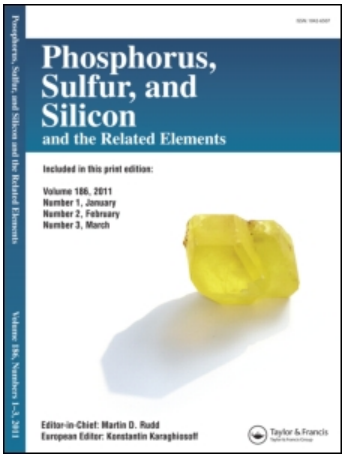

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PHYSICAL PROPERTIES OF CALCIUM PHOSPHATES GLASSES WITH VARIOUS CaO/P_2O_5 MOLE RATIOS

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Abstract

The physical properties of calcium phosphate glasses with various CaO/P_2O_5 mole ratios, Vicker's surface hardness, weight loss percentage after dipping Ringer's solution were investigated in this study. The best surface hardness of crystallized glass has an average hardness of 670Kg/mm^2 and the minmum weight loss percentage of crystallized glass were lower than 1%. The major crystalline phase that developed after heat treatment of these glasses was β-$Ca(PO_3)_2$ which characterized by X-ray powder diffraction method, and we found that the rod-like (3-5 μmD x 60 μmL) β-$Ca(PO_3)_2$ crystals from the photograph of SEM, the oxides composition of crystal was examined by EDS analysis also.

INTRODUCTION

Calcium phosphates ceramics are well known in bio-implant material applications because of their good biocompatibility, but the product of these ceramics is insufficient to make them useful as bone implant materials. Recently studies have been devoted to improving the mechanical properties of calcium phosphates.

β-$Ca(PO_3)_2$[1-2] fiber is expected to be for new composite-biomaterials. In this study, we tried to find the β-$Ca(PO_3)_2$ crystalline by changing the glass composition from traditional melting glass rod. The physical properties of calcium phosphates with various CaO/P_2O_5 mole ratios will be discussed.

MATERIALS AND METHODS

Batch mitures with CaO/P_2O_5 = O.73, 0.85, 1.0, 1.1 in mole ratio were prepared by using $Ca(H_2PO_4)_2 \cdot H_2O$, $CaCO_3$ and H_3PO_4. The composition of raw material were listed in Table 1. The mixtures were mixing with ethanol for 30 min. in a ball miller, after drying, the mixtures were melted for two stage in a platinum crucible at 850°C for 30 min.,and then 1250°C for 2 hour. The melts were poured onto a preheated graphite plate (280°C), then cooled to room temperature.

The glasses obtained from graphite plate were reheated at nucleation temperature maintaining 2 hours then at crystallization temperature for another various hours. The crystalline phases in the resultant products were identified by X-ray diffraction analysis (XRD).The morphologies of the crystalline were observed by scanning electron microscopy (SEM), and the chemical composition of crystalline were examined by EDS analysis. The physical properties such as Vicker's surface hardness, weight loss percentage after dipping in Ringer's solution for 14 days were examined also.

RESULTS AND DISCUSSION

The X-ray diffraction analysis of the resultant products, after various heat treatment procedure were showed as Figure 1, we found that the intensity of characteristic peak of β-$Ca(PO_3)_2$ enhancing with the duration time of heat treatment at crystal growth stage. The β-$Ca(PO_3)_2$ crystal was appeared at lower CaO/P_2O_5 mole ratio calcium phosphate glass.

The surface micrograph of the resultant product of glass D show as Figure 2. It was possessed the rod-like (3-5 μmD x 60 μmL) crystalline of β-$Ca(PO_3)_2$, the chemical composition of β-$Ca(PO_3)_2$ crystalline was examined by EDS, the energy-dispersive X-ray analysis of glass D was shown as Figure 3, the weight percentage composition of CaO/P_2O_5 was similared with the composition of β-$Ca(PO_3)_2$. We also found the glass-crystal interface show as Figure 4.

