

## Comparison of $\beta$ -tricalcium phosphate foam made using Mg stabilizer and by heat treatment

Kunio Ishikawa, Taro Nikaïdo, Kanji Tsuru

Department of Biomaterials, Faculty of Dental Science, Kyushu University

**Introduction:**  $\beta$ -tricalcium phosphate ( $\beta$ TCP) is widely used in clinical application as a bone replacement. Therefore,  $\beta$ TCP foam with fully interconnected porous structure allowing cell penetration and tissue ingrowth could be especially effective bone replacement. One of the methods to fabricate the foam is to employ the so-called ceramics foam method in which polyurethane foam is used as a template. Unfortunately,  $\beta$ TCP foam can not be fabricated directly using the ceramic foam method since the method require relatively high temperature and  $\beta$ TCP transforms to  $\alpha$ TCP at temperature higher than 1,180°C. In this investigation, Mg stabilizer addition and heat treatment of  $\alpha$ TCP foam was compared to find a method suitable for the fabrication of  $\beta$ TCP foam bone replacement.

**Methods:** In one method, polyurethane foam was immersed in suspension made with MgO, calcium carbonate and dicalcium phosphate dihydrate so that the amount of MgO became 0, 1, 2, 3, 4, 6 and 8 mol% with (Ca+Mg)/P molar ratio of 1.5. After drying, the foams were sintered at 1500°C for 5 h and cooled down to room temperature inside the furnace. In the other method,  $\alpha$ TCP foams made by sintering at 1500°C were heat treated at 600°C, 700°C, 800°C, 900°C, 1,000°C and 1,100°C for up to 300 h. Composition of the foam was analyzed with powder X-ray diffraction (XRD) and mechanical strength of the foams were evaluated using a universal testing machine in terms of compressive strength. Replacement of the foam to bone was evaluated by implanting the foam to the bone defect made at rat tibia..

**Results and discussion:** Fig. 1 summarizes the XRD patterns of the TCP foam containing 0-8mol% Mg when heated at 1,500°C for 5 h. When the TCP contains 0 or 1 mol% Mg, it transforms to  $\alpha$ TCP. The composition became mixture of  $\alpha$  and  $\beta$ TCP when the TCP contained 2 mol% Mg. When the TCP contains 3 mol% or higher Mg, the composition was  $\beta$ TCP. Since larger amount of Mg has no advantage, 3 mol% Mg may be enough for the stabilization of  $\beta$ TCP foam. Fig. 2 summarizes the transformation ratio of  $\alpha$  to  $\beta$ TCP when the  $\alpha$ TCP foam was heat treated at 600°C - 1,100°C. As shown, heat treatment at 900°C resulted in the fastest transformation from  $\alpha$  to  $\beta$ TCP. Heat treatment higher or lower than 900°C resulted in the slower transformation from  $\alpha$  to  $\beta$ TCP. No transformation was observed when the treatment temperature was 600°C or 1,100°C. The  $\alpha$ - $\beta$  transition temperature of TCP is 1,180°C. No transformation from  $\alpha$  to  $\beta$ TCP at 1,100°C may due to the limited driving force since 1,100°C is close to 1,180°C. Compressive strength of the  $\beta$ TCP was the same regardless of the Mg content or the heat treatment temperature and was approximately 50kPa. When the

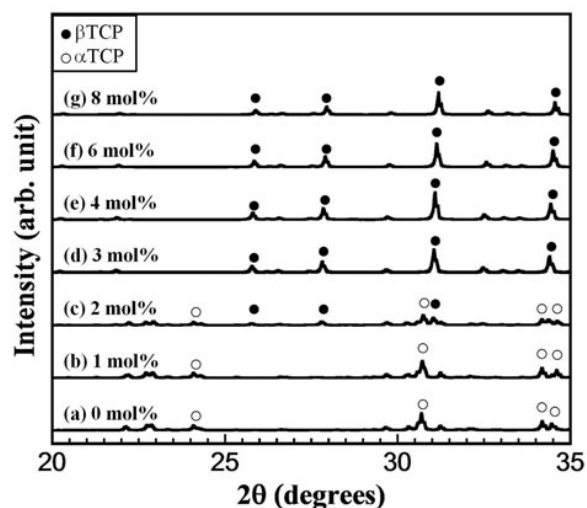


Fig. 1 Powder XRD patterns of ceramic foams containing different amount of Mg.

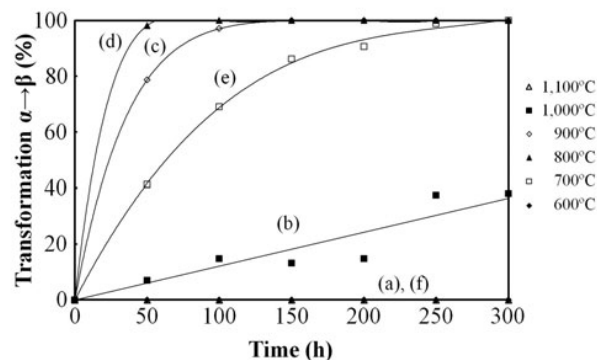


Fig. 2 Isothermal transformation curves for  $\alpha$ TCP to  $\beta$ TCP when  $\alpha$ TCP foams were heat treatment at 600°C (a), 700°C (b), 800°C (c), 900°C (d), 1,000°C (e) and 1,100°C (f) for various periods.

$\beta$ TCP foam was implanted in the bone defect made at tibia of rat,  $\beta$ TCP foam made by heat treatment was replaced to bone whereas  $\beta$ TCP foam made with Mg addition remained at the bone defect after 5 months. Mg may stabilize not only  $\beta$ TCP at high temperature but also solubility at the physiological condition.

**Conclusions:**  $\beta$ TCP foam that has structure similar to cancellous bone was fabricated based on so-called ceramics foam method using polyurethane foam as a precursor, and using Mg as stabilizer or heat treatment.  $\beta$ TCP foam made by heat treatment may be better for its use as bone replacement.