

PERFORMANCES EVALUATION OF A BIS-GMA RESIN-BASED COMPOSITE FOR DENTAL RESTORATION

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[Valutazione delle performances di un composito a base di resina Bis-GMA per restauro dentale]

ABSTRACT

The aim of this in vitro study was to evaluate the mechanical properties of a commercial Bis-GMA resin loaded with filler particles including Ba-Al-F-silicate glass (size 0.02-2.00 μm) and dispersed colloidal silica (size 0.02 to 0.07 μm). The study was conducted through the preparation of samples with thickness of 1.5 mm, length of 25 mm and width of 4.7 mm. Each samples of the resin composite was irradiated with the standard 20s polymerization mode using a LED lamp.

The experimental results of the flexural test and micro-hardness test evidenced that the layering technique influences the mechanical performances of the resin-based composite. Particularly, the samples evidence a sandwich structure with a core and two external skins characterized by different hardness and stiffness.

Key words: Resin composites, micro-hardness test, flexural test, resin polymerization, layering technique.

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Introduction

Resin-based composites have been introduced for the first time in the middle of 1960 as commercial materials for dental restoration, due to their excellent characteristics, such as mechanical properties, simple usability, aesthetic properties and biological compatibility⁽¹⁻⁵⁾. For these reasons, these resins have been widely used instead of traditional dental materials, which present problems such as cytotoxicity, corrosion and poor aesthetic properties⁽⁶⁻⁷⁾.

Such composites for dental use contain primarily a resin-based matrix, inorganic fillers and curing initiators. The matrix commonly used for resin composites in restorative dentistry is based on 2,2-bis [p-(2'-hydroxy-3'-methacryloxypropyl)-phenyl] propane (Bis-GMA)⁽⁸⁻⁹⁾, thanks to its high mechanical strength and low shrinkage value after curing. The addition of a large amount of inorganic particles to the polymeric structure, moreover,

allows an improvement of the mechanical performance⁽¹⁰⁻¹²⁾.

However, a specific experimental research could be useful in order to relate the performance of the composite with the layer increment technique of the product during the dental reconstructive phase. In example, the curing time and the number of layers are important parameters that affect the catalysis of the resin and consequently the mechanical properties of the final product.

In the present work the characterization of a resin composite, based on Bis-GMA reinforced with an inorganic (glass and colloidal silica) filler, was performed. The characterization was carried out by evaluating some of the most important mechanical properties of the material, such as the micro-hardness and flexural stiffness. In particular the variation of stiffness and hardness was evaluated at varying layers deposition. In addition, the correlation of these parameters has provided important

information on the layering technique in restorative phase.

Experimental part

Sample preparation

We used a commercial light-curing resin (Quadrant Universal LC, supplied by CAVEX HOLLAND BV). It is a Bis-GMA resin loaded with well dispersed fillers (Ba-Al-F-silicate glass, size 0.02-2.00 μm and dispersed colloidal silica, size 0.02 to 0.07 μm).

The uncured resin composite was placed in a silicone mold and irradiated with visible light by using a LED lamp (light-emitting-diodes) for a polymerization time of 20 s for each layer (about 1.5 mm). Preliminarily, according to ASTM D 3171, common physical parameters of the product were identified. The results are summarized in Table 1.

Filler Vol %	68.0
Filler Wt %	56.7
Density [g/cm ³]	2.075
Voids [%]	2.42

Table 1. Physical properties of the composite sample

Flexural test

Three point bending test was performed according to ISO 4049. The test was replicated 10 times to better identify the statically the mechanical performances. The flexural properties were measured using rectangular samples (length, $L = 25$ mm, thickness, $s = 1.5 - 3.0$ mm and width, $w = 4.7$ mm). The distance between supports, l , was 18.5 mm. The Young's modulus (E) and flexural strength (σ) were determined by the following expressions:

$$E = l^3 F / 4bd^3 y$$

$$\sigma = 3lF / 2bd^2$$

where l is the length between the supports, F is the applied load, b is the width of the specimen, d is the thickness of the specimen and y is the deflection.

