Effects of bioactive glass coating by electrophoretic deposition on esthetical, bending, and frictional performance of orthodontic stainless steel wire

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We investigated the surface modification of orthodontic stainless steel wire using electrophoretic deposition (EPD) of bioactive glass (BG). BG coatings were characterized by spectrophotometry, three-dimensional (3D) focal laser scanning microscopy and scanning electron microscopy. The mechanical properties of the BG-coated wires were estimated by nanoindentation, three-point bending and drawing friction tests. BG-coated specimens prepared at higher voltage showed higher values for both reflectance and L^* compared to those prepared at lower voltage. Specimens coated at higher voltage had significantly lower surface roughness than those coated at lower voltage, and their BG layers had higher hardness and elastic modulus values. In the three-point bending test, BG-coated wires produced significantly lower elastic modulus than non-coated wires. Most BG-coated specimens produced similar frictional forces to those produced by non-coated specimens. The surface modification technique applying EPD and BG coating to orthodontic stainless steel wire could be used to develop new esthetical orthodontic wire.

Keywords: Bending, Bioactive glass, Electrophoretic deposition, Friction, Nanoindentation

INTRODUCTION

Many orthodontic materials are formed from metals, which typically have superior mechanical properties compared with other materials. However, there are esthetic issues with metal orthodontic materials^{1,2}. More esthetically attractive orthodontic materials are desirable, especially for adult patients. Glass fiber-reinforced polymer wires have been investigated³⁻⁶, but are not yet widely used due to their brittleness and insufficient ability to withstand force³⁻⁵. Recently, coated archwires, including polymer-coated metal wires and rhodium-plated wires, have been developed^{2,7-12}. These are preferred by many patients and orthodontists for their improved esthetic qualities. However, polymer-coated wire loses a significant amount of its coating layer after 4–12 weeks in areas of archwire engagement within the oral cavity^{7,8}, which affects frictional properties¹¹ and bacterial adhesion⁹. One previous study⁸ reported that these coated wires generally showed less stiffness and produced lower forces compared with non-coated wires.

Acid etching of enamel surfaces for bracket bonding procedures has been accepted in modern clinical orthodontics since the mid-1960s^{12,13}; the enamel surface around bonded brackets is more susceptible to demineralization because these areas stagnate with plaque, making tooth-cleaning more difficult and

limiting the efficacy of natural self-cleaning mechanisms. Additionally, the mechanical properties of the enamel surface region decrease after bracket bonding with the etch-and-rinse adhesive system¹⁴, and irreversible alteration of the enamel can increase the risk of enamel micro-cracks forming during debonding procedures. Therefore, further demineralization of the enamel after bracket bonding should be prevented and, ideally, remineralization should be enhanced. One reasonable way to enhance the remineralization of tooth surfaces is to increase the calcium or fluoride concentrations of oral fluids^{15,16}. Various types of bioactive glass (BG) have been investigated since they were first reported¹⁷; the properties of BG include the ability to release ions (e.g., Ca, Na, Si) and form a hydroxyapatite layer, in addition to osteoinductive behavior, i.e., the ability to bond to both soft and hard tissues¹⁸⁻²¹. More recently, attention has focused on their modification to further enhance osteogenic behavior, or on the induction of further compositional changes to introduce additional multifunctional properties, such as antimicrobial activity²². Modification of metallic orthodontic material surfaces using BG could help to prevent the demineralization of tooth surfaces surrounding brackets and enhance remineralization after bracket debonding; these features are attractive in the clinical orthodontic setting.

Electrophoretic deposition (EPD) is a simple, rapid, and versatile coating technique whereby colloidal particles suspended in a liquid medium migrate under

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the influence of an appropriate electric field and are deposited onto an electrode, leading to film formation and coatings with high microstructural homogeneity and tailored thickness^{23,24}. Among the various techniques used for surface modification in the biomedical field, EPD is particularly attractive because it does not require expensive equipment and can be applied using colloidal BG particles to form complex-shaped orthodontic materials. A recent study²⁵ employed EPD to form thin BG layers on stainless steel plate specimens, and found that the amorphous structure of the BG layer was sustained to yield a satisfactory esthetic appearance and remineralization ability in artificial saliva. If the BG process can be applied to orthodontic wires, it may be beneficial in terms of preventing caries while maintaining a satisfactory appearance. The mechanical and frictional performance of BG should therefore be explored.

