CHAPTER I

INTRODUCTION

1.1 Hydroxyapatite as Bone and Tooth Implants

The concept of implanting a material into living systems to replace bone or tooth defects due to diseases, natural abrasions, or accidents is attractive in prosthetic and dental surgeries. Generally the body reacts to the foreign materials to get rid of them. The implant must, therefore, be chemically and physically inert to the tissues in order to effect the healing processes of the wound : chemically, the implant must not induce biological rejection and must not give rise to toxicity problems ; physically, they should have properties similar to those of the materials which they are replacing.

Ceramic materials, even though being known to be brittle, show a high potential for using as implant materials. This is because they show excellent biocompatibility and a superior tribological behaviour compared to metallic and plastic biomaterials. At the beginning much attention has been focused on porous ceramics, since pores distributing in such ceramics can allow for tissue ingrowth for retention and fixation. Most ceramics that have received trials are such oxides as TiO_2, Al_2O_3, $BaTiO_3$, etc. However, such ceramic implants, due to lack of suitable porosity, cannot provide satisfactorily ingrowth of tissues in many areas of dentistry and orthopaedics. In this context, "hydroxyapatite" (hereafter referred to simply as HAP) , which belongs to a family of minerals crystallizing in hexagonal symmetry which are found extensively in nature, is emerged as a particularly attractive candidate. Being of similar compositions and crystalline structure to bone and teeth, HAP, $Ca_{10}(PO_4)_6(OH)_2$, may provide the possibility of calcified tissue regeneration following its implantation, and accordingly effecting a permanent repair. Extensive investigations of HAP implantation in animals and clinical trials with patients, such as implanting in animal legs, implanting for tooth root replacement and for filling alveolar ridge, etc., show that there are tissue ingrowth around the edges and bridge over the implant, thus resulting in a strong attachment of the implant to the defect tissues at the interface without inflammation (1-3). Thus HAP has been proven to be one of the most biocompatible biomaterial and able to provide a biological bond to bone or teeth.

Many methods have been proposed for the synthetic preparations of HAP powders in pure and homogeneous form. The conventional preparation methods, such as the precipitation reaction,*

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*The process is carried out at normal temperature in a medium of sufficiently high pH and is occurred according to the reaction (4)

$$5Ca^{++} + 4PO_4^{3-} + H_2O \rightleftharpoons Ca_5(OH)(PO_4)_3 + HPO_4^{--}$$

the hydrothermal reaction* and the solid state reaction,** etc., are, however, all required considerable apparatus and time. Recently Sombuthawee has proposed a preparation method in which HAP powders can be obtained by a relatively simpler procedure compared to those of the conventional methods. Sombuthawee method***(5) involves in the preparation of HAP powders from cattle bone and then fabricated into a polycrystalline ceramics by means of normal sintering. The HAP thus prepared by Sombuthawee method is called a cattle bone material. It is tremendously cheaper than HAP prepared by other methods, thereby being a promising candidate for low cost HAP implants.

However, the principal limitations in the ultimate usefulness of HAP is its brittleness. Incipient cracks, which act as stress concentrators, can easily be initiated, particularly in the highly stress region. The incidence of crackings, even on microscale, can cause tremendous strength degradation, or even to total failure, if extraneous stresses reach a level at which

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*The process is occurred according to the reaction (5)

$$10Ca(NO_3)_2 + 6KH_2PO_4 + 14NaOH \xrightarrow[10\ days]{500^\circ C\ 360\ atm} Ca_{10}(PO_4)_6(OH)_3 + 6KNO_3 + 14NaNO_3 + 12H_2O$$

**The process is occurred according to the reaction (5)

$$3Ca(H_2PO_4)_2 \cdot H_2O + 7CaCo_3 \xrightarrow[H_2O + N_2]{1200^\circ C/3\ hrs} Ca_{10}(PO_4)_6(OH)_2 + 8H_2O + 7CO_2$$

***Sombuthawee method is described in detail in Chap. 2.

unstable crack growth can occur. The chief problem in design of HAP as stress-bearing implants is accordingly the containment of catastrophic formation and growth of microcracks.

1.2 Strength Evaluation of Ceramic Materials

1.2.1 Conventional Strength Approach

Investigations over the past decade on fracture properties of ceramic materials have indicated that brittleness is an inherent property of ceramics. Brittle fracture is essentially an atomistic process in which cohesive bonds are ruptured at the tip of a growing crack. The stress necessary to cause such rupture varies from material to material according to the type of interatomic potential function. Crack propagation in these materials is also influenced by the present of grain boundries, clevage planes, etc. Therefore, these properties determine toughness, and hence the intrinsic strength, of ceramics. Practically, strengths are found to be at least two orders of magnitude less than the calculated intrinsic strengths. Griffith(6) was the first to recognize that cracks are responsible for the observed low strength in glass specimens. Griffith laid down two major precepts in his formalism of fracture mechanics : (i) equilibrium extension of well-developed cracks is governed by a balance between driving forces (associated with mechanical energy release) and resisting forces (associated with creation of crack surface area) ; (ii) such cracks start from flaws which act as stress concentrators in the stressed material. Thus a general conclusion to be drawn from the Griffith theory is that strength is a measure of both intrinsic (material) and extrinsic (flaw) parameters.

It is as a direct consequence of this dependence on extrinsic as well as intrinsic parameters that strength data obtained from the conventional strength test, in which as-received materials are stressed to failure in either uniaxial tensile tests or bending tests, tend to a high degree of scatter (naturally occurring cracks are widely dispersed in size). Since it is usually not possible to locate and observe the evolution of critical crack prior to failure (the density of cracks being large, and the size small), priori predictions of strength are not easily made. It is this problem which has led to the adoption of statistical method to describe flaw-size variations. Unfortunately, while of considerable use for design purposes, the probabilistic approach offers little physical insight into the actual flaw processes. A large scatter in data also makes it difficult to evaluate the role of material properties in the characterizations of strength.

