

# REINFORCEMENT OF BONE CEMENT USING ZIRCONIA FIBERS WITH AND WITHOUT ACRYLIC COATING

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**INTRODUCTION:** Poly(methyl methacrylate) (PMMA or acrylic) has been used as a filler material between prosthesis and bone in cemented total joint arthroplasties. Failure of the PMMA mantle can lead to the loosening and ultimately to failure of the prosthesis [1]. Therefore, there is a need to increase the mechanical properties and fracture resistance of bone cement. Though there are numerous studies on fiber reinforcements of bone cement [2, 3], fibers made of zirconia have not been studied in cements. The advantage to using zirconia is that this material is already present in some acrylic bone cements as a radiopacifier (e.g. Palacos R). Therefore, the use of zirconia fibers for reinforcement of bone cement should not alter the biological response to the modified cement compared with standard cements. In this study, we investigate the use of short, high-strength, zirconia fibers with and without acrylic coating as reinforcements in bone cements. Specifically, the tensile, compressive and fracture properties of control and reinforced acrylic bone cements are investigated.

**METHODS:** Zirconia fibers (Advanced Cerametrics Inc., Lambertville, NJ) of two different diameters, ~15 $\mu$ m and ~30 $\mu$ m, were milled. The size distributions of the fibers were determined by optical microscopy measurements on 1000 fibers. The strength of some fibers was determined by tensile testing to failure on an Enduratec 3200 electromagnetic testing machine (Enduratec Inc., Minnetonka, MN). Briefly, the two ends of the fibers were potted to the grips using epoxy. Tensile testing was conducted, with a 3mm gage length, at a crosshead displacement rate of 0.3mm/sec to failure (n = 30/group).

Coating of individual zirconia fibers with PMMA was conducted using an emulsion polymerization technique. Briefly, broken fibers (50 gms) were stirred in a 2% sodium dodecyl sulphate (SDS) detergent solution at pH 2, containing 30% methanol. Benzoyl peroxide (1%) was added to the solution when the temperature is 60 °C, which allows the radicals formed by autocleavage of BPO to be adsorbed on the negatively charged fiber surfaces. MMA monomer (5 mL) was then added to the solution every 10 minutes for an hour. Polymerization of MMA initiates on the fiber surface through radicals adsorbed on the fiber. After 2 hrs, 45mL of acetone was poured and allowed to evaporate, following which fibers were filtered and vacuum dried to obtain acrylic coated fibers. The weight percent of acrylic coating was found through removal of coated acrylic using acetone as solvent. To determine if acrylic coating changed the mechanical properties of the fibers, another batch of longer fibers were subjected to coating. Following removal of coating, tensile tests were performed as before to evaluate mechanical properties (n = 30/group). Scanning electron microscopy was conducted on a Hitachi Field Emission microscope to determine fiber microstructure and evaluate acrylic coating.

For control cements, 10% by weight barium sulphate was blended into the acrylic beads (Zimmer Inc., IN). For reinforced cements, 14% by weight (2.3 vol. %) of 15 $\mu$ m zirconia fibers (with and without acrylic coating) and 30% by weight (5 vol. %) of 30 $\mu$ m zirconia fibers (with and without acrylic coating) were blended with pre-polymerized beads of PMMA in a commercial blender. The solids were mixed into 20mL of liquid monomer (MMA), stirred for 2 minutes and injected into sample molds under high pressure using a cement gun. Samples for tensile testing were manufactured by injecting into polysulphone molds. Samples for compression testing were made by injecting into steel molds while samples for fracture toughness tests were made in polyacetal molds. The samples were allowed to cure in the molds for 1 hr at 37 °C, after which they were removed from the molds and stored in a dry environment. Mechanical tests were conducted within 1 day of specimen manufacture on a screw driven ATS tensile testing machine (Series 910). Tensile, compression and fracture toughness tests were conducted at a rate of 0.3 mm/sec.

**RESULTS:** Scanning electron microscopy (SEM) of the milled 15 $\mu$ m zirconia fibers show that zirconia fibers (shown in Fig. a) are made by sintering nano-particles of zirconia. The length distribution of the milled fibers indicates a normal distribution with fiber lengths ranging between 10 $\mu$ m and 500 $\mu$ m. The average fiber lengths for the 15 $\mu$ m and 30 $\mu$ m diameter fibers were 233 $\mu$ m and 200 $\mu$ m (aspect ratios of 15 and 7), respectively.

Tensile testing of fibers indicated that the 15 $\mu$ m fibers had 16 % higher ultimate tensile strengths as compared to the 30 $\mu$ m fibers (397  $\pm$  96 MPa vs. 341  $\pm$  68 MPa, p< 0.01).

