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Original Article

Effect of coating on the color and surface hardness of the surface of dental ceramics

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ABSTRACT

Background: The aim of this study was to investigate whether boron nitride (BN) and BN–chromium (BN–Cr) coatings applied with the sputtering method would change the characteristics of hardness and discoloration of the ceramic veneer surface.

Materials and Methods: In this experimental study, thirty disc-shaped cores (10 mm in diameter and 0.8 mm in thickness) were prepared. Three different ceramic systems, IPS Empress (E) (Ivoclar Vivadent, Schaan, Liechntenstein), IPS e.max Press (EP) (Ivoclar Vivadent, Schaan, Liechntenstein), and Turkom Cera (T) (Turcom-Ceramic SDN-BHD, Kuala Lumpur, Malaysia), were tested, each with n = 10. The specimens were divided into two subgroups (n = 5) according to the coating on the veneering ceramic: BN or BN–Cr. The Vickers hardness and color differences (ΔE) values were measured before and after coating. Surface analysis was performed with X-ray diffraction, X-ray photoelectron spectroscopy, and scanning electron microscopic. The Wilcoxon signed-rank test was performed to compare hardness values. The Kruskal–Wallis test was performed to compare ΔE values among all groups. The *post hoc* tests was used after Kruskal–Wallis, with level of statistical significance set at $P \leq 0.05$.

Results: BN–Cr coating statistically significantly increased the hardness of all types of ceramics ($P < 0.05$). BN coating did not change the hardness statistically significantly ($P > 0.05$). The mean ΔE values indicated clinically noticeable (over the limit of 3.7) color change in all groups except for the EP-BN and T-BN groups.

Conclusion: Although BN–Cr coating in this thickness had a negative effect on color, it increased the hardness of the ceramics and could be useful in the palatal region.

Key Words: Ceramics, color, hardness, sputter coating

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INTRODUCTION

Although IPS e.max Press (EP) (lithium disilicate glass ceramic system) has the same composition as IPS Empress II, its physical properties and appearance have been improved.[1] Turkom-Cera was developed as a high-purity alumina (99.98%) with better fracture resistance than the other available ceramic systems.[2] Complete ceramic dental restorations are usually formed in layers.[3] They are produced with esthetic but weak veneer ceramics layered on dense and strong ceramic substructures. The most common cause of clinical failure is crack formation in the

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veneer ceramics.[4] Improvements at the surface level are often needed to prolong the lives of materials. According to Griffith's theory of brittle fracture,[5] ceramic breakage is caused by microcracks within the ceramic, rather than by atomic bond breakage. Microcracks in ceramics proliferate continuously under stress and are connected to each other. This makes the actual failure forces in ceramics lower than their theoretical values.[6] Some investigators have reported that if crack propagation conditions can be controlled, the mechanical properties of the ceramics can be improved to some extent.[7]

Functional coatings are the most significant of the surface improvement methods. Among these coatings, boron nitride (BN) has been cited as one of the most remarkable coatings because of its excellent mechanical and chemical characteristics.[8]

The cubic structure of BN (c-BN) offers high erosion resistance because of its excellent mechanical and chemical properties, making c-BN an important coating material with a wide range of application. High-degree hardness, low friction coefficient, excellent thermal conductivity, and high electrical and erosion resistance, together with chemical stability at high temperatures, are the best-known properties of c-BN. c-BN is the hardest material after diamond. The chemical stability of c-BN against oxygen and iron-based materials at high temperatures is even better than diamond.[8] As chromium (Cr) is a frequently used material in dentistry and has excellent mechanical properties,[9] we have experimentally prepared BN–Cr coating material to benefit from the positive properties of both Cr and BN. Günen *et al*.[10] found that there was an improvement in abrasion resistance as well as a decrease in friction coefficient with Cr coating. However, there is not enough research in this field.

