Original Article

Effects of a diamond-like carbon coating on the frictional properties of orthodontic wires

Takeshi Muguruma[a]; **Masahiro Iijima**[b]; **William A. Brantley**[c]; **Itaru Mizoguchi**[d]

ABSTRACT

Objective: To test the hypothesis that a diamond-like carbon coating does not affect the frictional properties of orthodontic wires.

Materials and Methods: Two types of wires (nickel-titanium and stainless steel) were used, and diamond-like carbon (DLC) films were deposited on the wires. Three types of brackets, a conventional stainless steel bracket and two self-ligating brackets, were used for measuring static friction. DLC layers were observed by three-dimensional scanning electron microscopy (3D-SEM), and the surface roughness was measured. Hardness and elastic modulus were obtained by nanoindentation testing. Frictional forces and surface roughness were compared by the Kruskal-Wallis and Mann-Whitney *U*-tests. The hardness and elastic modulus of the wires were compared using Student's *t*-test.

Results: When angulation was increased, the DLC-coated wires showed significantly less frictional force than the as-received wires, except for some wire/bracket combinations. Thin DLC layers were observed on the wire surfaces by SEM. As-received and DLC-coated wires had similar surface morphologies, and the DLC-coating process did not affect the surface roughness. The hardness of the surface layer of the DLC-coated wires was much higher than for the as-received wires. The elastic modulus of the surface layer of the DLC-coated stainless steel wire was less than that of the as-received stainless steel wire, whereas similar values were found for the nickel-titanium wires.

Conclusions: The hypothesis is rejected. A DLC-coating process does reduce the frictional force. (*Angle Orthod.* 2011;81:141–148.)

KEY WORDS: Orthodontic wire; Nickel-titanium; Stainless steel; Diamond-like carbon coating; Frictional properties; Nanoindentation test

INTRODUCTION

Friction (resistance to sliding) between the bracket and wire (archwire) during orthodontic tooth movement is an important factor in clinical orthodontics because a decrease in friction might shorten the treatment period and also improve anchorage control.[1] There are two types of friction: static and kinetic. Static friction occurs until the force is great enough to overcome the initial resistance to movement of the object; kinetic friction then opposes the continuation of movement.[1] The kinetic friction is irrelevant in orthodontic tooth movement because continuous motion along an archwire rarely, if ever, occurs.[1] Friction during clinical tooth movement depends on the size and shape of the wire,[2] the bracket type,[3,4] the bracket and wire materials,[5] the angulation of the wire relative to the bracket,[6] the type of ligation,[2] and whether the environment is wet or dry.[7] Kusy and Whitley[8] divided friction into three components: (1) friction, static and kinetic, due to contact of

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the wire with the bracket surface; (2) binding, created when the tooth tips or the wire flexes so that there is contact between the wire and the corners of the bracket; and (3) notching, when permanent deformation of the wire occurs at the wire-bracket corner interface.

Ever since self-ligating brackets were introduced a few decades ago, they have become widely used in clinical orthodontics. A previous study with an in vitro model showed that friction at an angulation of 0° was lower for self-ligating brackets than for a conventional bracket.[9] However, when the wire-bracket angulation increased, binding also increased, and the binding was similar with conventional and self-ligating brackets.

Recently, a plasma immersion ion implantation technique has become quite common in surface-coating to improve the mechanical properties and corrosion resistance of beta-titanium[10] and nickel-titanium alloys.[11] Ion-implanted nickel-titanium wire (Neo Sentally Ionguard, GAC International, Islandia, NY) and beta-titanium wire (Low Friction TMA, Ormco, Orange, Calif) are commercially available. Previous studies[12] with an in vitro model have shown that ion-implanted wires (nickel-titanium and beta-titanium) produce less frictional forces during tooth movement, although another randomized clinical trial on initial alignment[13] showed that there were no significant differences between ion-implanted nickel-titanium wire and nonimplanted nickel-titanium wire. Another surface-coating technique with diamond-like carbon (DLC), which offers excellent properties such as extreme hardness, low friction coefficients, and high wear-resistance[14] is becoming increasingly important in industrial applications, and the application of a DLC film to orthodontic appliances offers the potential of greatly improving frictional properties and corrosion resistance. A recent study showed that DLC films protect against the diffusion of nickel and its release at the surface of nickel-titanium archwires, and that these films are noncytotoxic in a corrosive environment.[14] Another research group investigated the effect of DLC coating on the static and kinetic friction of stainless steel, copper nickel-titanium, and beta-titanium wires, and found that DLC coatings of copper nickel-titanium and beta-titanium produced less frictional resistance than found for as-received wires.[15] However, they did not study the mechanical properties and morphology of the DLC layer, which greatly influences the frictional properties.

