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Effect of Dental Restorative Material Type and Shade on Characteristics of Two-Layer Dental Composite Systems

Abstract

The purpose of this study was to investigate the effects of shade and material type and shape in dental polymer composites on the hardness and shrinkage stress of bulk and two-layered restoration systems. For this purpose, some bulk and layered specimens from three different shades of dental materials were prepared and light-cured. The experiments were carried out on three types of materials: conventional restorative composite, nanohybrid composite and nanocomposite. Micro-indentation experiment was performed on the bulk and also on each layer of layered restoration specimens using a Vicker's indenter. The interface between the two layers was studied by scanning electron microscopy (SEM).
The results revealed significant differences between the values of hardness for different shades in the conventional composite and also in the nanohybrid composite. However, no statistically significant difference was observed between the hardness values for different shades in the nanocomposite samples. The layered restoration specimens of different restorative materials exhibited lower hardness values with respect to their bulk specimens. The reduction in the hardness value of the layered conventional composite samples was higher than those of the nanocomposite and nanohybrid composite specimens indicating more shrinkage stresses generated in the conventional composite restorations. According to the SEM images, a gap was observed between the two layers in the layered restorations.

Keywords

Shade effect, polymer dental materials, Layered restorative systems, Micro-indentation.

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1 INTRODUCTION

The demand for aesthetic, strength, life lasting and easy-to-use dental restorative systems has led to the development of polymer-based dental composite materials. At the present time, dental composites are widely used for filling the tooth cavities, veneering to mask discoloration, correcting contour, making dental implants and bonding orthodontic brackets . Dental composite materials consist of a polymeric matrix and inorganic ceramic filler particles which are generally produced by light curing. The polymeric matrix is flowable before curing, which makes the composite to fully penetrate into the tooth cavity. During the curing procedure, the polymerization is activated, which allows the resin matrix to solidify and to change its mechanical properties rapidly and significantly. Volumetric shrinkage also occurs in polymerization due to the decrease of intermolecular separations in the monomers of polymeric matrix (Li et al., 2011). In the restorative systems where the restorations are constrained along the interfaces, the polymerization shrinkage causes some shrinkage stresses in the composite and in the tooth. In these cases, shrinkage stress may result in pulling the material away from the cavity walls (Ferracane and Mitchem, 2003, Braga and Ferracane, 2004) which is the main reason for marginal debonding and then micro-leakage within the composite restorations (Davidson et al., 1984). Moreover, shrinkage forces on cusps produce cuspal deformation, enamel cracks and crazes (Marzouk and Ross, 1989) which cause reduction in the fracture resistance of the cusp (Wieczkowski Jr et al., 1988). Prediction or measurement of the shrinkage stresses is a difficult procedure because the each tooth cavity has small and complicated geometry and also there are many factors that affect the restorative composite properties. Up to now, some experiments have been developed to estimate the shrinkage stress of dental restorative composites (Ferracane, 2005, Gonçalves et al., 2008, Li et al., 2011, Simon et al., 2008).

Indentation experiments have received much attention from numerous researchers for determining the mechanical properties of different biomaterials, see for example (Şakar-Deliormanli and Güden, 2006, Kruzic et al., 2009, Ayatollahi and Karimzadeh, 2012, Karimzadeh and Ayatollahi, 2012, Towler et al., 2001, Karimzadeh et al., 2014, Oréfice et al., 2003). According to these studies, indentation tests are independent of the specimen geometry and size and give reliable results. Therefore, such tests can be considered as suitable alternatives for estimation of shrinkage stress and determination of the mechanical properties of restorations.

Recently, layering restoration techniques have been applied by various clinicians. The base of these techniques is restoring the lost tooth structure layer by layer using composite materials with different or similar shades. Two major advantages of layering restoration techniques are the creation of a more natural-looking restoration and the reduction in the magnitude of polymerization shrinkage stresses (Park et al., 2008, Li et al., 2011, Lee et al., 2007). Previous studies (Kwon et al., 2012, Park et al., 2008, Van Ende et al., 2013, Bicalho et al., 2013, Arakawa, 2010) have suggested that the layered or incremental filling method produces lower shrinkage stresses compared to the bulk filling method.

