

Mechanical Properties of a Dental Nano-Composite in Moist Media Determined by Nano-Scale Measurement

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Abstract—The aim of this study was to evaluate the influence of thermocycling on the mechanical properties of a commercial dental nanocomposite by using the nano-indentation experiment. The resin-based nanocomposite contained silica and zirconia nanofillers. Some disk specimens, each with the diameter of 10 mm and the thickness of 4 mm were prepared and the specimens were stored at various environmental conditions. Some of the samples were kept in the ambient condition, other samples were stored in distilled water at room temperature and other ones were thermocycled in distilled water for 1000 cycles between 5 °C and 55 °C. After preparation of samples, the nano-indentation test was applied on the prepared specimens by Triboscope setup. Then the modulus of elasticity and hardness of the test samples were calculated from the Oliver-Pharr's method using the data obtained from nano-indentation test. The present research showed that the samples stored in distilled water without thermocycling exhibited the highest elasticity modulus when compared to the other samples. In addition, the maximum hardness was observed for thermocycled specimens in distilled water.

Index Terms—Dental nanocomposite, thermocycling effect, nano-indentation test, hardness, elasticity modulus.

I. INTRODUCTION

Dental restorative materials are used in order to restore teeth, biologically, functionally and aesthetically. Amalgam and dental gold alloys have been used for more than 100 years as a dental restorative material in posterior teeth, and have achieved success in clinical matters. The reason is the similarity between the mechanical properties of these materials and the real teeth in various aspects [1], [2]; but the problem is their color which is different from the tooth making them aesthetically unsuitable for restoration of teeth. Therefore, dental composites were introduced as the tooth-colored restorative materials about 40 years ago. Dental composites are complex, tooth-colored filling materials composed of resin and reinforcing fillers that are used in different sizes. Although, there has been much improvement in dental restorative composites, their constitutive monomer systems have changed slightly. On the other hand, in the evolution of composite materials, their fillers changed fundamentally [3], [4] and led to introducing variety of composite restorative materials including: macrofilled composites, microfilled composites, hybrid composites, microhybrid composites and nanocomposites.

Manuscript received May 30, 2013; revised July 25, 2013.

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Researches conducted on dental composites indicate that strength, elasticity modulus, wear resistance, polymerization shrinkage and generally, the physical and mechanical properties of the composite are improved in direct relation to the amount of micro or nano fillers added [5]–[10].

Many efforts have been devoted to studies in the field of relationship between filler particles in composite resins and their properties. The experiment data provided by previous researches suggest that the filler plays an important role in some properties such as elastic modulus [11], polish retention [12]–[14], wear resistance [14], flexural strength [14]–[17], bond strength [18], color stability, translucency, opalescence [19] and fracture toughness [14], [20], [21]. Also, some other studies have shown the effects of filler size [8], [9], [22], [23] and filler shape [11], [20] on mechanical properties of dental composites.

The nanocomposites are relatively new composites consisting of filler particles in nanometric dimensions (between 20 to 75 nm) [14]. The main purpose of using fillers with nanometric dimension in these nanocomposites is the improvement in the strength, wear resistance and ability to polish. The materials used to restore teeth must have enough strength to resist again applied forces during chewing and remain strongly on the related part of the teeth.

Patient's oral conditions such as thermal gradient and humidity can have negative effects on the mechanical and physical properties of restorative materials. The most important reason for replacing dental restorative materials are fracture of restorative section and caries [1].

Thermocycling is an experimental process in which thermal conditions are simulated very similar to the real oral conditions such as thermal stresses created in the teeth and in the dental material during the treatment. The thermocycling simulations should be made in vitro and experimental condition just like the clinical conditions. In the thermocycling process, the test samples are embedded in hot and cold distilled water in order to simulate the temperature cycles in specified numbers and preset times. During the thermocycling process, the temperature gradient and the water absorbed by dental materials cause changes in their material properties. As reported in previous researches, thermocycling increases surface damage of composites and the thermal shock created among composite constitutions can produce microcracks [24]–[26]. Therefore, the assessment of the mechanical properties of dental material exposed to thermocycling process is important.

