

Chapter III

Experimental Procedure

This experiment concerned with inducing compressive surface stress of HAp by coating with CP glass that CP glass having a lower thermal expansion coefficient than the substrate. The experimental procedure consists of three parts as follow:

1. Preparation and ceramic processing of HAp
2. Preparation of CP glass
3. Strengthening of HAp by coating with CP glass

3.1 Preparation and Ceramic Processing of Hydroxyapatite

3.1.1. Preparation of HAp (MP)

MP was prepared by the method of Lorpayoon (1986). Selected parts of cattle leg bones were boiled in water to eliminate fat, calcined in an electric furnace at 700 °C for 3 hours to remove organic matters and ground with porcelain mortar to desired MP powder.

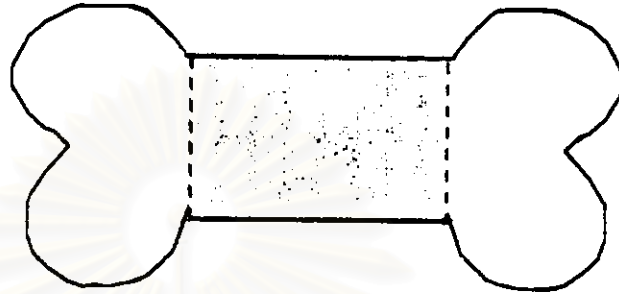


Fig. 3.1 Selected part of cattle leg bone

3.1.2. Attrition Milling of MP

The mixture of MP powder with 0.1% of dispex solution (as a dispersing agent), 0.5% of Zn stearate (as a lubricant) and 2% polyvinyl alcohol (as a binder) were put in an attrition mill with ZrO_2 balls in distilled water (water: powder = 1:1). Then the mixture was milled for 30 minutes at a speed of 600 rpm to obtain the desired powder.

3.1.3. Forming of MP

The MP obtained by attrition mill had a moisture content of about 15-20% and was a homogeneous matrix. It was aged over night and then extruded with a controlled rate of flow and pressure. The rod shape of MP had 6 cm in length and 0.45 cm in radius. The specimens were dried at 110 °C for 24 hours.

3.1.4 Calcination and Sintering of MP Rod Shape

MP rod shape specimens were calcined in an electric furnace to remove binder by heating up to 550 °C for 3 hours with a heating rate of 4 °C/min. After that all specimens were sintered at 1250 °C for 2 hours. Heating rate was 4 °C/min. The specimens were allowed to cool in the furnace.

3.2 Characterization of Hydroxyapatite

3.2.1 Chemical Composition and Impurities

The chemical composition and impurities of MP powder were analysed by XRF-borate fusion method. All of analyses were done by Mineral Assay and Services Co., Canada.

3.2.2 Phase Presence

The crystalline phase of HAp was detected by X-ray diffraction (XRD) method. The sample of milled MP powder was dried to remove moisture. Philips PW 1710 X-ray diffractometer used for identify crystalline phase using cobalt K_{α} radiation and iron filter. The intensity of 10° to $60^{\circ} 2\theta$ were measured with a scanning velocity of 0.05° per second.

3.2.3 Microstructure

Microstructure was observed by scanning electron microscopy (SEM) using a JEOL JSM 35 CF scanning electron microscope. The sintered specimens were cut a cross-sectional, etched with 0.25% H_3PO_4 for 20 seconds in an ultrasonic bath, washed with distilled water and dried. All specimens were sputtered with gold before placing on the target stub.

3.2.4 Water Absorption, Bulk Density and Apparent Porosity

The test method was based on ASTM test method C 373-72 (Reapproved 1982) in appendix B.

3.2.5 Thermal Expansion Coefficient

The coefficient of thermal expansion was measured by dilatometer using a Adamel Lhomary DI-24 dilatometer. Dilatometer was run from room temperature to 1250 °C with heating rate 10 °C per minute.

3.3 Strength Measurement of Hydroxyapatite

The sintered specimen were measured for their compressive and flexural strengths.