In addition to the conventional dental restorative composites, dental nanocomposites and nanohybrid composites have been recently introduced by changing the size and volume fraction of fillers in the dental composites. Dental nanocomposites contain nano-fillers with dimensions of 4 to 20 nm added to the composite resins. In the nanohybrid adhesives, in addition to nanometer particles, 0.2 to 1 micrometer particles are also added to the composite resins.

Tooth may have various colors depending on the patient's age and enamel thickness. It has been proven that aesthetic and durable restorations can be attained by using dental composites having physical properties similar to those of the natural enamel. Therefore, different shades of dental composite, nanocomposite and nanohybrid restorative materials are available to achieve desired properties of natural tooth structure. According to some research studies performed on different shades of dental restorative materials, the polymerization process of the dental restorations and their mechanical and physical properties are affected by the type of shades utilized (Aguiar et al., 2005, Cesar et al., 2001, Della Bona et al., 2007, Guiraldo et al., 2009). However, very few researches have been conducted on the mechanical properties of various shades of nanocomposite and nanohybrid composite.

In this study, the effects of material shade on hardness of three dental restorative polymers, i.e. conventional composite, nanocomposite and nanohybrid composite were investigated. In addition to hardness, the shrinkage stresses in two-layer restorative systems of these materials were studied. Besides, the interface between the two layers was inspected by scanning electron microscopy (SEM). The hypothesis is that (1) different shades of these restorative materials have various hardness values, and darker shades have higher hardness and (2) using different combinations of material shades affects the shrinkage stress in layered restoration systems.

2 MATERIALS AND METHODS

2.1 Materials

Three different types of dental polymer composites (i.e. conventional composite, nano-composite and nano-hybrid composite) with three different shades including enamel (A1), dentine (A4) and body (C2) shades were utilized. The characteristics of the used materials are indicated in Table 1.

Dental Material	Type of resins	Filler particles	Filler content	Shade
Composite	BIS-GMA[1], UDMA[2], BIS-EMA[3]	Zirconia/silica	60%V	A1,A4,C2
Nanohybrid composite	BIS-GMA, UD-MA,PEGDMA[4], TEGDMA[5]	Silica, Zirconia/silica	67.8%V	A1,A4,C2
Nanocomposite	BIS-GMA, UDMA, TEGD-MA, PEGDMA, BIS-EMA	Zirconia, silica	63.3%V	A1,A4,C2

Table 1: Characteristics of dental restorative composites used in the experiments.

2.2 Sample Preparation

Two categories of specimens, bulk and layered, were prepared for the tests. For the bulk category, nine cylindrical specimens of 5 mm diameter and 4 mm thickness were prepared from each type of material and shade. Each sample was light-cured using a LED light with minimum intensity of 400 mW/cm^2 according to its manufacturer's instruction.

For the layered category, a total number of 9 two-layer specimens were prepared from two shades of each restorative material. In the layered restoration systems, transparent restorative materials are commonly used for the outer layer. Thus, in the layered specimens at least one layer of enamel shade was used for each material type. For this purpose, a strip of enamel shade with 2 mm thickness was made and light-cured. Then, another shade of the same material with similar shape and thickness was applied on the first strip and light-cured. The light-curing procedure was performed on the materials in accordance with the manufacturer's instruction. Figure 1 shows some layered specimens prepared from different shades of each restorative material.

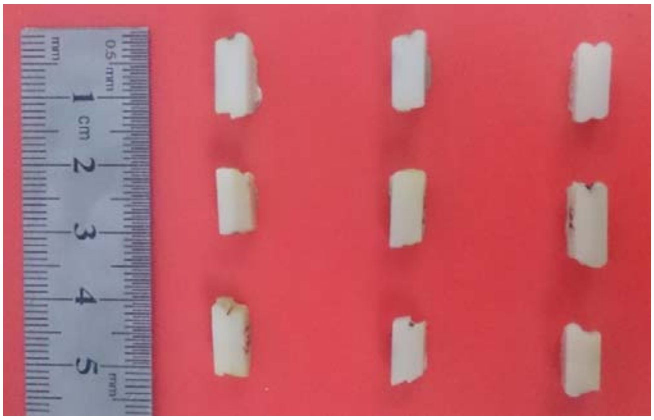

Figure 1: Layered specimens prepared from different shades of each restorative material.

