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Research Article

Evaluation of Material Characteristics of Nano Particle Coated and Uncoated Stainless Steel, Nickel-Titanium and Beta-Titanium Orthodontic Archwire - an *In-vitro* Study - ②

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ABSTRACT

Introduction & Objectives: The characteristics desirable in an orthodontic archwire are a large springback, low stiffness, good formability, high stored energy, biocompatibility, environmental stability, resilience and low cost.

The Aim is to evaluate mechanical properties of Nanoparticles coated stainless steel archwire and to compare its mechanical property with that of uncoated stainless steel, NiTi and Beta Titanium orthodontic archwire for the parameters: Tensile strength, Elongation measurements, Bending tests, Hardness tests, Microscopic examination & Friction test.

Methodology: A self-lubricating metal coating containing nanoparticles is demonstrated. A universal testing machine/ Instron is used for testing tensile Strength, elongation measurements, bending and frictional resistances. A Reichert Austria make micro hardness tester and optical microscope is used for hardness tests & evaluation of microstructure.

Results: Descriptive statistical analysis was carried out to explore distribution of various parameters across coated and uncoated groups which will be discussed through tables and graphs.

Conclusion:

- Tensile strength increased in nanoparticles coated archwires and coated SS showed the highest tensile strength.
- Coated archwires showed decrease in Elongation/Strain test.
- Bending test showed higher load deflection rates in coated wires and coated SS showed the highest load deflection rate.
- Coating significantly increases the Micro-hardness of the archwires.
- Frictional forces are reduced for coated archwires.
- SEM Study shows less surface irregularities on coated orthodontic archwires.

INTRODUCTION

Over the past 100 years, improvements in both mechanotherapy and treatment philosophy have led to major advancements in orthodontic patient care. Changes in the field of mechanotherapy have largely been made possible with the emergence of newer orthodontic materials. Archwire materials form a large part of this change, and selecting the appropriate archwire requires a thorough knowledge of archwire biomechanical and clinical applications. This knowledge requires proper characterization of archwire alloys to predict their outcome when used clinically [1]. Also, a number of wire alloys with a wide spectrum of mechanical properties have been introduced, adding versatility to orthodontic treatment. Frictional resistance generated at the bracket archwire interface is one of the important properties of the archwires that affect its clinical performance. Also as an ideal material has not been found, arch wires should be selected within the context of their intended use during treatment [2].

The mechanical properties of other wires from different alloys have been evaluated in several studies [3-5]. Hence, Knowledge of mechanical properties of several alloys may lead to the optimization of tooth movement [6]. Garner LD [7] reported that Stainless Steel (SS) provided significantly less frictional resistance than nitinol and TMA. They suggested that, when high stiffness was required during sliding mechanics, SS should be used over nitinol or TMA. Although various arch wire alloys are available for retraction of teeth, SS arch wires have always been the mainstay for this phase of treatment.

Nanoparticle coated archwires are a recent introduction in the field of orthodontic alloy archwire materials, and proper literature regarding the frictional properties of these are lacking. Firas Elayyan, et al. [8] reported that mechanical properties of ultraesthetic coated archwires produced lower loading and unloading forces than uncoated wires of same nominal dimensions.

The process of surface modification or coating is often a recommended practice to improve the biocompatibility and the need for lubrication arises when the tribological applications in mechanical

systems become severe and challenging to control friction and corrosion resistance of the archwires used.

Various traditional and well established surface engineering techniques such as painting, electroplating, galvanizing, thermal and plasma spraying and nitriding, carburizing, boriding are available. Also, other technologically advanced recent techniques are Physical Vapour Deposition (PVD), Chemical Vapour Deposition (CVD), Ion Implantation, Ion Beam Assisted Deposition (IBAD), ion beam mixing, laser treatment.

Nowadays, a multitude of options are available to select and specify a treatment or a combination of treatment to engineer the surfaces of various materials.

There are wide range of biomedical applications of nickel-titanium due to its Shape Memory Effect (SMA) such as in bone fixation appliances [9], in field of orthodontics and dentistry like in wires, distractors, palatal arches and endodontic files etc. other applications like in orthopedic field, vascular field, neurosurgical field and in surgical field.