In this article, BG particles were deposited onto orthodontic stainless steel wires using the EPD process under various conditions, and the BG coating was characterized esthetically, morphologically, compositionally, and mechanically using various methods. The effects of the BG coating on the bending and frictional properties of the wire were also investigated.

MATERIALS AND METHODS

Materials

As-received stainless steel orthodontic wires with cross-sectional dimensions of 0.017×0.025 inches (stainless steel archwire, 3M Unitek, Monrovia, CA, USA) were purchased and subjected to BG coating. As-received, preadjusted stainless steel orthodontic brackets (Mini Uni-Twin, 3M Unitek) for the upper canine teeth were used for friction testing. Non-coated specimens served as a control.

BG (45.0% SiO₂+24.5% Na₂O+24.5% CaO+6.0% P₂O₅) was prepared by melting raw materials in a platinum crucible at 1,550°C for 90 min using an electrically heated furnace (SSFT-1520, Yamada Denki, Tokyo, Japan). The molten glass was rapidly quenched by malleating (rolling) between two stainless steel plates (thickness: 10 mm). After cooling overnight, the glass was ground for 2 min in a vibrational rod mill (TI-200, CMT, Fukushima, Japan) to yield a particle diameter of *ca.* 100 µm. The powders were further milled using a high-pressure gas-milling apparatus (Nano Jetmizer, Aishin Nano Technologies, Saitama, Japan) under a grinding pressure of 1.4 MPa to provide particles with a median diameter (D₅₀) of 1.98 µm. Analysis of the BG by X-ray diffraction (XRD) confirmed its amorphous structure.

EPD process

Suspensions containing 20 g/L BG were prepared in 100 mL distilled water by dispersing the particles *via* magnetic stirring and sonication (UD-100, Tomy Seiko, Tokyo, Japan) for 600 s. The EPD cell included two parallel stainless steel wire specimens (length: 50 mm) as the deposition and counter electrodes; the distance

Fig. 1 Photomicrographs of non-coated and BG-coated wire specimens.

between electrodes was maintained at 3 mm. The coating was deposited using direct current (DC) or 1-kHz sine-wave alternating current (AC). Two voltages (10 and 15 V) were applied; the deposition time was 10 min for all conditions. After deposition, a coated specimen was gently removed from the suspension and dried at room temperature for 24 h before further characterization (Fig. 1). The thickness of BG-coated layers formed on the wire specimens was expected to be approximately 1–4 µm, based on the results of a previous investigation in which BG-coated stainless steel plate specimens were made using the same coating conditions²⁵.

Color measurements

The color of non-coated and BG-coated wire specimens was measured using a spectrophotometer (UV-2600, Shimadzu, Kyoto, Japan) with an integrated sphere (ISR-2600 Plus, Shimadzu). Diffuse reflectance measurements were performed in the range of 350–800 nm in 1-nm steps. Color was measured according to the Commission International de L'Eclairage (CIE) *L*^{*}*a*^{*}*b*^{*} color system (*n*=5)²⁶, which comprises a lightness scale, *L*^{*}, and two opponent color axes, *a*^{*} and *b*^{*}. Redness and greenness are represented by the *a*^{*} values, and yellowness and blueness are represented by the *b*^{*} values.

Coating surface characterization

The external surfaces of BG-coated wire specimens were examined using a three-dimensional (3D) confocal laser scanning microscope (VK-X200, Keyence, Tokyo, Japan; *n*=5). The mean surface roughness (*S*_a) was calculated at seven different areas of each specimen using the software supplied with the laser scanning microscope.

