

## Fracture properties of an acrylic bone cement

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This study investigated experimentally the fracture properties, i.e., the fatigue strength, the resistance to crack propagation and the fracture toughness, of an acrylic bone cement (Cemex<sup>®</sup> RX). The mean endurance limit was determined following the staircase method. The endurance limit was estimated at 9.2 MPa. The fatigue crack propagation rate was measured according to the ASTM E647 standard. The equation of the line fitting the crack growth per cycle ( $da/dN$ ) versus the stress-intensity factor range ( $\Delta K$ ), in a log-log graph, was used to calculate the empirical constants of Paris' law for the selected bone cement:  $da/dN$  (m/cycle) =  $3.56 \cdot 10^{-7} \cdot \Delta K$  (MPa·m<sup>1/2</sup>)<sup>5.79</sup>. This power-law relationship described well ( $R^2 = 0.96$ ) the growth rate in the stable crack growth region, i.e., in the mid  $\Delta K$  range. The fracture toughness  $K_{IC}$  of the bone cement was determined according to the ASTM E399 standard. The  $K_{IC}$  mean value was 1.38 MPa·m<sup>1/2</sup>. These experimental results provide the set of necessary inputs for numerical studies aimed to investigate the damage accumulation process in the mantle fixing cemented prostheses.

*Key words: biomaterials, cement, fracture properties, fatigue, fracture toughness*

### 1. Introduction

Polymethylmethacrylate (PMMA)-based bone cement is the most common, commercially available material used in the orthopaedic field to fix cemented prostheses to the hosting bone. The use of PMMA assures an optimal implant stability after the surgical session which should be guaranteed for the entire implant life.

Clinical data from Swedish Total Hip Replacement Register show that aseptic loosening has caused nearly 60% of the failures in cemented implants during the last 26 years [1]. Many causes may contribute to the complex phenomenon which causes implant loosening. Among others, one of the potential causes for aseptic loosening is the long-term mechanical performance of the cement mantle. It has to transfer loads, generated during daily activities, from the implant to the periprosthetic tissues. Therefore, in vivo

the cement mantle undergoes complex cyclic loadings. These loads cause cyclical stresses which may crumble the cement mantle [2], [3], promoting loosening of the prosthetic component [4]–[12].

The long-term behaviour of the cement mantle depends on the mechanical properties of the bone cement and on how it is stressed in vivo [5], [13]–[16]. The former are characteristics of the material itself, generally referred to as fracture properties [17]. However, the stress levels within the cement mantle are affected by prosthesis design, mantle thickness and quality, and support of the bone tissue surrounding the implant [9], [18]–[22]. The prosthesis-cement mantle-bone system can be investigated by means of Finite Element Models (FEMs). FEMs are used to calculate the stress within the mantle and to predict the fatigue damage under simulated physiological conditions [6], [10], [23]–[25]. These studies require the knowledge of the fracture properties of the bone cement.

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Received: November 25, 2007

Accepted for publication: February 15, 2008

Although there are many reports on the mechanical characteristics of bone cement [14], [26]–[31], a complete characterisation of the fracture properties of a commercial bone cement is missing. The aim of this study was to determine all the fracture properties, i.e., the fatigue strength, the resistance to crack propagation and the fracture toughness, of a PMMA-based radiopaque bone cement.

## 2. Materials and methods

### 2.1. Materials and specimens preparation

Cemex® RX (Tecres, Verona, Italy) was selected for this study. It is a PMMA-based bone cement. 9% barium sulphate was present in this formulation to assure the required radiopacity, as in most of the commercially available formulations of bone cements. The bone cement was mixed at a temperature of  $23\pm 1^\circ\text{C}$  and at a relative humidity ranging from 40 to 60%, in agreement with ISO 5833 recommendations. The bone cement was mixed in air and, once reached the doughing time, the dough was poured into the moulds to cast the specimens of defined dimensions (see figure 1). After 1 h of polymerization specimens were stored in saline solution at  $37^\circ\text{C}$  for 14 days before testing. Prior to testing both sides of each specimen were polished using 800-grid sand paper to adjust the thickness to the desired value, with an accuracy of 0.1 mm. Before testing, specimens were X-ray checked in order to reject all ones with macro-porosity (pore diameter  $>1$  mm) in the working region [17], [32]. Since this inspection was not possible for the 10-mm thick specimens, the fracture surface was examined after testing; if a macro-porosity was found on the crack surface, the specimen was discarded [31]. All the specimens were tested in the air at  $23\pm 1^\circ\text{C}$  with a material testing machine (MTS Mini Bionix 858, MTS System Corp., Minneapolis, MN). The frequency of cyclic loading was set at 4 Hz.

### 2.2. Fatigue testing

Fatigue tests were performed on dog-bone like specimens. The dimensions and geometry of the specimen were chosen in agreement with ISO 527-2.

