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EVALUATION OF THE MECHANICAL BEHAVIOR OF G-AENIAL RESTORATIVE COMPOSITES USING MULTI-CYCLING INDENTATION

Adriana-Madalina TURCANU (CONSTANTINESCU), Felicia STAN, Catalin FETECAU

Abstract: *During the mastication process, the restorative materials are exposed to dynamic loading as tooth cusps and hard foods cyclically indent these materials. In this work, the indentation technique is used to investigate the mechanical behavior of G-ænial under various multi-cycling nanoindentation conditions and to study the mechanical properties, such as elastic modulus and hardness. First, static indentation tests were performed to assess the effect of the holding time on the mechanical behavior of G-ænial. Then, a series of dynamic indentation tests were conducted in which the number of indentation cycles varied from 6 to 30. The experimental results indicated that both the static indentation hardness and elastic modulus decrease with increasing holding time from 0 s to 30 s. Moreover, the elastic modulus and indentation hardness of the G-ænial composite decreased with the increasing number of cycle loadings, indicating that the restorative composite becomes more susceptible to fatigue damage.*
Keywords: *restorative material, nano-indentation, elastic modulus, hardness, G-aenial.*

1. INTRODUCTION

Traditional materials such as amalgam, gold, and porcelain, traditionally used in dentistry, are now being replaced by composite resin dental materials, due to their improved adhesion, aesthetics, and physical properties.

A primary criterion for classifying composite resins is the size of the filler. Micro- and nano-sized fillers, such as colloidal and fumed silica, glass (borosilicate), and quartz (crystalline silica) are found in most composite resins [1, 2]. To create hybrid composite resins, organic fillers of various sizes are incorporated into their structure. Submicron-sized particles (40 nm) are also embedded within larger particles (10-50 μm) to enhance the properties of these materials [3, 4]. Therefore, to improve their efficiency, it is essential to evaluate the mechanical properties of these composite materials at the micro and nano scale.

During the mastication process the restorative materials are exposed to dynamic loading, as tooth cusps and hard foods cyclically indent these materials. As shown in the literature, two and three-body wear test can be used to simulate the phenomena that occur in the oral cavity during the mastication process [5, 6]. To simulate the chewing process, multi-cyclic indentation can be employed as an alternative [7, 8]. This process consists of repeatedly applying maximum force to the same location, while maintaining the loading and unloading rates.

After a series of indentation tests performed on different types of composites such as Charisma Diamond, Clearfil AP-X and Surefil, Cao *et al*. [9] demonstrated that each composite resin has a distinct performance, e.g. hardness, that depends on each formulation. Furthermore, it has been reported that the presence of fillers in the resin matrix influences both hardness and wear resistance of the analyzed composites. Nanofill composite exhibits better mechanical properties compared to composite resins [9].

Hirayama *et al.* [10] studied the indentation mechanical properties of flowable composites like Clearfil AP-X, Estelite, Filtek, and MI Flow, applying a holding time of 15 s to reduce the time-dependent elastic effects. The results demonstrated that the properties like hardness and elastic modulus of this composites are influenced by the filler and the properties of the resin matrix. Flowable composites exhibit low mechanical properties due to the properties of the resin matrix, which has a lower filler content than conventional composites [10].

G-aenial is a new class of resrorative composites that provides several benefits such as natural-looking, high-gloss restorations and increased resistance to leakage during the restoration process [11, 12]. However, as far as we know, this composite was not investigated using the multi-cycle indentation approach.

In this work, the viscoelastic properties of G-aenial are investigated by nano-indentation testing using a Berkovich indenter. To assess the behavior of G-aenial, static and dynamic indentation tests were first performed under various conditions. Then, the indentation hardness and elastic modulus were determined based on the Oliver and Pharr method [13] as a function of holding time and number of indentation cycles.

2. MATERIAL AND METHODS

2.1 Material and sample preparation

G-ænial A'CHORD (GC EUROPE N.V., Leuven, Belgium) is a hybrid composite that incorporates two types of prepolymerized resin fillers in its structure. The composition of this material includes fillers, photoinitiators, pigment and the base matrix (methacrylate monomers, silica, flour, fumed silica) [14].

