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Characterization of Hydroxyapatite Blocks for Biomedical Applications

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1. Introduction

There is an increasing demand for materials to be used in biomedical and dental applications. These materials are currently implemented in different forms, depending on the part of the body which needs repair. Biocompatibility, biofunctionality, and availability are three significant factors in selecting materials (L.L. Hench, 1998).

Historically, ceramics are the oldest materials in medical applications. Tricalcium phosphate (TCP) was used for repairing bone defects in the early 20th century (M. Bohner, 2000; T. Cuneyt, et al. 1997; J.G.J. Peelen, et al. 1987). Although, ceramics are brittle by nature, they have excellent compressive strength and a high wear resistance. Calcium phosphate compounds such as hydroxyapatite (HA), tricalcium phosphate (TCP), dicalcium phosphate dihydrate (DCPD), dicalcium phosphate anhydrous (DCPA), and tetracalcium phosphate (TTCP) (L.L. Hench, 1998; M. Bohner, 2000; T. Cuneyt, et al. 1997; J.G.J. Peelen, et al. 1987; C. Laverinia & J.M. Schoenung, 1991; M. Komath, et al. 2000; S.Takagi, et al. 1998) have almost the same chemical compositions as bone minerals. When these compounds are implanted into the living body (*in vivo*) for a period of time, they create a strong chemical bond with bone tissue (P. Luo, et al. 1998; H.H. Pham, et al. 1999).

In replacing bone defects, besides all compatibility parameters, the material should possess the same porosity as the bone. Bone has a complex structure with macro- and micro-pores. Pores are mostly interconnected to allow body fluids to carry nutrients and provide a medium where interfacial reactions between hard tissue and soft tissue can occur. An implant material should generally present similar properties to that of the bone. However, mechanical requirements dictate a high strength for implants which is associated with the elimination of some pores from them. As a result, reducing the porosity should result in an increase on the mechanical properties of HA as with any other ceramics. It is therefore important to find an optimum porosity to maintain the mechanical strength while pores provide the bone implant with an acceptable channel for nutrition to obtain the best implant properties (M. Jarcho, 1981; J.C. Le Hiec, et al. 1995).

The aim of the present work was to find the effect of sintering temperature on the microstructure, phase composition and the mechanical properties of hydroxyapatite ceramics.

2. Materials and methods

Medical grade hydroxyapatite powder was obtained from Sigma-Aldrich Chemical Company. The density of the powder was measured as 2.91 g/cm³ using ACCU-PYC 1330 (Micromeritics Gemini 2375). In order to determine the sintering temperature, a dilatometry test was performed. Also, the X-ray diffraction technique was employed using a Siemens, D500 diffractometer at each sintering temperature to estimate the probable phase transformations and the upper limit of the decomposition temperature. The particle size distribution was measured using a Fritsch Analysette22 system. The starting powder was uniaxially compacted at 86 MPa to form cylindrical shaped samples 55* 13 mm. The sintering was performed in air at 700-1300 °C with 1 hour soaking time. The rate of temperature increase was 10 °Kh⁻¹ while the cooling was carried out in the furnace. The mechanical properties of sintered bodies were examined by 3 and 4 point bending techniques using an Instron Universal Testing Machine 1196 was used with a cross head speed of 0.5 mm/min and maximum load application of 5 kN. Vickers hardness of specimens was measured using Vickers hardness testing device (Hardness Tester Akashi AVK cll) under 300 g loading at 20 seconds. Fracture toughness also was determined using Evans & Charles equation. The microstructures of the samples were studied under different sintering conditions using a Cambridge Stereosacn 360 scanning electron microscope.

