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Fatigue limit of Y-TZP reinforced with carbon nanotubes

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Purpose/aim: To compare the Cyclic Fatigue Limit (CFL) of a control yttria-stabilized tetragonal zirconia pollycristal (Y-TZP) with a composite produced by adding multi-walled carbon nanotubes (CNT) into Y-TZP.

Materials and methods: CNT were coated with zirconium oxide and yttrium oxide to form a powder (CNT/ZYO) using a hydrothermal co-precipitation method. Powders made of Y-TZP + (CNT/ZYO) were produced using 99 vol% of Y-TZP and 1 vol% of CNT/ZYO. CAD-CAM blocks (42.5 × 16.0 × 16.0 mm) were obtained by uniaxial pressing (67 MPa/30 s) of each powder in a steel matrix. These blocks were partially sintered at argon atmosphere (1100 °C/1 h/5 °C/s) and then sectioned to produce 14 bar-shaped specimens (3.0 × 4.0 × 25.0 mm/edges chamfered according to ISO6872:2008) of each material, which were sintered also in argon atmosphere (1.400 °C/4 h/5 °C/min). Density measured by Archimedes' method was used to calculate the relative density (RD), based on the theoretical values for both materials (6.06 g/cm^3). Flexural strength (FS) was measured in four-point bending with specimens immersed in water at 37 °C (inner and outer supports of 10 and 20 mm). CFL was determined in four-point bending, using the staircase method (10,000 cycles/5 Hz). In each cycle, the stress varied between the maximum stress (MS) and 50% of MS. The applied stress in the first specimen was 50% of FS. After 10,000 cycles, in case the specimen did not fracture, 10 MPa was added to the next specimen. RD and FS were analyzed by Student's *t* test (alpha = 5%). CFL was calculated according to: CLF = X0 + *d*(SUMini/SUMni ± 0.5), where X0 is the lowest stress value tested, *d* is the stress added or subtracted to each cycle and n is the number specimens that survived or failed in each stress level. The lowest stress level was computed as $i = 0$, and the next one was computed as $i = 1$, and so on. Fracture surfaces were fractographically analyzed.

Results: Specimens containing nanotubes showed significantly lower RD compared to the control ($p = 0.009$). Nanotube addition also caused a 50% significant decrease in FS ($p = 0.003$). However, the FS coefficient of variation for the control was higher (17%) compared to that of the composite (10%). CFL calculated for the control was 2.5 times higher than that of the composite. The %CFL (CFL in terms of percentage of the FS) was also higher for the control. Fractography indicated fracture origins associated to surface defects and porous regions related to nanotube agglomerates.

Conclusions: The processing method used to produce the composite Y-TZP/nanotubes needs to be improved since nanotube addition to Y-TZP caused a significant reduction of the relative density, strength and fatigue limit.

Material	Relative density (%)	Flexural strength (MPa)	CFL (MPa)	%CFL
Y-TZP (control)	98.6 ± 0.05^A	623.7 ± 108.8^A	439.0 ± 56.4	70%
Composite Y-TZP/ nanotubes	97.4 ± 0.03^B	299.4 ± 30.5^B	179.4 ± 22.5	60%

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Characterization and cellular response of 3D-scaffold functionalized with PLA nanofibers

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Purpose/aim: Characterized the physicochemical properties, microtopography and cellular response of a 3D scaffold functionalized with PLA nanofibers.

Materials and methods: Cylindrical 3D scaffolds (5 mm diameter and 20 mm height) were designed and fabricated by PLA 3D printing. The scaffolds were then functionalized with 6% PLA nanofibers by airjet spinning. Samples of each scaffold were evaluated by thermogravimetric analysis (TGA) to determine onset point (To) inflection point and (Tp) mass loss temperature (Tmax). All specimens were then evaluated by differential scanning calorimetry (DSC) to determine glass transition temperature (Tg) and melting point (Tm). Tg was confirmed by dynamic mechanical analysis (DMA). Microtopography was evaluated by scanning electron microscopy (SEM) at 100×, 500×, 2000× and 5000×. Human osteoblasts (hOB) were selected to perform cellular adhesion assay and cellular proliferation evaluation by MTT assay. Data were analyzed and compared using two-way ANOVA test.

Results: To, Tp and Tmax were comparable between both scaffolds, as well as Tg and Tm. 3D scaffold showed homogeneous non-porous microtopography, with well adapted printing patterns. The PLA nanofibers layer was well adapted