Fatigue limit of Y-TZP reinforced with carbon nanotubes


1 University City of São Paulo/UNICID, Brazil
2 Institute for Energetic and Nuclear Research/Ipen, Brazil
3 University of the State of São Paulo/UNESP, Brazil
4 Friedrich-Alexander-Universität Erlangen-Nürnberg, Germany
5 University of São Paulo, Brazil

**Purpose/aim:** To compare the Cyclic Fatigue Limit (CFL) of a control yttria-stabilized tetragonal zirconia polycrystal (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/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/cm³). 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: \( CLF = X_0 + d(SUMini/SUMni ± 0.5) \), where \( X_0 \) is the lowest stress value tested, \( d \) is the stress added or subtracted to each cycle and \( n \) is the number of 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.

<table>
<thead>
<tr>
<th>Material</th>
<th>Relative density (%)</th>
<th>Flexural strength (MPa)</th>
<th>CFL (MPa)</th>
<th>%CFL</th>
</tr>
</thead>
<tbody>
<tr>
<td>Y-TZP (control)</td>
<td>98.6 ± 0.05 a</td>
<td>623.7 ± 108.8 a</td>
<td>439.0 ± 56.4</td>
<td>70%</td>
</tr>
<tr>
<td>Composite Y-TZP/ nanotubes</td>
<td>97.4 ± 0.03 a</td>
<td>299.4 ± 30.5 b</td>
<td>179.4 ± 22.5</td>
<td>60%</td>
</tr>
</tbody>
</table>

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

F.C. Vazquez-Vazquez 1, D. Chavarria-Bolaños 2, D. Villalobos-Vega 2, J. Vega-Baudrit 3, A. Pozos-Guillén 4, D. Masuoka-Ito 5, M. Álvarez-Pérez 1

1 Universidad Nacional Autónoma De México, Cdmx, Mexico
2 Universidad De Costa Rica, San José, Costa Rica
3 Centro Nacional De Alta Tecnología, Laboratorio Nacional De Nanotecnología, San José, Costa Rica
4 Universidad Autónoma De San Luis Potosí, San Luis Potosí, Mexico
5 Universidad Autónoma De Aguascalientes, Aguascalientes, Mexico

**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 (Tb) 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.
on the 3D scaffold. The thickness and the distribution of PLA nanofibers were homogeneous in all the areas analyzed. The deeper nanofibers showed good adaptation and merge with printed scaffold. Functionalized scaffolds showed improved cellular adhesion and proliferation ($p < 0.05$) when compared with pure 3D scaffolds.

**Conclusions:** It was possible to functionalized 3D PLA cylindrical scaffolds, maintaining physical properties but improving the cellular response.

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**Bond strength of calcium-releasing self-adhesive cement after accelerated aging**

L. Chen, C. Gleave, B.I. Suh

Bisco Inc, Schaumburg, IL, USA

**Purpose/aim:** Calcium-releasing bioactive dental cements may help prevent or decrease dental secondary caries. However, it is still unknown whether they have stable bond strengths. The purpose of this study was to evaluate bonding durability of a calcium-releasing self-adhesive resin cement (TheraCem).

**Materials and methods:** Yttria-stabilized zirconia ceramic was sandblasted with alumina sand, rinsed and dried. Shear bond strength was tested using the notched-edge shear bond strength test method (ISO 29022:2013). Self-adhesive cement (TheraCem, Bisco; or RelyX UniCem2, 3M Oral) was placed on the substrate surface and self-polymerized (15 min/37°C). The specimens were then stored in water either at 37°C for 6 days (accelerated aging), or at 80°C for 6 days (accelerating aging), and tested by universal testing machine (Instron, crosshead-speed 1 mm/min). The data were analyzed statistically by one-way ANOVA and Student’s $t$-test.

**Results:** Mean shear bond strengths in MPa (standard deviation) are shown in **Table 1**. Means with different letters are statistically different ($p < 0.05$).

**Table 1**

<table>
<thead>
<tr>
<th>Resin cement</th>
<th>TheraCem</th>
<th>UniCem2</th>
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<tbody>
<tr>
<td>Initial (37°C for 24h), n=6</td>
<td>26.8 (8.9) a</td>
<td>18.7 (6.2) b</td>
</tr>
<tr>
<td>Aging (80°C for 6 days), n=7</td>
<td>19.1 (4.7) ab</td>
<td>6.0 (2.1) c</td>
</tr>
<tr>
<td>Mean bond value decrease</td>
<td>29% ab</td>
<td>64% c</td>
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**Conclusions:** The calcium-releasing self-adhesive resin cement, TheraCem, had a more stable bond than RelyX UniCem2.

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**Reciprocity studies on polymerization properties of BisGMA:TEGDMA based composites**

S.V. Palagummi 1, T. Hong 1, L. Jiang 2, E. Song 1, M.Y.M. Chiанг 1,∗

1 National Institute of Standards and Technology, Gaithersburg, USA
2 West China College of Stomatology, Sichuan University, China

**Purpose/aim:** Exposure reciprocity law has often been used as a rationale to adjust the curing duration based on the curing light intensity available to the dentist. This study examines the clinical validity of this rationale by real time simultaneous measurement of the polymerization properties for different light intensities, under a constant dose. These properties include shrinkage stress (PS), degree of conversion (DC) and temperature change (TC) due to exotherm and energy absorption of model dimethacrylate-based composites during the polymerization process.

**Materials and methods:** Mixtures of BisGMA:TEGDMA (7:3, 1:1 and 3:7) with commercial silanized micro-sized glass filler loading levels of 50 wt% and 75 wt% each were investigated. NIST SRI 6005 (standard reference instrument, a cantilever-beam based instrument) with an in situ near infrared spectrometer and a microprobe thermocouple was used for simultaneous measurement of PS, DC, and TC in real time. Light irradiance intensities were varied from 500 mW/cm² to 4000 mW/cm² while maintaining a constant dose of 10 J/cm² and 20 J/cm². Initially, a standard continuous curing mode was considered in this study. Measurements were carried out under three instrumental compliances, 0.33 m/N, 6.31 μm/N and 12.32 μm/N, that cover possible compliances of clinically prepared tooth cavities.

**Results:** The exposure reciprocity with respect to DC was observed for the composites, regardless the instrument compliance. The reciprocity with respect to PS was not followed under low compliance. However, the reciprocity with respect to PS at higher compliances was followed for the composites studied. The peak TC, which is independent of instrument compliance, increased significantly with intensity for all the composites tested.

**Conclusions:** Simultaneous and real-time measurements provide insight into inter-related kinetics of polymerization properties under various curing light intensities. Our preliminary results indicate that the exposure reciprocity studies should consider PS, DC and PE together to comment on its applicability for dental composites with varying constituents. More importantly, regarding the effects of reciprocity on PS with different curing light intensities, the relevant compliance of apparatus should always be considered.

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