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Mechanical characterization of optical glass fiber coated with a thin film of silver nanoparticles by nanoindentation

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ABSTRACT

Optical glass fibers coated with Ag nanoparticles are proposed as alternative material within a system of root dental fillings for endodontic therapy, due to their potential mechanical and antibacterial properties. A group of glass fibers filaments was covered with Ag nanoparticles prepared by the chemical reduction method and immersed in a solvent of tetrahydrofuran $(CH_2)_4O$ (THF). Nanoparticles achieve average size of 30 nm of Ag nanoparticles diameters. At 1 min of impregnation in THF on the surface of glass fiber cores, Ag nanoparticles formed thin films with better distributions with respect to the other impregnation times. Nano-mechanical properties such as hardness and elastic modulus were evaluated on the surface of optical glass fiber coated with Ag nanoparticles by nanoindentation using the continued stiffness measurements method. Hardness value was $H=3.6\pm0.17$ GPa and elastic modulus was $E=78.3\pm2.6$ GPa.

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1. Introduction

Gutta-percha points are still employed in current dentistry as conventional material to fill dental root canals during endodontics therapy; however, these materials manifest several inconvenient reactions *in-situ* like chemical incompatibility with adhesives restoratives and cementing materials derives from polymeric matrices. Gutta-percha points also manifest low sealing properties [1]. Optical fiber glasses tips covered with Ag nanoparticles are polymeric materials with light transmission properties and bacteriostatic qualities that may be proposed as a substitutive material with respect to gutta-percha points. Antibacterial effects of the Ag nanoparticles have been studied by different research works in the field of dental materials [2–5]. A large number of techniques have been used for the synthesis of nanoparticles [6]. Regarding the mechanical properties, dental filling materials undergo polymerization shrinkage that results in de-bonding tensions and gap formations along the canal walls. Inadequate polymerization is usually associated with poor mechanical properties; adequate polymerization increases the hardness, whereas moisture reduces it [7]. The mechanical and morphological degradation that human dentin undergoes after prolonged exposure in cola drinks was examined using nanoindentation and AFM. The elastic modulus of the dentin prior cola exposure was 18.28 ± 1.7 GPa, while after exposure it had decreased to 4.25 ± 1.4 GPa on the surface that was in direct contact with the drink, and to 13.38 ± 2.9 GPa on the surface that was in indirect contact with the drink [8]. In this work, on the surface of optical glass fibers, a group of glass fiber filaments was covered with Ag nanoparticles prepared by the chemical reduction method by impregnation in solvents of THF. Optical glass fibers coated with Ag nanoparticles are proposed as alternative material within a system of root dental fillings for endodontic therapy, due to their potential mechanical and antibacterial properties granted to the surface of glass fiber cores. Mechanical properties such as hardness and elastic modulus were evaluated on the surface of optical glass fibers coated with Ag nanoparticles in forming thin films by nano-indentation using the continued stiffness measurements method (CSM).

2. Materials and methods

Synthesis of silver nanoparticles by chemical reduction and AgNP's dispersion on polymeric materials: Multimode optical glass fibers with mechanically strippable acrylate coating (255 μm outer diameter) were covered with Ag nanoparticles by using a dipolar aprotic THF solvent at 1 min of impregnation.

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The synthesis was performed using silver nitrate ($AgNO_3$) and sodium borohydride ($NaBH_4$) as a reducing agent, which was added in a 3:1 ratio (mM) compared to the precursor ($AgNO_3$). The solution of 3 mM $NaBH_4$ was placed in an ice bath at a starting temperature of 4 °C for 24 h, under constant stirring at 300 rpm and the addition of 1 mM $AgNO_3$ solution at the rate of 50 drops/min with a peristaltic pump and treatment stirring. After 24 h the solution was centrifuged at 12,000 rpm/30 min. Subsequently, the nanoparticles were dried at room temperature and recovered. By impregnation in THF solvents, Ag nanoparticles were adhered to the optimal impregnation at 1 min.

Experiment of nanomechanical properties: Nanomechanical properties such as hardness (H) and elastic modulus (E) of glass fibers coated with Ag nanoparticles were evaluated by means of nanoindentation using the continuous stiffness measurement (CSM) method, employing a Nano Indenter G200 coupled with a DCM II head. The equipment was calibrated using a standard fused silica sample. Tests parameters were as follows: the constants of area function were $C_0=24.05$, $C_1=-178.33$, $C_2=6724.30$, $C_3=-24407.23$, and $C_5=18701.80$. The Berkovich diamond indenter with a tip radius of 20 ± 5 nm, depth limit of 140 nm, strain rate of 0.05 s^{-1}, harmonic displacement and frequency of 1 nm and 75 Hz respectively, and Poisson's ratio of $\upsilon=0.25$ was used. Residual indentation of samples was recorded by the AFM Nano Vision system attached to the nanoindenter system.

3. Results and discussion

Fig. 1a shows the synthesized Ag nanoparticles by the chemical reduction method that was taken at 100,000 ×. In this micrograph the shape, distribution and size of the Ag nanoparticles are clearly observed. The diameters achieve an average size of 30 nm Ag nanoparticle.