In fibers subjected to emulsion polymerization, SEM indicated that individual zirconia fibers were coated with acrylic (Fig. b). The strength of individual zirconia fibers did not change due to the coating process (p = 0.84). By coating the fibers, the apparent diameters of the 15  $\mu$ m thick fibers increased by 100% indicating that the fibers were uniformly coated with a thick acrylic coating (Fig 1b).

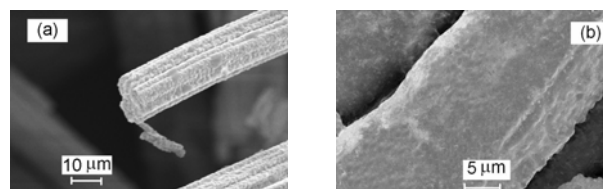


Figure 1) Representative images of the zirconia fiber before coating and after coating using the emulsion polymerization technique.

Table I	Elastic Modulus (GPa)	Ultimate Strength (MPa)	K <sub>IC</sub> (MPa m <sup>1/2</sup> )
Control (Barium sulphate)	3.01 $\pm$ 0.18 <sup>a</sup>	41.1 $\pm$ 6.3 <sup>a</sup>	1.47 $\pm$ 0.11 <sup>a</sup>
2% Zirconia, 15 $\mu$ m	3.76 $\pm$ 0.15 <sup>b</sup>	46.4 $\pm$ 4.2 <sup>a,b</sup>	1.79 $\pm$ 0.08 <sup>b</sup>
5% Zirconia, 30 $\mu$ m	4.30 $\pm$ 0.28 <sup>c</sup>	50.1 $\pm$ 5.9 <sup>b</sup>	2.08 $\pm$ 0.13 <sup>c</sup>
2% Acrylic Coated Zirconia, 15 $\mu$ m	3.65 $\pm$ 0.21 <sup>b</sup>	44.2 $\pm$ 5.3 <sup>a,b</sup>	1.82 $\pm$ 0.12 <sup>b</sup>
5% Acrylic Coated Zirconia, 30 $\mu$ m	4.25 $\pm$ 0.31 <sup>c</sup>	51.1 $\pm$ 6.5 <sup>b</sup>	2.10 $\pm$ 0.13 <sup>c</sup>

Similar letters indicate that the groups are similar

The mechanical properties of acrylic bone cements were improved by addition of zirconia fibers to acrylic bone cements. These zirconia volume fractions were chosen because they were the highest volumes that could be incorporated without increasing the solid: liquid ratios. Elastic modulus, ultimate strength and fracture toughness were improved by use of zirconia fiber reinforcements (Table 1) without significant decreases in ultimate strains. Similar results were observed in compression testing. Acrylic coating of fibers did not influence the tensile, compressive or fracture properties of bone cements significantly (Table 1). The improvement in mechanical properties was larger for the 30 $\mu$ m diameter fibers as compared to the 15 $\mu$ m fiber diameters. The largest improvements, namely, 24% in ultimate strengths and 43% in fracture toughness, were obtained with the 30 $\mu$ m diameter, acrylic coated fibers.

**DISCUSSION:** In this study, two novel methods are introduced for reinforcement of acrylic bone cement: (i) the use of zirconia in a fibrous form, as opposed to powder and (ii) coating of individual zirconia fibers with acrylic to facilitate bonding and to prevent leaching of particulate into articulating surfaces. The use of zirconia fibers resulted in improved mechanical properties of reinforced bone cements. Fibers used in this study exhibit 0.33-0.5 times the strength obtained by others [4], suggesting that larger increases in reinforcement efficiency could be obtained by improving fiber strength. The mechanical properties of bone cement were not significantly improved by the improved bonding between the acrylic and the fiber surface resulting from coating the fibers using the emulsion polymerization technique as there are little differences in mechanical properties when acrylic coated fibers are used as compared to uncoated fibers. This may be because of the lower molecular weights of PMMA in the coating. As increases in fracture toughness have been related to increased fatigue life<sup>2</sup>, these results suggest that the fatigue life of bone cements could be improved by using zirconia fibers. Moreover, the use of acrylic coated zirconia might prevent wear and other deleterious issues resulting from zirconia particles leaching from zirconia particle bone cements.

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**References:** (1) Lewis G, 1997, J. Biomed. Mat. Res. 38:155-182. (2) Topoleski LDT et al., 1998, Biomaterials. 19:1567-1577. (3) Gilbert JL et al., 1995, Biomaterials. 16:1043-1055. (4) Wegmann et al., 1998, CFI-Ceramic Forum International, 75:35-37.