3.3.1 Compressive Strength

The compressive strength of MP sintered specimens was measured using Shimadzu Universal Testing Machine model DSS-10T with the loading rate of 0.5 mm/min. Sintered specimens were cut with the ratio of the length to the cross-sectional width of about 2.0, polished with abrasive paper, dried at 110 °C for 24 hours and kept in a desiccator before strength testing.

Compressive strength was calculated from the equation below as:

$$\text{Compressive strength (Kg/mm}^2\text{)} = \frac{\text{Applied load (Kg)}}{\text{Cross-sectional area of specimen (mm}^2\text{)}}$$

The unit Kg/mm² can be converted into MPa unit by multiply with 9.81

3.3.2 Flexural Strength

Three point flexural strength testing was determined by using the same testing machine for compressive strength. Sintered specimen was supported at the ends of span and load was applied at the center with the loading rate of 0.1 mm/min.

3.4 Preparation of Calcium Phosphate Glass

The glass formulation was determined from chemical composition of MP powder. Each kind of the mixture of CP glass was composed of MP powder, sodium metaphosphate and phosphorus-pentoxide in the fraction of glass the formula. The composition of CP glass was listed as follows:

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Table 3.1 Composition of CP glass

Glass composition	Bone ash (g)	$(\text{NaPO}_3)_n (\text{HPO}_3)_n$ (g)	P_2O_5 (g)
C1 (0.02 mole Na_2O)	49.47	3.71	46.82
C2 (0.03 mole Na_2O)	48.92	5.53	45.55
C3 (0.04 mole Na_2O)	48.37	7.39	44.24
C4 (0.10 mole Na_2O)	45.02	18.32	36.66
C5 (0.15 mole Na_2O)	42.28	27.31	30.41
C6 (0.18 mole Na_2O)	40.66	32.65	26.69
C7 (0.20 mole Na_2O)	39.57	36.20	24.23

Starting materials were mixed and put in a crucible, melted in the electric furnace at 1100°C for 1 hour. The molten glass was poured into graphite mold and annealed in an electric furnace at 480°C for 3 hours. After that the glass specimen was cut for thermal expansion measurement by a dilatometer.

3.5 Strengthening of Hydroxyapatite by Coating with Calcium

Phosphate Glass

In this research, MP sintered specimens and mixture of CP glass in crucible were put into the furnace and heated up to 1100°C for 1 hour. Coating process was carried out by dipping the hot sintered specimens in the molten glass. After coating, a group of the specimens was kept under annealing at about 20°C higher than the T_g of CP glass for 2 hours and the other group was allowed to cool to room temperature in the furnace without annealing.

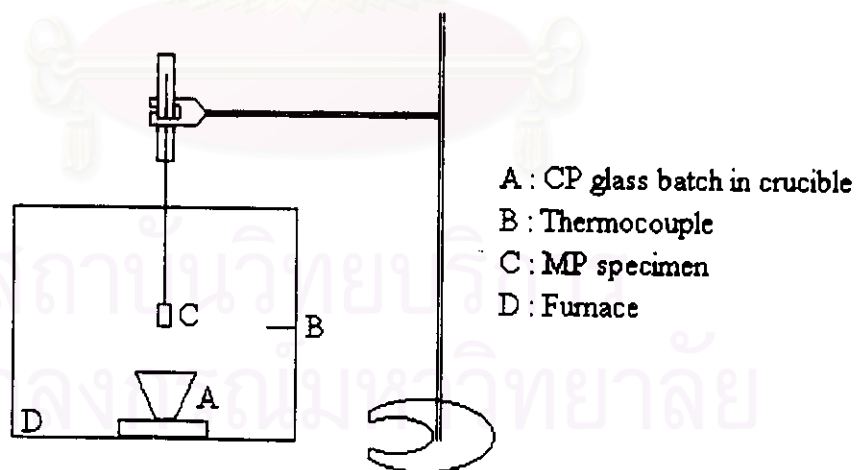


Fig.3.2 Illustration of coating process

3.6 Strength Measurement of Coated Hydroxyapatite

The compressive strength and flexural strength were measured by the same testing method as described in 3.3



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