the shear plane. The assumption was that during the scratch process limited plastic flow would occur along a shear plane, followed by the failure and removal of the tissue along the shear plane due to limited capability of