Tooth and dental restoratives are semi-translucent materials that allow partial light transmission, the color of which can be measured with spectrophotometers.[11-14] O'Brien *et al*.[15] interpreted clinical differences by classifying acceptable ΔE values. A value of ΔE ≥3.7 can be detected by 100% of observers and identifies a clinically detectable color difference. In cases in which the ΔE value is >3.7, the color difference has been interpreted as clinically incompatible.

The aim of this study was to investigate the effects of the BN and BN–Cr coatings on the surface hardness and color change of three different ceramic systems produced in a standard color (A1). The null hypothesis of this study was that the coating would not affect the color or the surface hardness.

MATERIALS AND METHODS

In this experimental study, ten discs (10 mm in diameter and 0.8 mm in thickness) each of IPS Empress and IPS EP were prepared with a framework thickness of 0.8 mm and a veneer thickness of 0.5 mm. IPS Empress and IPS EP discs were prepared by using the IPS Empress special wax (Elastiwax; Ivoclar Vivadent AG, Schaan, Liechtenstein) in accordance with the manufacturer's recommendations. Dental wax specimens were placed in phosphate-bonded investment material (IPS Empress, Esthetic Speed Investment, Ivoclar Vivadent AG). The pressing procedure for IPS Empress was about 20 min at 1075°C. Medium Opacity ingots (MO1) corresponding to A1 color in the Vita shade scale (Vita Zahnfabrik, Säckingen, Germany) for the IPS EP specimens were pressed at 960°C. Ten discs of Turkom-Cera alumina gel (Turkom-Ceramic SDN-BHD, Selangor, Malaysia) were used for Turkom-Cera frameworks and sintered at 1150°C for 5 min after 6 h of drying. Crystallization was completed by applying granulated glass (no. 2 Turkom-Cera Crystal Hardener; Turkom-Ceramic SDN-BHD) for 45–60 min at 1150°C. All specimens were measured by using a digital micrometer. The veneers were applied over the frameworks. The recommended veneering ceramics (in A1 color) were applied in thicknesses of 0.5 mm. IPS Empress Aesthetic Veneer (Ivoclar, Vivadent AG) for E; IPS e.maxCeram for EP; and VITA VM 7 for T. The E and EP specimens were fired in the EP600 vacuum porcelain furnace, and the T specimens were fired in the P300 vacuum porcelain furnace at the manufacturers' recommended firing temperatures for the recommended times.

BN coatings were done with a radiofrequency magnetron sputtering system. In sputter growth technique, a target, 2" radius and 0.250" thickness, is a material used for coating, which is prepared by the BN powders (99.99% pure, supplied from Graphene Supermarket, Calverton, USA). A BN–Cr target was also prepared by mixing the BN and Cr powders (Sigma Aldrich, Missouri, USA) with a weight ratio of 95%:5%. Simply during the sputtering process, the target, in this case BN or

BN–Cr, is bombarded by plasma of heavy ions, such as argon ions, to eject materials to be coated from the target surface. Growth was performed under 8 mTorr pressure. No substrate heating was applied. All the specimens were coated under the same growth conditions with a minimum thickness of 100 nm at room temperature.

Color measurements were performed using a spectrophotometer (Spectro Shade™ MICRO, Optic Research AG, Medical High Technologies). Measurements were made under the D65 standard lighting conditions corresponding to daylight, and the device was calibrated before each measurement. Measurements were performed on a standard white background (L = 92.9, a = 1.2, and b = −2.4), and the mean CIEL*a*b* value was obtained by measuring each specimen three times. The ΔE values of the ceramic specimens were calculated using the following formulae: $\Delta E = [(\Delta L^2)^{2} + (\Delta a^2) + (\Delta b^2)]$ ½ (ΔE = color difference), where ΔL = L2* − L1* (ΔL = brightness values); Δa = a2* − a1* (Δa = determined the difference in the red–green scale); and Δb = b2* − b1* (Δb = determined the difference in the green–yellow scale). The L2, a2, and b2 values represented the CIEL*a*b* values measured after coating the ceramic specimens, and the L1, a1, and b1 values represented the initially measured CIEL*a*b* values.[16]

The obtained data were analyzed using statistical software (PASW Statistics v18.0; SPSS Inc.) The data within all groups were subjected to Kolmogorov–Smirnov tests, and it was determined that they were not distributed normally. Wilcoxon signed-rank test was performed on the dependent samples to compare the hardness values obtained with different ceramic coatings. Kruskal–Wallis tests were performed to compare ΔE values among all groups, and the *post hoc* tests was used after Kruskal–Wallis (α = 0.05).