The purpose of this study was to investigate the effect of a DLC coating on the frictional properties of orthodontic nickel-titanium and stainless steel wires. Static friction of the as-received and DLC-coated wires was measured with a custom-fabricated friction-testing device for a conventional bracket and two types of self-ligating brackets. The wire surfaces were characterized by a three-dimensional scanning electron microscope (3D-SEM); the hardness and elastic modulus were investigated by nanoindentation testing. It was hypothesized that a DLC coating does not affect the frictional properties of orthodontic wires.

MATERIALS AND METHODS

Materials

Nickel-titanium wires with a diameter of 0.016 inch (Nitinol Super Elastic, 3M Unitek, Monrovia, Calif) and stainless steel wires with a diameter of 0.018 inch or cross-section dimensions of 0.019 inch × 0.025 inch (stainless steel archwire, 3M Unitek) were used in this study. One hundred twenty upper canine brackets for each of three types were used. The conventional stainless steel brackets (Victory Series, 3M Unitek) had a slot dimension of 0.022 inch, a mesiodistal width of 3.2 mm and no built-in torque or tip. The In-Ovation self-ligating brackets (GAC International) had a slot dimension of 0.022 inch, a mesiodistal width of 3.0 mm and no built-in torque or tip. The Damon Q self-ligating brackets (Ormco, Orange, Calif) had a slot dimension of 0.022 inch, a mesiodistal width of 2.8 mm, 0° torque, and +5° angulation.

DLC-coating Procedure

DLC films were deposited on the nickel-titanium and stainless steel wires using the plasma-based ion implantation/deposition (PBIID) method. All wires were fixed with a custom-made jig in the PBIID equipment (PEKURIS-HI, Kurita Seisakusho, Kyoto, Japan), and deposition was carried out at a target voltage of 5 kV under a pressure of 1.33×10^{-3} Pa and an acetylene gas atmosphere for 400 minutes.[16]

Friction Test

The static frictional force generated with each wire/bracket combination was measured under dry conditions and at room temperature (25°C) using a custom-fabricated friction-testing device attached to a universal testing machine (EZ Test, Shimadzu, Kyoto, Japan) with a load cell of 20 N (Figure 1a). The custom-fabricated friction-testing device was designed according to the descriptions by Redlich et al.[3] and Cha et al.[17] Each bracket was bonded with a nonfilled adhesive resin (Superbond, Sun Medical, Shiga, Japan) to a stainless steel plate using a bracket-mounting device (Figure 1b through d), which could give an accurate angulation to the nearest degree. In this study, an angulation of 0° or 10° was selected to position the bracket. The stainless steel plate with the bracket was attached to the friction-testing device, and