1.2.2 Controlled - Strength Approach

Due to the above mentioned difficulties associated with the conventional strength evaluation, the controlled strength technique has now emerged as an attractive alternative route to strength evaluation. This technique involves the introduction of a dominant crack of controlled size and shape into the strength test specimen so that it acts as a fracture-initiating crack in the subsequent strength test. The capacity of predetermining the scale and location of the controlled crack is a key element in the fracture mechanics analysis, or the evolutions of critical crack can be followed at all stages. The obtained strength data are also accurate and reliable.

Several ways of introducing controlled cracks for strength testing have been investigated. Of these, indentation with a sharp, fixed-profile diamond pyramid (Vickers or Knoop) has emerged as the most practical, requiring only access to a routine hardness-testing facility (7). The indentation crack is characterized by a well-defined geometrical pattern and can be accurately positioned on any prospective test surface. Most important, its scale is readily controlled to a high degree of reproducibility, via the indentation load.

Recent studies on a wide range of glasses and ceramics have demonstrated that a key feature in indentation crack evolution is the vital contribution of the residual stress field resulting from elastic-plastic mismatch at the boundary of the plastic or deformation zone which encases the sharp point and edges of the indenter body. As well as being the dominant driving force during the formation of indentation crack, this residual stress field continues to influence indentation crack during any subsequent load applications, with consequent deleterious effect in the strength characteristics (8). Strength testing of specimens containing indentation cracks accordingly reflect on more than just fracture toughness, the material hardness, which quantifies the irreversible component of the indentation field, also enters the description. With proper account of the residual stresses into the fracture mechanics analysis, numerous recent studies (9-11) have demonstrated that indentation fructure mechanics is a powerful tool for investigating the fracture properties of brittle materials.

Although the indentation cracks are artificially introduced entities, there is considerable evidence that they do indeed simulate the essential qualities of naturally occurring surface cracks in ceramic component (12) : cracks introduced by scratching, surface finishing process, and spurious particle contact are just a few pertinent examples. Thus the observations of indentation-crack evolutions, from initial formation to ultimate failure, offer considerable physical insight into damage processes associated with surface contact history as well as provide the insight into micromechanics of strength degradation due to such damage.

1.3 Objectives and Scope of This Study

In the present work, we aim to study the strength properties of cattle bone material using indentation - controlled strength method. The reasons behind this study have actually been provided in the various parts of the previous sections. However, they are repointed out here as follows : (i) the cattle bone material appears to be a promising candidate for low cost HAP implants for prosthetic and dental surgeries ; (ii) due to the brittleness of material, a reliable strength evaluation and material characterization need to be developed if the cattle bone material (which is a

HAP) is to used as a stress-bearing implant; (iii) the indentation controlled strength method is chosen as a means for strength evaluation because not only it can give a reliable and accurate strength data, but also because it can offer considerable physical insight into damage processes associated with the formation of surface crack, which have a surface - contact history and provide some physical insight into the micromechanics of strength degradation caused by such surface flaws.

Therefore the works to be carried out in this study will be as follows :

(i) Cow bone powders will be prepared and verified if they are single-phase HAP by X-ray diffraction method. They will then be pressed into a shape suitable for the bending test and finally fabricated into a polycrystalline ceramic by means of normal sintering at different temperatures and time. The X-ray diffraction method will again be used to verify if any second phase occurs during sintering.

(ii) The effect of sintering temperature and time on characteristics of cattle bone material will be investigated. Scanning electron microscope will be used to characterize their microstructures and the techniques defined in American Society for Testing and Material (ASTM) specification will be used to measure their apparent porosity and density.

(iii) The four-point bending test fixtures will be designed and constructed in accordance with ASTM specifications so that precise measurements of the strength of cattle bone material can be made.

(iv) The damage pattern produced by a standard Vickers diamond pyramid indenter into test surfaces of cattle bone material will be observed so that considerable insight into damage processes associated with Vickers indentation can be obtained. A high magnification optical microscope in reflected light, will be used to observe the damage pattern appearing on the specimen surface and a dye penetrant technique will be used to reveal the subsurface crack profile. The dimensions of crack and central impression will also be measured as a function of indentation load. These data will be useful in the fracture mechanics analysis of the ultimate indentation - controlled strength data. Moreover, toughness and hardness parameters of cattle bone material may be evaluated from these data. The postindentation slow crack growth will also be examined to check if the cattle bone material is susceptible to environmental assisted slow crack growth.

(v) Four-point bending tests will be performed on the indented specimen and failure stresses recorded as a function of indentation load. Specimens subjected to different sintering temperatures and time as well as to surface abrasions with different sizes of abrasive powders will be used so that the effect of sintering variables and surface finishing processes on strength of cattle bone material can be obtained. Moreover, the evolution of indentation crack during bending test will be investigated so that the physical insight into micromechanics of strength degradation of cattle bone material due to the indentation crack can be obtained.

(vi) Fracture mechanics analysis of the indentation - controlled strength data will be made so that the toughness parameter of cattle bone material can be evaluated.

Accordingly, the thesis will be set out as follows. The preparation and characterization of cattle bone material will be described in Chapter 2. In Chapter 3, background theory of indentation fracture mechanics will be provided so that some appreciation of the basic fracture mechanics involved will be gained. The survey of responses of various ceramics to the Vickers indentation will be given before the actual work on Vickers-produced deformation/ fracture patterns in cattle bone material is presented in this chapter. Chapter 4 deals with the indentation-controlled strength tests on cattle bone materials.

Finally, conclusions from this study will be pointed out in Chapter 5.