Nanoindentation and three-point bending testing of coating mechanical properties

The external surfaces of non-coated and BG-coated wire specimens were investigated by nanoindentation testing (ENT-1100a, Elionix, Tokyo, Japan). Specimens were fixed to the specimen stage with adhesive resin (Superbond Orthomite, Sun Medical, Shiga, Japan).

Nanoindentation testing was conducted at 28°C using a Berkovich indenter with a 10-mN peak load for *ca.* 1,000 nm depth analysis ($n=10$). Linear extrapolation methods (ISO Standard 14577) were used to determine the unloading curve between 95 and 70% of the maximum test force to calculate the elastic modulus²⁷⁻³⁰. The hardness and elastic modulus of the specimens were calculated using the software bundled with the nanoindentation apparatus.

Three-point bending test was conducted for non-coated and BG-coated wire specimens ($n=10$). A 12-mm span was chosen for the wire segments, in accordance with ANSI/ADA Specification No. 32. All specimens were loaded following the same protocol on a universal testing machine equipped with a 20-N load cell (EZ Test, Shimadzu) at room temperature (25°C). Each wire specimen was loaded to a deflection of 5.0 mm at a rate of 0.5 mm min⁻¹. Following the three-point bending test, representative specimens were sputter-coated with pure gold and inspected using a scanning electron microscope (SEM; SSX-550, Shimadzu) at 15 kV to observe BG layer detachment.

Drawing-friction testing of coating frictional properties

The frictional forces generated by each wire/bracket combination were measured under dry conditions at room temperature (25°C) using a custom-fabricated drawing-friction testing device attached to a universal testing machine (EZ Test, Shimadzu)³¹. Each bracket was bonded to a stainless steel plate with a non-filled adhesive resin (Superbond, Sun Medical), and a bracket-mounting device provided 0° or 10° angular positioning for the bracket. The stainless steel plate with the bracket was attached to a friction-testing device; a 4-cm wire segment was then bound to the bracket using an elastic ligature (Alastik Easy-To-Tie Ligatures, 3M Unitek). The upper end of the wire was fixed to a grip attached to the load cell, and the lower end of the wire was fixed to a 150-g weight. Each wire was drawn through the bracket at a crosshead speed of 10 mm min⁻¹ for a distance of 5 mm. The static frictional forces were determined from the load-displacement curves³². The sample size for each condition was 10 ($n=10$). Following the drawing-friction test, representative specimens were investigated using a stereoscopic microscope (SMZ 1500, Nikon, Tokyo, Japan).

Statistical analyses

Statistical analyses were performed using SPSS Statistics software (ver. 23J for Windows, IBM, Armonk, NY, USA). Mean values obtained in the experiments performed in this study were compared using one-way analysis of variance (ANOVA) followed by Tukey's or Games-Howell tests. Mean frictional forces were analyzed using two-way ANOVA; the two factors were coating procedure (non-coating, DC10V, DC15V, AC10V, and AC15V) and bracket torque (0° and 10°). For all statistical tests, significance was determined at a level of $p<0.05$. Data are presented as means and standard deviations.

RESULTS

Color measurements

Figure 2 shows the changes in representative diffuse reflectance curves for the wire specimens. Lightness (L^*) and opponent color (a^* , b^*) values are summarized in Table 1. Reflectance values (%) increased with increasing wavelength among all specimens. Non-coated wire specimens showed significantly higher reflectance values in the 350–800 nm range and L^* , because the polished bright surface acted as a mirror. Among BG-

Fig. 2 Diffuse reflectance curves of the non-coated and BG-coated wire specimens.