Fig. 1b and c shows the SEM micrographs of the optic fiber without and with Ag nanoparticles coated on the surface of the glass fiber cores, respectively. Fig. 1c clearly shows the good dispersion across superficial areas without cracks on the Ag thin film. Moreover, at 30,000 × zoom the coated fiber shows the distribution and addition of the Ag nanoparticles.

On the other hand, to measure the nano-mechanical properties of the optics fiber without Ag NPs the Oliver and Pharr method with controlled cycles was used [9]. The basic analysis of nanoindentation load–displacement curve (P–h) was established based on the elastic contact theory given by Sneddon [10] and Doerner [11].

The nanomechanical properties of the optics fiber without Ag NPs were stiffness $S=500$ N/m, very low hardness $H=0.058$ GPa=58 MPa and elastic modulus $E=2.9$ GPa.

Hardness (H) and elastic modulus (E) of Ag thin films, which were formed by the Ag nanoparticles, were calculated by the continuous stiffness measurement (CSM) method. The CSM method enables a continuous measurement of stiffness during loading, and not just at the point of initial unloads [12,13].

Fig. 2b and c shows the characteristics load–displacement curves of the optic fiber without and with Ag nanoparticles coated on the surface of glass fiber cores, respectively. In these typical curves we clearly see that the optic fiber without Ag nanoparticles coated is softer than with Ag nanoparticles coated, due to the residual displacement depth of hr=350 nm and hr=120 nm, respectively. On the other hand, Fig. 2a and d shows representative residual indentation images of the optic fiber without and with Ag nanoparticles coated on the surface of glass fibers, respectively. Fig. 2d shows the AFM micrograph in contact mode tacked in an area of 1 μm × 1 μm in the Ag thin film of the optic fiber where the nanoindentation was tested and Fig. 2e shows the prefill of an area residual after the nanoindentation test. This figure clearly shows

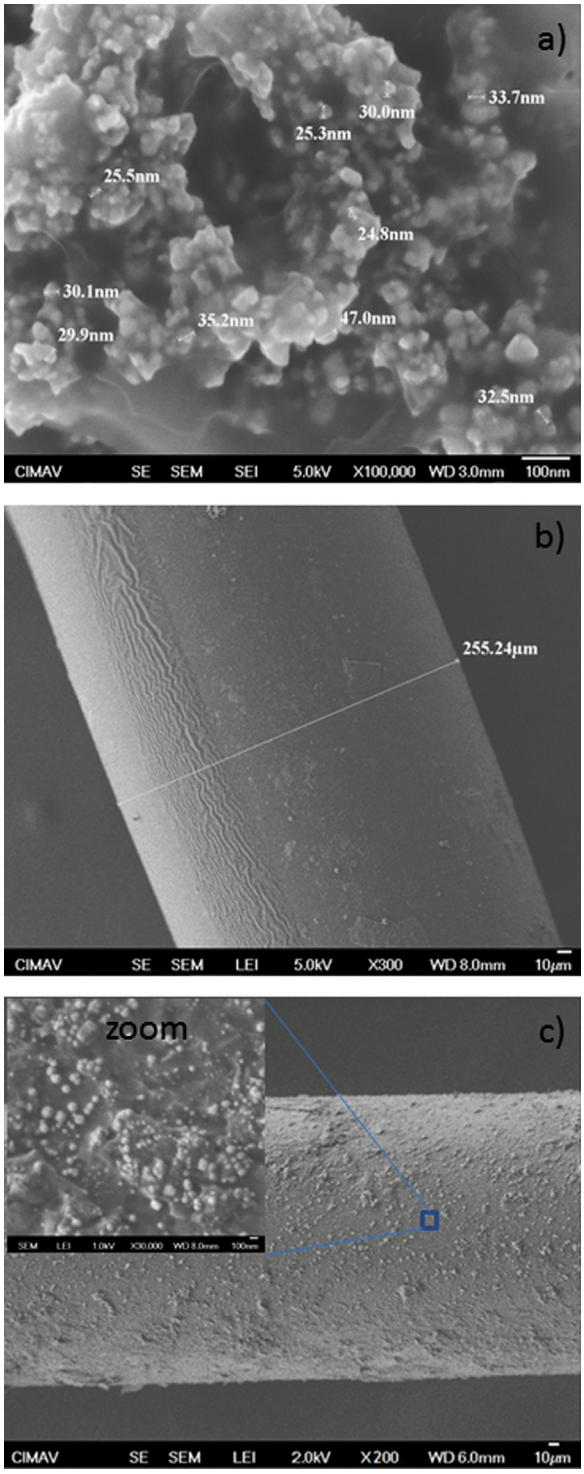

Fig. 1. SEM micrographs: (a) synthesized Ag nanoparticles by the chemical reduction method, and (b) and (c) of the optic fiber without and with Ag nanoparticles coated on the surface of glass fiber cores, respectively.

the homogeneity of the coated Ag nanoparticle. Additionally, it is possible to observe the size and depth of the nanoindentation.

Fig. 3a shows the mechanical behavior of the hardness *versus* displacement in the surface or penetration depth of the Ag thin film and optical glass fiber. This figure shows two regions. First is in the interval of the A–B (0–100 nm of h), that is with reference to the thin film coated by the Ag nanoparticles where the hardness has a value of $H=3.6\pm0.17$ GPa. The second region is the B–C (100–140 nm of h) where the hardness is influenced by the optic

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