The Vicker's surface hardness of the resultant products after heat treatment were shown in Table 2. The Vicker's surface hardness of resultant product of glass C appeared the highest Vicker's surface hardness, 670 Kg/mm^2. On the other hand, the density of these glass-ceramics were between 1.7 to 2.5 g/cm^3.

The weight loss percentage of the resultant products after heat treatment procedure, then dipping in Ringer's solution for 14 days were shown as Table 3, the weight loss percentage of the resultant product of glass D has a big weight loss percentage, the maximum value is 38.3%, but the weight loss percentage of the resultant product of glass B was lower than 1%.

CONCLUSION

In this work, the following conclusions can be drawn:

(1) The major crystalline phase of glass D that developed after heat treatment procedure was β-$Ca(PO_3)_2$, the shape of β-$Ca(PO_3)_2$ crystalline are rod-like type with 5μm diameter and 60μm in length.

(2) We obtained the β-$Ca(PO_3)_2$ crystalline by traditional melting and heat treatment method.

(3) The heightest Vicker's surface hardness of these glasses resultant was 670 Kg/mm^2.

(4) The physical properties of these glass-ceramics are in sufficient to make them useful as bone implant materials.

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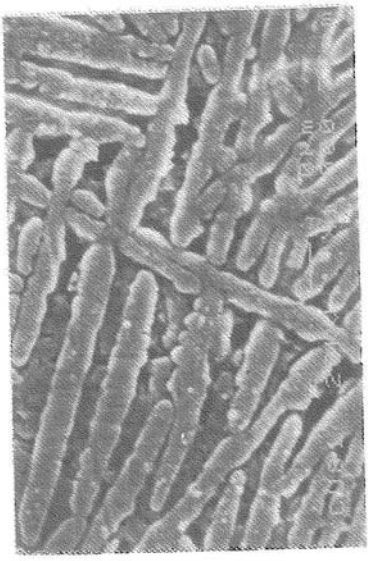

Figure 2. The surface micrograph of the resultant product of glass D.

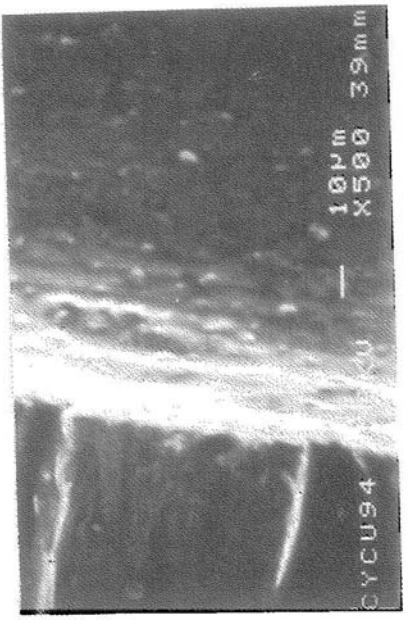

Figure 4. The glass-crystal interface of the resultant product of glass D.