In order to obtain a smooth surface, all samples were ground with 800 to 3000 grit sandpapers two days after their polymerization, and then polished by diamond suspension with meshes of 3 microns to 1 micron to make sure that the surface was sufficiently smooth.

2.3 Experiment

The micro-indentation experiment is a method for determining material hardness or resistance of material to penetration. It is also called micro hardness test. The micro hardness experiment can be utilized when test specimen is very small and when small regions in a composite sample or plating should be measured. This test uses an established method in which an indenter tip is pressed into specific sites of the material by applying an increasing normal load. When the penetration depth of the indenter tip or the indentation load reaches its pre-set maximum value, the normal load is reduced till partial or complete relaxation occurs.

In this study, micro-indentation experiment was performed on the bulk and layered samples by using a micro indentation instrument (SHIMADZU, Japan) and Vickers indenter. In the layered samples, the indentation was done at each layer. In the micro-indentation experiment, a normal load should be selected from the predefined load values of the instrument to make a perfect indentation hole without any crack or damage around it. The value of normal load depends generally on the properties of the sample material. In this study, a normal load of 490.3 mN was applied for all specimens to make a perfect indentation hole. The indentation hole was observed by a high resolution optical microscope with magnification of 40x.

2.4 Imaging

All layered specimens were analyzed by a Scanning electron Microscope (SEM) to investigate the interface between the two layers. A SEM instrument (Phenom Prox, Netherlands) was used to take some images of the interface. Each specimen was placed on a carbon double-side tape and an accelerated voltage of 15 kv was used for SEM.

3 RESULTS AND DISCUSSION

3.1 Micro Hardness

Hardness (H) could be calculated by dividing the normal load (F) by the area of the surface where load is imposed on (A).

$$H = \frac{F}{A} \quad (1)$$

When Vicker's indenter is applied, A is calculated from:

$$A = \frac{d^2}{2\sin(\alpha / 2)} \quad (2)$$

where d is the average diagonal length remained by the indenter and α is the angle between opposite faces of the indenter which is equal to 136° for the Vicker's indenter.

Table 2 shows the mean value and standard deviation (SD) obtained directly from the micro-indentation test for the hardness of each bulk sample.

	CA1[1]	CA4[2]	CC2[3]	NA1[4]	NA4[5]	NC2[6]	HA1[7]	HA4[8]	HC2[9]
Mean (GPa)	0.56	0.59	0.58	0.7	0.71	0.7	0.66	0.65	0.6
SD (GPa)	0.01	0.01	0.01	0.03	0.02	0.02	0.02	0.02	0.03

[1] CA1: composite restorative material with shade A1
[2] CA4: composite restorative material with shade A4
[3] CC2: composite restorative material with shade C2
[4] NA1: nanocomposite restorative material with shade A1
[5] NA4: nanocomposite restorative material with shade A4
[6] NC2: nanocomposite restorative material with shade C2
[7] HA1: nanohabrid composite restorative material with shade A1
[8] HA4: nanohybrid composite restorative material with shade A4
[9] HC2: nanohybrid composite restorative material with shade C2

Table 2: Mean value and standard deviation of micro hardness obtained for the bulk samples.

Comparing the results by one way ANOVA test shows that significant differences exist between the hardness of different shades in conventional composite (p-value=0.012) and in nanohybrid composite (p-value<<0.05) groups. No statistically significant difference is observed for the hardness values of different shades in the nanocomposite group. The Tukey HSD test was applied to evaluate pairwise differences between the mean values of hardness for the composite and the nanohybrid composite groups, because equal variances were tenable. The results indicate a significant difference between the mean hardness values of A1 and A4 shades in the composite group (p-value=0.009).

Moreover, in the nanohybrid restoration group significant differences are seen between A1 and C2 shades (p-value<<0.05) and between A4 and C2 (p-value=0.004) ones.

