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Coating of stainless steel archwires with silicon carbide & aluminum oxide nanoceramic particles: topographical & mechanical assessment.

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Abstract:

Introduction: *Orthodontists have been using metallic biomaterials extensively since its inception. Orthodontic devices have been made from a variety of metallic biomaterials, including titanium and its alloys, stainless steel, gold, and cobalt-chromium alloys, among others, since the 20th century. However, there are several disadvantages of these materials that the orthodontist may find problematic. Some of the most frequent drawbacks of employing metallic alloys in the production of orthodontic appliances are poor friction control, allergic reactions, and metal ionic release. The aim of the study was to investigate the effect of nanoceramic coating on the topographical and mechanical properties of stainless steel orthodontic wires.* ***Materials and methods:*** *0.019×0.025 inch gauged 30 stainless steel (St.St.) archwires were used as samples for study tests, which were divided into 3 groups as follows: SA: uncoated stainless steel wires; SB: stainless steel wires coated with silicon carbide; and SC: steel wires coated with aluminum oxide. Physical magnetron sputtering technique was used to nanocoat the wire samples with silicon carbide and aluminum oxide. Energy Dispersive X-ray Spectroscopy (EDX) was used to confirm the sample coating, Scanning Electron microscopic investigation (SEM) and optical microscopic investigation were used to examine the sample surface texture. Frictional force was evaluated by sliding wire samples through ceramic brackets using a universal testing machine. The wire-bending behaviour was evaluated through a three-point bending test using a universal testing machine.* ***Results:*** *EDX revealed that Samples were nanocoated with nanoceramic particles, fewer surface flaws and irregularities were noted on nanocoated samples. Significant decreases in the maximum force were recorded during the friction test for nanocoated samples compared with uncoated samples. No significant differences were recorded in the force during loading and unloading or the amount of permanent wire deformation after wire bending between coated and uncoated steel wire samples.* ***Conclusion:*** *Nanoceramic coating enhances surface texture and decreases the friction between orthodontic wires and brackets without significantly changing the wire bending behaviour.*

Key words: *Nanocoating, St.St archwires, Friction, 3point-bending test, Surface texture.*

Introduction

Orthodontists have been using metallic biomaterials extensively since its inception. Orthodontic devices have been made from a variety of metallic biomaterials, including titanium and its alloys, stainless steel, gold, and cobalt-chromium alloys, among others, since

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the 20th century. However, there are several disadvantages of these materials that the orthodontist may find problematic. Some of the most frequent drawbacks of employing metallic alloys in the production of orthodontic appliances are poor friction control, allergic reactions, and metal ionic release. [1] One approach to addressing these drawbacks and enhancing the mechanical, frictional,[2,3,4] surface roughness [2,3] bacterial adhesion [5],and corrosiveness [6] characteristics of archwires is coating.

The orthodontic archwires have been coated using various methods and materials. Coatings come in a variety of forms, depending on their intended use. Coating orthodontic archwires influences their surface characteristics, which in turn affect the properties of the archwire, such as surface roughness, bacterial adherence, and mechanical characteristics.[7] Recent developments in the field of physical vapor deposition techniques have made it possible to create a vast array of coatings with an amazing variety of characteristics.[8,9]

Physical vapor deposition technique has been widely employed in different industries. As it is offering an appropriate method for coating objects in a variety of complex designs with nearly unlimited types of coatings at a wide range of temperatures, that is allowing the coating of delicate objects as elastic ligatures and power chains beside metallic devices with uniform and thin film thickness.[8,9] These advantages allow a controllable coating procedure with various physical and biological properties that allow modification of the orthodontic appliances to meet the desired criteria.

Nanoceramics is the term for materials made of ultrafine ceramic particles, including silicon dioxide, silicon carbide, aluminum oxide, etc., with a diameter of less than 100 nm. The potential of nanoceramics to exhibit superior and distinct features above traditional bulk ceramic materials has drawn a lot of interest in them as potential materials.[10] Strength, toughness, controlled crystallinity, machinability, superplasticity, and other unique mechanical, surface, and processing properties are all presented by nanoceramics.[10] The effect of nanoceramic coating on behaviour of orthodontic wires seemed to be a point of worthy investigation, as few data had been published about this point. Accordingly, this study was conducted to highlight this aim. The null hypothesis was that, there were no topographical or mechanical differences between nanoceramic coated stainless steel archwires and uncoated stainless steel archwires.