3. Results and discussion

Figure 1 shows the particle size distribution. It is evident that the average size of the powder was around 4 micrometre. The particle size and specific surface area of the starting powder are the most important parameters affecting the sintering behavior of ceramics. By reducing the particle size and increasing the specific surface area, the same degree of sintering can be achieved at much lower temperatures.
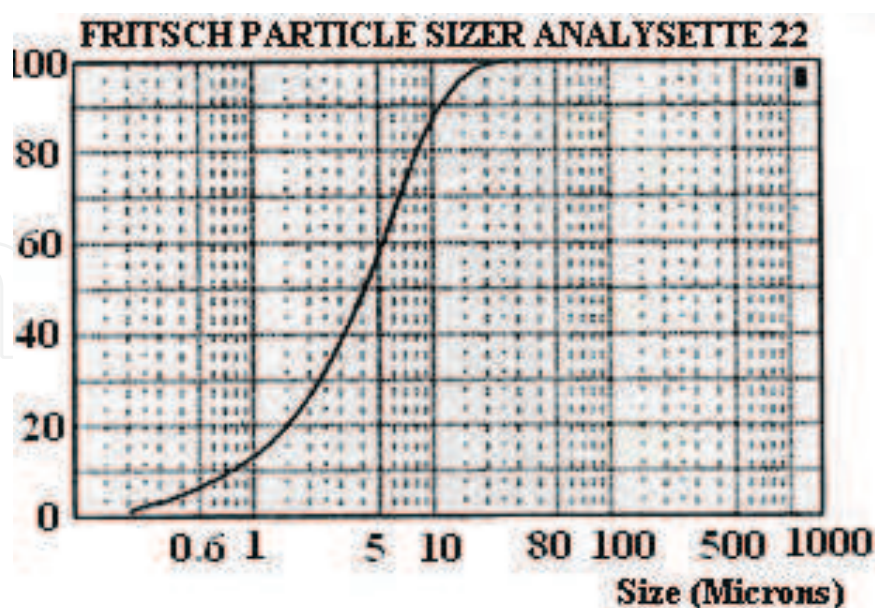

Fig. 1. Particle size distribution of the hydroxyapatite powder

The specific surface area of the starting powder was measured as 52.3 m²/g which compared with other commercial powders can be considered as an active powder. The

molar ratio of Ca/P is another important parameter which is 1.5 in tricalcium phosphate, 2 in tetra calcium phosphate, and 1.67 in stoichiometric hydroxyapatite (A. Siddharthan, et al. 2005). The molar ratio of the Ca/P in the present powder was measured with the ICP technique at around 1.62. A ratio less than 1.64 will be interpreted as the creation of pores and voids in a sintered body. A ratio higher than 1.67 means that the rate of absorption in vivo will be increased (P. Vincenzini, 1986).

In order to gain insights into the sintering behavior of hydroxyapatite, dilatometric tests were carried out. Figure 2 shows a typical curve which indicates the shrinkage starts at about 700 °C and the sintering temperature is estimated to be between 900 and 1300 °C (K.A. Hing, et al. 2000; A.J. Ruys, et al. 1995; B.J. Meenan, et al. 2000; L.L. Hench, 1991; M. Jarcho, et al. 1976; M.K. Sinha, et al. 2000; A.J. Ruys, et al. 1995; G. De With, et al. 1981; P. Landuyt, et al. 1995; M.Y. Shareef et al. 1993; J. Zhou, et al. 1993).
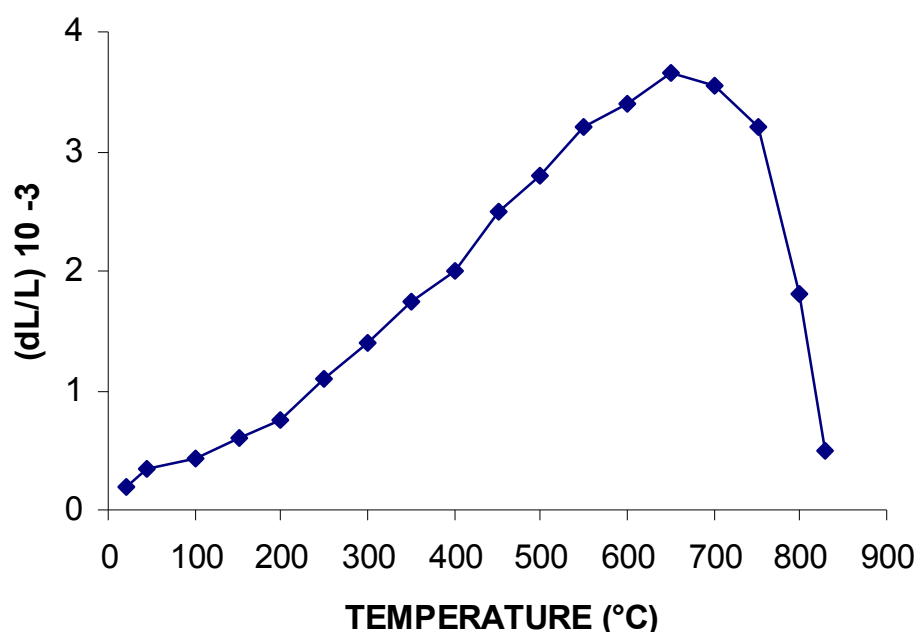