Based on Table 2, the highest value of hardness was obtained for the nanocomposite. This observation can be interpreted by the filler size and its volume fraction. According to previous research studies, hardness could be increased by applying smaller particles and higher filler contents (Oberholzer et al., 2003, Lodhi, 2006). The particle size of the nanocomposite is smaller than the other two materials and its volume fraction is higher than the conventional composite. Therefore, its hardness is also expected to be higher.

In the conventional composite group, the darker shade A4 was significantly harder than its lighter shade A1. While for the nanohybrid composite, higher hardness was obtained for the lighter shade A1 and for the nanocomposite group no significant difference was observed between the hardness values of different shades. Since the darker shade can absorb light and lighter shade spreads it (Sakaguchi et al., 1992, Aguiar et al., 2005), more hardness value was expected for the darker shade. It has been previously shown that shade may have different effects on the hardness of light-cured restorative composites. For example, Aguiar et.al. (Aguiar et al., 2005) found that darker shade of dental composite has lowest hardness value compared to the lighter shade. On the contrary, a higher hardness value was reported for the darker shade of dental composite in the research studies performed by Lodhi (Lodhi, 2006) and Pierce et.al. (Price et al., 2005). Previous studies have indicated that at each depth of light-cured restorative materials different factors have dominant effects on material hardness (Rueggeberg et al., 1993, Lodhi, 2006). The target layer in the present study is the top surface which is the nearest layer to the light-curing device. According to Rueggeberg et.al. evaluation (Rueggeberg et al., 1993), the influential factors at the top surface of the light-cured restorative materials, with respect to their importance are filler type, exposure duration and resin shade. Therefore, at the top surface, the shade is not of very important influence on hardness.

The size, load and distribution of fillers affect the light scattering and its absorption, hence they have influence on the polymerization shrinkage of restorations (Leonard et al., 2001, Ruyter and Øysæd, 1982, Aguiar et al., 2005). These factors vary greatly in different types of composites and their effects should be investigated separately with highly controlled conditions. As a result, the mechanical behavior of different material shades should be explored for the same types of restorative materials.

In terms of clinical applications, similar hardness values for different shades of the nanocomposite could be a good achievement for dental restorative behavior. However, the variations of hardness in the conventional composite and nanohybrid composite were also quite small for clinical applications.

3.2 Shrinkage Stress

Hardness is defined as material resistance against local plastic deformation. Therefore, hardness is affected by stress field around the indented material which has influence on the plastic deformation at this region (Withers and Bhadeshia, 2001, E., 1961, Tosha, 2002). Since residual stress influences the stress field, the hardness value would be changed in the presence of residual stress. Shrinkage stress generated due to the polymerization shrinkage in the restoration systems is a kind of residual stress which influences the hardness value.

In this part of research, hardness was measured in each layer of two-layer restorative systems made of different shades. By comparing the material hardness obtained for the bulk restorative materials with the corresponding value for the individual layer in the layered restoration specimen, the effect of shrinkage stress can be studied. The diagram of the hardness values of bulk samples and those obtained from each layer in the layered specimens has been exhibited in Figure 2.

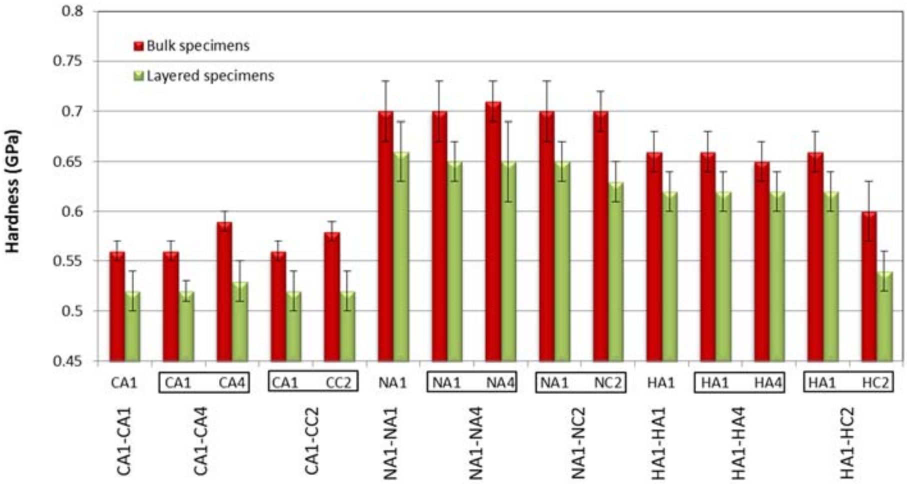

Figure 2: The hardness values of bulk samples and those obtained from each layer in layered specimens.