Nano-indentation experiment is a proper method to obtain the mechanical properties of small-sized samples and also surface properties of coatings. To measure the mechanical properties by common mechanical experiments, it is needed

to make large-scale samples which results in extra test expenses. On the other hand, determining surface or close to surface properties, by using common tests in macro and micro scales is not easily possible.

In this paper the mechanical properties of a specific dental nanocomposite is obtained by nano-indentation experiment. Also the effects of thermocycling process and different storage conditions on the mechanical properties of dental nanocomposite are investigated.

II. METHODS AND MATERIALS

In the present work, specimens were prepared from the Filtek Z350 XT (3M ESPE, Germany) nanocomposite. This resin-based nanocomposite contains a specified combination of individual nanoparticles and nanoclusters. Nanoparticles are discrete nonagglomerated and nonaggregated silica and zirconia fillers of 20 nm and 4-11 nm in size, respectively. Loose bound agglomerates of nano-sized silica/zirconia form nanocluster fillers. Since the nanocluster filler particles consist of loose bound agglomerates of nano-sized filler particles, the nanomer sized primary particles can be worn away during abrasion. Therefore, in comparison with traditional hybrid composites, the polish retention of cured composite will be increase. The Filtek Z350 nanocomposite consists of both nanoparticles and nanocluster fillers. The nanocluster particles increase filler loading, physical properties and polish retention of the nanocomposite [27].

The resin consists of Bisphenol A diglycidyl ether dimethacrylate (bis-GMA), urethane dimethacrylate (UDMA), triethyleneglycol dimethacrylate (TEGDMA), poly (ethylene glycol) dimethacrylates (PEGDMA) and Bisphenol A polyethylene glycol diether dimethacrylate (bis-EMA). For viscous adjustment, TEGDMA is used. Since UDMA and bis-EMA resins have higher molecular weight and, as a result, double bonds per unit of weight, they can affect the measurable viscosity, which will cause less shrinkage, age reduction and also somehow softer resin matrix [27].

Disc-shaped specimens of 10 mm diameter and 4 mm thickness were prepared from the Filtek Z350 nanocomposite. According to the manufacturer's instructions, both sides of samples were light cured for 20 seconds until the polymerization was carried out. Dimensional analysis applied to the samples used in the nano-indentation experiment showed that for a homogeneous material, the indentation test results were independent of all dimensions except depth [28], [29].

By comparing the dimensions of the samples used in this work and the indenter dimensions, it could be concluded that all samples used in this study for nano-indentation experiment, were homogeneous.

In order to evaluate the effect of thermocycling and storage conditions on the mechanical properties of dental nanocomposites, specimens prepared of nanocomposites were divided into three groups. One group of specimens were kept in vitro at room temperature (at 24 °C) and the next batch of specimens were stored at the distilled water, and the other specimens were thermocycled in distilled water for 1000 cycles between 5 °C and 55 °C. As suggested by Gale and

Darvell, a total number of 1000 cycles of thermocycling between 5 °C and 55 °C was applied on the samples kept in distilled water with a stopping time of 50 seconds in each of hot and cold environments [30]. According to a study by Brown *et al.* [31], ten cycles of thermocycling on a sample is equivalent to placing in the oral conditions for a day. Thus, 1000 cycles of thermocycling selected in this research is equivalent to putting the sample inside the patient's mouth for 100 days.

Previous researches demonstrated that high roughness of the sample surface will cause scattering of the hardness and elastic modulus values obtained from nano-indentation test [32]–[34]. To determine the level of acceptable smoothness for the nano-indentation test, ISO 14577 [35], [36] defines a reference based on the maximum indenter depth of the sample (h_{max}) which must be greater than 20 times of the mean surface roughness values (R_a) ($h_{max} > 20R_a$). In order to obtain a smooth surface for nano-indentation test, all samples were ground with 400 to 2500 grit sandpaper and then polished by diamond paste with meshes of 1 micron and 0.5 microns until the surface was smooth and glossy. The roughness values of samples were measured using atomic force microscopy (AFM) and the roughness analysis was performed on these images by Nanoscope ® II software.

