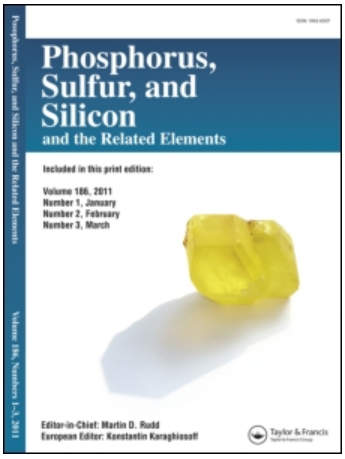

Phosphorus, Sulfur, and Silicon and the Related Elements

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

Directional Solidification of CaO-P205 Bioglass-Ceramics

J. C. Perng[a]; J. S. Lee[a]; C. W. Huang[a]
[a] Department of Chemistry, Chung-Yuan Christian University, Chung-Li, Taiwan

To cite this Article Perng, J. C. , Lee, J. S. and Huang, C. W.(1990) 'Directional Solidification of CaO-P205 Bioglass-Ceramics', Phosphorus, Sulfur, and Silicon and the Related Elements, 51: 1, 133 — 136
To link to this Article: DOI: 10.1080/10426509008040699
URL: http://dx.doi.org/10.1080/10426509008040699

Phosphorus, Sulfur, and Silicon, 1990, Vols. 51/52, pp. 133-136
Reprints available directly from the publisher
Photocopying permitted by license only

DIRECTIONAL SOLIDIFICATION OF CaO-P2O5 BIOGLASS-CERAMICS

J.C.PERNG , J.S.LEE and C.W.HUANG
Department of Chemistry, Chung-Yuan Christian University
Chung-Li, Taiwan, R.O.C.

Abstract Calcium phosphate glass-ceramics were prepared by reheating the glass rods between 675° and 750°C under a temperature gradient of ~100°C/cm.Floating zone method and silicon carbide furnace was made use to crystallizing the glass. The crystalline phases occurred as long fibers aligned parallel to the growth direction of the specimen.The spacings between the fibrous phases were smaller than 1 μm and very uniform.
Microstructure of the crystallized phases was examined by scanning electron microscope. Crystalline phases and crystallography were checked by X-ray diffraction.

1.INTRODUCTION

It was recently revealed by the present authors[1,3] that the directionally crystallized $Ca(PO_3)_2$ bioglass-ceramics have great potential of their high fracture strength and abrasion resistance. Biomaterials such as bone and dental implants must have both high mechanical strength and good biocompability with the tissue of the human body. These features can compensate for the low bending strength of other inorganic biomaterials, such as sintered hydroxyapatite and bioglass.[4]
When properly grown, directional solidification of eutectic ceramics have several intrinsic virtues: low porosity, microstructure stability up to temperatures approaching the eutectic temperature, and good bonding between the phases. The phases are uniformly distributed and their spacing, if small, might limit the size of microcracks. They are free from transverse grain boundaries which limit rupture strength.[7]
In this present study, formation of the unidirectional solidification glass-ceramic was investigated by observing the microstructure of the crystallized products. Crystallography was also studied by X-ray diffraction analysis.

2.Experimental methods

2.1.Starting Materials

The phase diagram of the system $CaO-P_2O_5$ as shown in Fig.1 reveals a eutectic at ~1280°C, a composition of 48 wt% CaO, and two terminal solid solutions. Mixtures with eutectic composition were made up from powders, e.q. $CaCO_3$ and $Ca(H_2PO_4)_2 \cdot H_2O$. Glasses were prepared by melting these raw materials at 1400° to 1500°C for 4 hours in a Pt crucible and subsequently quenching the melt. Rods about 8 cm in diameter were casted the melt into the graphite mold. X-ray analysis indicated the absence of crystalline phases in the quenched samples.