In Figure 2, the hardness of each individual layer in layered specimen has been shown by the right (green) bars and the hardness of bulk specimens has been indicated by the left (red) bars, as mentioned in the chart legend at the top corner of the figure. For each layered specimen, the data related to each of the two layers are written next to each other in a box beneath the horizontal axis, while the vertical axis shows the hardness values by the green bars. For those layered specimens in which the two layers are made of the same materials with the same shades, such as (CA1-CA1 or NA1-NA1 or HA1-HA1), the hardness of only one layer is shown in Figure 2, because both layers have equal hardness values.

It is seen from Figure 2 that the hardness of each individual layer in the layered specimens is less than the corresponding value of its bulk sample, which can be attributed to the polymerization shrinkage in the constrained samples causing shrinkage or contraction stresses. In the layered specimens, the shrinkage stress is created perpendicular to the interface of the two layers. The indentation test is also performed on the sample section which is not constrained. The indentation region and stress configuration are schematically depicted in Figure 3.

In Figure 3b, σ_r is the residual stress due to shrinkage of the sample materials and σ_i is the stress generated by the indenter during the micro-indentaion experiment. Because of Poisson's ratio effects, the tensile residual stress (σ_r) causes some contraction along Y and Z directions. Thus, less indentation force is required to make a specific amount of deformation in comparison to the no-stress conditions which occurs in the bulk specimens. This leads to a reduction in the measured hardness of the layers in the layered specimens compared to the corresponding values of bulk samples.

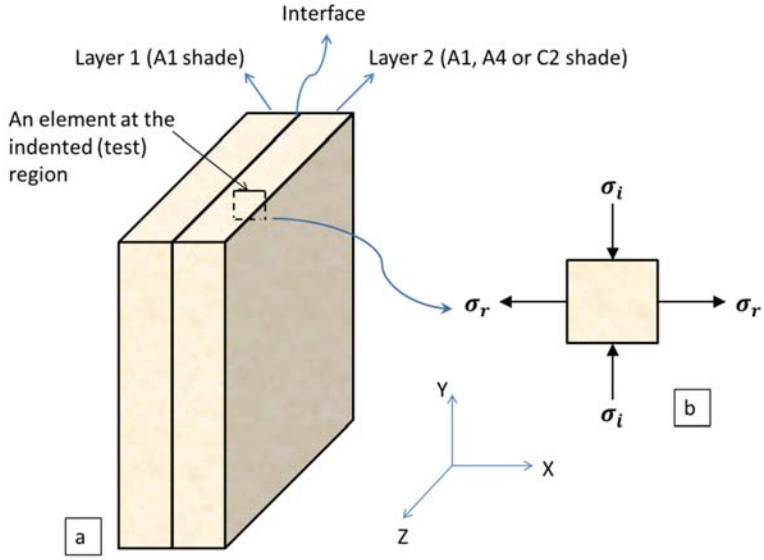

Figure 3: Schematic picture of stress configuration in a layered specimen at the indented or test region a) layered specimen with a square element at the indented (test) region and b) magnified square element with applied stresses caused by the indenter and shrinkage of the materials.

Since all conditions related to the sample preparation, the storage condition and the experimental procedure were carefully controlled, the material hardness of the layers in each layered specimen is predominately affected by the shrinkage stress. Although, the difference between the hardness values of layers and their bulk samples is not equal to the value of shrinkage stress, it can be used for comparing the effect of shrinkage stress in specimens of different materials and shades.

Comparing the hardness difference in the layered specimens of different restorative materials relative to their bulk samples indicates that more reduction has occurred in the hardness values of conventional composite specimens (around 7.1% to 10.3%) in comparison with the nanocomposite (around 5.7% to 10%) and the nanohybrid composite specimens (around 4.5% to 6.6%) all depending on the shades of layers. This implies that more shrinkage stress was generated in the conventional composite restorations.