Materials and methods

Ethical approval:

The Research Ethics Committee of the Faculty of Dentistry at Minia University approved this research proposal (Decision No. 892/2024).

Sample size calculation:

Before the study, the number of samples required in each group was determined after a power calculation according to data obtained from a previous study (**Elhelbawy et al., (2021)**)[11]. In that study, the mean friction value in uncoated stainless steel wires group was

1.79±0.61 newton(N), stainless steel wires coated with zinc oxide nanoparticles group was 0.82±0.26 N and stainless steel wires coated with Chitosan nanoparticles group 1±0.21 N. A sample size of 7 samples in each group was determined to provide 99% power for One-way ANOVA test at the level of 0.05 significance using G Power 3.1 9.2 software.

The sample:

0.019×0.025 inch gauged 30 stainless steel (St.St.) archwires (Ormco Corporation, California, USA) were used as samples for study tests. These wires were divided into three groups (ten wires in each group). Groups were coded as follows: SA: uncoated stainless steel wires, SB: stainless steel wires coated with silicon carbide, SC: stainless steel wires coated with aluminum oxide.

Nanocoating:

Nanocoating procedures were conducted in the faculty of nanotechnology, Cairo University, Egypt. The wires were unpacked and swapped with lint-free tissue to remove any dust or smudge remnants on the surfaces of the wires. Clean room gloves were used for manipulating wires. Wires were hanged above the target in the coating machine protoflex model 1600 magnetron sputtering system (Protoflex, USA) by using small pieces of wire that were shaped to hold the archwires and fixed in a perforated plate above the target material that was used for coating.

Physical Sputtering technique was used to nanocoat the wires by protoflex model 1600 magnetron sputtering system (Protoflex, USA). Silicon carbide (SiC) target (Protoflex, USA) with 99.5% purity was used for nanocoating the wires with SiC. Aluminum oxide (Al_2O_3) target (Protoflex, USA) with 99.99% purity was used for nanocoating the wires with Al_2O_3. The coating parameters were: Initial pressure: 10^{-5} barr, Deposition pressure: 10^{-3} barr, Sample rotation: 10 rounds per minute (RPM), Argon flow: 30 standard cubic centimeters (Sccm), Time: 90 minutes. After the coating procedure, the wires were carefully removed and packed separately in a plastic packet to prevent any further contamination.

Scanning Electron microscopic investigation (SEM)

Ten-mm wire segments were cut from archwires using a heavy-duty straight cutter (Miltex, Pakistan) and used as test samples. Samples were mounted in the scanning electron microscope for investigation (Figure 1). Scanning was conducted by Scanning Electron Microscope (SEM, Quanta FEG 250, FEI, Republic of Czech) in the National Research Center, Giza, Egypt. Each sample was investigated to determine any surface texture differences between coated and uncoated samples.

Energy Dispersive X-ray Spectroscopy (EDX)

Energy Dispersive X-ray Spectroscopy (EDX) was conducted to reveal the chemical composition differences between coated and uncoated samples. Elemental composition was inspected during Scanning Electron Microscopic investigation on the same samples used for the SEM test, as the SEM contained an integrated EDX unit.

Optical microscopic investigation

Thirty-mm wire segments were cut from archwires using a heavy-duty straight cutter (Miltex, Pakistan) and used as test samples. Samples were mounted in the optical microscope for investigation (Figure 2). Wires were investigated under an optical microscope, (Bx53M, Olympus, Japan). The surfaces of coated and uncoated wires were evaluated under the optical microscope to reveal any surface texture differences between the coated and uncoated wire samples.