The surface chemistry of a specimen from Group T, coated with BN–Cr, was investigated by X-ray photoelectron spectroscopy (XPS) [Figure 1]. The analyses were performed with a Specs Flex-Mod. Monochromatized AlKα X-rays with an excitation energy of 1486.7 eV were used. Signals were detected by using a two-dimensional CCD detector with a 150-mm hemispherical energy analyzer.

The surfaces of two specimens from the T group, with BN coating and no coating, were examined by X-ray diffraction (XRD) [Figure 2]. XRD measurement

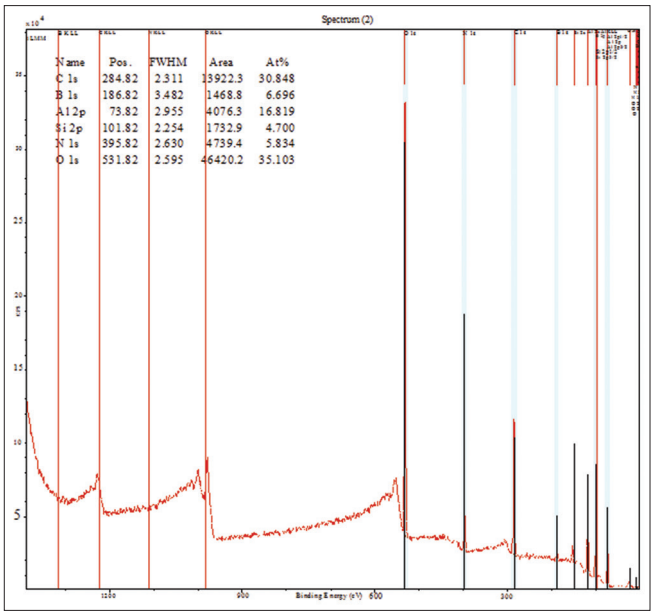

Figure 1: The result of X-ray photoelectron spectroscopy analysis.

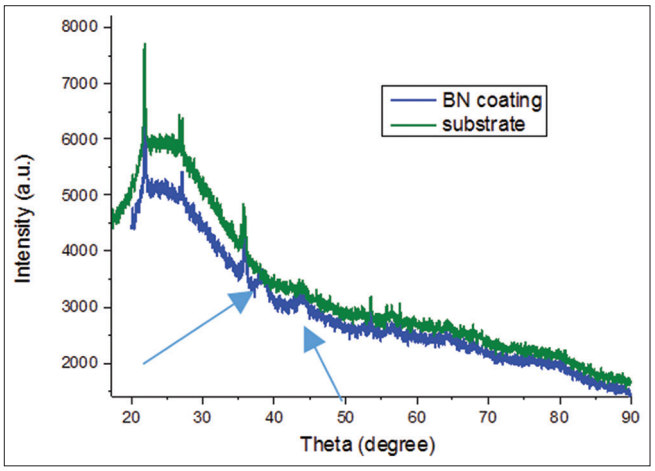

Figure 2: According to X-ray diffraction analysis, it can be said that the peak observed in 43.83 belongs to cubic boron nitrate.

was performed by a PANalytical Empyrean X-ray diffractometer with CuKα (λ = 1.5406 Å). Measurements were performed between 20°C and 90°C. Scanning electron microscopic (SEM) images [Figure 3] were made from a single specimen of each group after the specimens had been stored in distilled water for 24 h at 37°C. The initial Vickers hardness values (Shimadzu Dynamic HMV-G 21, Shimadzu Corporation) of all the ceramic specimens were then measured under a 0.98-N load with a 10-s waiting time prior to coating. Five different measurements were made from each specimen, and the arithmetic mean was obtained.[17] The hardness of each specimen was remeasured after the coatings were applied.