Several factors can affect the polymerization contraction and the shrinkage stress of dental restorative materials. The composition of resin matrix of a restorative material is one of the factors which influence the polymerization shrinkage. The resin matrix of nanocomposite and nanohybrid composite samples contains TEGDM which has higher shrinkage than other resins. The reported shrinkage for TEGDMA is about 12.5%, while its value for the BisGMA is equal to 5.2% and for typical resins is ranged between 2% and 3% (Labella et al., 1999, Gonçalves et al., 2011, Peutzfeldt, 1997, Stansbury, 1992). Therefore, more shrinkage would cause more stress in the constrained nanocomposite and nanohybrid composite samples compared to the conventional composite one.

The volume fraction of fillers has also a direct effect on the shrinkage stress (Calheiros et al., 2004, Chen et al., 2001, Condon and Ferracane, 2000). According to Table 1, the highest volume fraction among the examined materials belongs to the nanohybrid composite (67.8 %). Therefore, nanohybrid composite is supposed to have the lowest shrinkage stress between the three materials.

In restricted dental restorative materials, two factors must be considered. First, the level of restriction imposed on the material, which could be estimated by the percentage of composite surface that is bonded to the substrate in relation to its total surface area. Second factor is the compliance of bonding substrate (Braga et al., 2005). Since all restoration systems used in the present study are in the same shape, the percentage of constrained area has no effect on the variation of hardness. Hence the compliance of restorative layers which could be considered as the substrate in the layered restorative systems should be evaluated. Our previous study shows that the elasticity modulus of dental restorative nanohybrid composite is higher than the corresponding values for the nanocomposite and the conventional composite samples (Ayatollahi et al., 2015). Thus, the nanohybrid composite layer has less compliance compared to the other materials which increases the induced shrinkage stress in its layered specimens. Although the shrinkage stress of conventional composite is more than the other two materials, its compliance is more than the corresponding value for the nanocomposite and nanohybrid composite. It means that other factors have dominant effects on the shrinkage stress of nanocomposite and nanohybrid composite specimens.

The three tested restorative materials consist of different filler sizes which might affect their shrinkage stress. Satterthwaite et.al (Satterthwaite et al., 2012) showed that the shrinkage stress changes in a complex manner when the sizes of filler particles vary. However, more comprehensive studies on the effect of filler sizes with different combinations in their sizes and types are needed. Since in our study different factors such as resin matrix, substrate compliance and filler type change in the specimens, no judgment can be done about the effect of filler size on the shrinkage stress in different types of restorative materials.

As a result, in the restorative systems the interaction between their compliance, resin matrix component and filler volume fraction affects the shrinkage stress variation. The investigation of dominant factor depends greatly on the material type and the restoration system properties which should be found out from experiment and cannot be interpreted by knowing the general behavior of restorative systems. For example, in the nanohybrid two-layer restoration the volume fraction of fillers can be considered as a dominant factor while in the nanocomposite restoration the resin matrix component is the main factor.

According to Figure 4, different variations are seen between the hardness values of the bulk and layered samples when different shades of each material are used. As discussed in the previous section, the difference in the shades of light-cured restorative materials causes some variations in their polymerization rates and in the amount of polymerization shrinkage. Another consideration is that in the layered specimens, the indentation test was performed on a section of restoration layers which was perpendicular to the light-cured surface. The degree of polymerization is changed by the distance from the light-cure device, unless the light curing is performed carefully according to an optimum procedure which is usually the same as manufacturer instruction (Jeong et al., 2009, Lodhi, 2006, Shortall, 2005). In this study, all specimens were light-cured according to their manufacturer's instructions. Therefore, no significant variation was observed in the hardness values of layers through the distance from the light source.