Table 1 Commission International de L'Eclairage (CIE) lightness (L^*) and color (a^* , b^*) values for the specimens

	Non-coating		DC10V		DC15V		AC10V		AC15V		<i>p</i> value
	Mean	S.D.	Mean	S.D.	Mean	S.D.	Mean	S.D.	Mean	S.D.	
L^*	70.69 ^a	0.26	60.39 ^b	0.87	64.78 ^c	0.14	62.30 ^d	0.36	64.99 ^c	0.62	0.000
a^*	-0.04 ^a	0.02	-0.70 ^b	0.06	-0.18 ^c	0.07	-0.18 ^c	0.08	-0.27 ^c	0.05	0.000
b^*	4.09 ^a	0.08	7.44 ^b	0.66	6.42 ^{bc}	0.11	5.90 ^c	0.42	6.27 ^{bc}	1.34	0.000

Values are mean and standard deviation (S.D.), $n=5$. One-way ANOVA followed by Tukey-Kramer multiple range test. Identical letters indicate that mean values were not significantly different ($p<0.05$).

coated wire specimens, those prepared at higher voltage (15 V) showed higher values for both reflectance in the 550–800 nm range and L^* compared with those prepared at lower voltage (10 V), under both DC and AC conditions. Similar a^* and b^* values were obtained for all BG-coated specimens, with the exception of a^* and b^* values for one specimen prepared at 10 V DC.

Coating morphological features and surface roughness (S_a)

Figure 3 shows representative 3D images of the external

surfaces of non-coated and BG-coated wire specimens, obtained by confocal laser scanning microscope. All BG-coated specimens had similar surface morphology. Quantitative analysis confirmed that S_a values were significantly higher in all coated wire specimens than in non-coated wire specimens, and were significantly lower in specimens coated at higher voltage (15 V) than in those coated at lower voltage (10 V) under both DC and AC conditions (Table 2).

Fig. 3 3D images of the external surfaces of non-coated and BG-coated wire specimens obtained using a confocal laser scanning microscope.

Table 2 Average surface roughness (S_a values) of non-coated and BG-coated wire specimens

	Non-coating		DC10V		DC15V		AC10V		AC15V		p value
	Mean	S.D.	Mean	S.D.	Mean	S.D.	Mean	S.D.	Mean	S.D.	
S_a (μm)	0.00 ^a	0.00	0.79 ^b	0.12	0.46 ^c	0.13	0.75 ^b	0.05	0.61 ^d	0.19	0.000

Values are mean and standard deviation (S.D.), $n=20$. One-way ANOVA followed by Tukey-Kramer multiple range test. Identical letters indicate that mean values were not significantly different ($p<0.05$).

Table 3 Summary of mechanical properties of non-coated and BG-coated wire specimens

	Non-coating		DC10V		DC15V		AC10V		AC15V		n	p value
	Mean	S.D.	Mean	S.D.	Mean	S.D.	Mean	S.D.	Mean	S.D.		
Hardness (GPa) †	6.11 ^a	0.22	0.49 ^b	0.10	1.99 ^c	0.52	0.85 ^b	0.48	1.98 ^c	0.73	7	0.000
Elastic modulus (GPa) †	192.46 ^a	4.96	70.47 ^b	23.22	109.12 ^{cd}	23.39	84.32 ^{bc}	15.28	128.59 ^d	27.65	7	0.000
Elastic modulus (GPa) ††	166.40 ^a	1.32	155.00 ^b	2.49	152.63 ^{bc}	1.40	149.95 ^c	2.92	146.36 ^{bd}	4.81	10	0.000

Values are mean and standard deviation (S.D.) One-way ANOVA followed by Tukey-Kramer multiple range test. Identical letters indicate that mean values were not significantly different ($p<0.05$).

†: obtained by nanoindentation test (1,000 nm depth from surface), ††: obtained by three-point bending test.

Nanoindentation and three-point bending test results

Table 3 summarizes the mechanical properties of surface regions of the non- and BG-coated wire specimens. In nanoindentation testing, hardness and

elastic modulus were obtained for BG layers at a 1,000-nm depth from the specimen surface to characterize the mechanical properties of BG-coated layers, whereas elastic modulus was obtained for the entire specimen

Fig. 4 SEM photomicrographs taken following the three-point bending test.