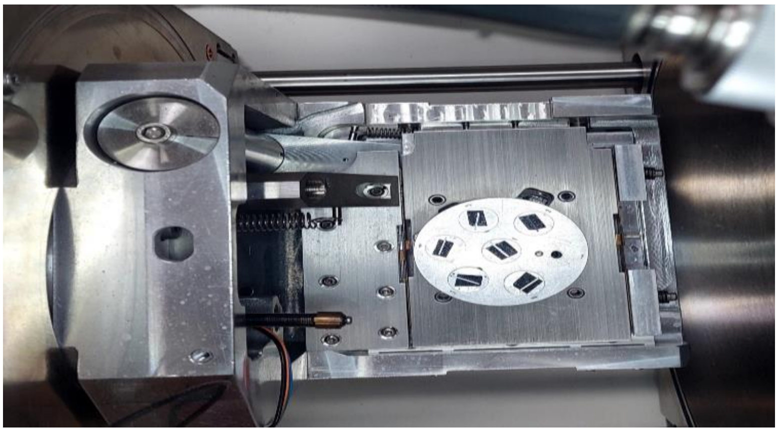

Figure 1: samples mounted in the examination chamber in the SEM and EDX device.

Figure 2: samples mounted under optical microscope.

Three-Point Bending Test

Previously cutted Thirty-mm of straight posterior segments were used as test samples. The test was conducted using a 50 KN (kilonewton) loading cell universal testing machine, (Instron, Instron corporation, USA). Custom-made two lower poles with a ten mm span between them were fixed on the lower fixture of the machine. The upper pole was fixed to the upper fixture of the testing machine and was positioned in the middle of the testing wire span, just passively immediately above the wire segment. The upper pole was moved vertically at 7.5 mm/minute with a maximum wire deflection of 3.1mm (Figure 3).[12] Forces were measured during the loading and unloading, and wire permanent deformation after unloading was measured.

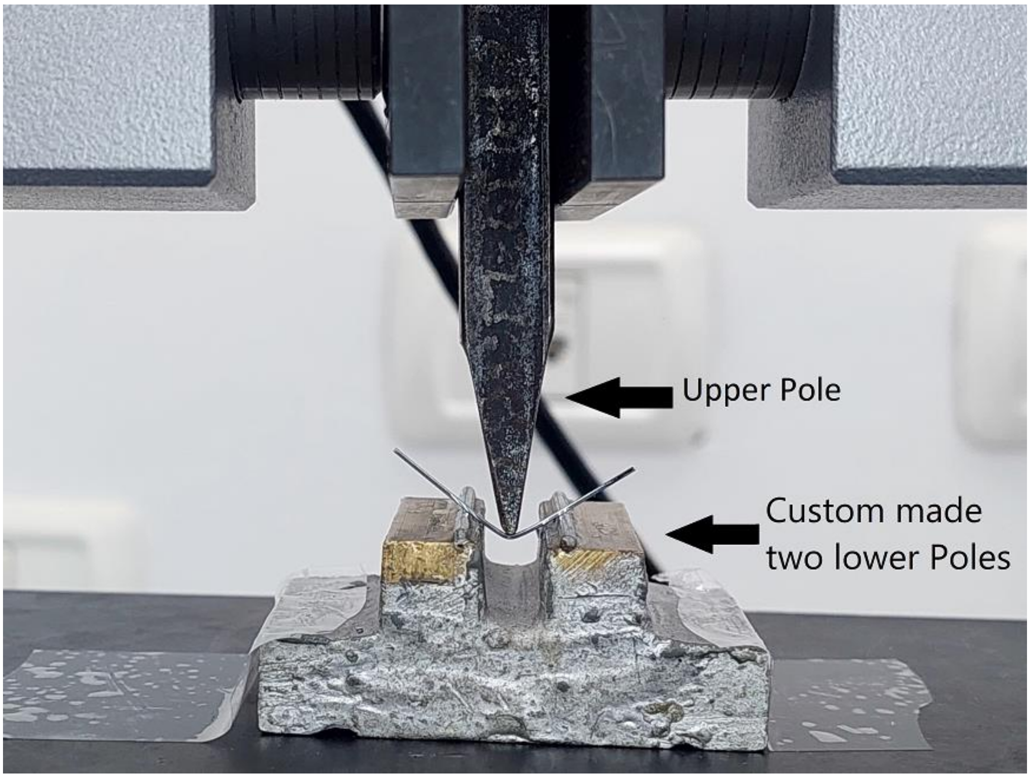

Figure 3: wire samples during three-point bending test.