Fig. 2. Dilatometry measurement for a hydroxyapatite sample.

XRD results of the sintered samples are shown in figure 3a to 3d. As it is evident from these patterns, within the sintering range of 700-1300 °C, no other phases could be detected by XRD and the results confirmed formation of hydroxyapatite phase. Also, the CaO phase was checked for in particular to be absent since this phase has been shown to have negative effects on the growth of bone cells (K.A. Hing, et al. 2000).

The Vickers hardness of specimens was measured through indentation method. The results showed rising hardness from 1.5 to 6.1 GPa with increasing sintering temperature from 1100 ° C to 1300 ° C that is shown in Figure 4. Fracture toughness of samples was also calculated from Evans & Charles equation was approximately between 0.5 and 0.85 MPam$^{1/2}$ at sintering temperature about 1200° C.

In order to make microscopic studies, samples were etched in 1% phosphoric acid and gold sputtered prior to study by SEM studies. Figure 5 shows scanning electron microscope images of hydroxyapatite sintered at different temperatures. In all cases, the sintered samples were highly polished with different grades of polish; the last one being 1 micrometre of diamond paste.
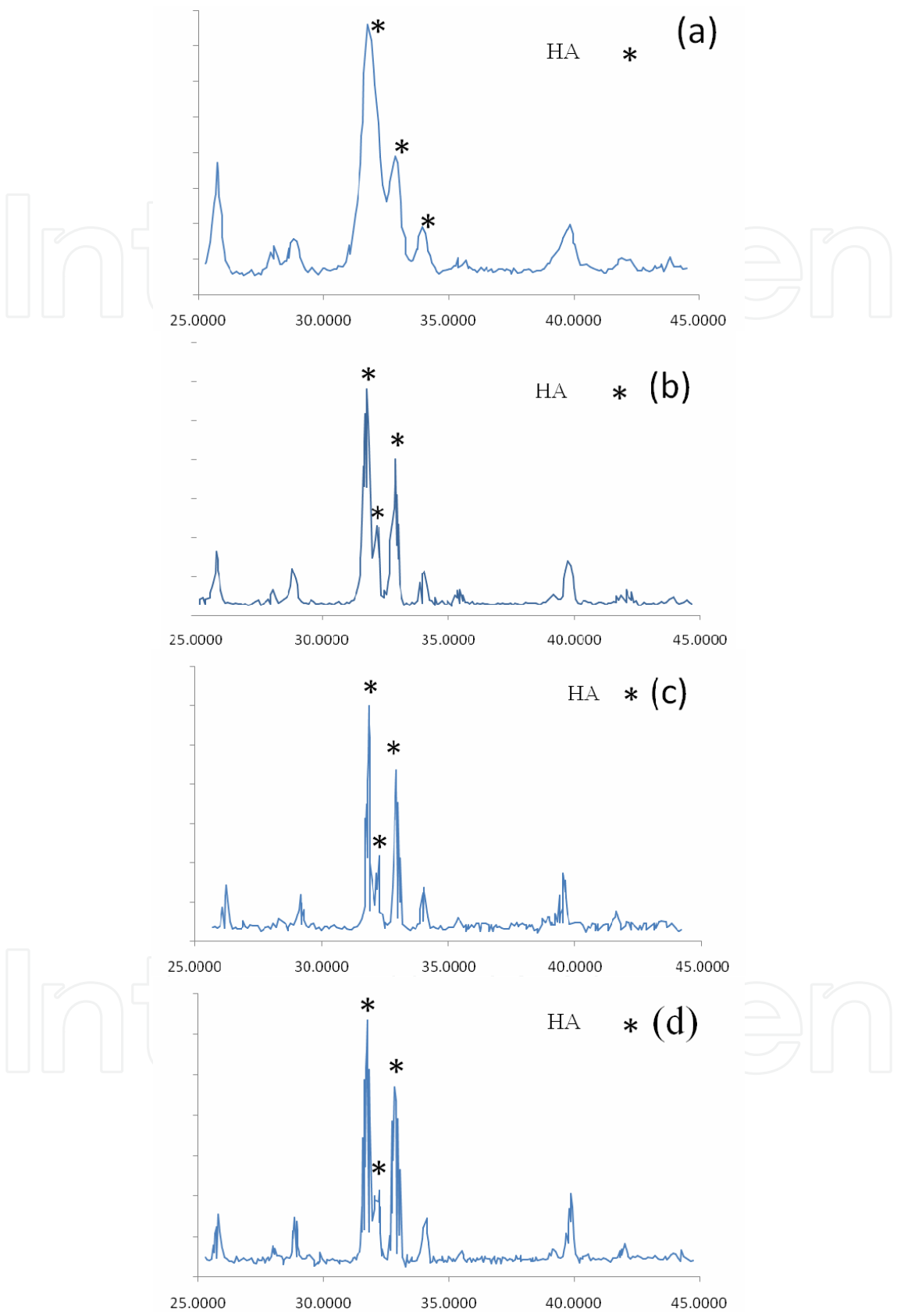
Fig. 3. X-ray diffraction pattern of: a) starting powder, b) fired at 900 °C for 1 hour, c) fired at 1100 °C for 1 hour, d) fired at 1300 °C for 1 hour.
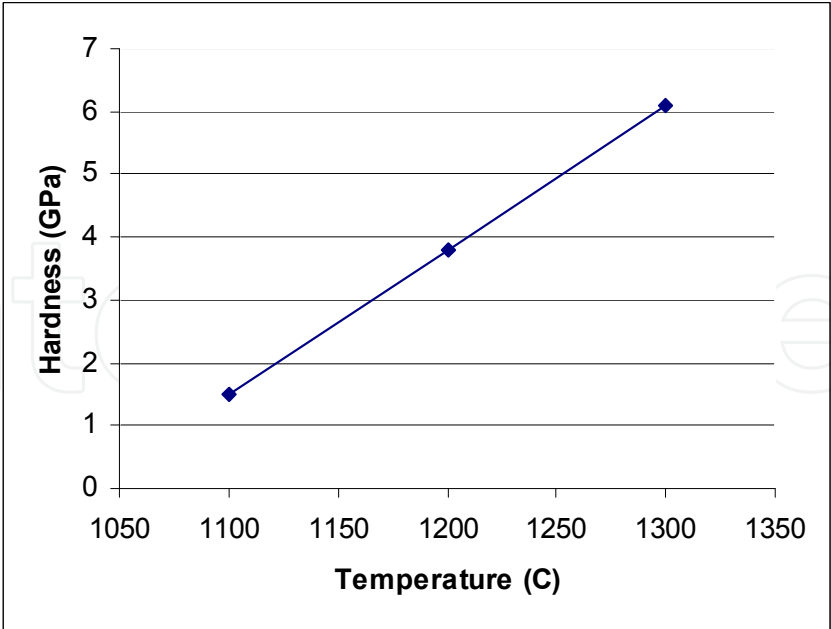
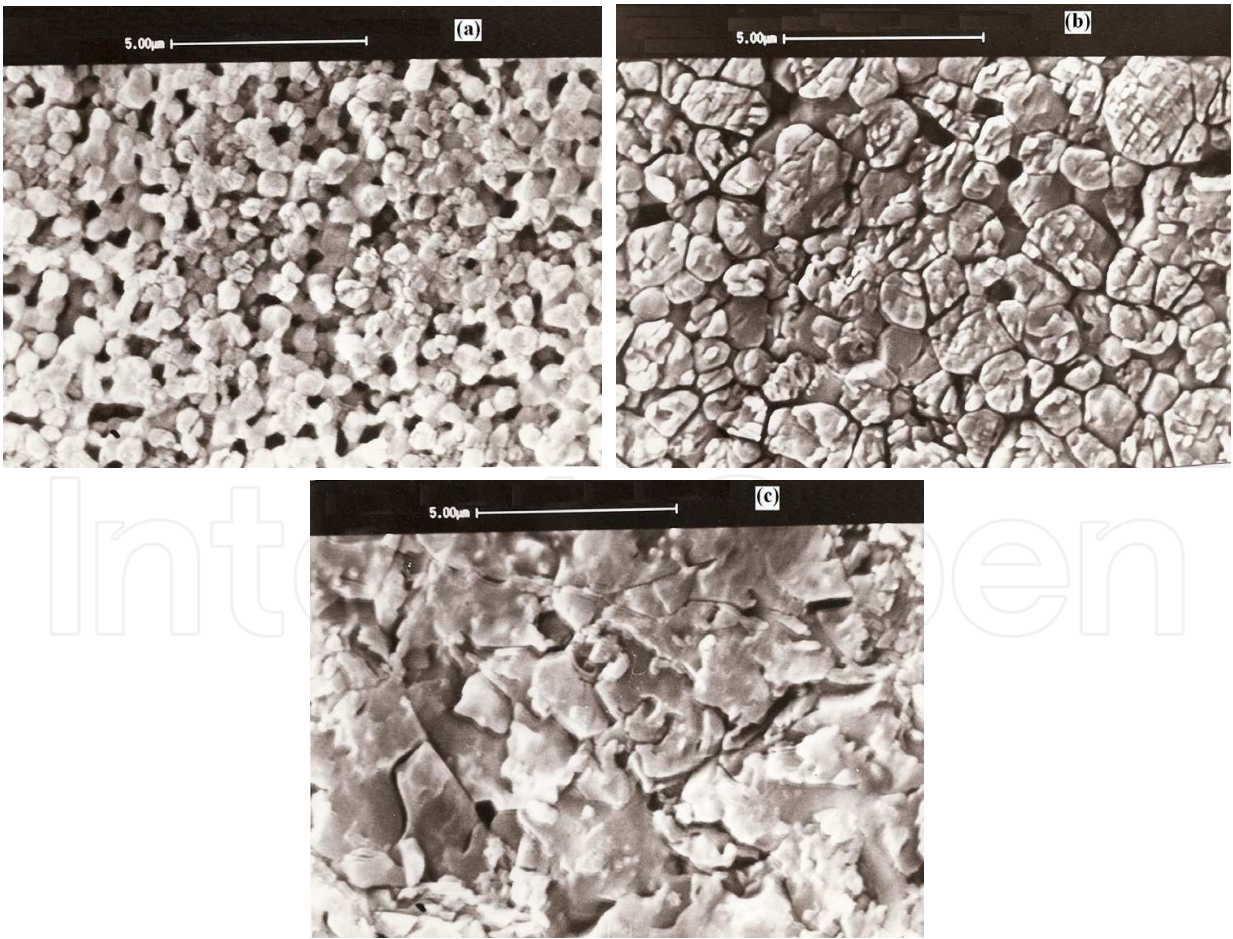Fig. 4. Vickers hardness changes with increasing temperature