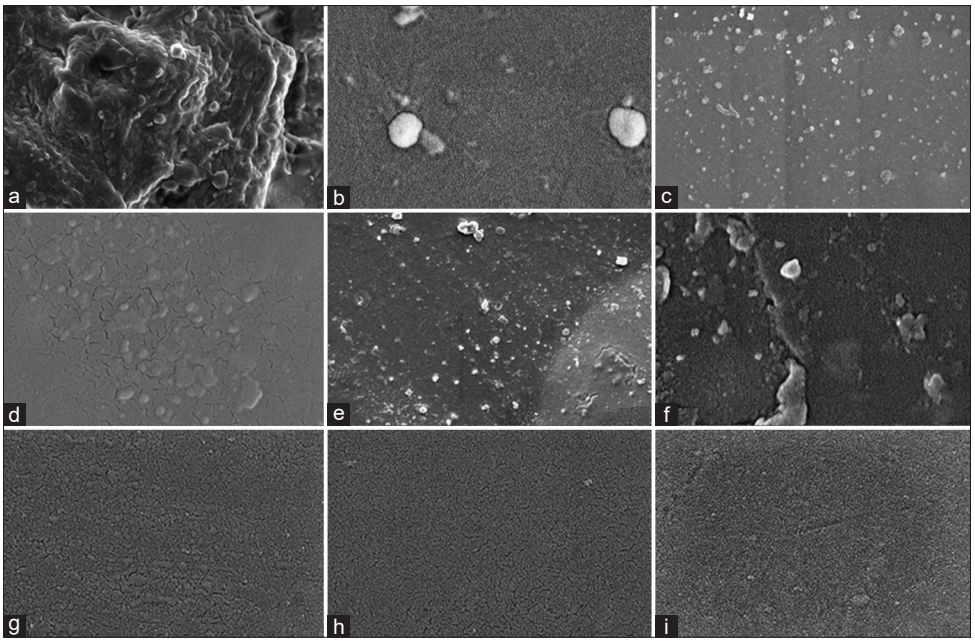

Figure 3: (a) Scanning electron microscopic image of boron nitride–chromium coating on E. (b) Scanning electron microscopic image of boron nitride–chromium coating on e.max Press. (c) Scanning electron microscopic image of boron nitride–chromium coating on T. (d) Scanning electron microscopic image of boron nitride coating on E. (e) Scanning electron microscopic image of boron nitride coating on e.max Press. (f) Scanning electron microscopic image of boron nitride coating on T. (g) Scanning electron microscopic image of control E. (h) Scanning electron microscopic image of control T. (i) Scanning electron microscopic image of control e.max Press.

RESULTS

The results of the Wilcoxon signed-rank test to compare the hardness values before and after coatings between the groups indicated that BN coating did not statistically significantly change the hardness ($P > 0.05$), whereas the BN–Cr coating increased the hardness statistically significantly in all types of ceramics ($P = 0.043$) [Figures 4, 5 and Table 1]. The mean ΔL, Δa, Δb, and ΔE obtained from all groups and the results of the statistical comparison are shown in Figure 6 (different letters equal significant differences and same letters equal nonsignificant differences). No significant differences were found between the groups as a result of the Kruskal–Wallis test, which was performed to compare ΔE values between the groups that BN–Cr coated ($P = 0.99$). The color darkened in all types of ceramics as a result of BN–Cr coating [Table 2 and Figure 7]. Regarding the mean ΔE values, it was determined that color change was higher than the clinically noticeable limit[15] of 3.7 in all groups that BN-Cr coated. Statistically significant differences were found between the groups as a result of the Kruskal–Wallis test, which was performed to compare ΔE values between the groups that BN coated ($P = 0.005$).