Similarly, stainless steel is one of the most commonly used materials in the manufacture of medical devices, and in particular stainless steel 304 and various metal alloys like Zr, Nb and Ti addition on injection molded 316L stainless steel for bio-applications like surgical purposes, specific grades of Stainless steel - 316 and 316L are predominantly used [10]. Stainless steel is commonly used in orthodontic archwires, wire coils, various speciality guide wires, currettes, screws / prostheses/ plates.

Lubricant is substance that reduces friction and wear at the interface of two materials. The lubricant at interface reduces the adhesive friction by lower the shear strength of interface. Various types of lubricants available are: Gaseous lubricants, Liquid lubricants, Semi-solid lubricants, Solid lubricants.

Thus, this current study demonstrates a self-lubricating metal coating containing nanoparticles i.e. Nanoceramics (commercially

available nanoparticle coating precursor solution) on uncoated orthodontic archwires which is economical and well established technique.

M Redlich, et al. [11] investigated that such coatings significantly reduce friction during tooth movement, and may decrease the adverse complications and might be biocompatible. Hence, our study is also aimed to evaluate the material characteristics of such nanoparticle coated and uncoated stainless steel, nickel-titanium and Beta-titanium for the parameters like tensile strength, elongation test/ Strain, bending test, hardness test, frictional resistance and surface topography.

MATERIALS AND METHODS

The experimental material required for the study was provided by the Department of Orthodontics at M. A. Rangoonwala Dental College, Pune, Maharashtra, India. The study comprised of six types of wires (Table I). The materials were classified into six major groups. Each group consisted of seven samples each of which was tested for tensile strength, elongation test, bending test, hardness measurements, friction test and surface roughness. Hence a total of 210 (uncoated and coated) wire samples were tested. Also all the groups used single wire sample for Scanning Electron Microscopy (SEM).

One of the most important and challenging part of our study was coating of uncoated orthodontic wires with nanoceramics (Figure 1) with a commercially available nanoparticle coating precursor solution (Progressive Electrochemicals Pvt. Ltd., India)

Commercially available uncoated straight length orthodontic wires of SS, TMA and NiTi (Figure 2) with a rectangular cross-section (0.019 X 0.025") shape, were coated with a uniform and smooth nanoparticle film using 100ml nanoceramics.

Commercially available uncoated straight length orthodontic wires of SS, TMA and NiTi were first cleaned under running water to discard any dust particles which will interfere with our coating procedure. The orthodontic wires were inserted into the nanoparticle bathtub for 30 min (Figure 3). Later the wires were removed from the tub and kept on a hanger where they were further painted with nanoparticle solution (Figure 4). The wires were then air dried with the help of drier for 2 minutes (Figure 5). The wires with the hanger were then placed in the hot air oven at 160°C for 3 minutes. (Figures 6,7). In this way the commercially available orthodontic wires were coated with nanoparticles.

The coated and uncoated wires were tested, by Instron universal testing machine to evaluate the tensile strength, elongation/strain test, bending test and lastly for the friction test. The coated and uncoated wires were evaluated for microhardness test using Vickers

Table 1: Composition of the study groups.	
Group	Wire used in the group
Group 1	Commercially available uncoated 0.019 X 0.025" stainless steel wire
Group 2	Nanoparticle coated 0.019 X 0.025" stainless steel wire
Group 3	Commercially available uncoated 0.019 X 0.025" NiTi wire
Group 4	Nanoparticle coated 0.019 X 0.025" NiTi wire
Group 5	Commercially available uncoated 0.019 X 0.025" TMA wire
Group 6	Nanoparticle coated 0.019 X 0.025" TMA wire
Group 1 - Group 6 were evaluated for 7 times for 5 different parameters.	

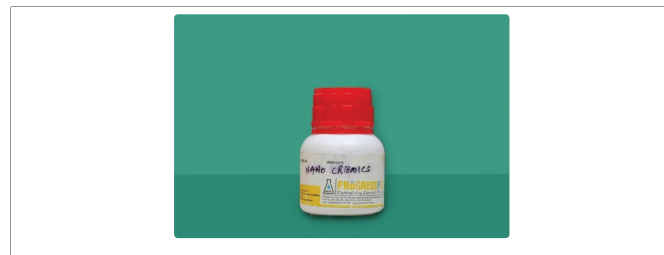


Figure 1: Nanoceramics - Commercially available Nanoparticle Coating Precursor Solution.