Table 4 Static frictional forces (gf) of the non-coated and BG-coated wire specimens

	Non-coating		DC10V		DC15V		AC10V		AC15V		<i>p</i> value
	Mean	S.D.	Mean	S.D.	Mean	S.D.	Mean	S.D.	Mean	S.D.	
0 degree	130.22 ^a	11.72	145.41 ^{ab}	18.66	145.11 ^{ab}	9.09	162.19 ^b	14.83	140.00 ^{ab}	16.30	0.035
10 degree	198.44 ^a	13.82	363.63 ^{ab}	98.79	278.59 ^{ab}	21.82	349.81 ^b	100.35	305.00 ^{ab}	28.58	0.000

Values are mean and standard deviation (S.D.), $n=5$. One-way ANOVA followed by Tukey-Kramer multiple range test. Identical letters indicate that mean values were not significantly different ($p<0.05$).

Before friction test

After friction test at bracket angulations of 0°

After friction test at bracket angulations of 10°

Fig. 5 Stereomicroscope photomicrographs taken following the drawing-friction tests. Original magnification $\times 96$.

by three-point bending. Nanoindentation testing results showed that hardness and elastic modulus values of the BG layers were significantly lower than those of non-coated specimens, and significantly higher among specimens coated at higher voltage (15 V) than among those coated at lower voltage (10 V). According to the three-point bending test, BG-coated wire specimens produced significantly lower elastic modulus than non-coated wires. SEM images taken following three-point bending tests confirmed that none of the BG coatings were damaged after three-point bending and that they sustained good interfacial adhesion (Fig. 4).

Drawing-friction test results

Table 4 summarizes the static frictional forces determined by drawing-friction testing of the non-coated and BG-coated wire specimens under dry conditions. Two-way ANOVA results showed that coating procedure (non-coating, 10 V DC, 15 V DC, 10 V AC, 15 V AC) and bracket angle (0°, 10°) were statistically significant factors affecting the static frictional forces (not shown). One-way ANOVA and Tukey's test showed no significant difference in frictional force (at 0° bracket angle) between non-coated and BG-coated wire specimens, except for one BG-coated wire specimen (10 V AC). When the bracket angle was increased from 0° to 10°, the frictional force obtained from BG-coated specimens (10 V DC, 10 V AC) produced significantly greater frictional forces than the non-coated wire specimen. Stereomicroscope photomicrographs taken after drawing-friction tests confirmed that none of the coatings for all wire specimens were damaged or removed upon frictional testing at bracket angulations of 0° (Fig. 5). After drawing-friction tests at bracket angulations of 10°, partially removed coating layers were observed in areas rubbed by the bracket slot.

DISCUSSION

In the present study, thin BG coating layers with a milky-white appearance were formed on stainless steel wire specimens using an EPD process with a BG suspension. Quantitative color measurements showed that the EPD process at higher voltage (15 V) under both DC and AC conditions produced higher values for both reflectance (%) in the range of 550–800 nm and L^* (mean: 64.78 for DC, 64.99 for AC). The range of L^* values measured for BG coating layers (60.39–64.99) was greater than that previously reported for ceramic and plastic brackets (36.2–50.3)³³, although the color values (a^* : -0.70 to -0.18, b^* : 5.90 to 7.44) of the BG coating layers were similar (a^* : -1.3 to 3.8, b^* : -2.9 to 11.2). The color of orthodontic appliances, such as brackets and archwires, should ideally match that of natural teeth, although natural tooth color varies according to race, gender, and age³⁴. A previous study measured CIE $L^*a^*b^*$ color values using the Vita Lumin Vacuum shade guide (A3.5, B1, B3, C4)³³, the color selection scale most widely used in dentistry; the values ranged from 43.2 to 61.4 for L^* , from -1.6 to 6.8 for a^* , and from 13.2 to 28.8 for b^* . The L^*

and a^* values obtained in the present study were similar, although our mean b^* value was smaller than that of the Vita Lumin Vacuum shade guide. Therefore, BG coatings formed by the EPD process in the present study likely produced a clinically acceptable color appearance.

