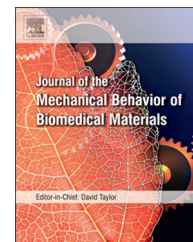


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## Research Paper

# Mechanical and physical properties of calcium silicate/alumina composite for biomedical engineering applications



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## ARTICLE INFO

## Article history:

Received 2 June 2013

Received in revised form

12 October 2013

Accepted 21 October 2013

Available online 7 November 2013

## Keywords:

Calcium Silicate

Alumina

Mechanochemical synthesis

Hardness

Fracture toughness

Young's modulus

## ABSTRACT

The focus of this study is to investigate the effect of  $\text{Al}_2\text{O}_3$  on  $\alpha$ -calcium silicate ( $\alpha$ - $\text{CaSiO}_3$ ) ceramic.  $\alpha$ - $\text{CaSiO}_3$  was synthesized from  $\text{CaO}$  and  $\text{SiO}_2$  using mechanochemical method followed by calcinations at  $1000^\circ\text{C}$ .  $\alpha$ - $\text{CaSiO}_3$  and alumina were grinded using ball mill to create mixtures, containing 0–50 wt% of  $\text{Al}_2\text{O}_3$  loadings. The powders were uniaxially pressed and followed by cold isostatic pressing (CIP) in order to achieve greater uniformity of compaction and to increase the shape capability. Afterward, the compaction was sintered in a resistive element furnace at both  $1150^\circ\text{C}$  and  $1250^\circ\text{C}$  with a 5 h holding time. It was found that alumina reacted with  $\alpha$ - $\text{CaSiO}_3$  and formed alumina-rich calcium aluminates after sintering. An addition of 15 wt% of  $\text{Al}_2\text{O}_3$  powder at  $1250^\circ\text{C}$  were found to improve the hardness and fracture toughness of the calcium silicate. It was also observed that the average grain sizes of  $\alpha$ - $\text{CaSiO}_3$  / $\text{Al}_2\text{O}_3$  composite were maintained 500–700 nm after sintering process.

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## 1. Introduction

Calcium Silicate ( $\text{CaSiO}_3$  or  $\text{CaO-SiO}_2$ ) ceramics are widely used for load-bearing bone substitutes in clinical applications due to their significant biocompatibility, high osteoconductivity and their biological apatite similarity to human hard tissues (Beheri et al., 2013; Lin et al., 2005; Yu et al., 2012). Also, pure calcium silicate ceramics are greatly induced formation of a hydroxyapatite (HA) layer on calcium silicate surface after soaking in simulated body fluid (SBF) and in saliva medium (De Aza et al., 1994; Siriphannon et al., 1999,

2002a). These layers help to form the chemical bonds between the bioceramics and bone tissues.

In fact, calcium silicate ( $\text{CaSiO}_3$ ) was introduced as a biomaterial for hard tissue repair substituent since 1970s, when Hench and coworkers invented Bioglass<sup>®</sup>. As yet several forms of silicate glasses and glassceramics like bioactive glasses (Kokubo, 1991; Rahaman et al., 2011) and calcium silicate based glass ceramic (Cho et al., 1997; Liu et al., 2004) have been explored. Previous studies revealed that the biomaterials containing  $\text{CaO-SiO}_2$  act excellent bioactivity and were found to bond to living bone and soft tissue, through the development of a biologic HA layer on their surface (Siriphannon et al., 2002b).

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However, the foremost limitation of ceramic powders is their low fracture toughness that restricts some restriction of biomedical application such as load bearing implant application (De Aza et al., 1997; Encinas-Romero et al., 2008; Mehrali et al., in press). Therefore, one approach to improve the mechanical properties of bioactive ceramic materials is by incorporate the alumina as a ceramic second phase dispersed in a ceramic matrix. Some researchers in regards to the approach have investigated the alumina reinforcement role in HA and also have demonstrated that alumina particles could improve toughening and hardness (Huang and Nicholson, 1993; Ji and Marquis, 1992). Alumina, which is mostly categorized as a bioinert material, has been broadly investigated as a reinforcement agent for ceramics (Mobasherpour et al., 2009). Good flexural strength, excellent resistance to dynamic and impact fatigue, resistance to subcritical crack growth and excellent compressive strength are obtained with average grain sizes  $<4\text{ }\mu\text{m}$  and purity  $>99.7\%$  (Askari et al., 2012). However, just several researches have been done on the effective role of bioinert materials such as alumina and zirconia on the fracture toughness and hardness of calcium silicate, similar to what has been done in this study.

In this study, the effects on the mechanical properties of calcium silicate, namely micro hardness, fracture toughness and Young's modulus, by changing the composition (based on different weight ratios of alumina) and sintering temperatures, were investigated.

## 2. Material and method

### 2.1. Synthesis of calcium silicate powder

Calcium silicate powders have been synthesized using mechanochemical process. The main advantage using this method is a good crystallinity and small crystallites without using high temperatures for synthesizing (Nasiri-Tabrizi et al., 2009).