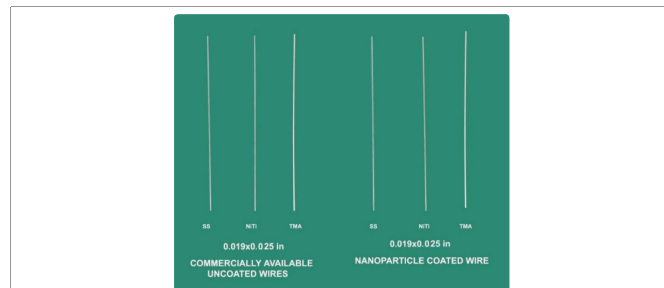


Figure 2: Orthodontic wires - SS, Niti and TMA.



Figure 3: Nanoparticle bathtub.

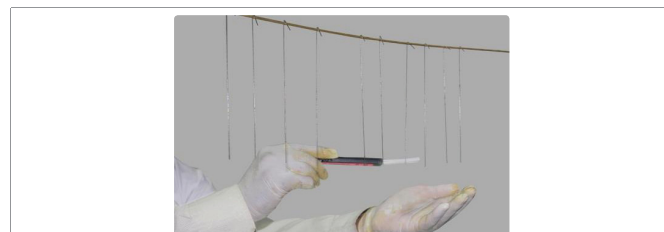


Figure 4: Nano particle painting.



Figure 5: Air drier.

microhardness testing machine to test the resistance to indentation on the surface. Scanning Electron Microscopy (SEM) was used to assess the surface topography of the archwires. The scanning electron

microscope used for our study was based on the technology of Environmental Scanning Electron Microscopy (ESEM).

STATISTICAL ANALYSIS

The collected data was statistically analysed using statistical package for social sciences (SPSS Version 11.5, Inc. Chicago, USA) for MS Windows.

The statistical significance was carried out using independent sample 't' test after confirming the underlying normality assumption. One-Way Analysis Of Variance (ANOVA) with Post-Hoc Tukey's correction for multiple group comparisons is used to test the inter-group comparisons of the wires studied.

The *p* - values less than 0.05 are considered to be statistically significant [S: Significant, NS: Non significant]. All the hypotheses were formulated using two tailed alternatives against each null hypothesis.

RESULTS

Upon Tensile strength evaluation, The results reveal that the mean tensile strength values of each wire assembly as follows (Tables 2,3 & Graph 1) Group 1 = 1875.5 ± 66.5 Mpa, Group 2 = 1898.7 ± 49.1 Mpa , Group 3 = 1005.0 ± 72.3 Mpa, Group 4 = 1229.5 ± 30.3 Mpa, Group 5 = 1080.1 ± 60.7 Mpa, Group 6 = 1052.1 ± 37.5 Mpa.

Secondly, Elongation/ Strain results gave the mean values of each wire assembly as follows (Tables 4,5 & Graph 2) Group 1 = 7.01 ± 0.85, Group 2 = 6.22 ± 0.48, Group 3 = 24.89 ± 2.39, Group 4 = 23.95 ± 1.48, Group 5 = 9.00 ± 0.79, Group 6 = 7.43 ± 0.56.

Next, a three-point bend test was performed to evaluate the load deflection properties. The mean values of each wire assembly were (Tables 6,7 & Graph 3) group 1 = 16.61 ± 0.76, group 2 = 19.48 ± 0.95, Group 3 = 4.59 ± 0.39, Group 4 = 6.28 ± 0.28, Group 5 = 10.33 ± 0.15, Group 6 = 11.89 ± 0.50.

Micro hardness tests were performed to test the resistance to



Figure 6: Wire with a hanger in hot air oven.