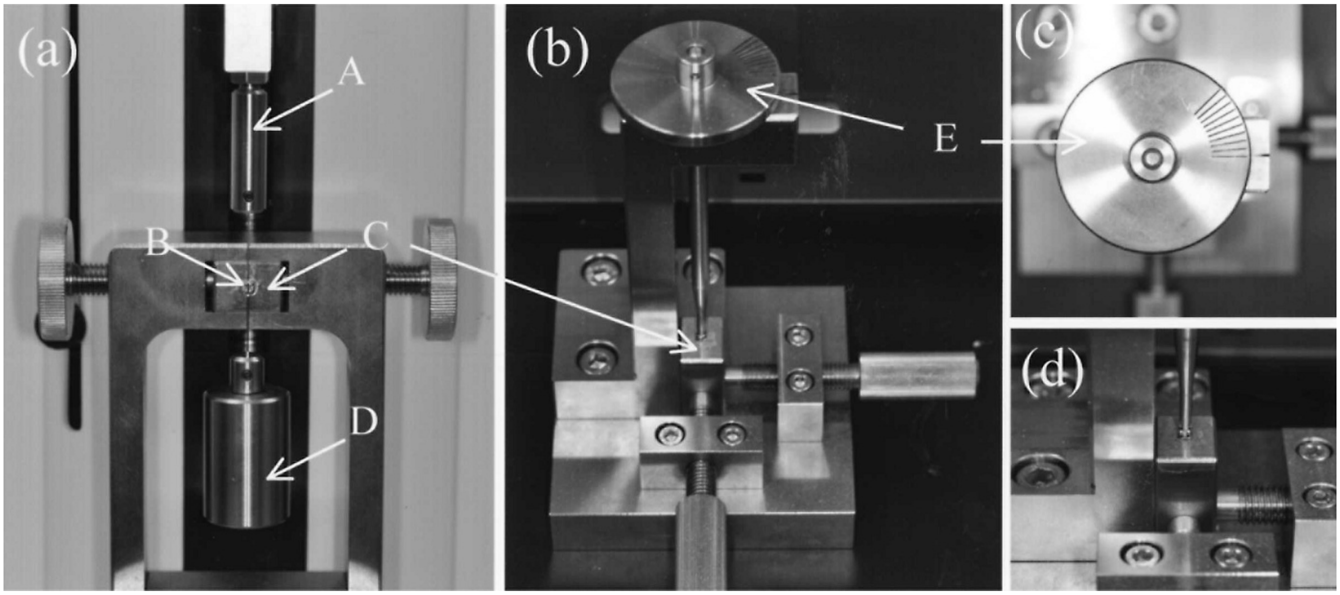

Figure 1. Friction-testing system. (a) Custom-fabricated friction-testing device attached to a universal testing machine. (b), (c), and (d) Bracket-mounting device. A, grip; B, bracket/wire combination specimen; C, stainless steel plate; D, weight (150 g); and E, angle measurement device.

a 5-cm segment of wire was then ligated to the bracket with an elastomeric ligature (AlastiK Easy-to-Tie Ligatures, 3M Unitek), except that the self-ligating brackets were tested in a closed position. The upper end of the wire was fixed in a grip that was 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 cross-head speed of 10 mm/min for a distance of 5 mm. The static frictional force was determined from load-displacement curves.[1] The sample size for each bracket/wire combination and for the two bracket angulations (0° or 10°) was 10. A total of 360 brackets were used in this study.

3D-SEM Observation and Measurement of the Surface Roughness

To observe the DLC layer in cross-section, representative DLC-coated nickel-titanium and stainless steel wires were encapsulated in epoxy resin (Epofix, Struers, Copenhagen, Denmark) and polished using a series of silicon carbide abrasive papers and a final slurry of 0.05-μm alumina particles; argon ion etching was carried out using an ion shower apparatus (EIS-200ER, Elionix, Tokyo, Japan). The DLC layers were observed by 3D-SEM (ERA-8900, Elionix) with four-channel secondary electron detectors.

To observe and characterize the external surfaces of the as-received and DLC-coated wires, all specimens were sputter-coated with platinum and examined using 3D-SEM. The R_a value, the arithmetic mean of the height of peaks and depth of valleys from a mean line, was used for values of the surface roughness. The R_a at five different areas for each wire was calculated by software that was supplied with 3D-SEM.

Nanoindentation Test

The external surfaces of the as-received and DLC-coated wires were fixed to the specimen stage for the nanoindentation test using adhesive resin. All nanoindentation testing was carried out at 28°C (ENT-1100a, Elionix) using a Berkovich indenter. The measurement points were selected using the optical microscope and charge-coupled device (CCD) camera coupled to the nanoindentation test apparatus. Each test consisted of three segments: 10 seconds for loading to the peak value, a 1-second hold at the peak load, and 10 seconds for unloading. Two peak loads, 5 mN and 100 mN, were used for measurements. The maximum depth of indentation, hardness, and elastic modulus were calculated by software that was supplied with the nanoindentation apparatus, using equations in ISO Standard 14577.[18] The sample size for each wire was 10.