Fig. 5. Scanning electron micrographs of hydroxyapatite sintered for one hour at a) 1100 °C b) 1200 °C and c) 1300 °C.

As is evident from figure 5(a), when the samples were fired at 1100 °C, hardly any changes in grain size could be observed. Sintering at 1200 °C results in grain growth in the microstructure of samples as is shown in figure 5(b). The grain growth is also associated with a reduction in the apparent porosity which is a favorable condition as far as the mechanical strength of the part is concerned

Figure 5(c) shows an electron micrograph of a sample sintered at 1300 °C. Further grain growth is evident and a higher mechanical strength is expected for this sample which is in accordance with bending strength results.

The mechanical properties of sintered bodies were examined by 3 and 4 point bending techniques after samples polished with emery paper and diamond paste. An Instron Universal Testing Machine 1196 was used with a cross head speed of 0.5 mm/minute and maximum load application of 5 kN. In fact, in many cases, the mechanical properties of the samples were improved when they were fired at higher temperatures as is demonstrated in figures 6 a and b.
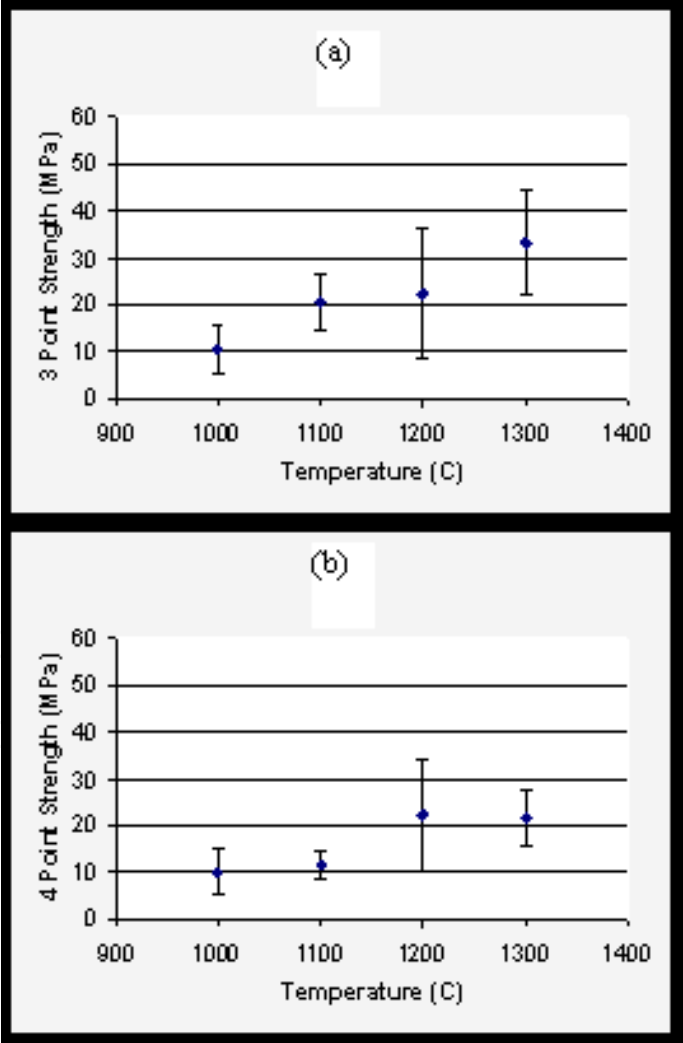

Fig. 6. Bending strength against sintering temperature a) 3 point and b) 4 point.