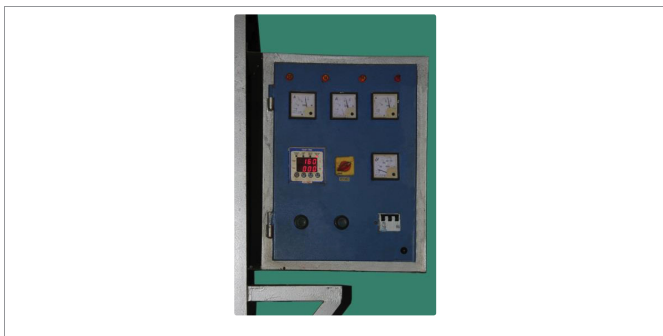


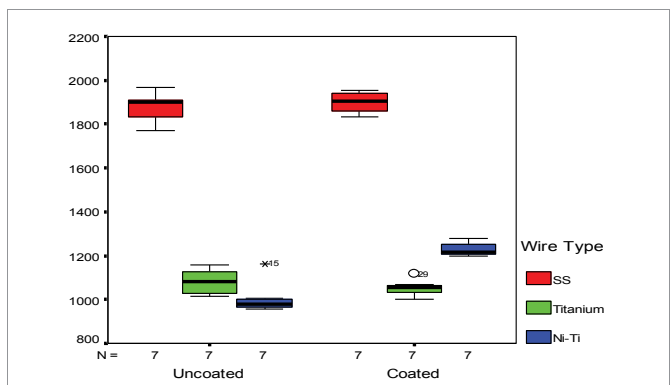
Figure 7: Hot air Oven at 160°C.

Table 2: Intra - group comparison of Tensile strength (MPa).

Wire type	Uncoated wire (n = 7)	Coated wire (n = 7)	<i>P</i> - value (Uncoated v Coated)
SS	1875.5 ± 66.5	1898.7 ± 49.1	0.472 (NS)
Ni-Ti	1005.0 ± 72.3	1229.5 ± 30.3	0.001 (S)
Titanium	1080.1 ± 60.7	1052.1 ± 37.5	0.319 (NS)

Table 3: Inter - group comparison of Tensile strength (MPa).

Wire type	Uncoated wire (n = 7)	Coated wire (n = 7)
SS	1875.5 ± 66.5	1898.7 ± 49.1
Ni-Ti	1005.0 ± 72.3	1229.5 ± 30.3
Titanium	1080.1 ± 60.7	1052.1 ± 37.5
Inter-group comparisons (<i>P</i> -values)		
Ss wire v Titanium wire	0.001 (S)	0.001 (S)
Ss wire v Ni-Ti wire	0.001 (S)	0.001 (S)
Titanium wire v Ni-Ti wire	0.117 (NS)	0.001 (S)



Graph 1: The Inter - group distribution of Tensile strength (Box - Plot).

Table 4: Intra - group comparison of Elongation (%).

Wire type	Uncoated wire (n = 7)	Coated wire (n = 7)	<i>P</i> - value (Uncoated v Coated)
SS	7.01 ± 0.85	6.22 ± 0.48	0.052 (NS)
Ni-Ti	24.89 ± 2.39	23.95 ± 1.48	0.391 (NS)
Titanium	9.00 ± 0.79	7.43 ± 0.56	0.001 (S)

Table 5: Inter - group comparison of Elongation (%).

Wire type	Uncoated wire (n = 7)	Coated wire (n = 7)
SS	7.01 ± 0.85	6.22 ± 0.48
Ni-Ti	24.89 ± 2.39	23.95 ± 1.48
Titanium	9.00 ± 0.79	7.43 ± 0.56
Inter-group comparisons (<i>P</i> -values)		
Ss wire v Titanium wire	0.063 (NS)	0.068 (NS)
Ss wire v Ni-Ti wire	0.001 (S)	0.001 (S)
Titanium wire v Ni-Ti wire	0.001 (S)	0.001 (S)

indentation on the surface [12]. The measurements quantify the resistance of a material to plastic deformation. The results gave the mean values of each wire assembly as (Tables 8,9 & Graph 4) Group 1 = 379.29 ± 5.13, Group 2 = 443.43 ± 37.63, Group 3 = 331.21 ± 4.85,

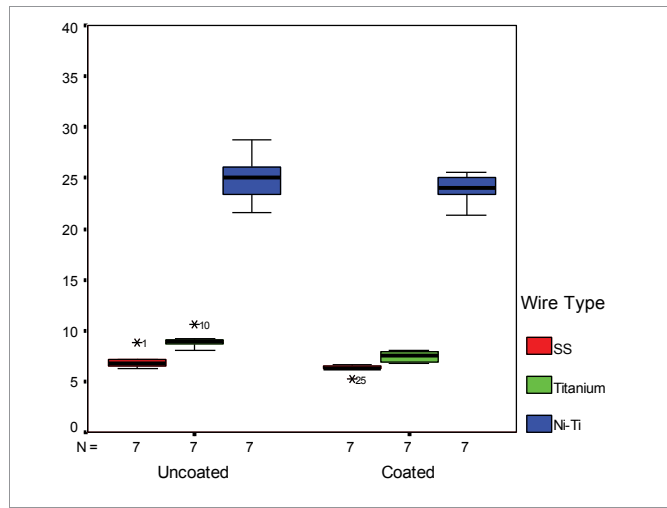
Group 4 = 364.07 ± 12.21, Group 5 = 322.07 ± 9.45, Group 6 = 387.00 ± 6.85.