Statistical Analysis

Statistical analysis was performed using the Statistical Package for Social Science software (version 16.0J for Windows, SPSS Inc, Chicago, Ill). The data were examined for normality with the Levene's test. Since the data for the mean static frictional force were not normally distributed, values were compared using the Kruskal-Wallis test and the Mann-Whitney *U*-test. Significance was predetermined at $P < .0167$. Since the data for surface roughness were also not normally distributed, mean values were compared by the Mann-

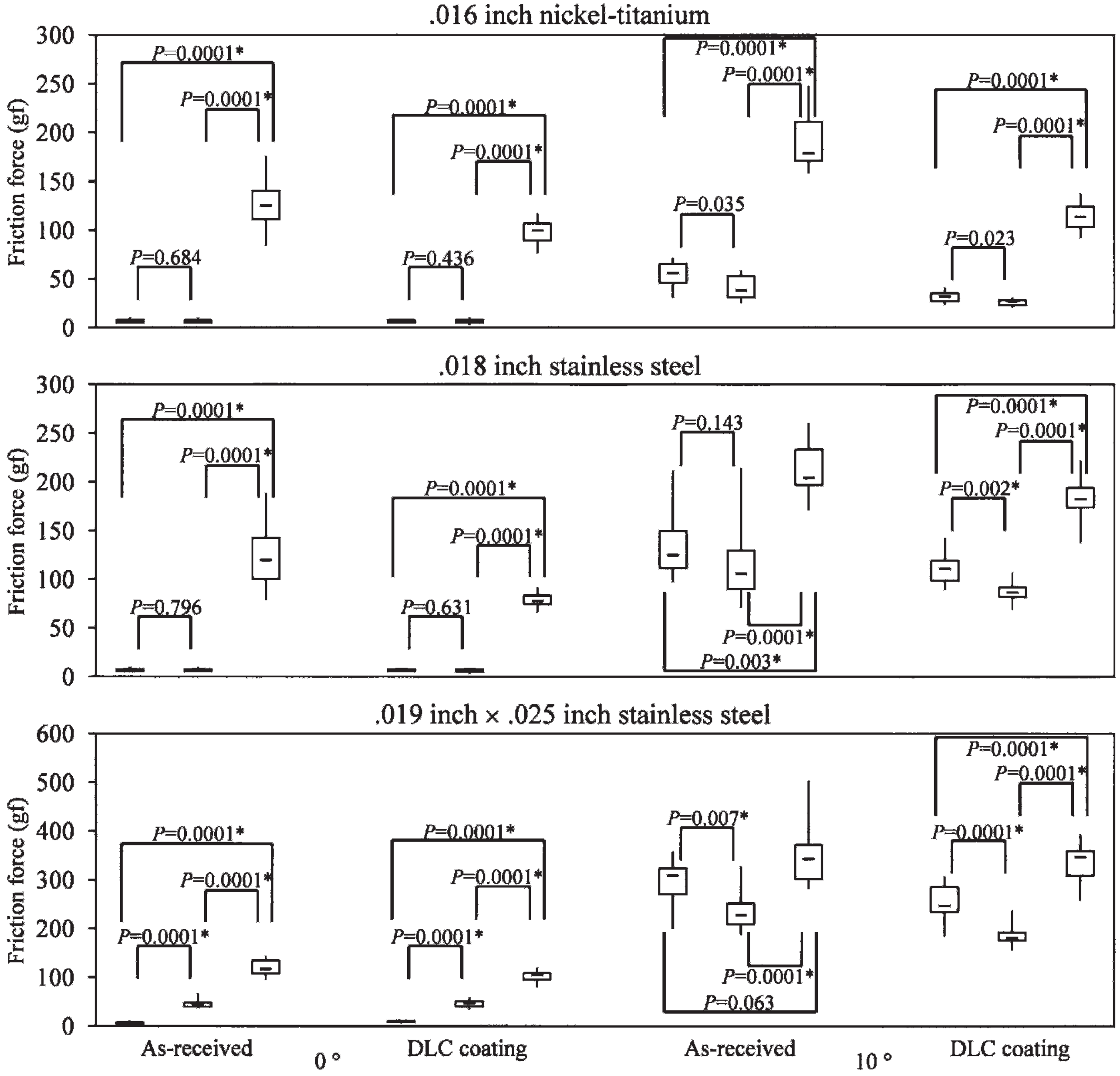

Figure 2. Static frictional forces for angulations of 0° and 10° for conventional and self-ligating brackets with as-received and DLC-coated wires. Comparisons are presented for each bracket in the three types of wires. Left side, Damon Q; middle, In-Ovation; and right side, Victory brackets. * $P < .0167$ by Mann-Whitney *U*-test; ns indicates nonsignificant.

