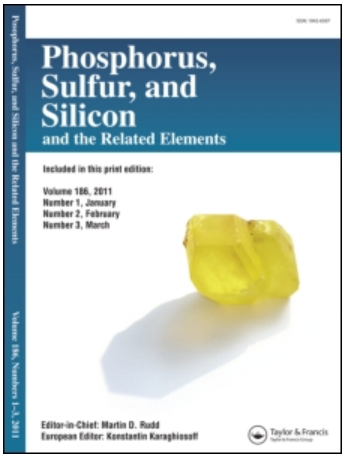

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Synthesis of Calcium Phosphates by Gas-Solid Reaction of CaO-P 2 O 5

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SYNTHESIS OF CALCIUM PHOSPHATES BY GAS-SOLID REACTION OF CaO-P_2O_5

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Calcium phosphates are useful materials in fertilizer, dentifrice, biomaterials, etc., and have been popularly synthesized by a wet method, a hydrothermal method, a flux method, and a solid-state reaction method. However, there has been no report on synthesis by chemical vapor deposition (CVD) method or gas-phase reaction.

In the present study, calcium phosphates were synthesized by gas-solid reaction of CaO solid and P_2O_5 gas. The CaO/P_2O_5 ratio of starting materials was changed from 5.06 to 0.64, and the reaction was carried out at 400–1100°C. These calcium phosphates were investigated using X-ray diffractometry (XRD) and scanning electron microscope (SEM). By gas-solid reaction of CaO-P_2O_5, β-MET only was synthesized at 400–500°C, β-MET, HA, and β-PYR at 600–700°C, and β-MET, HA, β-PYR, and β-TCP above 800°C. Synthesized calcium phosphates were independent on the CaO/P_2O_5 ratio of starting materials but dependent on the reaction temperature.

Keywords: Calcium phosphates; CaO-P_2O_5 system; gas-solid reaction

INTRODUCTION

Calcium phosphates are useful materials as fertilizer, dentifrice, biomaterials, etc., and have been popularly synthesized by a wet method, a hydrothermal method, a flux method, and a solid-state reaction method.[1,2] However, there has been no report on the synthesis of calcium

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phosphates by a chemical vapor deposition (CVD) method or gas-phase reaction, though the CVD has been applied to fabricate thin film in the electric field.

In the present study, calcium phosphates were synthesized by reacting CaO solid with P_2O_5 gas, which is vaporized above 350°C.

MATERIALS AND METHODS

Materials

CaO (99.9%) and P_2O_5 (98.0%) powder were used as starting materials. A reaction tube made of quartz (ϕ 13 mm × 100 mm) was used as a reaction chamber.

Methods

CaO and P_2O_5 powders with 5.06–0.64 of CaO/P_2O_5 were separately put into the reaction tube, as seen in Figure 1. The tube was heated at 400–1100°C in air for 1 hour.

After the reaction, all products were investigated using X-ray diffractometry (XRD, Rigaku Corp., RINT2200H), and scanning electron microscopy (SEM, JEOL, JSM-5310LVB).

RESULTS AND DISCUSSION

Figure 2 shows a SEM photo after the reaction by CaO/P_2O_5 with 0.64 at 900°C. In this photo, natural shape crystal cannot be seen.

Table I shows synthesized calcium phosphates by gas-solid reaction. β-calcium metaphosphate (β-MET) only was synthesized at 400–500°C, β-MET, hydroxyapatite (HA) and β-calcium pyrophosphate (β-PYR) at 600–700°C, and β-MET, HA, β-PYR, and β-tricalcium phosphate

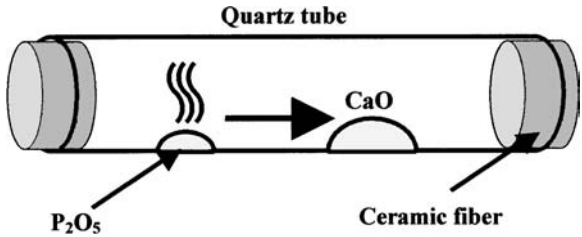

FIGURE 1 Scheme of reaction tube.

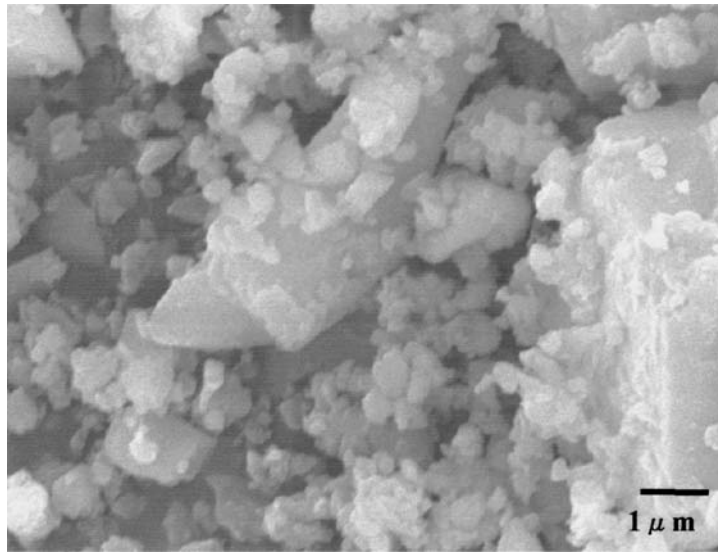

FIGURE 2 SEM photo of powder by gas-solid reaction.

(β-TCP) above 800°C. The calcium phosphates were independent on CaO/P_2O_5 of starting material. From these results, these reactions indicate as follows.

400–500°C: $CaO + P_2O_5 \rightarrow \beta$-MET
600–700°C: β-MET + $CaO \rightarrow \beta$-PYR
β-PYR + $CaO \rightarrow$ HA
800–1100°C: HA + β-PYR $\rightarrow \beta$-TCP

By these reactions, multilayers of calcium phosphates were formed above 800°C.

TABLE I Calcium Phosphates by Gas-Solid Reaction

	CaO/P_2O_5				
	5.06	2.54	1.26	0.84	0.64
400–500°C	●	●	●	●	●
600–700°C	●■▽	●■▽	●■▽	●■▽	●■▽
800°C	○▽	●■○▽	●■○▽	●■○▽	●■○▽
900°C	●■○▽	●■○▽	●■▽	●■○▽	●■○▽
1000°C	■○▽	●■○▽	●■○▽	●■○▽	■○▽
1100°C	■○▽	■○▽	■○▽	■○▽	■○▽

●: β-MET, ■: β-PYR, ○: β-TCP, ▽: HA.

CONCLUSIONS

- By gas-solid reaction of $CaO\text{-}P_2O_5$, β-MET only was synthesized at 400–500°C, β-MET, HA, and β-PYR at 600–700°C, and β-MET, HA, β-PYR, and β-TCP above 800°C.
- Calcium phosphates synthesized by gas-solid reaction were independent on CaO/P_2O_5 ratio of starting materials but dependent on reaction temperature.

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