Later, friction tests were performed to test the frictional resistance of a coated and uncoated wire. The results gave mean frictional values of each bracket/wire assembly as follows (Tables 10,11 & Graph 5) group 1 = 171.43 ± 15.19, group 2 = 91.43 ± 4.76, group 3 = 220.71 ± 10.58, group 4 = 138.57 ± 11.07, group 5 = 474.29 ± 14.84, group 6 = 370.00 ± 7.64.

Last part of our study was to compare the surface topography of coated and uncoated orthodontic wires. SEM analysis of the uncoated and coated straight wire with the nanoparticles with X 500 magnification of group 1-6 (Figure 8-13) revealed that the orthodontic wires coated with nanoparticles had less surface irregularities than the uncoated wires.

DISCUSSION

In orthodontics, many studies have evaluated the mechanical properties and surface characteristics of archwire alloy that uses experimental testing models that include two or three archwire alloys.



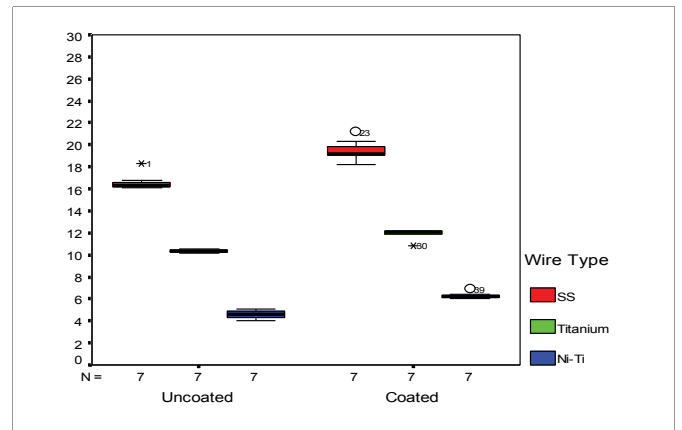
Graph 2: The Inter - group distribution of Elongation (Box - Plot).

Table 6: Intra-group comparison of Bending test [Flexural Modulus (MPa)].

Wire type	Uncoated wire (n = 7)	Coated wire (n = 7)	P-value (Uncoated v Coated)
SS (x 10 ⁴)	16.61 ± 0.76	19.48 ± 0.95	0.001 (S)
Ni-Ti (x 10 ⁴)	4.59 ± 0.39	6.28 ± 0.28	0.001 (S)
Titanium (x 10 ⁴)	10.33 ± 0.15	11.89 ± 0.50	0.001 (S)

Table 7: Inter-group comparison of Bending test [Flexural Modulus (MPa)].

Wire type	Uncoated wire (n = 7)	Coated wire (n = 7)
SS (x 10 ⁴)	16.61 ± 0.76	19.48 ± 0.95
Ni-Ti (x 10 ⁴)	4.59 ± 0.39	6.28 ± 0.28
Titanium (x 10 ⁴)	10.33 ± 0.15	11.89 ± 0.50
Inter-group comparisons (P-values)		
Ss wire v Titanium wire	0.001 (S)	0.001 (S)
Ss wire v Ni-Ti wire	0.001 (S)	0.001 (S)
Titanium wire v Ni-Ti wire	0.001 (S)	0.01 (S)



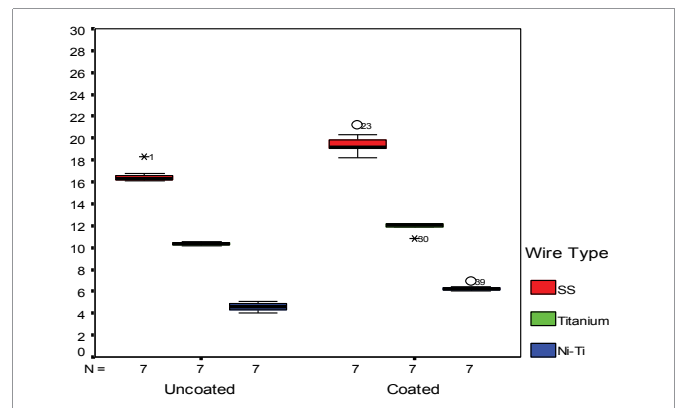
Graph 3: The Inter - group distribution of Bending test [Flexural Modulus (MPa)] (Box - Plot).