Working part dimensions were: length ( $l$ ) 80 mm, width ( $w$ ) 10 mm, and thickness ( $t$ ) 4 mm (figure 1).

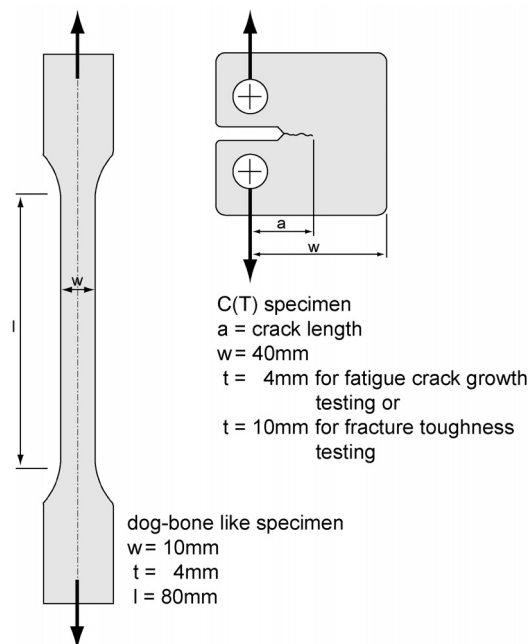


Fig. 1. The dimensions of the dog-bone like specimen and the C(T) specimen;  $t$  stands for the specimen thickness

Fatigue testing was carried out at selected load levels until specimen fracture or runout took place (test completed). Runout was fixed at 10 million cycles. Preliminary testing was performed above the roughly estimated endurance limit. This series continued decreasing the load level until a specimen did not fail during the test, i.e., reached 10 million cycles. At this point the up-and-down scheme of the staircase method started and continued until 15 specimens were tested in the failure–not failure region. The data collected were used to determine the median endurance limit [33].

### 2.3. Fatigue crack propagation testing

The crack propagation rate was measured according to the method based on ASTM E647. Standard compact-type (C(T)) specimens were moulded. Specimen dimensions were: width ( $w$ ) 40 mm, and thickness ( $t$ ) 4 mm (figure 1). A razorblade was used to produce a pre-crack in the specimen notch before subjecting the specimen to cyclic loading. A sinusoidal tensile load between 0 and 60 N was applied. Before testing, Krak Gages (Mod. B20CE, Rumul, Switzerland) were attached to both sides of the specimen to allow the measurement of the crack length ( $a$ ) during the test. The number of load cycles ( $N$ ) were recorded at each crack length increment

of  $0.4 \pm 0.1$  mm. Five test repetitions were performed. A linear regression was used to fit the data, plotted in a log–log graph of the crack growth per cycle ( $da/dN$ ) versus the stress-intensity factor range ( $\Delta K$ ). The regression equation was used to calculate the empirical constants  $C$  and  $n$  of the Paris' law ( $da/dN = C \cdot (\Delta K)^n$ ). This power-law relationship describes the growth rate in the stable crack growth region of the log–log graph, referred to as region II [33].

## 2.4. Fracture toughness testing

The fracture toughness was determined according to the method based on ASTM E399. Specimen dimensions were: width ( $w$ ) 10 mm and thickness ( $t$ ) 10 mm (figure 1). Preliminary, the specimens were pre-cracked by applying a cyclic load. To maintain the pre-crack growth rate in the order of  $10^{-3}$  mm/cycle, four decreasing load levels were chosen. The crack length was monitored by means of an extensometer attached to the specimen mouth. Pre-cracking was stopped when the  $a/W$  ratio fell in the range of 0.45–0.55. Then the specimen was preloaded with 100 N and subjected to a monotonic tensile test at a crosshead rate of 10 mm/min. The load and the corresponding crack opening were recorded throughout the test to calculate the critical stress intensity value ( $K_{IC}$ ) according to the ASTM standard. Experimental series continued until five valid specimens were tested.

## 3. Results

### 3.1. Fatigue testing

15 specimens were tested in the failure–not failure region. Six specimens completed the test. As runout was the less frequent event, its occurrence was used to estimate the mean endurance limit. The specimen fraction not-failed at 360 N was 60%, at 370 N was 40%, while all specimens failed at 380 N. On the basis of these experimental data, the mean endurance limit was estimated at 9.2 MPa.

### 3.2. Fatigue crack propagation testing

A set of fatigue crack growth data versus stress-intensity range was collected for each specimen. All

the five sets are plotted in figure 2 together with the regression line. The coefficient of determination was  $R^2 = 0.96$ . From the equation of the regression line the constants  $C$  and  $n$  of the Paris' law were calculated:  $C = 3.56 \cdot 10^{-7}$  (m/cycle  $\cdot$  (MPa  $\cdot$  m $^{1/2}$ ) $^{-n}$ );  $n = 5.79$ .