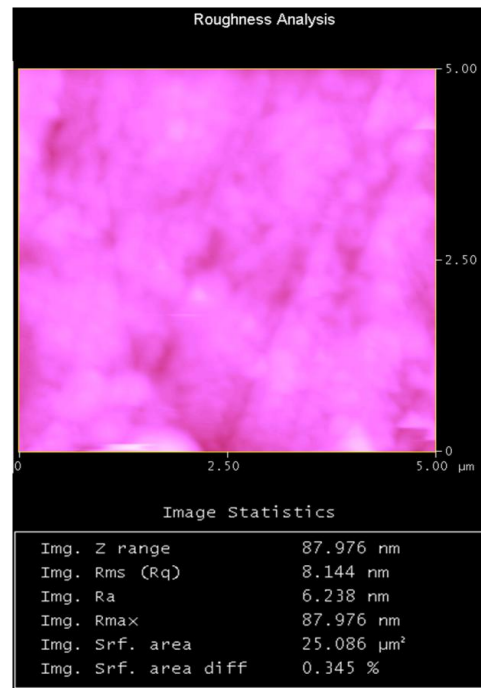


Fig. 1. AFM images from sample surface before nano-indentation testing and results of roughness analysis.

As shown in Fig. 1, the typical mean roughness value of a test sample was 6.238 nm. The mean roughness values of all samples were measured using the described method. The highest value of mean roughness among all samples was 10.798 nm. Therefore, according to the maximum average roughness value of the samples and the criterion introduced by ISO 14577, it can be concluded that if the indenter depth is greater than 215.96 nm, the remained roughness of surface will not be effective on the nano-indentation experiment results. When the smoothness of sample surface reached acceptable level for nano-indentation test, all samples were maintained in ambient temperature (at 24 °C) for one hour

before performing the nano-indentation experiment.

III. EXPERIMENT

The nano-indentation test was performed by using Triboscope system (Hysitron Inc., USA) based on International Standards Organization (ISO) 14577 standard and Berkovich indenter. The Berkovich indenter has an average radius of curvature of about 150 nm and is primarily used for bulk materials and thin films with thicknesses greater than 100 nm. The tip of the testing instrument was calibrated by Oliver-Pharr method (ISO 14577 [37], [38]) and the same method was utilized for analyzing the experimental data. The nano-indentation test uses an established method in which an indenter tip is pressed into specific sites of the material to be tested by applying an increasing normal load. When the penetration depth of the indenter tip achieves a pre-set maximum value, the normal load is reduced until partial or complete relaxation occurs. In this study, an indentation load of 750 μN with constant rate of 15 $\mu\text{N s}^{-1}$ was applied. Previous studies [39], [40] illustrate that the penetration depth of the indenter affects the measured mechanical properties of materials and in most cases, after a depth of 200 nm the measured properties became almost stable. Indeed, the indentation depth should be deep enough to minimize the surface effect. Meanwhile, the indentation depth should also be less than 10% of the film thickness when the sample is mounted on a hard substance; otherwise the obtained value is usually larger than the amount it should be, due to the effect of the support [41]. Therefore, the maximum indentation depth in our experiments was limited to 250 nm. The Oliver and Pharr method is dependent of the unloading segment of the load-displacement curve and assumes that only the elastic displacements are recovered. However, with this assumption, an error occurs in determination of the mechanical properties of polymers due to their time and rate dependent behavior. Therefore, in order to eliminate the error in viscous materials, it is common to hold the indenter at the maximum load for a period of time [42], [43]. In the current study, a holding time of 10 s was selected to minimize the likely error due to the time dependent behavior of the material. According to the explained method, at least five indentations on the randomly selected sites of the samples were performed at a temperature of 24 °C. The AFM images were used for data analysis, before and after the indentations.