Table 8: Intra-group comparison of Microhardness (HV).

Wire type	Uncoated wire (n = 7)	Coated wire (n = 7)	P-value (Uncoated v Coated)
SS	379.29 ± 5.13	443.43 ± 37.63	0.001 (S)
Ni-Ti	331.21 ± 4.85	364.07 ± 12.21	0.001 (S)
Titanium	322.07 ± 9.45	387.00 ± 6.85	0.001 (S)

Table 9: Inter-group comparison of Microhardness (HV).

Wire type	Uncoated wire (n = 7)	Coated wire (n = 7)
SS	379.29 ± 5.13	443.43 ± 37.63
Ni-Ti	331.21 ± 4.85	364.07 ± 12.21
Titanium	322.07 ± 9.45	387.00 ± 6.85
Inter-group comparisons (P-values)		
Ss wire v Titanium wire	0.001 (S)	0.001 (S)
Ss wire v Ni-Ti wire	0.001 (S)	0.001 (S)
Titanium wire v Ni-Ti wire	0.054 (NS)	0.182 (NS)



Graph 4: The Inter - group distribution of Micro hardness (HV) (Box - Plot).

This study evaluated mechanical properties of Nanoparticles Coated and uncoated Stainless steel, NiTi and Beta Titanium orthodontic arch wire. A comparison between the properties was also performed to provide an insight into their use in each stage of orthodontic mechanotherapy. The bracket wire assembly included MBT 0.022” slot stainless steel bracket ligated with 0.009” stainless

steel ligature and wires tested were uncoated 0.019 X0.025” SS wires, NiTi wire, TMA wire and nanoparticle coated 0.019 X0.025” SS wire, NiTi wire and TMA wire.

M Redlich, et al. [11], who concluded that the wires coated with WS2 nanoparticles might offer a novel opportunity to substantially reduce friction during tooth movement. A few tests undertaken to evaluate the toxicity of the fullerene-like nanoparticles have provided indications that they might be biocompatible. In this study with an extension to the above research, we have tested new nanoparticle coating based on a novel coating methodology. The coating methodology which is used in our study can also be performed at the clinical chairside and coating procedure gives a colorless film which does not alter the appearance of the wire.

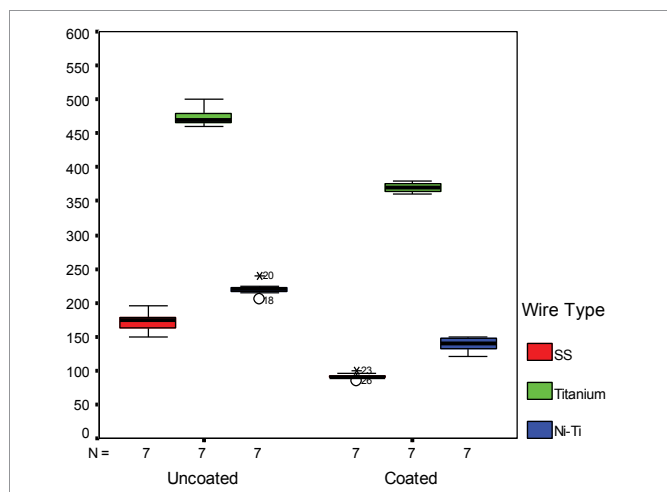
On the basis of our study, for the tensile strength, the Coated SS wire was the strongest alloy with highest values for tensile strength, and thus coated SS wire can sustain a much higher maximum load than NiTi and TMA wires. Also, coated NiTi wire (group 4) showed significant increase in tensile strength, followed by coated SS wire. This indicates the clinical performance of wire in terms of working

Table 10: Intra-group comparison of Frictional Resistance (gms).

Wire type	Uncoated wire (n = 7)	Coated wire (n = 7)	P-value (Uncoated v Coated)
SS	171.43 ± 15.19	91.43 ± 4.76	0.001 (S)
Ni-Ti	220.71 ± 10.58	138.57 ± 11.07	0.001 (S)
Titanium	474.29 ± 14.84	370.00 ± 7.64	0.001 (S)

Table 11: Inter - group comparison of Frictional Resistance (Gms).