Friction test

A model of two rectangular steel rods was used on one of the two rod. Three brackets were bonded and used as a holder for the archwire segment during testing. On the other rod, a ceramic bracket was bonded and used to test the friction between the wire segment and bracket. Brackets were initially bonded by using orthodontic bracket adhesive (DMP, Greece). bracket was bonded on the same straight line by using a straight wire segment before curing the composite for 40 seconds. Cyanoacrylate adhesive (Amir Alpha, Egypt) was used to enforce the adhesion between the bracket and steel rod.[13,14]

Forty-mm straight posterior segments were cut from archwires and used as samples. The test was conducted using a 5KN (kilonewton) loading cell universal testing machine, (Instron, Instron corporation, USA). Steel rods were fixed to the machine fixtures with caution to make the wire segment passively engaged in the bracket on both rods to be parallel to each other on the same straight line (Figure 4). The steel rods were positioned 1 mm apart, and then the wire segment was engaged in the bracket on both rods by using elastomeric ligatures (American Orthodontics, USA). Instron machine pull the upper steel rods with 5 mm/minute speed as the wire slides across the ceramic bracket slot on the lower rod. The wire segment was allowed to slide 5 mm, and force was measured during the procedure. [13.14]

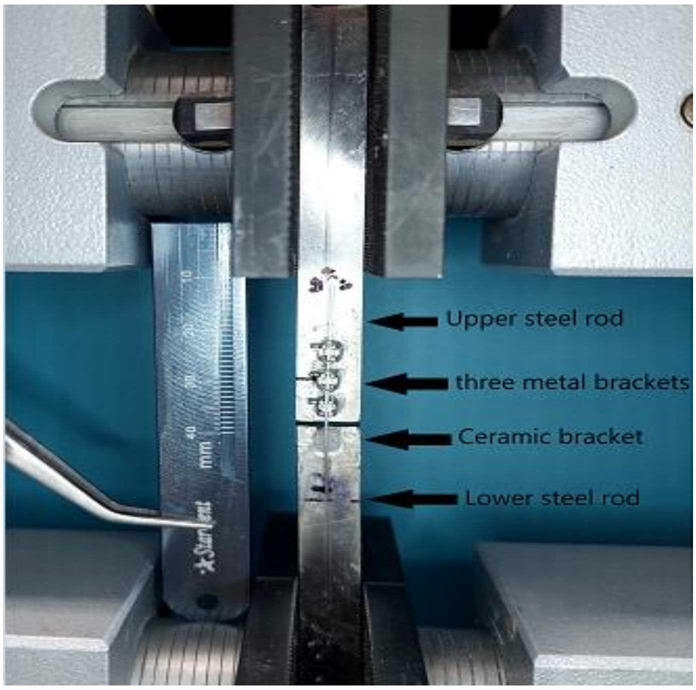

Figure 4: wire sample during friction test.

Statistical analysis:

Statistical analysis was performed with SPSS 20 (Statistical Package for Social Science, IBM, USA), Graph Pad Prism (Graph Pad Technologies, USA), and Microsoft Excel 2016. All data was presented as minimum, maximum, mean, and standard deviation. All data was explored using Shapiro wilk and Kolmogorov-Smirnov test (Table 1). It was revealed that the significance level (P-value) was shown to be insignificant as P-value > 0.05, which indicated that data originated from normal distribution (parametric data) in the groups. One Way ANOVA test was used to compare the effect of different subgroups followed by Tukey`s Post Hoc test for multiple comparisons.