4. Conclusions

The present study revealed that the sintering of hydroxyapatite at 1100- 1300 °C results in an essentially porous body which can be used as an implant. XRD diffraction patterns of the sintered ceramics proved that no additional phase formation takes place even at elevated temperatures.

Mechanical measurements proved that the bending strength of the sintered bodies were between 7-44 MPa which improved proportional with sintering temperature. Microstructural studies showed that while the grain size of the bodies sintered at 1100 °C remained basically comparable to the particle size of the starting powder, ceramics fired at 1200 to 1300 °C showed an increase in grain size in line with the increased temperature.

5. References

Bohner M., *Injury Int. J. Care Injured.* 31 (2000): S-D37-47.

Cuneyt T., Korkusuz F., Timucin M. and Akkas N., *J. Mater. Sci.: Med.* 8 (1997) 91-96.

De With G., Van Dijk H.J.A., Hattu U., and Prijs K., *J. Mater. Sci.* 16 (1981) 1592-1598.

Hench L.L., *J. Am. Ceram. Soc.* 74 [7] (1991) 1487–1510.

Hench L.L., Bioceramics. *J. Am. Ceram. Soc.* 81[7] (1998)1705-1728.

Hing K.A., Gibson I.R., Revell P.A., Best S.M. and Bonfield W., *Key Eng. Mater.* 192-1 (2000) 373-376.

Jarcho M., Bolen C.H., Thomas M.B., Bobick J., Kay J.F. and Doremus R. H., *J. Mater. Sci.* 11 (1976) 2027-2035.

Jarcho M., *Clin Orthop* (1981) 259–78.

Komath M., Varma H. K. and Sivakumar R., *Bull. Mater. Sci.*, Vol. 23, No. 2 (2000) 135–140.

Landuyt P., Li F., Keustermans J. P., Streydio J. M., Delannay F. and Munting E., *J. Mater. Sci. Mat. Med* 16 (1995) 8-13.

Laverinia C. and Schoenung J.M., *Ceramic Bulletin;* Vol. 70, No.1 (1991) 95-100.

Le Hiec J.C., Schaeverbeke T., Clement D., Faber J. and Le Rebeller A., *Biomaterials* 16 (1995) 113-118.

Luo P., Liu N. and Thelen M., Proc. *24th Annual Meeting of the Society for Biomaterials,* Vol. 21 (1998) 278.

Meenan B.J., Boyd A., Love E. and Akay M., *Key Eng Mater* 192/196 (2000) 15–18.

Peelen J.G.J., Rejda B.V. and De Groot K., *Ceramurgia Int.* 4 (1987) 71-74.

Pham H.H., Luo P., Genin F. and Dash A.K., AAPS *Pharm Sci. 1999;1(suppl):S-373.*

Ruys A.J., Brandwood A., Milthorpe B.K., Dickson M.R., Zeigler K.A. and Sorrell C.C., *J. Mater. Sci: Mat. Med.* 6 (1995) 297-301.

Ruys A.J., Wei M., Sorrell C.C., Dickson M.R., Brandwood A. and Milthorpe B.K., *Biomaierials* 16 (1995) 409-415.

Shareef M.Y., Messer P.F. and van Noort R., *Biomaterials* 14 (1993) 69-75.

Siddharthan A., Seshadri S. K. and Sampath Kumar T. S., *Trends Biomater. Artif. Organs*, Vol 18 (2) (2005) 110-113.

Sinha M.K., Basu D., Sen P.S., *Interceram,* Vol. 49, No.2 (2000) 102-105.

Takagi S., chow L.C. and Ishikawa K., *Biomaterials,* 19 (1998) 1593-9.

Vincenzini P. (Ed.), *Proceedings of the International Symposium on Bioceramics (BIOTEC)*, Milan, Italy Vol. 39 (1986) Elsevier.

Zhou J., Zhang X., Chen J., Zeng S. and Groot K., *J. Mater. Sci: Mat. Med* 4 (1993) 83-85.

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