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ScienceDirect
Ceramics International 41 (2015) 12340–12348
CERAMICS INTERNATIONAL
www.elsevier.com/locate/ceramint

Determination of elastic modulus, tensile strength and fracture toughness of bioceramics using the flattened Brazilian disc specimen: analytical and numerical results

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Received 30 May 2015; received in revised form 12 June 2015; accepted 13 June 2015
Available online 20 June 2015

Abstract

Ceramics and bioceramic composites are materials characterized with strong but quite low crack-resistance properties. Indeed, their application is hindered by their brittleness and low ability to deform, and the accurate measurement of their fracture toughness can often be challenging. This measurement along with that of tensile strength and elastic modulus were conducted using a novel configuration of the Brazilian test. The flattened Brazilian specimens, which are in the shape of discs having parallel flat ends, are subjected to compression for the determination of opening mode I fracture toughness K_{IC}. The experiments were conducted using tricalcium phosphate–fluorapatite composites which were tested by compressive loading on the parallel flat ends. The loading angle corresponding to the flat end width is about $2\alpha = 20^\circ$ to guarantee crack initiation at the specimen center according to the Griffith criteria. Finite element program, called ABAQUS, is used for the numerical modeling for finding stress intensity factors. The effects of fluorapatite additives and fracture toughness were studied. The fracture toughness values of tricalcium phosphate–fluorapatite composites were found to increase with the increase in fluorapatite addition until an appropriate value. It is shown that there is a good agreement between the experimental, analytical and numerical results.

Keywords: B. composite; C. Toughness; C. Mechanical properties; Flattened Brazilian test; Numerical modeling

1. Introduction

Hydroxyapatite (Hap) and β tricalcium phosphate (β-TCP) which are the most frequently used bioceramics due to their biocompatibility and bioactivity can be designed under an appropriate shape and size [1–3]. These bioceramics have attracted considerable interest for orthopedic and dental functions [1]. But their application at high load bearing conditions was restricted owing to their brittleness, poor fatigue resistance and strength. Moreover, the majority of bioceramics have low mechanical properties such as hydroxyapatite, tricalcium phosphate (β-TCP) and fluorapatite (Fap).

The development of the composite material industry poses the necessity for determining the stress intensity factor (SIF) of bioceramic cracking. Researchers are still studying to find a simple and accurate standard method to determine fracture toughness which is an important parameter to determine the stress required to drive a preexisting crack which generally exists in materials. However, the International Society for Rock Mechanics (ISRM) has suggested some methods to determine fracture toughness, listed in [4], examples include (1) Cheveron Bend (CB) specimens, (2) Short Rod (SR) specimens and (3) Cracked Chevron Notched Brazilian Disc (CCNBD).

Some methods were previously used to find mode I fracture toughness, K_{IC}, like Modified Ring (MR) test [5,6], Diametral Compression Method [7], semi-circular core in three point bending (SCB) [8] and finally Brazilian disc test (BDT) [9,10].

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http://dx.doi.org/10.1016/j.ceramint.2015.06.063
0272-8842/