Whitney *U*-test ($P < .05$). Since the data were normally distributed for the maximum depth of indentation, hardness, and elastic modulus, mean values for the as-received and DLC-coated wires were compared using Student's *t*-test ($P < .05$).

RESULTS

Table 1 and Figure 2 show static frictional forces for angulations of 0° and 10° for conventional and self-ligating brackets and for as-received and DLC-coated wires. When the angulation was increased to 10°, the frictional forces increased in all of the bracket-wire combinations, and the DLC-coated nickel-titanium and stainless steel wires showed significantly less frictional force than as-received wires, except for some wire/bracket combinations. Both self-ligating brackets showed significantly less frictional force than the conventional bracket at 0° angulation. When angulation was increased to 10°, both self-ligating brackets had significantly less frictional force than the conventional bracket for the as-received wires, except for the Damon Q bracket/as-received 0.019 inch × 0.025 inch stainless steel wire combination ($P = .063$).

Table 1. Static Frictional Forces for 0° and 10° Angulations of Conventional (Victory) and Self-Ligating Brackets (Damon Q, In-Ovation) to As-Received and DLC-Coated Wires[a]

			Friction Force (gf)				
			As-Received		DLC-Coating		
Angulation, Degree	Bracket	Wire	Mean	SD	Mean	SD	*P* Value
0	Damon Q	.016-in NiTi	6.63	2.15	6.12	1.32	.796
		.018-in SS	7.39	1.88	6.88	1.23	.631
		.019-in × .025-in SS	6.63	1.78	9.43	1.23	.003
	In-Ovation	.016-in NiTi	6.88	1.72	6.88	2.42	.971
		.018-in SS	7.14	1.61	6.37	1.8	.481
		.019-in × .025-in SS	46.65	8.92	46.65	7.11	.739
	Victory	.016-in NiTi	125.68	26.14	98.66	13.17	.011
		.018-in SS	123.13	33.59	79.03	7.88	.0001
		.019-in × .025-in SS	120.58	16.04	101.97	11.71	.015
10	Damon Q	.016-in NiTi	54.55	14.08	31.61	5.79	.001
		.018-in SS	139.19	40.7	110.89	15.91	.063
		.019-in × .025-in SS	298.01	45.82	254.42	37.19	.019
	In-Ovation	.016-in NiTi	41.3	11.82	26	3.56	.0001
		.018-in SS	117.52	43.64	88.21	11.41	.063
		.019-in × .025-in SS	235.04	41.76	187.88	22.1	.002
	Victory	.016-in NiTi	191.45	30.2	113.95	14.47	.0001
		.018-in SS	211.08	29.55	182.78	22.48	.043
		.019-in × .025-in SS	353.84	68.57	336	42.96	.739

[a] DLC indicates diamond-like carbon; NiTi, nickel-titanium; and SS, stainless steel.

Figure 3 shows representative SEM photomicrographs for cross-sectioned DLC-coated stainless steel and nickel-titanium wires. The figure clearly shows that the thin DLC layers on the wire surfaces have a thickness of approximately 0.5 μm.

Figure 4 shows 3D-SEM images of stainless steel and nickel-titanium wires. The as-received and DLC-coated wires had similar surface morphologies for both the stainless steel and nickel-titanium wires. On the other hand, the surface morphology of the nickel-titanium wires was rougher than that of the stainless steel wires. Quantitative analysis of the surface roughness shown in Table 2 confirmed that the nickel-titanium wires had greater surface roughness than the stainless steel wires, and the DLC-coating process did not affect the surface roughness ($P = .548$).

