

Mechanical behaviour of porous hydroxyapatite

Li-Hong He^{a,b}, Owen C. Standard^c, Tiffany T.Y. Huang^{a,b}, Bruno A. Latella^d,
Michael V. Swain^{a,b,*}

^a Biomaterial Research Unit, Faculty of Dentistry, University of Sydney, Australia

^b Westmead Centre for Oral Health, Westmead Hospital, Westmead, Sydney, Australia

^c School of Materials Science and Engineering, The University of New South Wales, Australia

^d Australian Nuclear Science and Technology Organization, Australia

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Abstract

The aim of the study was to investigate the role of microstructure and porosity on the mechanical behaviour of sintered hydroxyapatite. Hydroxyapatite disks with four different porosities were used in this investigation. With a nanoindentation system, elastic modulus, hardness, contact stress–strain relationship, energy absorption and indentation creep behaviour were investigated. The elastic modulus and hardness of hydroxyapatite exhibited an exponential relationship (e^{-bP}) with the porosity P , which is similar to Rice's finding with the minimum solid area model. High porosity samples showed more substantial inelastic behaviour, including higher energy absorption, no linear elastic region in the contact stress–strain curve and some indentation creep behaviour. We conclude that porous microstructure endows hydroxyapatite with inelastic deformation properties, which are important in a material for bone substitution usage.

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1. Introduction

There is an increasing demand for synthetic bone and hard tissue replacement materials due to the lack of autograft materials and health risks of allografts. From biological and physiological perspectives, biomaterials for tissue replacement interact with the surrounding in vivo environment chemically, mechanically and morphologically. A successful biomaterial for reconstruction purposes must be similar in the above aspects to the tissue that it is replacing. Chemically, hydroxyapatite (HA) is the main inorganic component of human hard tissues, including bone and teeth. Morphologically, porous HA has been developed to mimic the porous nature of trabecular bone and has been widely used as a bone replacement material. The advantages of the porous structure are that it is light in

weight and that the porous network provides appropriate space (approximately $>50 \mu\text{m}$) for the ingrowth of the bone tissue and accelerates the replacement of the material by bone tissue [1,2]. Mechanically, the major shortcoming of HA is that the mechanical properties are not very comparable with bone tissue, especially the inelastic behaviour. On the one hand, HA has a much higher elastic modulus ($\sim 120 \text{ GPa}$ [3]) than bone ($<30 \text{ GPa}$ for trabecular bone [4]); on the other hand, HA is a typical brittle material while bone exhibits considerable inelastic behaviour [5,6]. The big difference in elastic modulus between HA and bone, and lack of inelastic ability of HA lead to greater stress concentration and fracture at the bone–material interface. Furthermore, from a materials science point of view, much is still unknown about the role of porosity on the properties of porous medical materials, including on what scale and in what form the pores influence the mechanical behaviour of the material, and how the biomechanical performance of porous medical materials can be improved by optimizing the microstructure.

* Corresponding author. Address: Biomaterial Research Unit, Faculty of Dentistry, University of Sydney, Australia. Tel./fax: +61 02 9351 8375. E-mail address: mswain@mail.usyd.edu.au (M.V. Swain).

Extensive research has been done on the mechanical properties of HA, including elastic modulus, hardness and strength, with respect to porosity and pore shape [7–11]. However, until now, no studies have investigated the influence of porosity on the contact deformational response of the porous materials, including the stress–strain, energy absorption and creep behaviour. In this paper, the mechanical behaviour of porous hydroxyapatite was examined using nanoindentation in order to determine the deformation mechanisms with respect to the microstructure. The aim of the work was to illustrate the role of porosity on the mechanical behaviour of the bulk materials.