Wire type	Uncoated wire (n = 7)	Coated wire (n = 7)
SS	171.43 ± 15.19	91.43 ± 4.76
Ni-Ti	220.71 ± 10.58	138.57 ± 11.07
Titanium	474.29 ± 14.84	370.00 ± 7.64
Inter-group comparisons (P - values)		
Ss wire v Titanium wire	0.001 (S)	0.001 (S)
Ss wire v Ni-Ti wire	0.001 (S)	0.001 (S)
Titanium wire v Ni-Ti wire	0.001 (S)	0.001 (S)



Graph 5: The Inter - group distribution of Frictional Resistance (Gms) (Box - Plot).

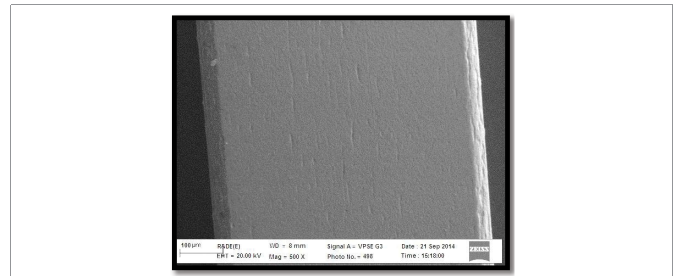


Figure 8: Sem image for Group 1.

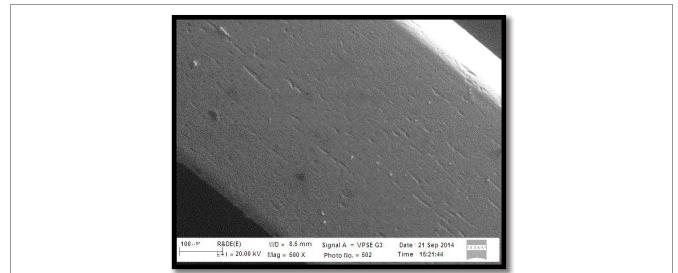


Figure 9: Sem image for Group 2.

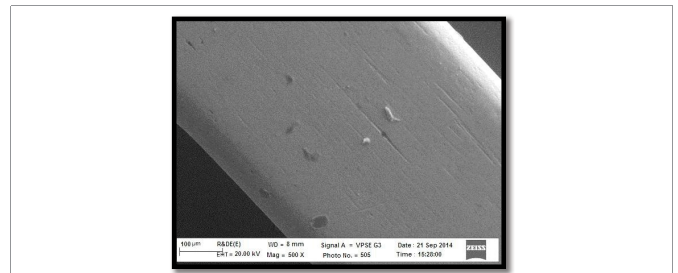


Figure 10: Sem image for Group 3.

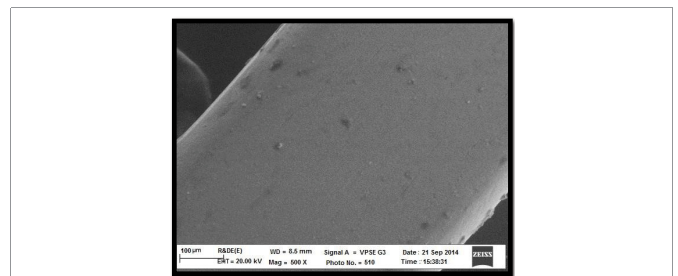


Figure 11: Sem image for Group 4.

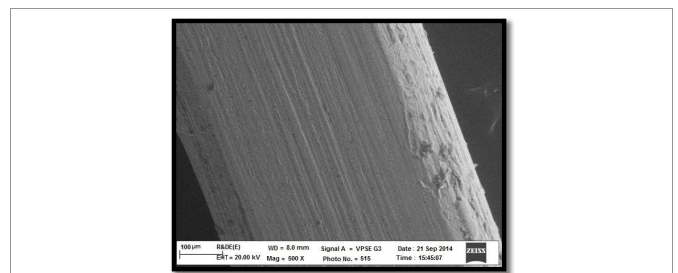


Figure 12: Sem image for Group 5.

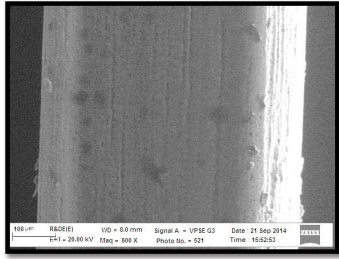


Figure 13: Sem image for Group 6.

range. But, coated TMA showed decrease in tensile strength as compared to uncoated TMA wire.

