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Evaluation of surface-modified orthodontic wires by different concentration and dipping duration of titanium oxide (TiO₂) nanoparticles

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Abstract

OBJECTIVE: To evaluate in-vitro surface characteristics and frictional properties of orthodontic stainless steel and beta-titanium archwires after surface modification with different concentrations and coating time of titanium oxide (TiO₂) nanoparticles by Sol-gel dip coating method.

MATERIALS AND METHODS: The experiment was carried out with 4 different concentrations (1:2, 1:4, 1:6, and 1:8) and three different dipping durations (24 hours, 48 hours, and 72 hours) over ten main test groups of SS and TMA archwires with uncoated wires acting as control in both dry and wet conditions. Phase analysis and surface characterization of TiO₂ was analyzed by X-ray Diffractometry, surface evaluation with the help of scanning electron microscopy (SEM), and frictional characteristics were evaluated.

RESULTS: Among all the concentrations 1:6 ratio with 48 hours of dipping duration showed better surface characteristics. A statistically significant difference in frictional coefficient was observed in both SS and TMA wires than their respective controls ($p = 0.001$). Intragroup comparison among SS and TMA groups showed that groups with 1:6 ratio and 48 hours dipping duration had least frictional coefficient in both dry and wet conditions ($p = 0.001$). Intergroup comparison between SS and TMA showed that SS group had significantly reduced friction than TMA ($p = 0.001$) except in few groups.

CONCLUSION: TiO₂ nanoparticle with a concentration ratio of 1:6 and 48 hours dipping duration is recommended for surface modification of orthodontic archwires.

Keywords:

Coating, friction, nanoparticles, orthodontic wires, surface modification

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Introduction

Selection of an appropriate archwire requires thorough knowledge of the archwire's biomechanical and clinical applications.^[1] Moreover, among the properties of an alloy that alter the behavior of the archwire, surface characteristics play a crucial role. Surface topography of an archwire can affect its mechanical characteristics, corrosion

behavior, friction, aesthetic appearance, and biocompatibility.^[2] The retarding frictional force develops due to the surface roughness of the archwire. Nanoengineering-based surface modification can be used to reduce the surface roughness of orthodontic wires. Studies have demonstrated a reduction in the surface roughness and friction on the coating of archwires such as diamond-like carbon,^[3] Teflon,^[4] and fullerene-like nanoparticles.^[5] Anuradha *et al.*^[6] reported that sputter coating with titanium on archwires reduces surface roughness.

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Furthermore, other studies have focused on bacterial adhesion based on the photocatalytic activity of TiO₂.^[7,8] Liu *et al.*^[9] reported that the corrosion resistance of the composite archwire might be substantially upgraded after coating with the TiO₂ nanocrystal thin film. Owing to its beneficial characteristics such as biological stability, antibacterial property, and high frictional resistance, titanium dioxide (TiO₂) has been receiving considerable attention in recent years compared with other nanoparticles used for the surface modification of orthodontic archwires. To the best of our knowledge, rarely any study has evaluated the surface roughness and frictional resistance of surface-modified orthodontic wires by using different concentrations of TiO₂ nanoparticles at different coating times.

Materials and Methods

This study was conducted at the Department of Orthodontics in collaboration with the School of Materials Science and Technology. The study was approved by the institutional ethical committee (DEAN/2019/EC/1155). Preformed straight-length rectangular stainless steel (SS) and beta-titanium (TMA) archwires (Ormco. Corp) were used in this study. The study was performed using 260 specimens of orthodontic wires, with each being 65 mm in length. The specimens were divided into 10 main test groups. The groups containing uncoated SS and uncoated beta-titanium wires acted as the control group for their respective experimental group. Each group except the control group (Group 1-ST and Group 2-TT) was divided into three sub-groups based on the dipping duration of the respective colloidal solution of TiO₂ [Table 1]. The nanocrystalline anatase phase of TiO₂ with different ratios (1:2, 1:4, 1:6, and 1:8) was prepared [Table 2]. The sol-gel method was used for coating at room temperature without any heat treatment. Before deposition, the bare SS and TMA wires were cleaned in dilute H₂SO₄ and absolute ethanol solution. The substrates were immersed in the coating solutions of different concentrations and withdrawn at a series of three different immersion timings of 24 hours, 48 hours, and 72 hours. After withdrawal from the solution, the wires were dried in an oven at 60°C for 5 min. The formation and phase analyzes of obtained TiO₂ were performed using the Rigaku X-ray diffractometer (XRD). Surface morphology of the uncoated and coated SS and TMA wires was examined through scanning electron microscopy (SEM; Nova Nano SEM 450). Tidy's protocol^[10] was adopted to determine frictional characteristics. All the tests were conducted in both dry and wet (artificial saliva) conditions by using an Instron universal testing machine (Instron corp., model number- 3379). The bracket and wire were replaced after sliding each sample to ensure the similarity of conditions for all the samples.