Table (1): Normality Data Exploration of the groups using Kolmogorov–Smirnov test and Shapiro–Wilk test:

	Group	Load deflection.		Friction	
		P-value	**Indication**	**P-value**	**Indication**
St.St wires	SA	0.72 (ns)	Normal data	0.53 (ns)	Normal data
	SB	0.36 (ns)	Normal data	0.44 (ns)	Normal data
	SC	0.81 (ns)	Normal data	0.29 (ns)	Normal data

-Shapiro wilk and Kolmogorov-Smirnov tests.
Ns; Insignificant Difference as P>0.05.

Results

Energy Dispersive X-ray Spectroscopy (EDX)

EDX analysis (Figure 5) for uncoated steel wire samples revealed that there was no Aluminum which is a component of aluminum oxide or silica, which is a component of silicon carbide. EDX analysis of steel wire samples coated with silicon carbide revealed the presence of the silica element (Si) which is a component of silicon carbide. EDX analysis for steel wire samples coated with aluminum oxide revealed the presence of aluminum element (Al) which is a component of aluminum oxide.

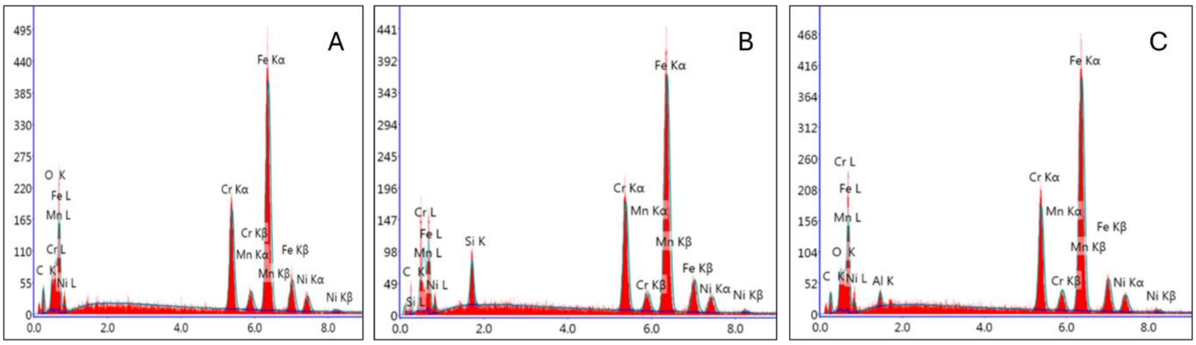

Figure 5: EDX analysis for uncoated steel wire samples. A:uncoated sample. B:sample coated with SiC. C: sample coated with Al_2O_3

Scanning Electron microscopic investigation (SEM) & Optical microscopic investigation

Scanning Electron Microscope (SEM) (Figure 6) and Optical Microscopic Scanning (Figure 7) revealed that the flaws and irregularities on the coated samples, especially those coated with silicon carbide are less prominent than those on uncoated samples. Also, a bluish surface shade of silicon carbide Coated samples that can be noted by optical microscopic scanning.

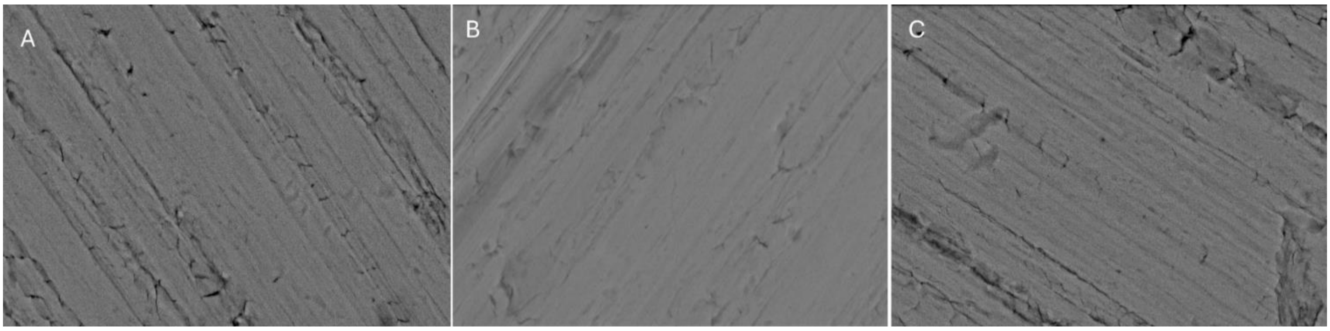

Figure 6: scanning electron microscope of stainless steel wire samples. A:uncoated sample. B:sample coated with SiC. C: sample coated with Al_2O_3