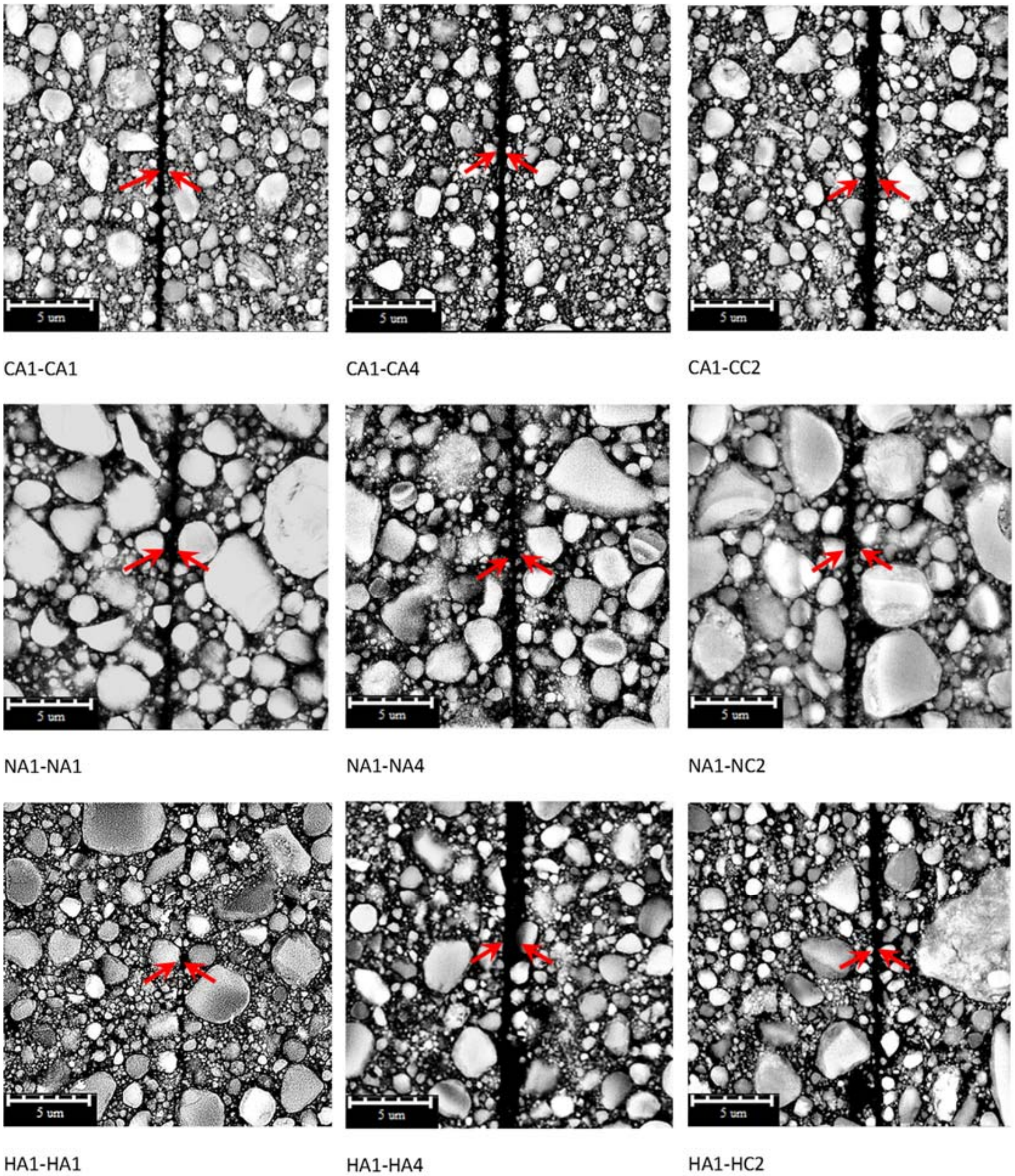

Figure 4: SEM images of the layered restoration systems.

It was seen that, A1-C2 layered specimens of all materials possess relatively higher hardness reduction at the C2 layer in comparison to the other two shades. Therefore, using the combination of shades A1 and C2 is not recommended for the layered restorations in clinical applications. Moreover, in terms of the shrinkage stress, using either of the nanocomposite and the nanohybrid composite materials could be a better choice for two-layer dental restorations compared to the conventional composite samples. Although the hardness of nanocomposite is more than that of the other two restorative materials, the shrinkage effect causes higher reduction in its hardness when used in two-layer specimens compared to the layered specimens of nanohybrid composite. Meanwhile, the nanohybrid composite has shown greater hardness than the conventional dental composite and its hardness reduction in the layered specimens is also lower than that of the conventional dental composite. Consequently, considering its higher hardness and lower shrinkage effect, nanohybrid composite can be recommended for the layered restoration treatments.