The mean values and standard deviations for the maximum depth of indentation h_{max}, hardness, and elastic modulus obtained in the nanoindentation tests on the wire surfaces (5 and 100 mN peak load) are listed in Table 3. The mechanical properties obtained with a 5 mN peak load are mainly for the DLC layers because the h_{max} of the 5 mN peak load was less than

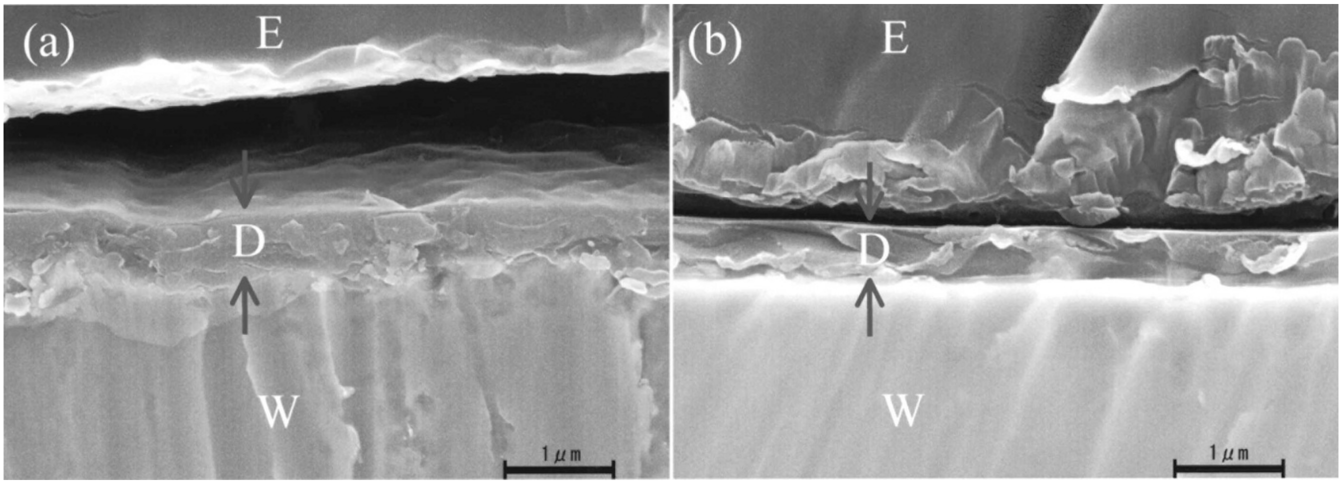

Figure 3. SEM photomicrographs of cross-sectioned (a) stainless steel and (b) nickel-titanium wires. DLC layers formed on the wires are evident. Original magnification 20,000×. E indicates epoxy resin; D, DLC layer; and W, wire.