Elongation/Strain is measured for the ductility of steel. It is overall deformation (elastic or plastic) of a material as a result of tensile force application [12]. When a material is tested for tensile strength it elongates a certain amount before fracture takes place. Upon elongation test, this nanoceramic coating decreased the ductility i.e. ability of a material or arch wire to deform under tensile stress, to a statistically significant difference.

A three-point bend test performed to evaluate the load deflection properties, which is the most important parameter determining the biologic nature of tooth movement [13]. This test was chosen mainly because of its close simulation to clinical application, reproducible results, and the ability to differentiate wires with super elastic properties. Therefore, load deflection of the coated wires was slightly increased (group 2, group 4 and group 6).

Vickers Micro hardness tests are performed to test the resistance to indentation on the surface and is been used in mechanical engineering to determine the hardness of a material to deformation and thus Nanoceramics coating enhance the resistance of a material to plastic deformation [12].

The microhardness of coated SS (group 2) had the hardest surface among all the groups. Coating proved to have a statistically significant effect on the hardness of each orthodontic archwire alloy. Thus, coated SS was the hardest alloy compared to uncoated SS, followed by coated TMA compared to uncoated TMA and finally coated NiTi was superior as compared to uncoated NiTi. Frictional resistance of every nanoceramic coated archwire specimens was decreased as compared to uncoated wire specimens. Thereby, this could have important clinical application during initial alignment & retraction phase of orthodontic treatment.

Hence, we found that the nanoparticle coating on 0.019 X 0.025” SS, NiTi and TMA wires produced significantly better tensile strength, decreased elongation/ ductility, Increased hardness, lower level of frictional resistance and less surface irregularities than uncoated 0.019 X0.025” SS, NiTi and TMA archwires respectively. This nanoceramic coated wires may represent a valid alternative to commercially available uncoated wires with more desired ideal wire characteristics.

In a study conducted by Chembath, et al [14], Titania coating was successfully developed on chemically treated NiTi alloy by sol-gel process. The electrochemical behavior of sol-gel-coated NiTi surface was similar to that of pure titanium. The passive layer formed on the surface was stable until 0.96 V and displayed good corrosion resistance than chemically treated or bare NiTi. This confirms the improvement in pitting resistance of NiTi surface due to sol-gel Titania coating.

Similarly, in a recent study by Zhang, et al. [15], evaluated the difference of the structure, adhesion, wear and corrosion properties of the DLC coating on different substrates (stainless steel, CoCrMo or titanium alloy). They found that the structure of the DLC coating on these substrates was similar. The DLC coating on titanium alloy showed the best adhesion than that on stainless steel and CoCrMo alloy, because the chemical bond energy holding Ti atom (of titanium alloy surface) and C atom (of DLC) together is the strongest. The failure mode of DLC coatings on 316L stainless steel and CoCrMo alloy in the friction test was coating delamination.

The limitations of this research are that it was carried out in vitro, in a passive configuration. Tipping, torquing forces and the functional forces of the stomatognathic muscles can also affect the mechanical properties during space closure; however these factors need to be studied.

With further improvements in the coating method and its suitability for use in the oral cavity, mechanical properties of the orthodontic arch wire alloys could be enhanced, which would further enhance anchorage control and also reduce the treatment duration.

CONCLUSIONS

- Tensile strength increased in nanoparticle coated archwires and coated SS showed the highest tensile strength. Thus this coating could improve the clinical performance or efficacy in terms of working range.
- The study showed decrease in Elongation/Strain Test. Thus, a decrease in ductility was seen for the coated wires over uncoated archwires.
- The bending test showed higher load deflection rates for the coated arch wires as compared to uncoated wires. Also, among the groups, load deflection of the coated SS was the highest. This would not be beneficial in a clinical situation where engagement of the wire in the bracket requires low deflection rates for a malaligned tooth, which would then deliver controlled forces to the tooth and supporting tissues.
- Coating the arch wires significantly increases the Microhardness. Among the coated archwires, SS was the hardest and NiTi had the lowest value. Thus, nanoceramic coating enhances the resistance of a material to plastic deformation.
- Frictional forces were reduced for coated archwires. Therefore, can improve the inter-relation between the bracket and the coated archwires during orthodontic tooth movements.

The SEM showed less surface irregularities on coated orthodontic archwires, hence decreasing the frictional resistance of coated wires.

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