In order to obtain optimal samples for indentation testing, a polylactic acid (PLA) mold was 3D-printed using a Prusa i3MK3S (Prusa Research, Prague, Czech Republic) printer. The material was deposited layer by layer in the mold, with each layer being dried in a Transformer UV lamp for 60 s (Fig. 1). Then, the top surface of the sample underwent a grinding and finishing process.

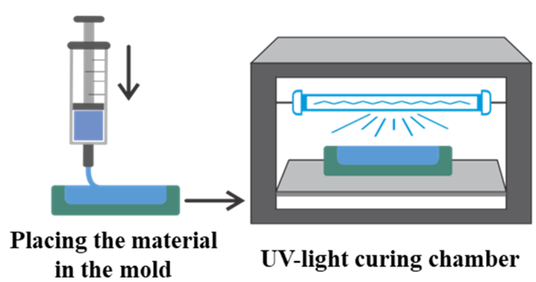

Fig. 1. Schematic representation of the deposition of G-aenial material in the matrix.

2.2 Nano-indentation testing

Static and multi-cycling indentation tests were carried out using the NHT nano-indentation tester (Anton Paar GmbH, Graz, Austria) with a Berkovich tip (Fig. 2).

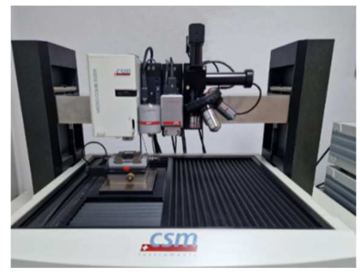

Fig. 2. NHT nano-indentation tester.

To investigate the behavior of the G-aenial restorative composite, three types of indentation tests were performed, as presented in Fig. 3: (1) a 2-step indentation test (Fig. 3a) with linear loading to the maximum load followed by linear unloading; (2) a 3-step indentation test (Fig. 3a) involving linear loading to the maximum load, holding, and linear unloading; and (3) a multi-cycle indentation test (Fig. 3b).

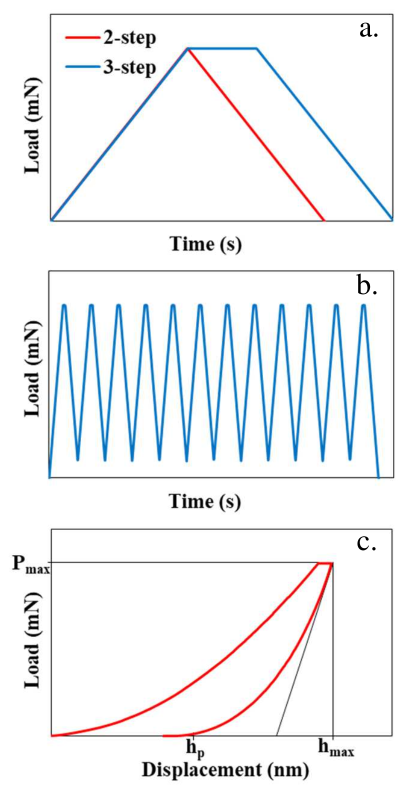

Fig. 3. Schematic representation of the indentation loading profile for a. 2- and 3-step indentation, b. multi-

cycle indentation and c. load-displacement indentation curve.

The multi-cycle indentation process consists of applying the maximum force, P_{max}, for a number of times in the same location on the indented sample and determining the mechanical properties following the contact between the indenter and the sample [7, 8].

Figure 3c represents the load-displacement curve resulting from the 3-step indentation test. The maximum displacement, $h_{\max}$, is achieved at the end of the holding segment. After unloading, some residual indentation displacement, h_p, occurs due to plastic deformations during the loading stage [15, 16].

The holding stage during the 3-step indentation test plays an important role in reducing the time-dependent phenomena that may develop during the loading stage [9, 15, 17]. During the holding stage, the indented material deforms to some extent, so that the creep deformation becomes negligible during the unloading stage. However, since the indentation depth increases with time during the holding stage, it is recommended that the holding time be in the range of 10 to 60 s, thus reducing the influence of the creep deformation on the slope of the unloading curve and, in turn, on the reduced modulus [15, 17].