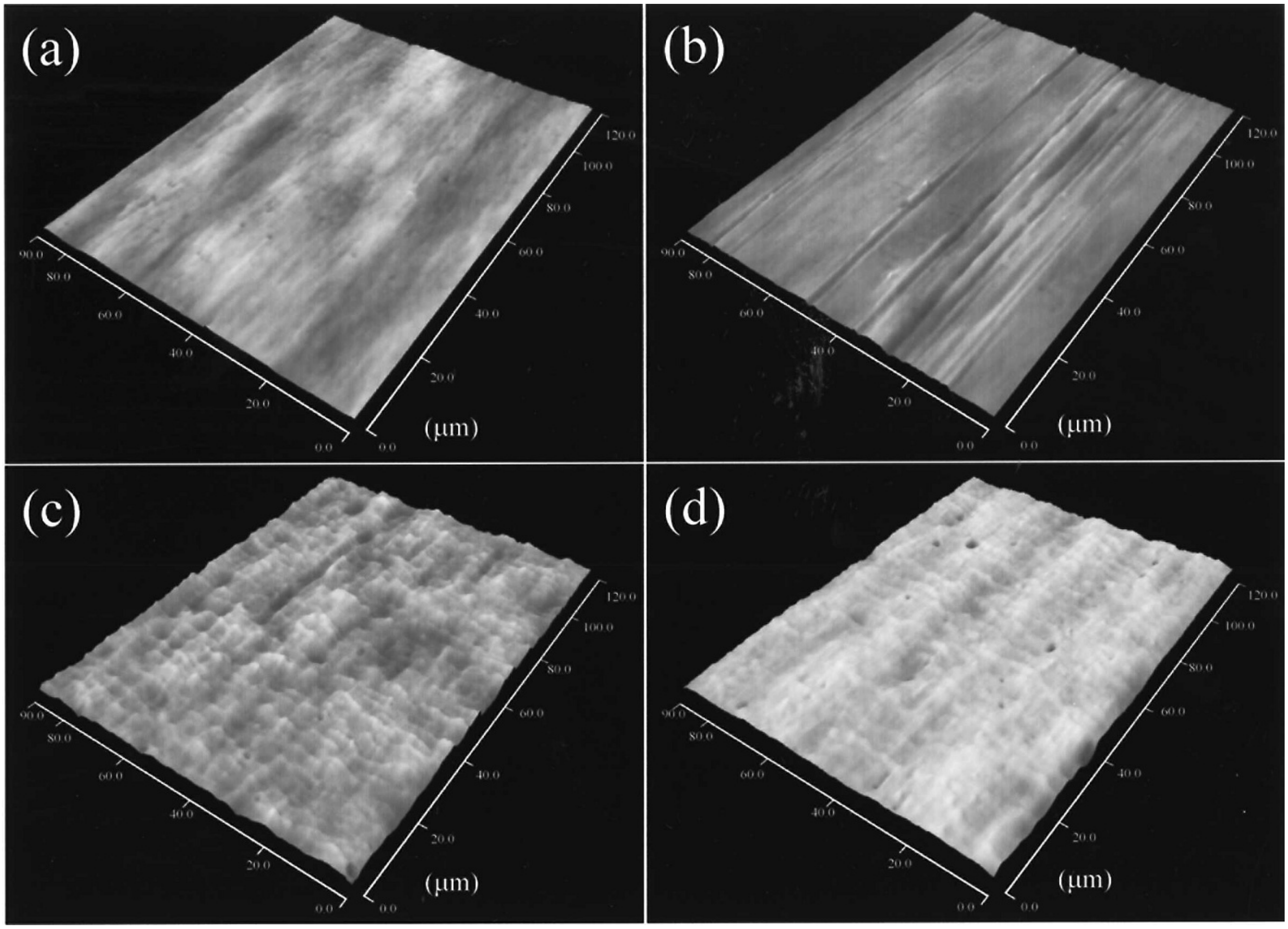

Figure 4. Images of stainless steel and nickel-titanium wires obtained with four-channel 3D-SEM. (a) As-received stainless steel wire. (b) DLC-coated stainless steel wire. (c) As-received nickel-titanium wire. (d) DLC-coated nickel-titanium wire. Original magnification 1000×.

300 nm for both stainless steel and nickel-titanium wires. The hardness for the DLC-coated stainless steel and nickel-titanium wires obtained with the 5 mN load was much higher than that of the as-received stainless steel ($P = .0001$) and nickel-titanium wires ($P = .0001$). On the other hand, the elastic modulus for the DLC-coated stainless steel wire obtained with a 5 mN load was less than that of the as-received stainless steel wire ($P = .0001$). The hardness and elastic modulus for nickel-titanium wires obtained with a 100 mN load were similar for DLC-coated and as-received wires.

Table 2. Average Surface Roughness (R_a Value) for Stainless Steel and Nickel-Titanium Wires

	Surface Roughness, μm				
	As-Received		DLC-Coating		
Wire	Mean	SD	Mean	SD	*P* Value
Stainless steel	0.035	0.01	0.032	0.004	.548
Nickel-titanium	0.094	0.009	0.088	0.013	.548

DISCUSSION

Frictional force between the bracket and the wire during orthodontic tooth movement is an important factor in clinical orthodontics, and if the frictional force can be decreased, the efficiency of tooth movement can be improved. The frictional forces increased when the angulation was increased for all of the bracket-wire combinations in this study, and this is consistent with previous studies.[3,17] In this study, although the stainless steel wire showed smoother and harder surface characteristics than the nickel-titanium wire, the stainless steel wires had greater frictional forces than the nickel-titanium wires. The stainless steel wires had wider cross-section dimensions and a higher value of the elastic modulus than the nickel-titanium wires, and this should have affected binding and notching.