2. Materials and methods

2.1. Manufacturing procedure

Commercial high-purity HA powder (E. Merck, D-6100 Darmstadt, Germany) was used as the starting material in this study. This powder has been characterized previously [12] and was shown to consist of agglomerates ($>2\ \mu\text{m}$ diameter) of nanometre-sized primary particles ($\sim 50\text{--}100\ \text{nm}$ diameter) with open pores between the primary particles. The HA powder was uniaxially pressed at 100 MPa in a hardened-steel die using a hydraulic press to form disks, each nominally 12.8 mm diameter and 3 mm thick. A saturated solution of stearic acid in acetone was used as a die lubricant, the die being cleaned and lubricated between pressings. Pressed disks were placed onto a layer of the HA powder on an alumina tile and then sintered in an electric furnace (Ceramic Engineering Furnace Manufacturers, Marrickville, NSW, Australia) at 900, 1100, 1200 or 1250 °C for 2 h in air to produce a range of bulk densities.

2.2. Density and porosity measurement

The bulk density, apparent solid density, apparent porosity and closed porosity of the sintered HA disks were determined by hydrostatic weighing according to AS1774.5 [13]. Briefly, the disks were dried at 110 °C and allowed to cool in a desiccator before their dry masses were measured. The disks were then infiltrated with deionized water under vacuum ($\sim 0.3\ \text{kPa}$) for 3 h. The mass of each specimen immersed in water was then recorded and the saturated mass was determined in air by carefully removing excess water from the surface with a damp cloth. The above density and porosity values were calculated from the dry, immersed and saturated masses and assuming a theoretical density of $3.16\ \text{g cm}^{-3}$ for the hydroxyapatite [14].

2.3. Surface preparation

Using an automatic machine (RotoPol-22, Struers, Copenhagen, Denmark), the surfaces of samples for microstructural characterization and nanoindentation testing were ground on successively finer grades of SiC paper, polished using successively finer grades of diamond paste and

finally polished with $0.05\ \mu\text{m}$ alumina paste. Following washing and drying, the samples were stored under laboratory conditions (room temperature, $\sim 40\%$ relative humidity).

2.4. Microstructural characterization

The microstructure of the sintered HA disks was examined by scanning electron microscopy (SEM; Philips XL-30, Netherlands). All samples were sputter-coated with gold and observed with a secondary electron detector. As-polished surfaces were used to examine the pore morphology of the sintered HA: pore size was calculated from micrographs as an equivalent diameter using image analysis. Fracture surfaces were used to examine grain structure: grain size was calculated from micrographs by the linear intercept method using image analysis.

2.5. Nanoindentation testing

Indentation experiments were performed using a nano-indentation system (Ultra Micro-Indentation System, UMIS-2000, CSIRO, Australia). Polished specimens were mounted on metal bases with wax using a paralleling machine (Leitz, Wetzlar, Germany). The mounting base contained a strong magnet to ensure stable contact with the UMIS test base.

Using a calibrated Berkovich indenter, force–displacement load–unload curves were measured for each specimen for maximum loads of 50, 100, 200 and 400 mN. At least five indents were made at each load level on each sample. Using the Oliver–Pharr method [15], the elastic modulus and hardness were calculated (by the UMIS software) as a function of penetration depth, h_p , for each indentation. It is known from finite element calculations that the Poisson's ratio of porous ceramics does not change significantly as a function of porosity [8] and, therefore, a constant value of 0.25 was used in all calculations.

Using a calibrated spherical-tipped indenter ($10\ \mu\text{m}$ nominal radius), force–displacement curves were measured for each specimen from 1 to 350 mN. In the case of the spherical contact condition, Tabor [16] showed that contact stress σ and strain ε_r can be estimated as $\sigma \approx H/3$, where H is the contact pressure, and $\varepsilon_r \approx 0.2 \tan \beta$, where β is the angle between the indenter flank and the original surface. At small depth for spherical contact ($a \ll R$), $\tan \beta \approx \sin \beta = a/R$, where a is the contact radius and R is the radius of spherical tip. Based on Tabor's empirical relationship and the more recent work by Lawn and colleagues [17–19], H – a/R curves can be used to indicate the stress–strain relationship of a material. In our previous paper [20], we developed a method to calculate the H – a/R relationship of a sample from its nanoindentation-derived force–displacement curves. Using this method, the H – a/R curves of the hydroxyapatite samples were determined from the force–displacement load curves, thus enabling

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