Table 1 presents the test parameters used to determine the mechanical properties. All tests were performed by applying a maximum force of 100 mN. Since the G-aenial is a composite material, for each static mode, 12 indentations were performed in the form of a 4 x 3 matrix, at a distance of 0.4 mm from each other (Fig. 4), to analyze the homogeneity and repeatability of the properties.

Table 1

Testing parameters.

Number of cycles	Holding time (s)	Loading/unloading speed (mN/s)
1	0	100
1	10	100
1	30	100
6	10	100
12	10	100
18	10	100
24	10	100
30	10	100

The indentation hardness and elastic modulus were determined from the load-displacement curves using the standard Oliver and Pharr method [13].

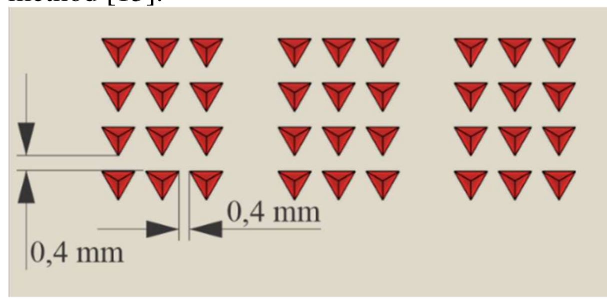

Fig. 4. Schematic representation of the indentation matrix.

The indentation hardness, H, is given by the ratio of the maximum applied force to the contact area developed under the action of the respective force [13, 16]:

$$H = \frac{P_{max}}{A_p} \tag{1}$$

where: P_{max} is the maximum applied force; A_p is the projected contact area.

The elastic modulus, E_{IT}, considers the elastic deformations of the indenter and the material to be studied, and is calculated using the following equation [11, 14]:

$$E_{IT} = \frac{1-\upsilon^2}{\frac{1}{E_r^*} - \frac{1-\upsilon_i^2}{E_i}} \tag{2}$$

where: E_r is the reduced modulus of elasticity; E_i =1140 GPa and υ_i =0.07 is the Young's modulus and Poisson's coefficient of the diamond indenter, respectively, and υ=0.3 is the Poisson's coefficient of G-aenial composite [14].

2.3. Statistical analysis

The values reported in this paper are the means of 12 independent replicates ± standard deviation. The effects of holding time and the number of indentation cycles on indentation properties were analyzed using one-way analysis of variance (ANOVA) with Tukey pairwise comparison at 0.05 significance level (p-value < 0.05). The statistical analysis was performed with Minitab 19 statistical software [18].

3. RESULTS

Figure 5 presents the load-displacement curves of the G-aenial at a maximum force of

100 mN for different holding times (0 s, 10 s, and 30 s). The load-displacement curve obtained from the 2-step indentation tests (0 s) indicates the presence of a small "nose" during the initial part of the unloading curves, indicating that the restorative material exhibits creep deformation at a higher rate than elastic recovery [16, 17]. Therefore, the assumption of elastic unloading is not applicable for the indentation of G-aenial [17]. However, the "nose" effect disappears with increasing holding time, as can be seen in Fig. 5.

Figure 6 presents a typical load-displacement curve under multi-cycle indentation for 30 indentation cycles with a 10 s holding time.

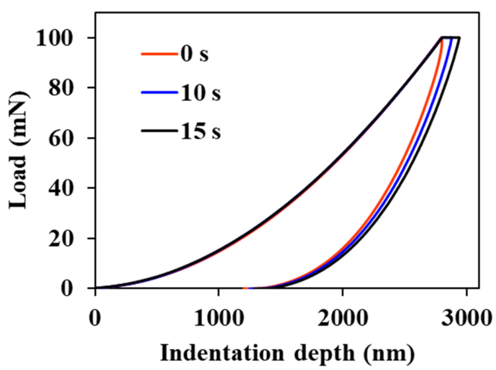

Fig. 5. Indentation load – displacement curve for G-aenial at diferent holding times.