IV. RESULTS

A. Modulus of Elasticity

Based on the continuous load–displacement data obtained from a complete cycle of loading and unloading during the nano indentation test and by using the Oliver- Pharr method, the effective modulus of elasticity is measured. A sample load–displacement curve measured from the nano-indentation test is shown in Fig. 2. Elasticity modulus of samples can be calculated by using the following equation [38]:

$$\frac{1}{E_{eff}} = \frac{1-v^2}{E} - \frac{1-v_i^2}{E_i} \quad (1)$$

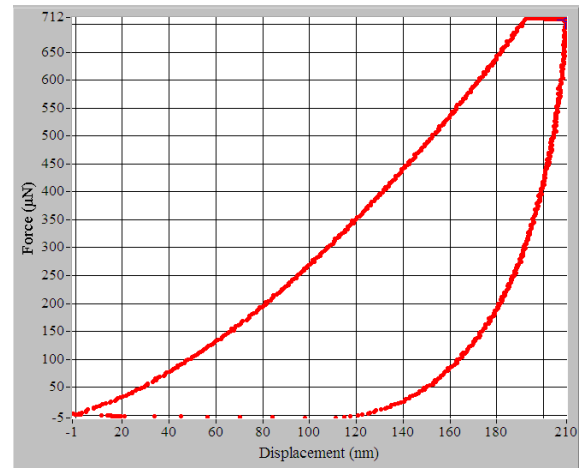


Fig. 2. Load-displacement curve for nanocomposite stored in ambient condition.

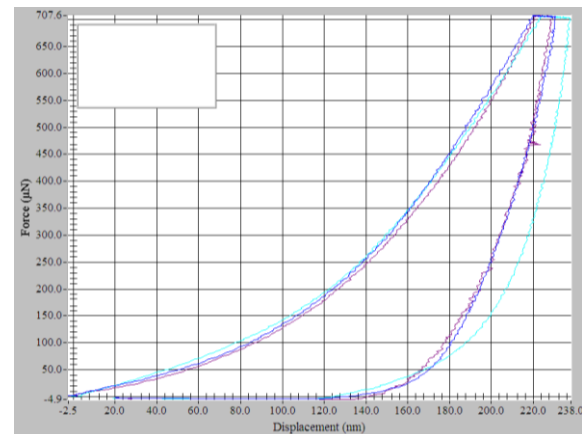


Fig. 3. Repeatability of Load-displacement curves obtained from the nano-indentation test.

where E and ν are the elasticity modulus and Poisson's ratio of samples, E_i and ν_i are elasticity modulus and Poisson's ratio of the indenter tip, respectively, and E_{eff} is the effective elasticity modulus of the material obtained from the nano-indentation test. The AFM images of all indentation tests were studied and the elasticity moduli of all samples were calculated by using Eq. (1) and considering $\nu = 0.31$ [44], $\nu_i = 0.07$ and $E_i = 1140$ GPa. The values used for ν_i and E_i are based on the technical data available from the related Triboscope system. It is noteworthy that no radial crack around the indentation hole was observed. Cracking around the hole will cause modulus and hardness results measured from nano-indentation tests which will not be reliable, because in this case, a large fraction of the force applied to the indenter is consumed for the formation and growth of cracks instead of material deformation [38]. As mentioned earlier, the hardness and elastic modulus of the specimens were calculated without observing any cracks around the indentation hole. The mean modulus value obtained from the samples is given in Table I. It should be noted that the assessment of force-displacement curve achieved from nano-indentation tests for every sample, represents good repeatability for nanocomposite samples used in this experiment. (Fig. 3)

B. Hardness

Material hardness is defined as the resistance of material against surface deformation caused by external load. This

mechanical property can be calculated via dividing the normal load by the projected area of the surface on which it is imposed. The hardness or the normal hardness (H_n) can be calculated in the nano-indentation test from [45]:

$$H_n = \frac{F'_n}{A} \quad (2)$$

where F'_n and A are the maximum load for a given indentation and the projected area of the contact surface between the specimen and the indenter, respectively. In this study, in order to calculate F'_n and A in Eq. (2), the load-displacement curve obtained from the nano-indentation test and also the Oliver-Pharr method was utilized [38]. The mean hardness value measured from the samples is given in Table I.

TABLE I: VALUES OF ELASTICITY MODULUS AND HARDNESS MEASURED FROM NANO-INDENTATION TESTS

Specimen	Modulus of elasticity (GPa)	Hardness (GPa)
Nanocomposite stored in ambient condition	10.99±0.41	0.38±0.01
Nanocomposite stored in distilled water	15.07±0.91	0.47±0.05
Thermocycled nanocomposite in distilled water	12.07±0.15	0.61±0.01

V. DISCUSSION

The results obtained from nano-indentation experiments indicated that the samples stored in distilled water without thermocycling exhibited the highest elasticity modulus compared to other samples. Moreover, the maximum hardness was observed for specimens which were thermocycled in the distilled water. Table I, shows the mean value of the elasticity modulus and hardness of all samples.