Micro-hardness test

Micro-hardness tests were performed by using a FUTURE-TECH MICROHARDNESS TESTER FM-300 e. Hardness is defined as the resistance of a material to indentation or penetration. It has been used to predict the wear resistance of a material and its ability to abrade or be abraded by opposing tooth structures. We used a Vickers indenter and a com-

pressive load of 50g. The Vickers hardness, HV, was calculated as average of 20 measurements for sample.

Results and discussion

The three points bending test was used to examine the mechanical behavior of such material, used for dental restoration, because this test is able to give information about the compressive (adjacent to the point of application of the load) and tensile (on the opposite side of the sample) behavior of the composite material. Figure 1 shows the Young's modulus E (gray curve) and the flexural strength σ (black curve) as a function of the deformation ϵ of a reference sample.

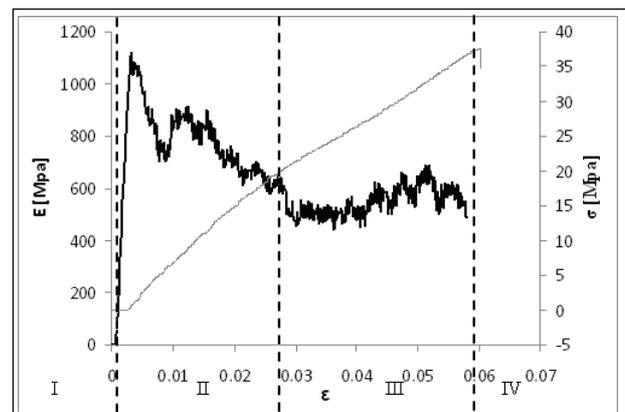


Figure 1: Young's modulus E (gray curve) and flexural strength σ (black curve) vs. deformation ϵ of a reference sample.

Four regions can be identified:

I. Zone I is related with the experimental set-up and it has not a real physical meaning.

II. In zone II it is observed a drastic increase of the elastic modulus up to a maximum value is reached at about 1100 MPa. Afterwards the modulus progressively decreases.

III. In zone III a local plastic deformation of the sample occurred. The sample evidenced a constant elastic modulus of about 570 MPa.

IV. The zone IV identifies the fracture failure of the sample.

A similar behavior was observed for all tested samples. By analyzing the experimental data a bimodal elastic behavior is clearly evidenced, related directly with the resin polymerization. "In fact the composite structure is characterized by a core region with good stiffness and strength and two soft skins with very poor mechanical performances". The plastic collapse of the skins is responsible of

the transition from zone II to zone III.

With the purpose to evaluate the geometrical thickness of the stiff and soft layers a micro-hardness map of the cross section of the composite samples was carried out. The results for a two-layer sample are reported in figure 2. The two composite laminae can be clearly distinguished. The layer I received two curing steps (20 second each) despite layer II that received only one curing step.

The middle region of layer I (dark zone in the map with high hardness values, HV ~ 110) is very hard compared with that of layer II (light gray zone in the map, HV ~ 90); moreover both layers have an external soft skin with poor polymerization, evidenced by the low micro-hardness values, (dark zone with low hardness values, HV ~ 30).

From the map reported in figure 2, these discriminated regions can be dimensionally evaluated.

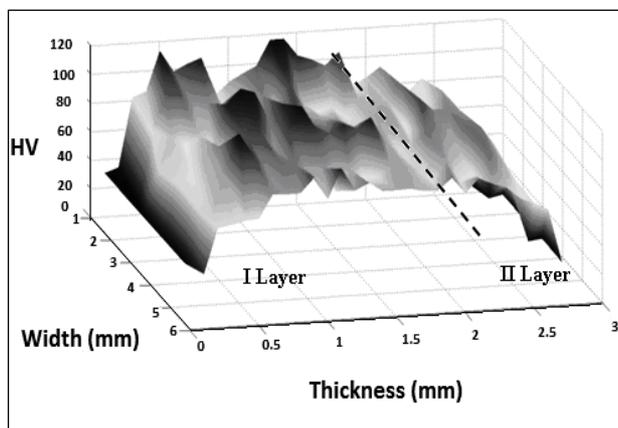


Figure 2: 3D map of micro-hardness HV of a two-layer reference sample.