Results

Figure 1 shows the diffraction pattern of 1:6 TiO₂ nanoparticles coated on SS and TMA wires. The diffraction patterns obtained were similar for all the four different colloidal solution concentration. The very first observation of the diffraction pattern is broad diffraction peaks indicating the TiO₂ crystallites to be in a nanometre range. Results of phase analysis, showed that there was 83% of anatase phase and 17% of rutile phase of TiO₂. Further, the particle size of the anatase phase in all concentrations has a similar size of 7 nm. In the Figure 2a, we can observe the rough surface morphology of Group 1-ST, that is, uncoated SS wire. The uncoated SS archwire surface is full of pits and wedges adding to a rough surface. In Figure 2b and c, the microphotographs of Group 2-ST₂₄, Group 2-ST₄₈, and Group 2-ST₇₂ which were coated with the solution ratio 1:2, smooth surface morphology without any pits and voids are evident. In Group 2-ST₇₂, slight irregularity was observed [Figure 2d]. In Group 3-ST₇₂, more irregularity was observed as compared with Group 2-ST₂₄ and Group 2-ST₄₈ [Figure 2e]. Group 2-ST₄₈ showed relatively smoother surface when compared with other groups. In Figure 2f, we can observe a smooth surface morphology of Group 4-ST₄₈. In addition, irregular surface with pits were observed in Group 4 ST₇₂. [Figure 2g]. In Figure 2h, we can observe a irregular surface morphology with pits and voids of Group 5-ST₂₄. For the concentration ratio of 1:4 and 1:6, 24 hours duration was insufficient to form a homogeneous nanofilm of TiO₂. Figure 3a, shows roughest surface morphology of Group 6-TT, that is, uncoated TMA wire. Figure 3b and c represents the SEM images of Group 7-TT₄₈ and Group 7-TT₇₂. We can observe a smoother surface morphology of TMA wire coated with 1:2 ratio. Figure 3d-f, reveals the SEM images

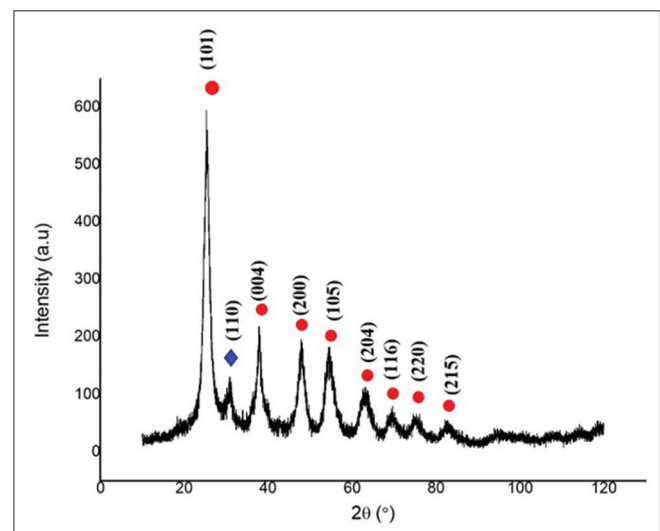


Figure 1: XRD pattern of the colloidal solution with the concentration ratio of 1:6. (Solid blue diamond represents rutile phase and solid red circle represents anatase phase)