It seems that two factors can have influence on mechanical properties of samples exposed to thermocycling process or keeping in distilled water. These two factors respectively are:

- 1) The effect of humidity on nanocomposite structure.
- 2) Thermal stresses generated due to thermal gradient in thermocycling process among structural constitutions of the nanocomposite.

The negative influence of water on composites can be explained by two different mechanisms. First mechanism is the role of water molecules in changing the material behavior from elastic state to plastic state which will increase the matrix volume of the nanocomposite. According to this mechanism the stiffness of the material decreases. The second mechanism is the dissolution of composite components in the water.

Previous studies have shown that maintaining the restorative composites and nanocomposites in humid environment have different influences on their mechanical properties including elasticity modulus, depending on temperature and time of storing in water. In some of previous investigations [46], [47], storing the dental composite

samples in water for a definite time and specified temperature have led to increase in the elasticity modulus, whereas in others [48] decreased the elasticity modulus. Whereas in some reported cases [49], [50] storing the dental composites in humid ambient have had no impact on elasticity modulus.

A review of the results obtained in this research and in the previous studies [46]–[50] demonstrates that the influence of water on the mechanical properties of dental nanocomposite does not follow a specific law and depends on material type and storage conditions.

Based on the results obtained in this paper, it can be concluded that fillers existing in Filtek Z350 nanocomposite have major influences in the elasticity modulus and hardness values; because even softening of nanocomposite matrix due to water penetration had no effect on the decrease on the stiffness and hardness of the nanocomposite. However, one of the suggested effects of nanocomposite can be the higher water uptake and resultant degradation of resin–matrix interface due to the large surface to volume ratio in the nanoparticles [51].

As mentioned before, creation of thermal stresses in the nanocomposite components generated by thermocycling process is another factor which influences the mechanical properties of samples. The elasticity and hardness values obtained from this study indicate that thermal stress can increase the stiffness and hardness of the nanocomposite. Some previous studies indicate that water sorption, solubility and diffusion coefficients increase due to rise of temperature [52]. Janda *et al.* [50] illustrated that thermocycling in water ambient can have various effects on the flexural modulus of restorative composites. They indicated that thermocycling in some restorative components lead to reduction of flexural modulus and in some others have no influence on the flexural modulus. Tirado *et al.* [53] indicated that thermocycling has negative effects on the hardness of some dental composites. It should be mentioned that none of the pervious researches have pointed to thermocycling effect on restorative nanocomposite and according to the present results it can be said that 1000 cycles of thermocycling which is equivalent to 100 days of remaining restoration in mouth [30] increase the elasticity modulus and hardness of the dental nanocomposite.

The elasticity modulus investigated in this study before and after thermocycling is very close to the dentine elasticity modulus (8.7-11.2 GPa) [54]. This is clinically important, because if the restorative elasticity modulus is lower than the elasticity modulus of tooth, the restorative material deforms more than its working conditions in dental substrate against applied stresses. This may lead to damage in the dental substrate around the restorative material or to its separation from the tooth surface.

VI. CONCLUSION

In this paper elasticity module and hardness was measured for a specific type of nanocomposite using nano-indentation experiment. The effects of two maintenance conditions and 1000 cycles of thermocycling in distilled water on the nanocomposite were studied. The results indicate that:

- 1) The elasticity modulus of Filtek Z350 nanocomposite is in the range of dentine elasticity modulus.

- 2) The samples stored in distilled water without thermocycling exhibited the highest elasticity modulus compared to other samples. Increase in elasticity modulus due to thermocycling process or storing in distilled water is in a way that the nanocomposite still remains in the range of dentine elasticity modulus.
- 3) The maximum hardness was observed for the samples which were exposed to thermocycling process in distilled water. Therefore, it can be concluded that thermocycling leads to increased hardness of the Filtek Z350 nanocomposite.

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