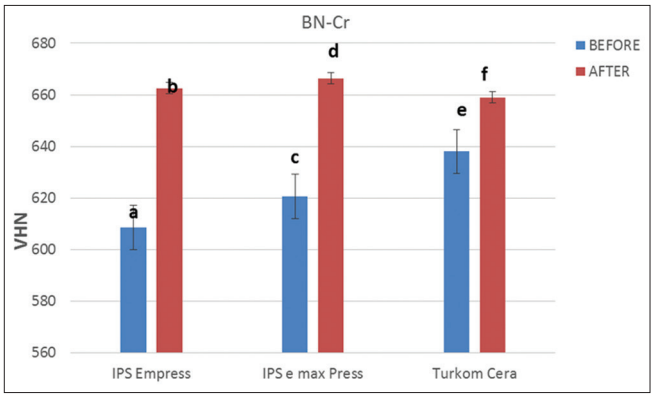

Figure 4: Vickers hardness values before and after boron nitride–chromium coating.

Differences were found between T-BN and E-BN groups [Table 2 and Figure 8]. Regarding the mean ΔE values, it was determined that color change was higher than the clinically noticeable limit[15] of 3.7 in E-BN group. It was determined that color change was not higher than the clinically noticeable limit[15] of 3.7 in the EP-BN and T-BN groups. In summary, it was determined that the BN–Cr coating contributed to the hardness, whereas the BN coating does not contribute to it.

As a result of XPS analysis, the appearance of B and N elements shows that the coating was made. In

Table 1: The results of the Wilcoxon signed-rank test

Coating type	Ceramic type	Hardness 1		Hardness 2		*Z*	*P*
		Mean±SD	Median (minimum-maximum)	Mean±SD	Median (minimum-maximum)		
BN-Cr	E	608.6±0.89	608 (608-610)	662.6±16.32	662 (643-683)	−2.023	0.043
	EP	620.6±9.66	619 (608-635)	666.4±19.63	668 (645-688)	−2.023	0.043
	T	638±16.32	645 (618-652)	659±21.48	673 (635-676)	−2.023	0.043
BN	E	608.8±1.3	608 (608-611)	574.6±64.19	589 (501-666)	−1.219	0.223
	EP	622.8±6.98	622 (612-629)	607.2±37.37	617 (544-642)	−0.674	0.5
	T	648.4±25.74	660 (619-676)	632.4±26.77	624 (608-676)	−1.214	0.225

BN: Boron nitride; BN-Cr: BN-chromium; SD: Standard deviation

Table 2: The results of the Kruskal-Wallis test

Coating type	Ceramic type	Delta E		χ^2	*P*	*Post hoc*
		Mean±SD	Median (minimum-maximum)			
BN-Cr	E	64.74±7.22	66.7 (53.5-71)	0.02	0.99	-
	EP	65.12±7.06	68.7 (54.2-71.2)			
	T	66±4.1	65.1 (60.2-70)			
BN	E	4.3±0.5	4.3 (3.7-4.8)	10.637	0.005	T-E
	EP	2.76±0.84	2.4 (2.1-4.2)			
	T	1.9±0.32	1.9 (1.5-2.3)			

BN: Boron nitride; BN-Cr: BN-chromium; SD: Standard deviation; E: Empress; ES: e.max Press; T: Turkom-Cera

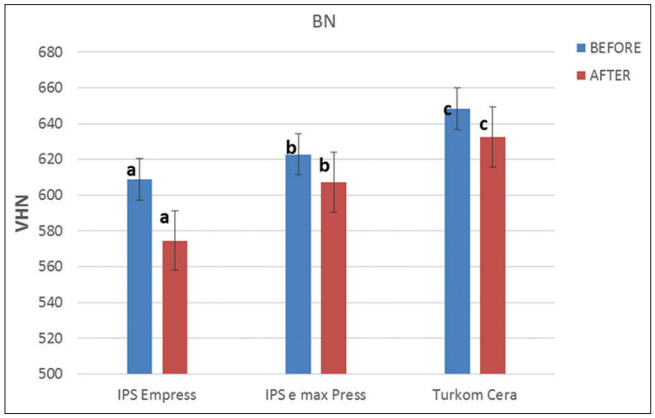

Figure 5: Vickers hardness before and after boron nitride coating.