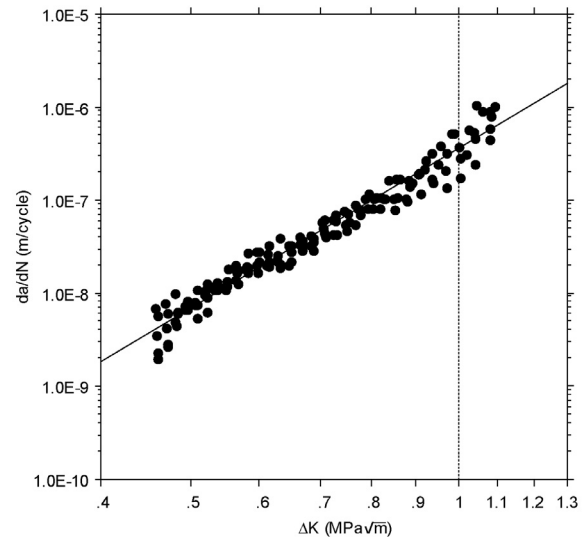


Fig. 2. A linear line fitting the fatigue crack growth data for the bone cement investigated

### 3.3. Fracture toughness testing

Seven C(T) specimens were tested. Two of these were excluded since a macro-porosity was found on the fracture surface, and therefore the value of  $K_{IC}$  was not calculated from the experimental data. The mean value of  $K_{IC}$  calculated for the five valid specimens was  $1.38$  MPa  $\cdot$  m $^{1/2}$ . The coefficient of variation for  $K_{IC}$  of these five specimens was 3.6%.

## 4. Discussion

This study was aimed to assess the fracture properties of a commercial PMMA-based bone cement. The fracture properties characterise the material behaviour under cyclic loads. These data are necessary as input parameters in FEMs investigating the damaging process in the cement mantle due to load generated during physiological activities.

The fracture properties of a bone cement may depend on chemical formulation of the material [34]–[37], on the procedure for moulding the specimen (i.e., on the final quality of the specimen) [38]–[42],

and on the testing procedure [17], [43], [44]. The first consideration would require that this study should be performed for each cement formulation whose mechanical behaviour is going to be modelled in a FEM. Because of a great number of bone cement brands currently available on the market for orthopaedic applications [37], this approach is not possible. The bone cement investigated in this study was selected as “representative” of a standard PMMA-based bone cement. This formulation contains barium sulphate as radiopacifier, the most common compound added to gain material radiopacity. Barium sulphate, together with benzoyl peroxide (a polymerisation catalyst), *N, N*-dimethyl-*p*-toluidine (a polymerisation accelerator), and hydroquinone (a MMA stabiliser), is generally present in the bone cement formulation [37]. Referring to the quality of the specimens, all three procedures considered the specimen inspection and a rejection criterion: specimens showing macro-porosities were rejected, in agreement with that proposed by other authors [17], [31], [45]. Similarly, the specimens were seasoned for 14 days before testing to assure a complete polymerisation of the material [46]–[48]. Last, the experimental procedures used in this study were preliminary validated [31], [32] and/or were already used by other authors to determine some of the fracture properties of a bone cement [49], [50].

On the basis of this rationale, few data reported in the literature can be compared with the present one. To the authors’ knowledge, no data have been published about the crack growth rate for the bone specimen investigated. In a previous study, an estimation of 9.7 MPa for the endurance limit was reported [51]. However, in that study, the stress value of the slope segment of the Wöhler curve corresponding to 2 million cycles was assumed as a rough prediction of the endurance limit. In this study, the mean endurance limit was estimated defining the runout at 10 million cycles, therefore at a higher fatigue life. This may explain the difference of 5% between the two estimated mean endurance limits. Another study reported fatigue data for the same bone cement tested at higher stress levels [52]. In that study, the endurance limit was estimated at 12.9 MPa, although the authors stated that their procedure might overestimate the real value. Also the calculated fracture toughness was by 33% lower than that reported for the same formulation by LEWIS et al. [53]. In this study, a fatigue-cracked C(T) specimen was tested, while in the study cited, the fracture toughness was determined using Chevron-notched short-rod specimen obtained machining the slot but no fatigue pre-crack was reported. Therefore, the difference may be due to both the effective shape

of the crack tip [31] and the different experimental procedures [43].

Considering different cement formulations, the results found in the present study are in agreement with those reported for a not commercial bone cement, similar to the present one except for the barium percentage [54]. Not considering the important effect of the cement formulation on its fracture properties, it can be highlighted that the fracture properties measured in this study were within the range reported in the literature for PMMA-based cements both for the endurance limit [27], [55] and for the fracture toughness [31], [35], [56]–[61], although none of the studies cited considered all the fracture properties of the bone cement.

In conclusion, the fracture properties of a PMMA-based bone cement were experimentally determined. These properties are the necessary inputs to any numerical studies aimed to investigate the damage accumulation process in mantles fixing cemented prostheses.

## Acknowledgements

This work was partially funded by Tecres SpA. The authors thank Luigi Lena for the illustrations and Caroline Öhman for editorial assistance.

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