In the present study, BG-coated wire specimens produced significantly lower elastic modulus according to the results of a three-point bending test (146–155 GPa) compared with the non-coated wire (166 GPa). This finding is consistent with a previous study⁸. The most likely reason is that the work-hardened structure of stainless steel may have been annealed during the EPD coating process. An alternative reason is that the cross-sectional dimension of the BG-coated wires might have been reduced due to the corrosion of stainless steel surfaces during the EPD process. Fortunately, the reduction in elastic modulus was only 7–12%. SEM images taken after the three-point bending test confirmed that none of the BG coatings had been damaged by three-point bending, and that they had sustained good interfacial adhesion (Fig. 4). Therefore, minimum influence should be expected in clinical tooth movement due to orthodontic forces from the BG-coated wires.

A previous study²⁵ in which EPD was applied to form BG layers on stainless steel plate specimens showed that the etched enamel surfaces of the BG layers had remarkable remineralization ability. Amorphous calcium phosphate (ACP) is an outstanding material for supply mineral³⁵. A previous study³⁶ comparing remineralization potential between ACP and BG demonstrated that both were able to remineralize early enamel caries. Previous BG studies have reported significant acid-neutralizing properties due to the ability of BG to release a variety of alkali ions (*e.g.*, Si, Na, and Ca), which exhibit antibacterial activity^{37,38}. Similar remineralization and acid-neutralizing capabilities can be expected for BG layers formed on stainless wire specimens in the present study, and these properties may mitigate enamel demineralization and accelerate the remineralization of etched enamel; however, further study is required to fully explore these properties.

The frictional force between the bracket and archwire during tooth movement is a primary issue in orthodontics. If the frictional force can be decreased, then the efficiency of tooth movement can be improved^{31,32}. These frictional properties are attributed to multiple factors such as surface roughness, hardness, elastic modulus, and the cross-sectional dimensions of the orthodontic wires and brackets^{31,32}. A recent study reported that commercially available esthetic coating wire influences the frictional properties of the wire¹¹; esthetic polymer coating may increase frictional resistance due to increased wire-binding at the edge of the bracket. In the present study, BG-coated specimens (10 V DC, 10 V AC) produced significantly greater frictional forces than the non-coated wire specimen when the bracket angle was increased from 0° to 10°. The BG layer formed on the surface of wire specimens produced significantly lower hardness (0.5–2 GPa) than the surface of non-coated wire (6.1

GPa), according to the results of a nanoindentation test. High frictional resistance for BG-coated specimens may be due to their low surface hardness, which may increase wire-binding between the bracket and wire. However, similar frictional forces were produced from BG-coated and non-coated wire specimens when EPD was conducted at higher voltage (15 V) under both DC and AC conditions. One possible reason for this finding is that specimens coated at higher voltage (15 V) had significantly higher hardness and elastic modulus and smaller S_a values than wire specimens coated at lower voltage (10 V) for both DC and AC conditions, which may decrease wire-binding at the edge of the bracket. Additionally, the BG coating layer formed on specimens at the higher voltage (15 V DC, 15 V AC) had better quality, density, and mechanical properties, which can decrease wire-binding. The BG coating formed at the higher voltage had favorable esthetic character and acceptable mechanical properties. Therefore, higher voltage (15 V) may be more suitable for the EPD coating process. More research is needed to investigate which coating conditions yield improved mechanical properties and better interfacial adhesion, as partially removed coating layers were observed after the drawing-friction test.

CONCLUSIONS

The findings of this study are summarized as follows:

1. The BG coating process using EPD had little influence on bending and friction performance. BG coating formed at higher voltage (15 V) under both DC and AC provided acceptable quality, favorable esthetic character, and good mechanical properties.
2. The surface modification technique using EPD and BG for orthodontic stainless steel wire offers the potential to develop new orthodontic metallic wires with satisfactory esthetic appearance and remineralization ability, without being cytotoxic.

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