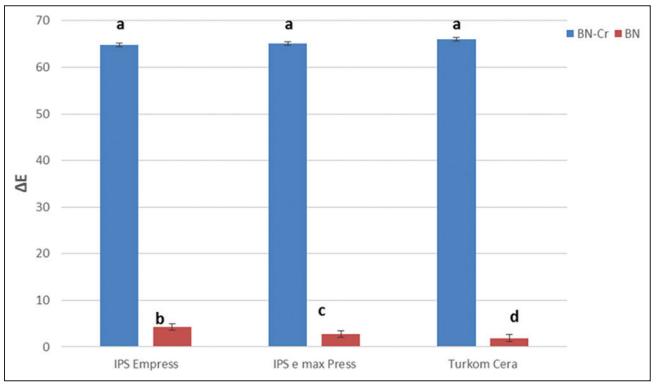

Figure 6: Color change before and after coatings.

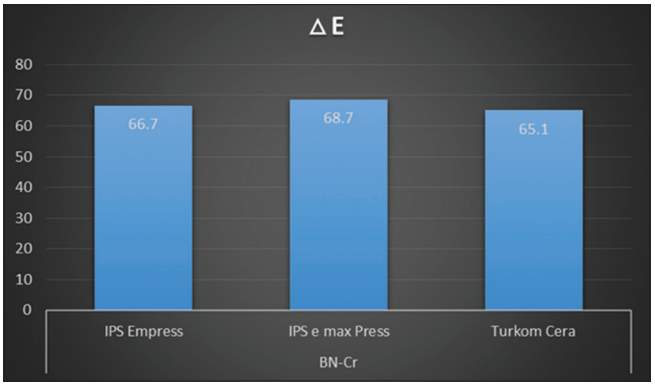

Figure 7: ΔE values between the groups that boron nitride-chromium coated.

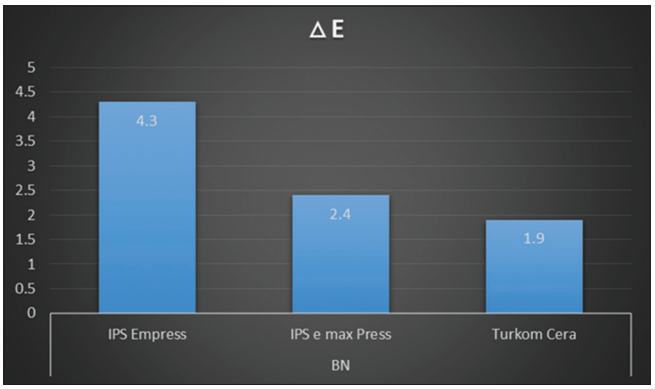

Figure 8: ΔE values between the groups that boron nitride coated.

the XPS analysis, elements such as Al and Si, which are in the content of the ceramic material, may also surface due to impact during the coating process due to their high diffusion. C and O elements are

caused by surface contaminants. The binding energy of advantageous C element is used to correct the spectrum. Almost stoichiometrics BN films have been obtained as seen from the inset table.

According to XRD analysis, it was determined that sharp peaks came from substrate. In contrast, the graph shown on the blue line shows two very broad peaks. BN peaks appear. The results of SEM analysis before and after the coating show that the coatings were made successfully.

DISCUSSION

BN, a ceramic material, is currently widely used because of its high hardness, erosion resistance, and durability. Its structure resembles diamond, and it is resistant to high temperatures (up to 1200°C) and pressure (~1 GPa).[13] BN also has excellent thermal conductivity.[18] However, cBN is rarely condensed by using conventional sintering methods because of its strong covalent structure and the tendency of its structure to transform into a soft hexagonal BN (hBN) phase.[19] Long known to increase surface hardness and provide adequate protection against erosion,[20] boron has become one of the alternative surface-hardening methods. In recent years, scientific research has focused on boron, and new areas are emerging for the use of boron compounds.[20]