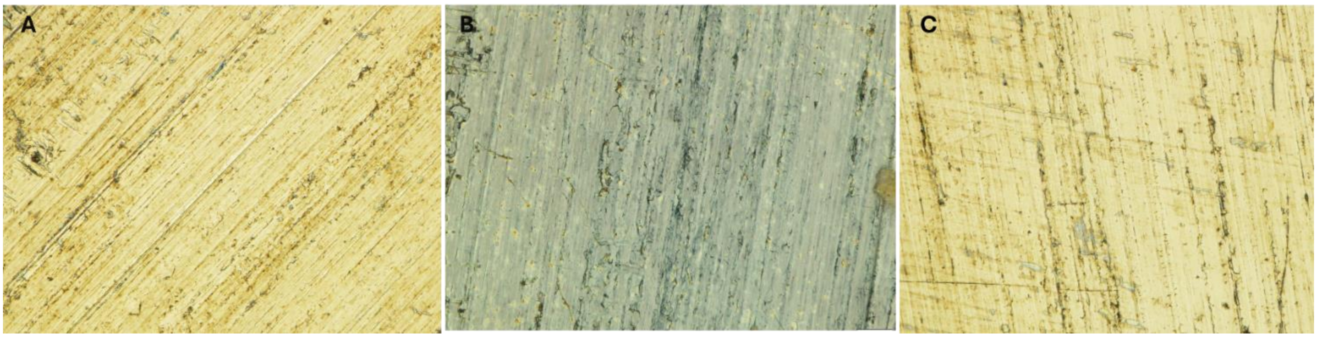

Figure 7: Optical Microscopic Scanning of stainless steel wire samples. A:uncoated sample. B:sample coated with SiC. C: sample coated with Al_2O_3

Friction test

The friction test revealed a significant decrease in the maximum force recorded during the test for nanocoated samples compared with uncoated wire samples, which record a higher frictional force. No significant differences were recorded in frictional force between silicon carbide and aluminum oxide coated wire samples (Table 2).

Three-point bending test

The three-point bending test revealed that there were no significant differences recorded in the force during loading and unloading or the amount of permanent wire deformation after wire bending between coated and uncoated wire samples (Table 3).

Table (2): Descriptive results of steel wire samples maximum friction force (newton (N)), comparison between different subgroups:

Maximum load (N)	N	Minimum	Maximum	Mean	Standard Deviation	P value (One Way NOVA test)
Uncoated samples	10	0.4747	0.6970	0.5652 a	0.0893	< 0.0001*
Silicon carbide coated samples	10	0.3161	0.4541	0.3722 b	0.0455	
Aluminum Oxide coated samples	10	0.2631	0.4689	0.3819 b	0.0699	

-One Way ANOVA test and Tukey`s Post Hoc tests.
**Highly significant difference as P<0.001.*
Means with different superscript letters were significantly different as P<0.05.
Means with the same superscript letters were insignificantly different as P>0.05.

Table (3): Mean and standard deviation of Steel wire samples loading and unloading force(Newton(N)) and permanent deformation(Millimeter(mm)) during wire bending:

Parameter	N	Uncoated samples		Silicon carbide coated samples		Aluminum oxide coated samples		P value
		M	SD	M	SD	M	SD	
0.5mm wire deflection (load (N))	10	19.951 a	0.308	20.021 a	0.2925	20.028 a	0.2536	0.85 ns
1mm wire deflection (load (N))	10	29.101 a	0.436	29.068 a	0.3023	29.375 a	0.2878	0.22 ns
1.5mm wire deflection (load (N))	10	29.743 a	0.563	29.720 a	0.3682	29.929 a	0.3276	0.38 ns
2mm wire deflection (load (N))	10	28.725 a	0.611	28.745 a	0.4096	28.872 a	0.3169	0.81 ns
2.5mm wire deflection (load (N))	10	27.056 a	0.520	27.120 a	0.4102	27.243 a	0.3218	0.70 ns
2.5mm wire deflection (unload (N))	10	10.230 a	0.358	10.110 a	0.2616	10.091 a	0.4540	0.74 ns
3mm wire deflection (load (N))	10	25.047 a	0.466	25.075 a	0.3143	25.237 a	0.2757	0.57 ns
3mm wire deflection (unload (N))	10	20.234 a	0.228	20.072 a	0.1981	20.201 a	0.3089	0.45 ns
permanent deformation (mm)	10	2.058 a	0.021	2.062 a	0.0125	2.062 a	0.0221	0.85 ns

-One Way ANOVA test
**Highly significant difference as P<0.001.*
Means with the same superscript letters were insignificantly different as P>0.05.

Discussion

Using surface coating has been widely investigated in the orthodontic field for many purposes. Enhancing the topographical surface properties, decreasing the bacterial accumulation, enhancing the esthetical appearance of orthodontic appliances, and decreasing friction, especially during sliding mechanics are the major aims of these studies.[1,2,3,4,5] In this study, the major aim was to investigate the changes in surface topographic properties, behaviour of wires during bending, and friction of orthodontic wires when they were coated with nanomaterials.

Many materials were used for coating orthodontic wires. In this study, silicon carbide and aluminum oxide were used as nanocoating materials. Biocompatibility and cytotoxicity are great debate issues when nanomaterials are used for medical interventions. Silicon carbide and aluminum oxide are two of the most biocompatible materials with no cytotoxic effects on human body tissues.[15,16,17,18]

Silicon carbide has been used for many medical purposes, such as medical orthopedic implants and protective coats for medical implants[15]. **Pourchez et al.[16]** concluded that no cytotoxic effect of the investigated silicon carbide nanoparticles was recorded. **Chen et al.[17]** concluded that after testing two different structured silicon carbide nanomaterials, no cytotoxic effect was recorded. **Radziun et al.[18]** tested the cytotoxicity of aluminum oxide nanoparticles on mammalian cells, and no cytotoxic effects were noted.

In this study, the magnetron sputtering physical vapor deposition technique was used to coat orthodontic wires with aluminum oxide and silicon carbide nanomaterials. This method has the advantage of having a uniform coating layer all over the wire surface as the wire sample rotates during coating.[9]

Arici et al.[19] coated orthodontic wires with aluminum oxide and reported that a uniform surface coating on the wire sample was formed by using the magnetron sputtering physical vapor deposition technique, which aligns with the results of this study. Coating was performed at room temperature in a vacuum chamber filled with inert gas to prevent changing the physical properties of the coated wires due to heat or chemical reactions[8].

In this study, the presence of aluminum oxide and silicon carbide nanocoating was evaluated by scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDX) which were used to identify the chemical composition of the samples. Scanning electron microscope and optical microscope used to evaluate the surface topographic features of coated and uncoated wire samples revealed the smoother and less surface roughness of coated samples with both aluminum oxide and silicon carbide nanoparticles compared to uncoated samples.

The findings of this study coincided with **Arici et al.[19]** findings, which noted smoother and less roughness of samples coated with aluminum oxide nanoparticles. On the same side, **Lin et al.[20]** investigated Al_2O_3 atomic layer deposited films on NiTi shape memory alloys for biomedical applications and revealed the formation of a smooth coating layer of aluminum oxide.

On the other hand, **Liu et al**.[21] investigated the NiTi alloy coated with aluminum oxide by using the micro-arc oxidation technique involving an electrolyte-containing sodium aluminate solution. They investigated the surface roughness, frictional behaviour and corrosion resistance of the samples and concluded that aluminum oxide is formed on the sample surface with a significant increase in surface roughness, an improvement in frictional behavior and a decrease in corrosion resistance.