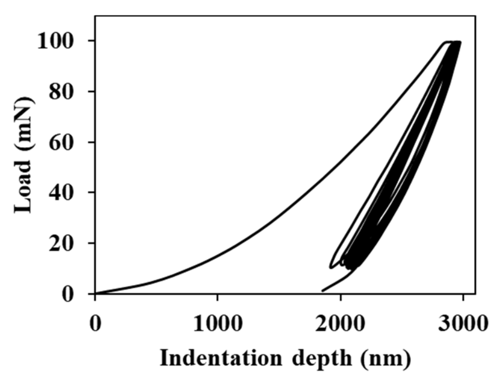

Fig. 6. Indentation load-displacement curve for G-aenial for 30 indentation cycles with 10 s holding time.

Figures 7a and 7b present the influence of the holding time on the indentation hardness and elastic modulus, respectively. It can be observed that the nano-indentation hardness of the G-aenial composite decreased from about 672 MPa to 642 MPa with an increase in holding time from 0 s to 30 s, respectively. However, statistical analysis of the experimental data indicates that the effect of holding time on the nano-indentation hardness is not statistically significant. On the other hand, the elastic modulus decreased about 15% with increasing holding time from 0 to 30 s (Fig. 7b), and the effect of holding time is statistically significant. This correlates with the presence of inelastic deformation during the unloading phase as indicated in Fig. 5.

Figure 8 presents the variation of mechanical properties (e.g., indentation hardness and elastic modulus) with the number of indentation cycles. The indentation hardness of the G-aenial composite (Fig. 8a) decreased from about 700 MPa to 625 MPa with increasing the number of cycles from 6 to 30, respectively. ANOVA Table 2 shows that the effect of the number of cycles is statistically significant ($p<0.05$). However, after 6 cycles, the decrease in the hardness is minimal.

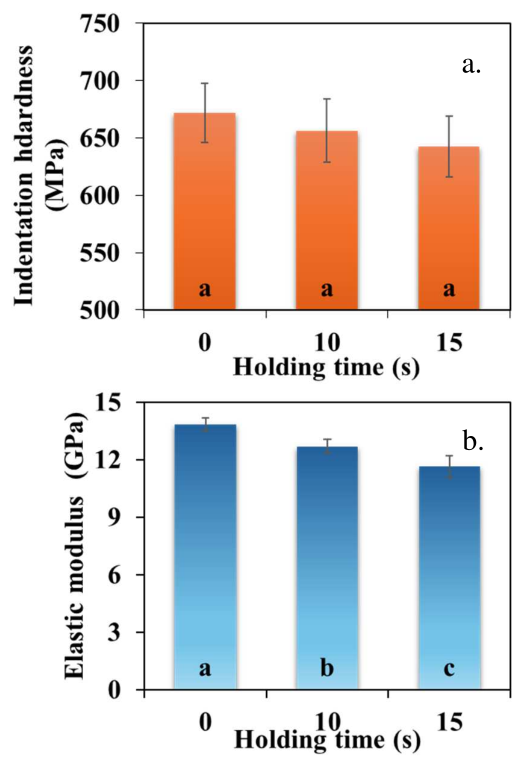

Fig. 7. The effect of holding time on a. indentation hardness and b. elastic modulus of G-aenial. (Identical letters indicate that the means are not statistically significant).

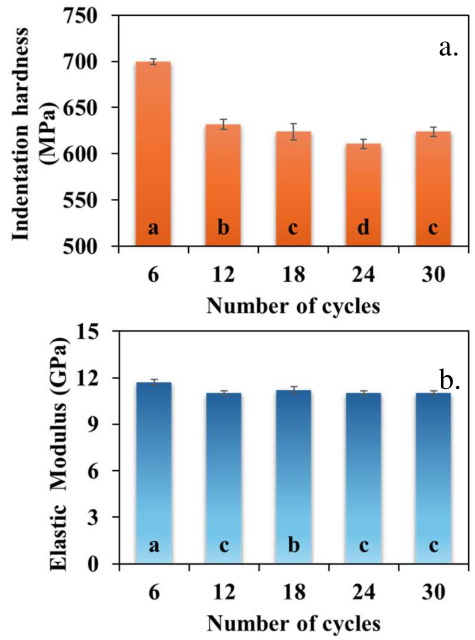

Fig. 8. The effect of number of cycles on a. indentation hardness and b. elastic modulus of elastic modulus of G-aenial. (Identical letters indicate that the means are not statistically significant).