Table 1: Groups of coated and uncoated SS and TMA wires

Group Name	Main group with description	Sub-group with description
Group 1-ST	Control group–Uncoated SS wire	No sub-groups
Group 2-ST	Experimental group–Coated SS wire with 1:2 ratio	Group 2-ST ₂₄ –dipping duration of 24 hours Group 2-ST ₄₈ –dipping duration of 48 hours Group 2-ST ₇₂ –dipping duration of 72 hours
Group 3-ST	Experimental group–Coated SS wire with 1:4 ratio	Group 3-ST ₂₄ –dipping duration of 24 hours Group 3-ST ₄₈ –dipping duration of 48 hours Group 3-ST ₇₂ –dipping duration of 72 hours
Group 4-ST	Experimental group–Coated SS wire with 1:6 ratio	Group 4-ST ₂₄ –dipping duration of 24 hours Group 4-ST ₄₈ –dipping duration of 48 hours Group 4-ST ₇₂ –dipping duration of 72 hours
Group 5-ST	Experimental group–Coated SS wire with 1:8 ratio	Group 5-ST ₂₄ –dipping duration of 24 hours Group 5-ST ₄₈ –dipping duration of 48 hours Group 5-ST ₇₂ –dipping duration of 72 hours
Group 6-TT	Control group–Uncoated beta titanium wire	No sub-groups
Group 7-TT	Experimental group–Coated beta titanium wire with 1:2 ratio	Group 7-TT ₂₄ –dipping duration of 24 hours Group 7-TT ₄₈ –dipping duration of 48 hours Group 7-TT ₇₂ –dipping duration of 72 hours
Group 8-TT	Experimental group–Coated beta titanium wire with 1:4 ratio	Group 8-TT ₂₄ –dipping duration of 24 hours Group 8-TT ₄₈ –dipping duration of 48 hours Group 8-TT ₇₂ –dipping duration of 72 hours
Group 9-TT	Experimental group–Coated beta titanium wire with 1:6 ratio	Group 9-TT ₂₄ –dipping duration of 24 hours Group 9-TT ₄₈ –dipping duration of 48 hours Group 9-TT ₇₂ –dipping duration of 72 hours
Group 10-TT	Experimental group–Coated beta titanium wire with 1:8 ratio	Group 10-TT ₂₄ –dipping duration of 24 hours Group 10-TT ₄₈ –dipping duration of 48 hours Group 10-TT ₇₂ –dipping duration of 72 hours

Table 2: Concentration ratios of different solutions

Solution	Ratio	Quantity (in ml)
Colloidal solution no. 1	1:2 (1 part of solution mixture and 2 parts of distilled water)	33.3 ml solution mixture and 66.7 ml of distilled water
Colloidal solution no. 2	1:4 (1 part of solution mixture and 4 parts of distilled water)	20 ml solution mixture and 80 ml of distilled water
Colloidal solution no. 3	1:6 (1 part of solution mixture and 6 parts of distilled water)	16.70 ml solution mixture and 83.30 ml of distilled water
Colloidal solution no. 4	1:8 (1 part of solution mixture and 8 parts of distilled water)	12.5 ml solution mixture and 87.5 ml of distilled water

of Group 10TT₂₄, Group 10TT₄₈, and Group 11TT₄₈. All these groups showed better surface morphology with a relatively smoother surface. Figure 4c and d suggest that at the least concentration ratio (1:8), even 48 hours and 72 hours of dipping duration was insufficient to form a homogenous covering. Rather we can observe increased crystallization and peeling of TiO₂ layer on SS wire. At highest concentration (1:2), 24 hours of dipping duration is insufficient to form a homogeneous nanofilm of TiO₂ over TMA archwires. The surface morphology was rough with voids and uncoated areas [Figure 4e-f]. On an average, groups of concentrations (1:2, 1:4, and 1:6) except 1:8 showed better surface characteristics [Figure 4a-f]. Among these three concentrations, all the groups with 48 hours of

dipping duration exhibited good surface characteristics. Group 4-ST₄₈ has the least frictional resistance in both dry and wet conditions among all the groups of SS wire while in TMA, Group 9-TT₄₈ has the least frictional resistance in both conditions [Table 3]. Intragroup comparison between Group 2-ST₄₈ and Group 4-ST₄₈ among SS wires was not significant ($p = 1.000$) in both dry and wet conditions. Rest all other intragroup comparison of SS wires have significant difference among each other ($p = 0.001$) in both dry and wet conditions. Intragroup comparison among TMA wires showed that there was no significant difference between Group 7-TT₄₈ and Group 8-TT₄₈ ($p = 1.000$) in both dry and wet conditions and also there was no significant difference between Group 7-TT₄₈ and Group 9-TT₄₈ ($p = 1.000$) and between Group 8-TT₄₈ and Group 9-TT₄₈ ($p = 1.000$) only in wet condition. But during dry condition both of the above intragroup comparison showed significant difference ($p = 0.002$ and 0.001). Rest all other intragroup comparison of TMA wires showed significant difference ($p = 0.001$) [Table 4]. Intergroup comparison between SS and TMA showed that there was significant difference among all the groups ($p = 0.001$) except Group 2-ST₄₈ and Group 7-TT₄₈ ($p = 0.481$ -dry and 1.000 -wet) in both the conditions [Table 5].