During tooth sliding, 40-50% of the applied force was dissipated to overcome the friction between the orthodontic wire and brackets.[22,23] Light force is optimal to prevent any adverse effects such as periodontal damage, root resorption, unwanted tooth movement, and loss of anchorage. Many surface treatment methods were conducted to decrease the friction during tooth movement. Coating orthodontic wire with nanoparticles is one of the more advanced methods.[24]

In this study, **Mantel assembly**[13,14] was used to measure friction with the modification of using a ceramic bracket instead of the conventional metal bracket. Great esthetic demands have been raised, especially in adult patients. According to patient desire, ceramic brackets are used to provide a more esthetic appearance. Ceramic brackets produce more friction than conventional metal brackets. [25] Elastic ligature is used for wire ligation as it provides easier, faster, and more standard pressure on the investigated wire than steel ligature, which produces varied force depending on the tightening degree produced by the operator. 0.019×0.025-inch gauged wires were used as it is recommended working wires for many biomechanics techniques as space closure during sliding mechanics or closing loops. [26]

This study revealed that coating orthodontic wires with aluminum oxide and silicon carbide nanoparticles decreased the friction between the orthodontic wire and brackets significantly. The result of this study is well aligned with the results of **Arici et al.**[19] and **Liu et al.**[21] as they concluded that friction is decreased in samples coated with aluminum oxide nanoparticles compared to uncoated samples.

On the same side**, Karandish et al.**[27] concluded that stainless steel wire samples coated with zinc nanoparticles exhibited less friction during the friction test and more loading force during the three-point bending test than uncoated samples. **Elhelbawy et al.**[11] noticed a decrease in the friction force in the stainless steel wire samples coated with zinc oxide and Chitosan nanoparticles compared to uncoated samples.

On the other hand, **Arici et al.**[19] reported insignificant differences in the friction force between nanocoated samples with chromium nitride (CrN) and uncoated samples. **Golshah et al.**[28] concluded that there was an insignificant decrease in the friction of TMA wire samples coated with zirconium oxide nanoparticles compared to uncoated samples.

In this study, the loading deflection rate is measured according to the three-point bending test, which is a precise, reliable, and standard method to understand the behaviour of the wire during bending[29]. This is critical in sliding mechanics because the resistance during the movement of the active unit is not only dependent on the friction between the orthodontic wire and the bracket; wire bending also plays a major role in movement resistance.

During space closure by sliding mechanics tooth start movement by small tipping movement until bracket slot corner is bending the wire the movement will stop until tooth is uprightening so sliding movement actually is cycle of tipping and uprightening movement called a binding-and-release phenomenon[30]. In frictionless mechanics, the force delivered by closing loops greatly depends on the load deflection rate of the orthodontic wire.[31]

In this study, no significant differences were recorded in loading and unloading forces. This can be attributed to the large dimension of the wire compared to the thin nanocoating layer. Force during loading can be recorded in .5mm, 1mm, 1.5mm, 2mm, 2.5mm, and 3mm deflection, while force during unloading can be recorded only in 2.5 mm and 3 mm deflection as the wire was permanently deformed, so no force was applied by the wire above the point of permanent deformation.

These findings point to the ability of the wire to preserve its original load deflection rate after the nanocoating process. that can be attributed to the coating process that is performed at room temperature in a vacuum chamber filled with inert gas to prevent any changes in the physical properties and chemical composition of the coated wire. Also, the thin coating layer does not affect the bending behaviour of the wire while enhancing the surface topographic features and decreasing friction during orthodontic treatment. According to the results of this study, there were differences between the nanocoated wire samples and the uncoated wire samples, so the null hypothesis was rejected.

Conclusions:

Coating orthodontic stainless steel archwires with silicon carbide and aluminum oxide nanoceramic materials has enhanced surface texture by decreasing surface flaws and irregularities and has decreased the friction between orthodontic wires and brackets without significant changes in the wire bending behaviour. No significant differences were recorded in friction or bending behaviour between silicon carbide and aluminum oxide nanocoated wire samples.

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