2.2.Decomposition and solidification method

Decomposition of the noncrystalline single phase into two crystalline phases may occur on reheating the quenched material to a temperature at which the rate of crystal growth is detectable. In general, the orientation of the crystalline decomposition products is random. We obtained aligned structures by decomposition in a temperature gradient.[5,6]

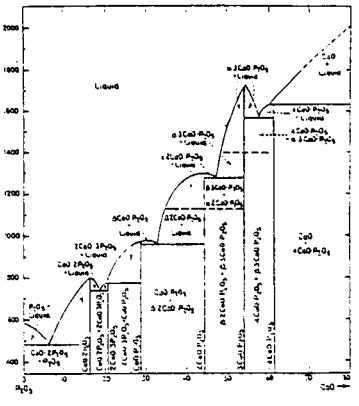

Fig.1.The phase diagram of the system $CaO-P_2O_5$

In eutectic solidification experiments the glass rod is moved from a region of low temperature to one of high temperature. The interior layout of solidification furnace was shown in Fig. 2. The thermal gradient of the furnace, determined with a PR13% thermocouple, could be varied from ~30 to ~150°C/cm. Nucleation occurs in the hot zone and the growth direction of the rods is towards the high temperature side. Provided the moving rate is not too high, there will be some temperature in the gradient, where the growth rate equals the moving rate of the rod.

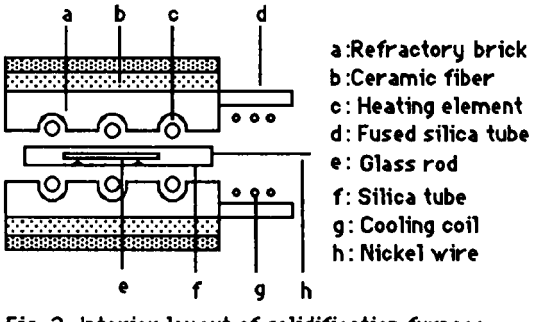

Fig. 2. Interior layout of solidification furnace

2.3.Instrumental analysis

The resulting rods were sectioned, polished, and etched with dilute HCl for metallographic and scanning electron microscopy. X-ray diffraction technique method was used to study relative orientation

3. Result and discussion

The examination of the microstructures which were produced confirmed the composition of the eutectic at 51.2 wt% P_2O_5 for which the best results were obtained and for which the lowest liquid temperature was observed. Well aligned structures were obtained even at high pulling rates (41mm/hr) indicating the presence at rate of a flat solid-liquid interface on the microscopic scale. The fact that even at off-eutectic compositions aligned microstructure were observed.
For moving rates too high (>45mm/hr), the parallelism of the lamellar crystals was lost, which means that the velocity of the transformation isotherm could not keep up with the moving rate. In the region where parallelism is obtained, the velocity of the transformation isotherm may be higher than the moving rate.

3.1. Growth rate(R) and Temperature gradient(G)

An increase in G/R markedly affects the overall microstructure of a eutectic because an increase in G or a decrease in R decreases the constitutional supercooling. The fully aligned microstructure desired for directionally solidification eutectics can be obtained only when solidification occurs under plane-front condition. Plane front growth occurs when there is no supercooling and, on a microscopic scale, the interface advances as a plane surface.

3.2. Eutectic microstructures

All these eutectic specimens exhibited a polycrystalline lamellar structure that consisted of rod like $2CaO.P_2O_5$ and $3CaO.P_2O_5$ phases. The rods were aligned parallel to the growth direction in the center portion of each sample as shown in the longitudinal section of Fig.3. The lamellar interfaces of both types as shown in Fig.3 A and B were observed in longitudinal sections perpendicular to the growth axis of the specimen.
Two kinds of lamellar structure were observed in this figure. The grain about 3 and 1μm in diameter were observed on opposite side of the center of the specimen. Eutectic grain had boundaries but no growth defect such as mismatch boundaries and terminations were observed on longitudinal sections of all specimens. There were the larger in diameter and the smaller in lamellar spacing of these crystal grains.