3.3 SEM Analysis

Each layered sample was inspected by a SEM instrument to study the interface between the two layers of the layered restoration systems and to investigate the quality of their interfacial seal. Figure 4 shows the SEM images of the interface zone in all the layered specimens wherein the gap between the two layers is pointed by red arrows.

According to Figure 4, an interfacial gap exists between the two layers in all specimens, and the thickness of gap varies for different samples. The interfacial gap formation can be attributed to the poor bond strength between the two restoration layers which was not strong enough to resist against the polymerization shrinkage of the resin-based dental restorative materials and could not provide an adequate interfacial seal for these restoration systems (Erickson, 1992, Samet et al., 2006). Table 3 presents the mean values of the gap thickness measured from the SEM images.

	CA1-CA1	CA1-CA4	CA1-CC2	NA1-NA1	NA1-NA4	NA1-NC2	HA1-HA1	HA1-HA4	HA1-HC2
Mean (μm)	0.53	0.55	0.78	0.54	0.62	0.58	0.38	1.16	0.61
SD (μm)	0.12	0.15	0.17	0.16	0.18	0.11	0.1	0.4	0.17

Table 3: The mean value and standard deviation of the gap thickness for each layered restoration specimen.

Based on Table 3, the gap thickness of the sample which consists of two nanohybrid composite with A1 shade (HA1-HA1), is lower than the other specimens. Since the same preparation procedures were applied to all samples, this observation could be due to the stronger bonding between the two layers of the HA1-HA1 specimen. Thus, better seal is obtained by using the A1 shade of nanohybrid composite for the layered restoration systems. This is in agreement with the results of previous studies which have shown the hybrid restorative composites generated fewer voids than the conventional dental restorative composites (Kugel and Perry, 2002, Samet et al., 2006). Indeed, the possibility of interfacial gap occurrence in layered restorations is reduced when fewer voids are created. However, it is noteworthy that from the esthetic aspect, restoring only by one shade of dental restorative material can not satisfy the natural optical properties of teeth.

Although the gap thicknesses in the restorative systems used in our study are less than the values reported in the previous studies for the gap thickness between the restorative and tooth (Oztas and Olmez, 2005, da Silva Telles et al., 2001) or for the interlayer gaps (Samet et al., 2006, Deyhle et al., 2010), appropriate methods should be used to reduce this gap further. Because this region is susceptible for the bacterial stagnation which leads to secondary caries (Li, 2012). Moreover, the presence of a gap in restoration systems may increase the potential for mechanical failures by the bacterial growth (Moorthy et al., 2012). To improve the bond strength between the composite restorative systems, using a bonding agent is recommended in order to make a strong chemical bonding between the two restorative layers.

4 CONCLUSION

Micro-indentation technique was used to measure the hardness values of three polymer restoration materials of different shades. The experiments were performed both on bulk materials and on two-layer restorative systems. The results indicated that the effect of material shades on hardness was dependent on the type of restorative material used. Since there are several factors involved in the degree of polymerization and the mechanical properties of the dental restorations, separate comparisons should be performed on each material. In the two-layer restorative systems, the shade and the material type affected the shrinkage stress of different restorative layers, but the level of influence was again dependent on the materials used. More reductions occurred in the hardness values of the conventional dental composite specimens (around 7.1% to 10.3%) in comparison to those of the nanocomposite (around 5.7% to 10%) and the nanohybrid composite (around 4.5% to 6.6%) all depending on the shades of layers. As a result of this study, the nanohybrid composite can be recommended for layered restorations in clinical applications, because of its higher hardness (equal to 0.6 GPa to 0.66 GPa depending on the shade) and lower shrinkage effect in comparison to the conventional composite and nanocomposite dental materials. While the SEM images showed a gap between the two layers in the layered restorations, the thinnest gap with a thickness about 0.38 μm, was observed in the nanohybrid layered specimens.

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