Commercial calcium oxide (CaO) (Ajax chemicals industries) and silicon oxide ( $\text{SiO}_2$ ) (Sigma-Aldrich Inc.) were used in  $\text{CaSiO}_3$  preparation. For reaction to occur, aforementioned materials were grounded on a planetary-mill (Retsch PM 100) with the stoichiometric proportionality between the oxides as given in reaction (1). Milling was accomplished using the rotational speed of 500 rpm. The ratio of ball to powder was assumed to be 20/1. 60 of min milling with 10 min interval pauses were performed through the process to avoid overheating. Total duration of the process to perform the reaction 1 was 50 h. Finally, the white powders were calcined at  $1000\text{ }^\circ\text{C}$  for 2 h to obtain  $\alpha\text{-CaSiO}_3$ . Previous researches demonstrated that  $\alpha\text{-CaSiO}_3$  is less degradable than  $\beta$  phase which is more applicable in implantation (Liu et al., 2008; Shirazi et al., 2013; Wu et al., 2007). Hence the mechanochemical technique to make submicron ( $<1\text{ }\mu\text{m}$ ) calcium silicate powder is done as



### 2.2. Fabrication of composites and sintering

The prepared calcium silicate was mixed with  $\alpha\text{-Al}_2\text{O}_3$  (99.95%, Alfa Aesar, Ward Hill, MA, USA) powders with an average crystal and particle size of  $0.3\text{ }\mu\text{m}$  using ball milling in

ethyl alcohol for 24 h with zirconia balls as media to break the  $\text{CaSiO}_3$  agglomerates. Mixing of  $\text{CaSiO}_3$  and alumina was done in different ratios (5, 10, 15, 25 and 50 wt%) in order to peruse the influence of  $\text{Al}_2\text{O}_3$  content in calcium silicate mechanical and physical properties. The powder mixtures ( $\text{CaSiO}_3/\text{Al}_2\text{O}_3$ ) were uniaxial compressed at 10 t and then cold isostatic press (CIP) was applied into rectangular-prism shape specimens ( $30 \times 8 \times 2.5\text{ mm}^3$ ) under a pressure of 250 MPa for 20 min. Subsequently all bulk samples were sintered at two different temperatures;  $1150\text{ }^\circ\text{C}$  and  $1250\text{ }^\circ\text{C}$  for 5 h with  $5\text{ }^\circ\text{C/min}$  heating rate. Three samples per each combination were fabricated with aforementioned process.

### 2.3. Characterization of $\alpha\text{-CaSiO}_3/\text{Al}_2\text{O}_3$ composites

The sintered bulks were grounded, polished, and thermally etched for 2 h in a furnace at  $1100\text{ }^\circ\text{C}$  in air atmosphere for microstructural analysis. The porosity sintered materials was measured by the Archimedes method. The microstructure and phase composition of the calcined powders and sintered samples were determined by field emission scanning electron microscopy (FESEM; CARL ZEISS-AURIGA 60 microscope) and X-ray diffraction (XRD; EMPYREAN, PANALYTICA) using  $\text{Cu K}\alpha$  radiation. XRD was performed over a range of  $5^\circ < 2\theta < 80^\circ$  with a step size of  $0.015^\circ$  and a time per step of 20.02 s. The relative density was calculated by comparing the measured data to the theoretical densities of  $\text{CaSiO}_3$  ( $2.905\text{ g/cm}^3$ ) and  $\text{Al}_2\text{O}_3$  ( $3.969\text{ g/cm}^3$ ).

### 2.4. Mechanical properties evaluation

Vickers hardness was measured by the indentation-strength method at room temperature using an applied load of 2 kgf with a dwell of 15 s. (MITUTOYO-AVK C200-AKASHI CORPORATION). Five indentations run per sample were carried out to determine the average value of mechanical properties. Fracture toughness was then determined through using the following equation (Anstis et al., 1981; Lawn et al., 1980; Shetty et al., 1985):

$$K_{IC} = 0.0937 \times \left( \frac{H_v \times P}{4L} \right)^{(1/2)} \quad (2)$$

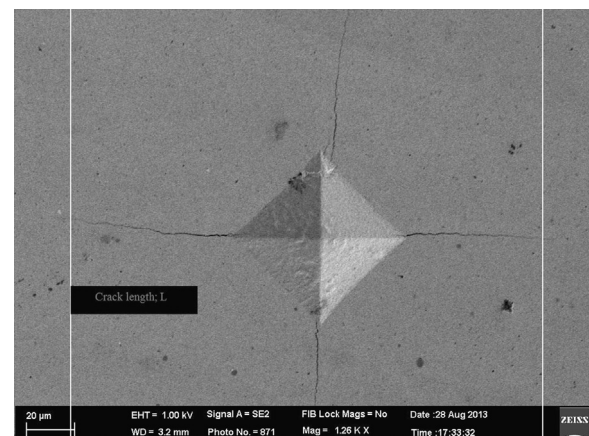


Fig. 1 – FESEM micrograph of the indentation-induced crack propagation.

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