Discussion

Resistance during tooth movement may occur due to

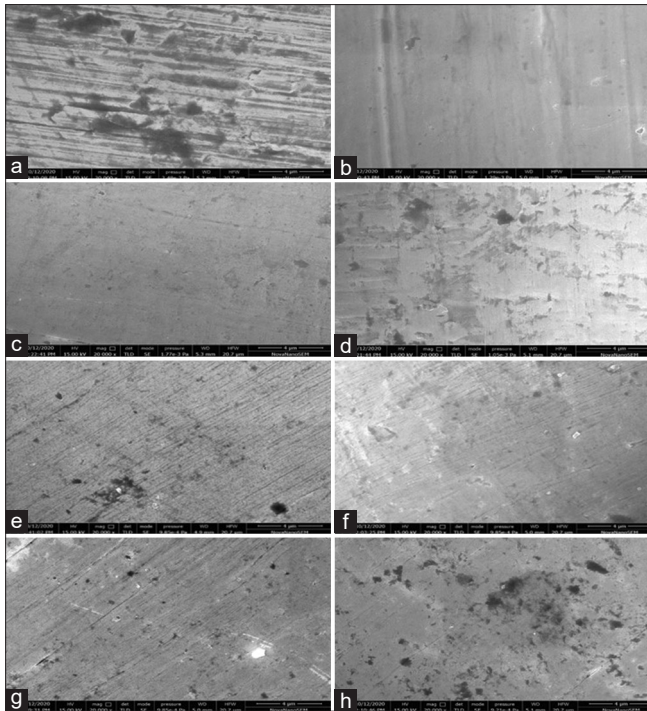


Figure 2: Scanning Electron Microscopic images of uncoated and TiO₂ coated SS archwires. (a) SEM image of Group 1-ST (uncoated SS wire), (b) SEM image of Group 2-ST₂₄, (c) SEM image of Group 2-ST₄₈, (d) SEM image of Group 2-ST₇₂, (e) SEM image of Group 3-ST₇₂, (f) SEM image of Group 4-ST₄₈, (g) SEM image of Group 4-ST₇₂, (h) SEM image of Group 5-ST₂₄

physical^[11] or biological factors.^[12] Use of nanoparticles in various studies has led to the development of many orthodontic wires with improved frictional characteristics.^[3-6] The antibacterial effect of TiO₂-coated wires has been evaluated without considering frictional properties.^[7,8] Various physical and chemical approaches have been used to prepare TiO₂ thin films, including sol-gel^[13] and pulsed laser deposition.^[14] Hosseingholi *et al.*^[15] synthesized nanocrystalline TiO₂ sols through the hydrolysis of tetra-isopropyl orthotitanate at different pH values and room temperature. We used the Sol-gel method, which is the most facile and cost-effective route that not only omits the requirement of high temperatures, toxic chemicals, and expensive equipment but also offers several advantages such as the better homogeneity of the structure, high purity of starting materials, and possibility to control the porosity and structure of a fixed, three-dimensional network of gels with better coating stability.^[16] The ratio of 1:1 (1 part of TiO₂ and 1 part of deionized water) was not observed in the colloidal state and thus discarded. XRD findings indicated the presence of an equal size of TiO₂ nanoparticles among different concentrations. Approximately 83% of the nanocrystalline structure of TiO₂ was in the anatase phase, as indicated by Hosseingholi *et al.*^[15] In contrast to the findings of our study, a previous study reported that the rutile phase exerts a greater effect on improvement in surface characteristics.^[17] However, another study