Table 2

ANOVA for Indentation hardness (MPa).

Source	DF	Seq SS	Adj SS	Adj MS	F-Value	P-Value
Cycle	4	38554	38554	9638.6	271.28	0
Error	85	3020	3020	35.53		
Total	89	41574				

Under dynamic loading, the elastic modulus of the G-aenial composite decreased from about 12 GPa to 11.00 GPa with increasing number of cycles from 6 to 30, respectively. Statistical analysis of the elastic modulus indicates that the effect of the number of cycles is statistically significant (Table 3). However, Tukey pairwise comparison indicates that the effect of cycles is less significant after 12-18 cycles, as shown in Fig. 8b. It should be noted that the elastic modulus after 6 cycles is about 8% smaller compared to the static elastic modulus (e.g., one cycle).

Table 3

ANOVA for Elastic Modulus (GPa).

Source	DF	Seq SS	Adj SS	Adj MS	F-Value	P-Value
Cycle	4	3.143	3.143	0.78568	33.19	0
Error	85	2.012	2.012	0.02367		
Total	89	5.155				

4. CONCLUSION

In this study, the mechanical properties of G-aenial restorative composite were characterized by nanoindentation using static and dynamic multi-cycling tests. Both the static indentation hardness and elastic modulus decrease with increasing holding time from 0 s to 30 s; however, the effect on the indentation hardness is not statistically significant. The elastic modulus decreased by about 15% with increasing holding time from 0 to 30 s. Statistical analysis confirmed that the holding time has a significant influence on the elastic modulus due to the time-dependent response of the composite. The elastic modulus and indentation hardness of the G-aenial composite at 10 s holding time decreased with increasing number of cycle loadings, indicating that the restorative composite becomes more susceptible to fatigue damage. Future work will focus on investigating the effect of other parameters on this restorative material, such as loading/unloading rate, maximum load, or the influence of the polishing process.

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Evaluarea comportamentului mecanic al compozitului de restaurare G-aenial utilizând tehnica de indentare în cicluri multiple

În timpul procesului de masticație, materialele de restaurare sunt expuse la o încărcare dinamică, deoarece cuspiții dinților și alimentele dure indentează ciclic aceste materiale. În această lucrare, tehnica de indentare este utilizată pentru a investiga comportamentul mecanic al compozitului de restaurație G-ænial în diferite condiții de nanoindentare cu cicluri multiple și pentru a determina proprietățile mecanice, cum ar fi modulul de elasticitate și duritatea. Inițial, au fost efectuate teste de indentare statică pentru a evalua efectul timpului de menținere asupra comportamentului mecanic al G-ænial. Apoi, au fost efectuate o serie de teste de indentare dinamică în care numărul de cicluri de indentare a variat de la 6 la 30. Rezultatele experimentale au indicat că atât duritatea statică, cât și modulul de elasticitate scad odată cu creșterea timpului de menținere de la 0 s la 30 s. Mai mult, modulul de elasticitate și duritatea compozitului G-ænial au scăzut odată cu creșterea numărului de cicluri, ceea ce indică faptul că materialul de restaurare devine mai susceptibil la deteriorarea prin oboseală.

Cuvinte cheie: *material restaurator, nano-indentare, modul elastic, duritate, G-aenial.*

Adriana-Madalina TURCANU (CONSTANTINESCU), PhD student, Chemist, “Dunărea de Jos” University of Galati, Center of Excellence Polymer Processing, madalina.constantinescu@ugal.ro.

Felicia STAN, PhD, Professor, “Dunărea de Jos” University of Galati, Center of Excellence Polymer Processing, felicia.stan@ugal.ro.

Catalin FETECAU, PhD, Professor, “Dunărea de Jos” University of Galati, Center of Excellence Polymer Processing, catalin.fetecau@ugal.ro, Corresponding author,