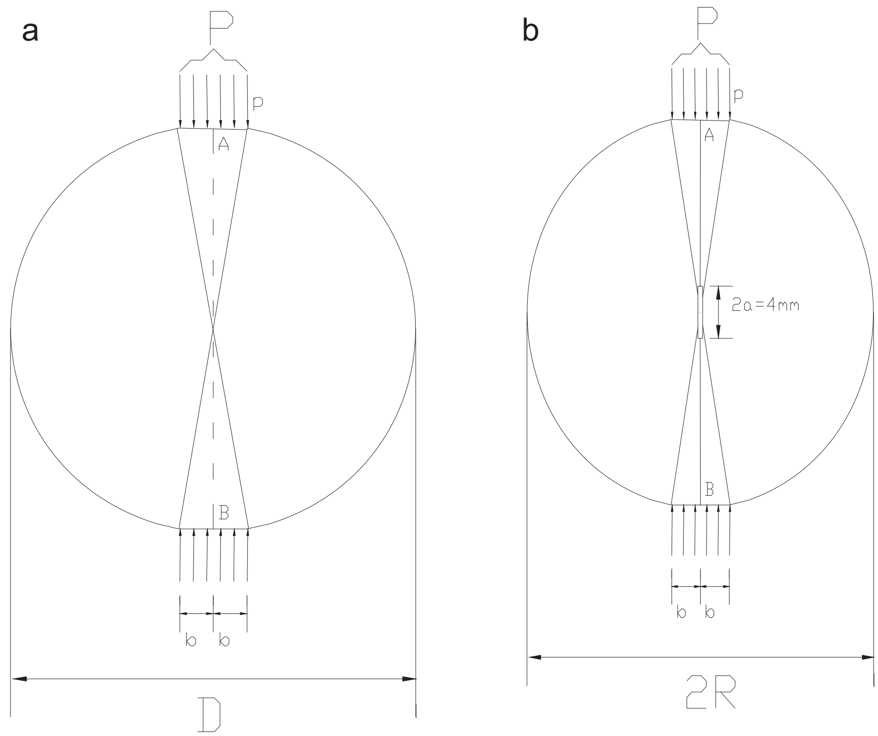

Fig. 1. (a): Flattened Brazilian specimen, (b): flattened brazilian specimen with a central straight-through crack (CSTFBD).

In order to define the elastic modulus, tensile strength and fracture toughness of biomaterials, the flattened Brazilian test is ideal for the specimens in the pure mode I fracture and the well-known test configurations for determining the parameters previously mentioned. The disc specimen presented in Fig. 1 is designed by introducing two equal-width parallel planes in the sample, which are prepared specifically for load application.

In just one test, the flattened Brazilian test assists for proving the tensile strength, elastic modulus and opening mode fracture toughness K_{IC}. The loading angle conforming to the flat end width 2α must be greater than a critical value ($2\alpha \geq 20^{\circ}$) in order to guarantee crack initiation at the center of the disc [11]. The obtained numerical results are compared with the experimental and analytical ones.

In this study we used the commercial tricalcium phosphate (β-CTCP) reinforced with the fluorapatite (Fap) with different additive amounts (13.26 wt%, 19.9 wt%, 26.52 wt%, 33.16 wt % and 40%) sintered at 1300 °C. The objective is to determine the stress intensity factors for modified Brazilian test with analytical formula and numerical simulation. A range of specimen geometries having various crack length (a) were modeled and analyzed with ABAQUS finite element program. Fracture toughness values with varying geometric parameters were analyzed. Experiments were conducted on flattened Brazilian specimens with crack length « $2a$ », flat loading (2α) end and widths ($2b$).

2. Materials and methods

In order to elaborate CTCP-Fap, the used materials were the commercial tricalcium phosphate (Fluka) and synthesized fluorapatite. The Fap powder was synthesized by the precipitation method [13]. A calcium nitrate ($Ca(NO_3) \cdot 4H_2O$, Merck) solution was gradually added to a boiling solution, including diammonium hydrogenophosphate ($(NH_4)_2 \cdot HPO_4$, Merck) and ammonium fluorine (NH_4F, Merck), with continual magnetic stirring. During the reaction, the pH was adjusted to the same level (pH 8–9) by adding ammonia. The obtained precipitate was filtered and washed with deionised water, and then desiccated at 70 °C for 12 h. The obtained fluorapatite was not quite pure, it rather contained some impurities such as pyrophosphate which was observed by the SEM micrographs.

The approximate representatives Fap – β-CTCP were [{13.26 wt%, 86.74 wt%} {19.9 wt%, 80.1 wt%},{ 26.52 wt%, 73.48 wt%}, {33.16 wt%, 66.84 wt%} and {40 wt%, 60 wt%}]. The estimated quantities of each powder were milled with absolute ethanol and treated by ultrasound machine for 20 min. The milled powder was dried in a low-temperature oven at 80 °C to eliminate the ethanol and generate a finely divided powder. Powder mixtures were molded in a metal mold and uniaxially pressed at 67 MPa to form cylindrical compacts with 30 mm in diameter and about 5 mm in thickness. The green compacts were sintered in a horizontal resistance furnace (Pyrox 2408) at 1300 °C for 1 h 30 min. The heating and cooling rates were 10 and 20 °C min^{-1}, respectively. A LLOYD model test machine was used for the flattened Brazilian test for the measurement of elastic modulus and tensile strength.

In this study, we used two different geometries for the samples construction. Actually, for the flattened Brazilian disc (FBD): $D=30$ mm, $t=5$ mm and $2b=5$ mm (the flat for $2\alpha=20^{\circ}$) where D is the diameter, t the thickness and $2b$ is the width of the flat end (see Fig. 1a). Besides, for the flattened