About the layer I, the external soft skin has a thickness of about 0.25 mm, the hard core is thick about 1.75 mm, while the layer II has a thinner hard core (about 1 mm) and a thicker soft external skin (about 0.375 mm). Table 2 summarizes the mechanical performances of a mono-layer composite sample.

	Average	Standard deviation
Sigma compr [MPa]	157.76	30.43
sigma flex [MPa]	31.54	5.7
E flex [MPa]	854.91	152.28
HV [MPa]	99.72	5.22

Table 2: Mechanical performances of a mono-layer composite sample.

Conclusions

The experimental results, carried out on a commercial BIS-GMA resin reinforced with an inorganic filler, evidenced that the layering technique, necessary to realize the dental reconstruction, influences the mechanical performances of the resin composite. After the curing, the samples evidence a sandwich structure with a core with very high mechanical properties and two external skins with low properties. The application of cure times higher allows obtaining a stiffer resin composite, reducing the soft skin layers.

Consequently, as a result of this structural anisotropy, the stiffness and strength of the specimen is lower than in full cure conditions (where no-soft skin layers can be observed). From the dental point of view, the mechanical behavior of the reconstructed tooth is the result of the combination of the performance of different layers, characterized by different hardness and stiffness. This information may be useful in order to provide a correct procedure of deposition and curing of the resin composite during dental reconstruction. In this sense, a more detailed research will be developed to verify the stress distribution for a dental multilayer system in order to be able to predict the final performance of a real reconstructed tooth.

References

- 1) J.Z. Luo, R. Seghi, J. Lannutti. "Effect of silane coupling agents on the wear resistance of polymer-nanoporous silica gel dental composites". *Mater. Sci. Eng. C* 5 (1997) 15.
- 2) K.S. Wilsona, K. Zhang, J.M. Antonucci. "Systematic variation of interfacial phase reactivity in dental nanocomposites". *Biomaterials* 26 (2005) 5095.
- 3) L. Musanje, J.L. Ferracane. "Effects of resin formulation and nanofiller surface treatment on the properties of experimental hybrid resin composite". *Biomaterials* 25 (2004) 4065.
- 4) H.H.K. Xu, J.B. Quinna, D.T. Smith, J.M. Antonucci, G.E. Schumacher, F.C. Eichmiller. "Dental resin composites containing silica-fused whiskers – Effects of whisker-to-silica ratio on fracture toughness and indentation properties". *Biomaterials* 23 (2002) 735.
- 5) Y. Xia, F.M. Zhang, H.F. Xie, N. Gu. "Nanoparticle-reinforced resin-based dental composites". *J. Dent.* 36 (2008) 450.
- 6) H.H.K. Xu, F.C. Eichmiller, J.M. Antonucci, G.E. Schumacher, L.K. Ives. "Dental resin composites containing ceramic whiskers and precured glass ionomer particles". *Dent. Mater.* 16 (2000) 356.

- 7) V.M. Karbhari, H. Strassler. "Effect of fiber architecture on flexural characteristics and fracture of fiber-reinforced dental composites". Dent. Mater. 23 (2007) 960.
- 8) J.R. Condon, J.L. Ferracane. "Reduced polymerization stress through non-bonded nanofiller particles". Biomaterials 23 (2002) 3807.
- 9) D.A. Bindslev. "The environmental effects of dental amalgam". Adv. Dent. Res. 6 (1992) 125.
- 10) Tinschert J, Zvez D, Marx R, Anusavice KJ. "Structural reliability of alumina-, feldspar-, leucite-, mica- and zirconia-based ceramics". J Dentistry 2000; 28: 529-35.
- 11) Willems G, Lambrechts P, Braem M, Celis JP, Vanherle G. "A classification of dental composites according to their morphological and mechanical characteristics". Dent Mater 1992; 8: 310-9.
- 12) Leinfelder KF. "New developments in resin restorative systems". J Am Dent Assoc 1997; 128: 573-81.

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