Although the mechanical properties of bioactive ceramics are determined according to their crystal structures, some academicians have summarized the crack propagation behavior and fracture mechanism of ceramics. They found that improving the mechanical properties of the ceramic is possible to a certain extent by controlling the conditions of crack propagation.[21] Furthermore, in order to increase the fracture resistance of bioactive ceramics and to improve their mechanical performances, the resistance of ceramics to crack propagation and thus the fracture energy should be increased. Crack defects on the surface of and within the ceramic should be reduced, thus leading to reduction of stress concentration at the crack end.[22] The addition of graphene and boron nitrate nanotubes (BNNTs) to ceramics increases their fracture strength and fracture toughness.[23]

Chen *et al*.[24] and Gao *et al*.[25] reported that BNNTs had no adverse effect on cellular viability and proliferation in *in-vitro* cytotoxicity studies. Even though dental ceramics are highly esthetic, their high brittleness and low strength have limited their applications. In the last few years, low-dimensional nanomaterials (LDNMs), including carbon nanotubes (CNTs), graphene, and BNNTs, have attracted attention because of their biocompatibility and excellent mechanical properties. Because of these characteristics, LDNMs have become potential nanofillers for strengthening bioactive ceramics.[26] However, until now, maintaining the structural stability of LDNMs during the manufacturing of ceramic composites has been challenging because of the length of processing. Studies of dental ceramics in which BN coating was applied by using a sputtering method are lacking. In the present study, we used a coating approach to improve the surface hardness of the veneering porcelain on representative ceramic frameworks. The sputtering coating approach is a noninvasive method of surface modification to improve the surface hardness of silicate-based veneering ceramics. Hardness is a valuable property affecting the polishability, occlusal erosion resistance, and laboratory procedures of the ceramic material.[27] If the hardness of the veneering ceramic can be increased by strengthening procedures, its fracture resistance can be increased, thus avoiding material failures.[28] In the present study, an increase in superficial hardness was attempted by coating a thin layer on the veneering ceramic because materials with high hardness exhibit high abrasion resistance and are difficult to polish.

The magnetron sputtering method, based on the physical vapor deposition (PVD) principle, stands out among other coating methods, primarily because of its low coating temperature. The PVD magnetron sputtering method is widely used for coating all crystal forms of BN as a thin film.[20,29] BN, with a crystal structure as a result of the coating, may increase the hardness of porcelain more than BN with an amorphous structure. In the present study, the temperature was not increased while the coating was applied. An increased coating temperature may increase the crystal structure of BN, a topic for future study.

In another study, to increase the bond strength between fibers and ceramic matrix composites, coating of fibers with h-BN by chemical vapor deposition is presented.[30] One recent study found that CNT-reinforced Cr oxide coatings (reinforced with 8% CNT by weight and 92% Cr oxide by weight) increased microhardness by 30% and reduced porosity by 20%.[31] In a recent study, the physical

properties of nickel-chrome (Ni–Cr) spinel infrared radiation coating, plasma spraying on metal substrate were investigated. The results showed that the Ni–Cr spinel coating material has excellent infrared radiation properties. It is stated that the coating prepared by plasma spraying has infrared thermal stability and adhesion strength and can meet the standard of use.[32]

Limitations of this study include the limited number of test methods and veneer ceramic materials used to determine the mechanical properties. Moreover, the development of new strategies to ensure the structural stability of LDNMs should be pursued. It was difficult to maintain the structural stability of BN during the production of ceramic composites because of the length of the process.[23] As a result, the use of LDNMs in ceramics is at an early stage, and more research into this promising research area is needed.

CONCLUSION

According to the results of this study, the BN–Cr coating on the ceramic surface may increase surface hardness, but may be less clinically useful as the color change is excessive. Nevertheless, it may be useful in the palatal region. The BN coating does not change the color and remains esthetically successful, but does not increase the hardness of the ceramic. Its other physical properties should be evaluated before it can be recommended for clinical application.

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Conflicts of interest

The authors of this manuscript declare that they have no conflicts of interest, real or perceived, financial or non-financial in this article.

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