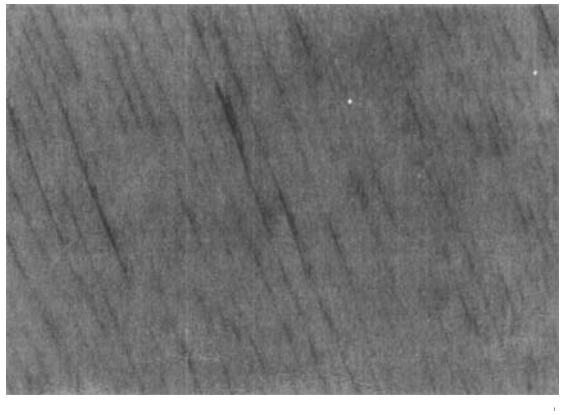

Fig.3. The longitudinal sections prependicular tothe growth axis of the specimen.(X1200,by SEM)

3.3.Crystallographic orientation

The crystallographic orientation of the calcium phosphate crystal of the unidirectional solidification specimens was determined by an X-ray diffraction technique. Ploished specimens cut parallel and perpendicular to the pulling axis were irradiated with a copper Ka beam of X-ray. As the same time, the specimen of bulk crystallization with random orientation crystals were detected.

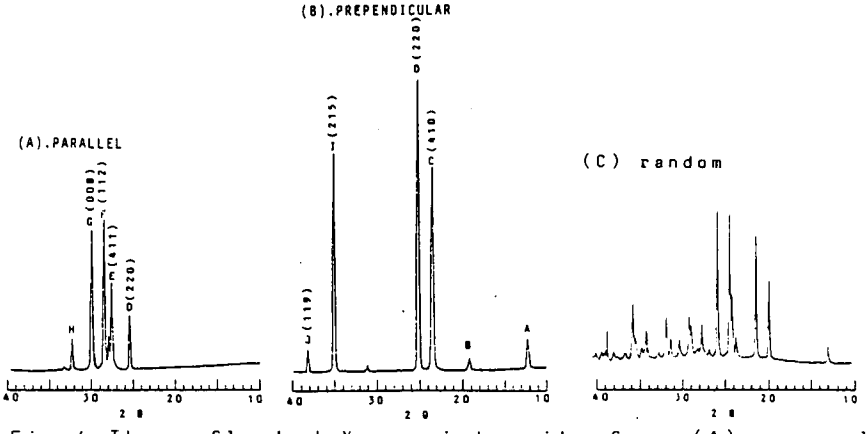

Fig.4.The reflected X-ray intensity from (A) a sample cut parallel and (B) perpendicular to the pulling axis and (C) random diffraction of powder sample.

Fig.4 A and B are part of the reflected X-ray intensity from specimen cut parallel and perpendicular to the pulling axis. The diffracted intensity from a specimen with a random arrangement of crystals was shown in Fig.4 C,the Bragg reflections from this specimen are less intense because a lower crystallinity of this sample was irradiated compared with the directional solidified sample.[2]

A comparison of Fig.4A and 4B shows that the reflection index are all different except the index (220). The sum of these reflections equal the random reflections of Fig.4C. A consideration of these data show that the calcium phosphate crystals are crystallographically aligned with one direction.

4.Conclusion

We have produced calcium phosphate bioglass-ceramics by unidirectional solidification which had high mechanical strength by fibrous crystals.

Reference

1.F.M.A.Carry and W.A.Cense,J. Crystal Growth,24/25(1974)551.
2.D.I.H.Atkinson and P.W.Mcmillan,J. Mater. Sci.,12(1977)443.
3.D.Michel, Y.Rouaux and M.Perez y jorba,ibid.,15(1980)61.
4.Y.Abe, M.Hose and T.Kasuga,Commun. Am. Ceram. Soc.,C-189(1982).
5.Y.Abe, T.Kasuga and H. Hosono,ibid.,C-142(1984).
6.F.L.Kennard, R.C.Bradt and V.S.Stubican,J. Am. Ceram. Soc.,59[3-4]566(1973).
7.R.L.Ashbrook,ibid.60[9-10]428(1977).