CHAPTER 1

INTRODUCTION

1.1 Bioceramics

A BIOMATERIAL is defined as any substance, other than a drug, or combination of substances, synthetic or natural in original, which can be used for any period of time , as a whole or as a part of a system which treats, augments, or replaces any tissue, organ or function of the body. "

A BIOCERAMIC is defined as a ceramic used as a biomaterial. All materials elicit response from living tissues. Four types of responses are possible. (1) The material is toxic and the surrounding tissue dies. This material, obviously, should not be used as a biomaterial. (2) The material is non-toxic and biclogical inactive and a fibrous tissue capsule of varying thickness forms around the material, or in the case of a bone implant, the optical microscope rnay show direct apposition of bone to the material. In case of ceramics this could be classified as a Nearly lnert Bioceramic. (3) A third possibility. the materiai is non-toxic anc. biological active and an interfacial bond forms between the material and . tissue. Such a material would be referred to as a Surface-Active Biomaterial. (4) A fourth possibility would be that the material is non-toxic and dissolves with the surrounding tissue replacing the dissolved material. Such material would be classified as a Resorbable Biomaterial.

The potential of ceramics as biomaterials relies upon their compatibility with the physiological environment. The compatibility of bioceramics is the result of the fact that they can be composed of ions commonly found in the physiological environment (calcium, potassium, magnesium, sodium, etc.) and of other ions also showing very limited toxicity to body tissue (aluminum and titanium).

Inert bioceramics (oxides or carbon compositions) undergo little or no chemical change during long-term exposure to the physiological environment. Even in those cases where these bioceramics may undergo some long-term chemical or mechanical degradation, the concentration of degradation product in adjacent tissue is easily controlled by the body's natural

regulatory mechanisms. Tissue response to inert bioceramics involves the formation of a very thin, several micromeres or less, fibrous membrane surrounding the implant material.

1.2 Hydroxyapatite as Bioceramics

Bone and teeth are biologically created ceramics, therefore manmade,ceramics are being developed and increasingly applied as skeletal and dental replacements or augmentations. While porcelian enamels have a long history as dental replacements as early as the 1930s, it was not until the mid-1960s that significant research on modern ceramics as prosthetic materials was initiated. Ceramics continuely available for use in protheses and dental sugeries, e.g. Al_2O_3, ZrO_2, are attached to the host system through mechanical interlocking, thus resulting in the loosening problem due to elastic mismatch between the host bone and implant.

In1971, Monroe et al. (1) suggested hydroxyapatite ($Ca_{10}(PO_4)_6OH_2$) for use for bone and tooth implants. A major interest for hydroxyapatite (later will be referred simply as HAp) arises from the fact that HAp crystals are the inorganic constituent of vertebrate hard tissue, comprising 60 to 70 precent of bone and 98 percent of dental enamel. HAp has now been proven to be the most biocompatible material, nonbioresorbable, and surface active (2), i.e. bony tissue can bond directly onto the implant so giving excellent fixation. Thus, HAp is a challengeable bioceramics for being used as medical implants instead of metals, plastics and other ceramics conventionally available. These include the use for bone substitution, hip joints, middle ear reconstruction, jaw augmentation and tooth root replacement.

Due to its poor behaviour towards tensile stress, the applicability of HAp at present is restricted to the so-called small surgery such as oral, ear, subjected to mechanical loads no other than compressive stresses. Incipient cracks, which act as stress concentrators, can easily be initiated, particularly for loading situations in which considerable bending would be brought to be on the implant. The incidence of cracking,even on microscale, which cannot be seen by naked eyes, can cause tremendous strength degradation, or total failure if extraneous stresses reach a level at which unstable crack growth can occur (3). Understanding of how microcracks

evolve in HAp is critical to the prevention of catastrophic failure and to the development of HAp with optimal fracture properties.

The fracture toughness, K_{IC}, which is the most important parameter characterising the resistance of material to crack extension due to the many possible stresses impossed on the crack during its actual use, is presumed to be a single value quantity for HAp. This thus leads to a prediction that failure should occur spontaneously from some preexistentcrack, C_o , when a critical applied stress, σ_c, reaches, such that the strength varies to the inverse square root of the initial crack size $\sigma_o \sim K_{IC}/C_o^{1/2}$. This prediction along with the notion that the size of the strength controlling cracks generally scales with microstructural dimensions has resulted in a strong movement towards systematic efforts of achieving HAp with minimal grain sizes and porosity. However, even with HAp recently beingsintered to nearly full density (4), its tensile strength was still found to be only about 100 MPa which is still insufficient to be used for load-bearing situation where considerable bending and torsional forces would be brought to bear on the implant.

1.3 Objectives and Scope of This Work

In this thesis, fracture propertics of HAp will be examined, with the main aim of gaining a physical insight as to how strength properties may be improved through microstructure adjustments. The motivation comes from the recent discoveries that a wide range of ceramics exhibit the rising fracture toughness with crack extension, the so-called T-curve behaviour, rather than a single-valued quantity (5). In the ziconia-based ceramics, the principal factor established as the underlying T-curve mechanism is the phase transformation from tetragonal to monoclinic under the influence of the applied stress. The transformation events are generally taken to be confined within a process zone about the advancing crack tip. Such a process zone travels with the extending crack tip and thereby dissipates energy from the loading system (6-8). In simple monophase, nontransformable ceramics like alumina and glass ceramics, the principal mechanism of T-curve behaviour is grain-localized bridging at the crack interface behind the advancing crack tip. In this case, intact grains bridge the crack walls

and apply closure forces across the crack that shield the crack tip from the applied stress-intensity field. The cumulation of bridging tractions over the interface with continued propagation leads to a rising toughness curve (9). This recent realization that many ceramics display a rising toughness with continued crack extension rather than a single value toughness, gives an indication that in many cases a processing philosophy based on the achievement of ceramics of minimal grain size and porosity be reexamined. The magnitude and shape of the resultant T-curve are sensitive to the microstructure. This strong influence of microstructure leads to the potential for manipulation of crack resistance properties through controlled processing which can improve strength properties, by enhancing the toughening mechanism of the crack-interface bridging restraints.

Accordingly the scope of this thesis will be as follows:

(i) Some essential equipments for conductiving the processing and mechanical testing will be constructed.

(ii) HAp powders will be synthesized from the precipitation reaction between $Ca(NO_3)_2.4H_2O$ and $(NH_4)_2HPO_4$. The obtained powders will be identified by X-ray diffraction and fourier transform infrared spectrophotometer techniques.

(iii) The prepared powders will be single-end die press into a disc shaped pellet with dimensions suitable for the biaxial strength tests. The pellets will then be wet-bag isostatic press for the removal of defects produced in the single-end die press. The pellets will finally be densified by sintering at a suitable temperature and time.

(iv) The density of the obtained test piece will be measured using Archimedes principles. Its microstructure will be characterized using X-ray diffraction, fourier transform infrared spectrophotometer, and scanning electron techniques.

(v) The complete mechanical properties of the obtained test pieces will be evaluated using the indentation techniques.

(vi) An new set of test-pieces will be fabricated so as to obtain a more suitable microstructure. Examples of the possible modification are inclusion of special additives in the starting HAp powders and control heat treatments to promote desirable changes in the material

microstructure, e.g. elongated grains, or dispersing Y_2O_3-ZrO_2 uniformly in HAp matrix to promote phase-transformation tougthening.

(vii) This new set of test-pieces will be characterized and subjected to mechanical testings and evaluation for the strength properties.