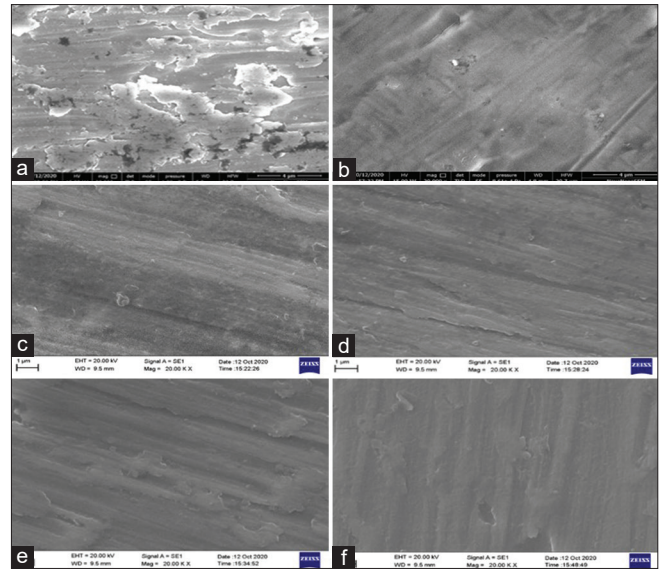


Figure 3: Scanning electron microscopic images of uncoated and TiO₂-coated TMA archwires (a) SEM image of Group 6-TT (uncoated TMA wire), (b) SEM image of Group 7-TT₄₈, (c) SEM image of Group 7-TT₇₂, (d) SEM image of Group 8-TT₂₄, (e) SEM image of Group 8-TT₄₈, (f) SEM image of Group 9-TT₄₈

indicated that both the anatase and rutile phases of TiO₂ can improve surface characteristics equally.^[18] With a decrease in the TiO₂ concentration, the duration required to form a smooth and uniform nanofilm increased. Moreover, at a ratio of 1:8, crystallization and peeling of TiO₂ nanoparticles were observed. The 24-hours dipping duration was insufficient to form a uniform TiO₂ nanofilm. Only a higher ratio (1:2) and 72-hours dipping duration led to the peeling of the TiO₂ nanofilm surface, as observed through SEM. All the coated wires of SS and TMA exhibited a significantly decreased frictional coefficient compared with their respective uncoated controls ($p = 0.001$). Intergroup comparison indicated that most of the SS wire groups demonstrated significantly decreased friction compared with TMA wire groups except in a few groups (Group 2-ST₄₈ and Group 7-TT₄₈). TMA wires contain 70% titanium, which makes their surface rougher than that of SS wires, resulting in an increase in friction. This finding is in accordance with those of previous studies.^[19,20] At a higher concentration (1:2), TMA required 48 hours of dipping to form a smoother TiO₂ nanofilm with uniform deposition. TiO₂-coated SS/TMA wires under wet conditions consistently exhibited the lowest frictional resistance values. This finding is supported by a study using fullerene-like nanoparticles.^[21] SS/TMA coated archwires with a ratio of 1:6 and a dipping duration of 48 hours demonstrated better results with respect to both surface characteristics and frictional resistance compared with other ratios (1:2 and 1:4). Lower concentrations of TiO₂ (<1:6) did not produce promising results. A dipping duration of 48 hours was optimal because <48 hours led to irregular coating and >48 hours led to the

Table 3: Mean values of coefficient of friction for SS and TMA wires

Groups (SS)	Coefficient of friction (μ) Mean \pm SD		Groups (TMA)	Coefficient of friction (μ) Mean \pm SD	
	Dry condition	Wet condition		Dry condition	Wet condition
Group 1-ST	0.611 \pm 0.006	0.500 \pm 0.043	Group 6-TT	0.743 \pm 0.014	0.687 \pm 0.003
Group 2-ST ₄₈	0.281 \pm 0.010	0.233 \pm 0.004	Group 7-TT ₄₈	0.456 \pm 0.024	0.292 \pm 0.002
Group 3-ST ₄₈	0.436 \pm 0.019	0.377 \pm 0.031	Group 8-TT ₄₈	0.439 \pm 0.007	0.289 \pm 0.005
Group 4-ST ₄₈	0.253 \pm 0.013	0.228 \pm 0.002	Group 9-TT ₄₈	0.364 \pm 0.016	0.250 \pm 0.007

Table 4: Statistical analysis of intra-group comparison between SS wires and TMA wires