DLC coatings are becoming increasingly important in industrial and biomedical applications.[19,20] Hardness is often the most noted property of DLC coatings. It can be challenging to determine the true mechanical properties of a material that exists only as a thin

Table 3. Summary of Mechanical Properties for As-Received and DLC-Coated Wires Obtained by the Nanoindentation Test

Wire	Peak Load, mN		As-received Mean	As-received SD	DLC-coating Mean	DLC-coating SD	*P* Value
Stainless steel	5	h_{max}, nm	157.37	6.17	146.75	5.03	.001
		Hardness, GPa	11.62	0.99	17.58	1.67	.0001
		Elastic modulus, GPa	277.39	16.33	194.44	9.09	.0001
	100	h_{max}, nm	958.88	56.59	891.42	24.17	.005
		Hardness, GPa	6.15	0.84	7.18	0.52	.004
		Elastic modulus, GPa	143.26	11.21	158.65	2.1	.0001
Nickel-titanium	5	h_{max}, nm	253.12	13.22	215.34	4.39	.0001
		Hardness, GPa	4.72	0.56	9.11	0.51	.0001
		Elastic modulus, GPa	82.87	6.27	74.87	2.84	.002
	100	h_{max}, nm	1273.95	46.91	1275.61	76.99	.954
		Hardness, GPa	3.73	0.32	3.85	0.54	.538
		Elastic modulus, GPa	63.56	3.44	60.28	4.97	.104

coating less than a few micrometers thick. The recent development of the nanoindentation technique, which offers nanometer-scale resolution, has allowed such measurements to be successfully performed.[21] Most DLC films are harder than most metallic materials, and DLC coating by the PBIID gives hardness values ranging from 6 GPa to 20 GPa depending on the deposition conditions.[19,20] In this study, the hardness values of the DLC layer on the stainless steel (17.6 GPa) and nickel-titanium wires (9.1 GPa) were determined by the nanoindentation test and were significantly higher than the hardness values of the surface layers on the as-received stainless steel (11.6 GPa) and nickel-titanium wires (4.7 GPa). This study clearly demonstrated that the DLC-coating process reduces frictional force. The harder surface of the DLC-coated wires not only reduces friction but also reduces the effects of binding and notching. In addition, the DLC layer on the stainless steel and nickel-titanium wires showed a lower elastic modulus than the surface layer on the as-received wires. DLC-coated wires with a lower elastic modulus might show superior flexibility, which is a desirable characteristic of an orthodontic wire. Further research is needed to evaluate the flexibility of the DLC-coated orthodontic wires.

The basic advantages of self-ligating brackets involve the elimination of certain utilities or materials such as elastomeric modules, along with the process or tools associated with their application.[4] In addition, it has been proposed that due to bracket-wire engagement, light forces and reduced friction can be attained with a desirable outcome on the rate of orthodontic tooth movement.[22] However, a recent study concluded that a self-ligating bracket was no better during initial alignment than a conventional bracket.[4] In the present study, both self-ligating brackets produced significantly less frictional forces in both angulations (0° or 10°) than the conventional bracket. The self-ligating brackets produced almost 0 g for 0° angulation, excluding the In-Ovation bracket/0.019 inch × 0.025 inch stainless steel wire combination. However, this condition (0° angulation) never occurs clinically. When angulation was increased to 10°, the In-Ovation bracket showed a lower rate of increase in the frictional force than the Damon Q bracket, even though the In-Ovation bracket has a wider mesiodistal width than the Damon Q bracket. The results of this study showed that a self-ligating bracket/DLC-coated wire combination is beneficial for orthodontic tooth movement.

CONCLUSIONS

- The surfaces of nickel-titanium and stainless steel orthodontic wires can be successfully modified by the PBIID method to create a DLC layer.
- The DLC-coating process reduces the frictional force for these wires in brackets.
- The DLC layer has a higher hardness value than the as-received wires.
- Self-ligating brackets produce less frictional force than the conventional stainless steel bracket.

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