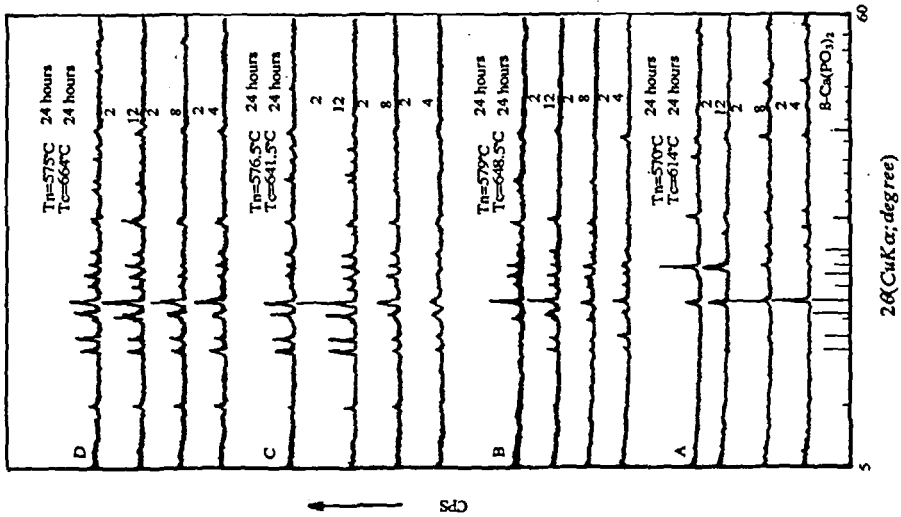

Figure 1. X-ray diffraction pattern of the resultant products.

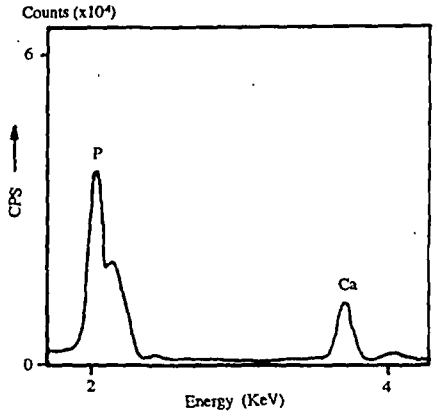

Figure 3. Energy-dispersive X-ray analysis of ß-$Ca(PO_3)_2$ crystalline

ELMT	APP.CONC	ERROR(WT%)
CaK: 0	3.433	.040
P K: 0	6.234	.091

ZAF CALCULATIONS

20.00 KV TITL= .00 ELEV =40.00 AZIM = .00 CONSIN =1.000

Spectrum: *INITIAL START-UP*

Last elmt by STOICH.,NORMALISED

ELMT	ZAF ratio	%ELMT	Error	ATOM.%	%OXIDE	FORMULA
CaK :	0.956	20.597+-	.240	11.324	28.820	CaO
P K :	1.151	31.062+-	.454	22.101	71.180	P_2O_5
O K :	0.000	48.341+-		66.575		
TOTAL		100.000		100.000	100.000	

Table 1 Composition of calcium phosphate glasses.

Glass	Composition		
	$Ca(H_2PO_4)\cdot H_2O$	$CaCO_3$	H_3PO_4
A	90.9	9.1	0
B	100	0	0
C	73.9	0	26.1
D	53.8	0	40.2

Table 2. Vicker's surface hardness and density of resultant product

Sample	Duration Time of Nucleation (hours)	T_n(°C)	Duration Time of Crystallization (hours)	T_c(°C)	Hardness (kg/mm^2)	Density (g/cm^3)
A	2	570	0	614	-	2.27
A	2		4		212	2.21
A	2		8		230	2.23
A	2		12		235	2.23
A	24		24		257	2.23
B	2	579	0	648.5	-	2.30
B	2		4		331	2.22
B	2		8		352	2.18
B	2		12		378	2.19
B	24		24		405	2.21
C	2	576.5	0	641.5	-	2.41
C	2		4		427	2.16
C	2		8		483	2.22
C	2		12		672	2.25
C	24		24		583	2.27
D	2	575	0	664	-	1.99
D	2		4		304	2.00
D	2		8		343	2.01
D	2		12		372	2.00
D	24		24		457	1.78

Table 3. Weight loss percentage of resultant product after dipping in Ringer's solution for 14 days

Sample	Duration Time of Nucleation (hours)	T_n(°C)	Duration Time of Crystallization (hours)	T_c(°C)	Wt. loss % of resultant product
A	2	570	4	614	2.2
A	2		8		5.3
A	2		12		4.3
A	24		24		9.7
B	2	579	4	648.5	0.8
B	2		8		0.6
B	2		12		0.6
B	24		24		0.2
C	2	576.5	4	641.5	6.2
C	2		8		8.5
C	2		12		8.1
C	24		24		7.1
D	2	575	4	664	23.7
D	2		8		27.0
D	2		12		21.4
D	24		24		38.3