Comparison groups (SS)	P		Comparison groups (TMA)	P	
	Dry condition	Wet condition		Dry condition	Wet condition
Group 1-ST and Group 2-ST ₄₈	0.001	0.001	Group 6-TT and Group 7-TT ₄₈	0.001	0.001
Group 1-ST and Group 3-ST ₄₈	0.001	0.001	Group 6-TT and Group 8-TT ₄₈	0.001	0.001
Group 1-ST and Group 4-ST ₄₈	0.001	0.001	Group 6-TT and Group 9-TT ₄₈	0.001	0.001
Group 2-T48 and Group 3-ST ₄₈	0.001	0.001	Group 7-T48 and Group 8-TT ₄₈	1.000	1.000
Group 2-T48 and Group 4-ST ₄₈	1.000	1.000	Group 7-T48 and Group 9-TT ₄₈	0.002	1.000
Group 3-T48 and Group 4-ST ₄₈	0.001	0.001	Group 8-T48 and Group 9-TT ₄₈	0.001	1.000

Significant at the $P \leq 0.05$

Table 5: Statistical analysis of inter-group comparison between SS and TMA wires

Comparison groups	P	
	Dry condition	Wet condition
Group 1-ST and Group 6-TT ₄₈	0.001	0.001
Group 2-ST ₄₈ and Group 7-TT ₄₈	0.481	1.000
Group 3-ST ₄₈ and Group 8-TT ₄₈	0.001	0.001
Group 4-ST ₄₈ and Group 9-TT ₄₈	0.001	0.001

Significant at the $P \leq 0.05$

crystallization and peeling of the nanofilm layer. Keerthi *et al.*^[22] suggested that the smoothness of TiO₂-coated wires was lost at the end of 1 month of intraoral use. Improper concentration and coating time of TiO₂ may be responsible for the loss of coating. The variation in results may be due to the intraoral environment (in-vivo) in the previous study which is not the exact replica of our in-vitro experimental condition, although we used both dry and wet conditions.

Conclusion

Optimal concentration and coating time required for surface modification are elaborated in detail and concluded that:

- The optimum concentration of TiO₂ ranges from 1:2 to 1:6 with a dipping duration of 48 hours.
- Increasing or reducing the TiO₂ concentration did not affect the nanoparticle size of TiO₂.
- Frictional forces decreased in almost all the coated wires of SS and TMA except in some cases with a dipping duration of <48 hours.
- Saliva could reduce friction in both the TiO₂-coated wires (SS and TMA).
- TMA wire relatively required increased dipping durations even at higher concentrations.

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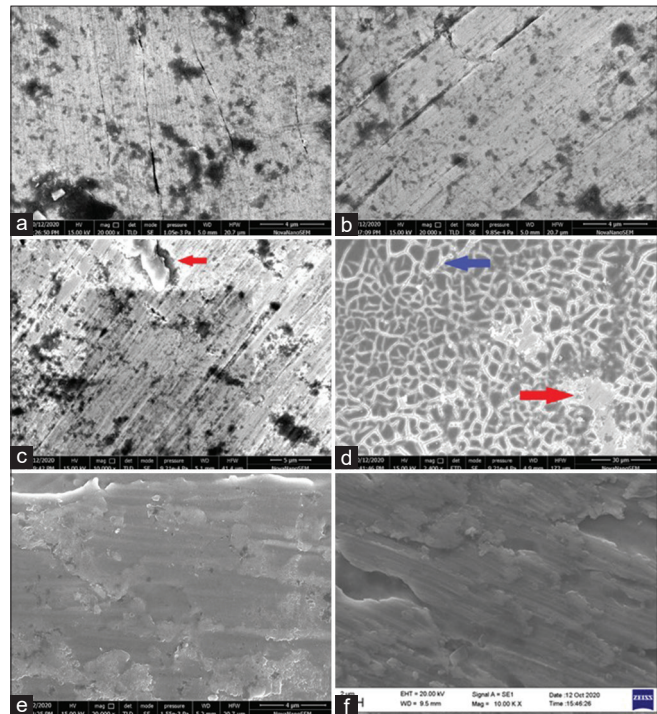


Figure 4: Scanning electron microscopic images of poor coating of TiO₂ on SS and TMA archwires (a) SEM image of Group 3-ST₂₄, (b) SEM image of Group 4-ST₂₄, (c) SEM image of Group 5-ST₄₈ (red arrow indicates peeling of TiO₂ coating), (d) SEM image of Group 5-ST₇₂ (blue arrow indicates crystallization of TiO₂ nanoparticles and red arrow indicates uncoated bare wire surface), (e) SEM image of Group 7-TT₂₄, (f) SEM image of Group 9-TT₂₄

Conflicts of interest

There are no conflicts of interest.

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