

## Brushing-Induced Surface Roughness of Two Nickel Based Alloys and a Titanium Based Alloy: A Comparative Study - *In Vitro* Study

B L Guruprasanna Acharya<sup>1</sup>, Ramesh Nadiger<sup>2</sup>, Bharathraj Shetty<sup>3</sup>, G Gururaj<sup>4</sup>, K Naveen Kumar<sup>5</sup>, D D Darshan<sup>6</sup>

### Contributors:

<sup>1</sup>Assistant Professor, Department of Prosthodontics, Malabar Dental College and Hospital, Mallappuram, Kerala, India; <sup>2</sup>Professor, Department of Prosthodontics, Sri Dharmasthala Manjunatheshwara College of Dental Sciences, Sattur, Dharwad, Karnataka, India; <sup>3</sup>Assistant Professor, Department of Prosthodontics, AB Shetty Memorial Institutes of Dental Sciences, Mangalore, Karnataka, India; <sup>4</sup>Assistant Professor, Department of Pedodontics, Navodaya Dental College and Hospital, Raichur, Karnataka, India; <sup>5</sup>Assistant Professor, Department of Conservative Dentistry and Endodontics, KVG Dental College and Hospital, Sullia, Karnataka, India; <sup>6</sup>Assistant Professor, Department of Oral Medicine and Radiology, KVG Dental College and Hospital, Sullia, Karnataka, India.

### Correspondence:

Dr. Acharya BL. Department of Prosthodontics, Malabar Dental College and Hospital, Mallappuram, Kerala, India. Email: guruacharyasdm@gmail.com

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### Abstract:

**Background:** Alloys with high nickel content have been increasingly used in dentistry. Alloys have high corrosion rates when exposed to chemical or physical forces that are common intra orally. Titanium is the most biocompatible materials for crowns, fixed partial dentures and implants in the present use, but paradoxically the self-protective oxide film on the titanium can be affected by excessive use of the most common preventive agents in dentistry. Therefore, this study is undertaken in order to draw attention toward the potential effect of prophylactic brushing in a saline medium.

**Materials and Methods:** Forty-five wax patterns in equal dimensions of 10 mm × 10 mm × 2 mm were cast in titanium (Grade II) and nickel-chromium. Of the 45 wax patterns, 15 wax patterns were used for preparing cast titanium samples and 30 wax patterns were used for preparing cast nickel-chromium samples and polished. These samples were divided into three groups of 15 samples each. They are brushed for 48 h each clinically simulating 2 years of brushing in a saline tooth paste medium. The surface roughnesses of the samples were evaluated using profilometer, scanning electron microscopes and energy dispersive spectroscopy. Results were subjected to statistical analysis.

**Results:** The statistical analysis of the Rz and Ra surface roughness values were calculated. Significant difference of surface roughness was present in the titanium samples compared to that of the machine-readable cataloguing and Wirrolloy (nickel-chromium) samples after the study. To know the difference in the values of all samples before and after, Student's paired *t*-test was carried out.

Results showed that there is a significant change in the Rz and Ra values of titanium samples.

**Conclusion:** The present findings suggest that, prophylactic brushing with the fluoridated toothpaste have an effect on the surface roughness of titanium and also to a certain extent, on nickel-chromium. Therefore, careful consideration must be given to the selection of the toothbrushes and toothpastes with the medium abrasives in patients with these restorations.

**Key Words:** MAARC, titanium, wirrolloy

### Introduction

Metals have been used as biomaterials for many centuries. In 1565, gold plate was reported to be used to repair the cleft palatal defects. Gold alloys and their substitutes are formed by a casting process developed by Taggart in 1907. Since then, cast restorations have been routinely used in dentistry.

With the advances in the 1960s and a significant increase in the price of gold in the 1970s, alternative alloys such as palladium alloys and base metal alloys were developed. The allergenic and carcinogenic properties of the base metal alloys used in the dentistry, especially nickel and beryllium alloys have fuelled controversies. The safety concerns and possibility of the adverse health effects from the exposure of the certain elements in the biomaterials have been raised more often in the recent years, kindling concerns among the public. The evolution of the titanium applications to the medical and dental implants has dramatically increased in the past few years because of its excellent biocompatibility, corrosion resistance, and desirable physical and mechanical properties.<sup>1</sup>

With development in the dental casting technique for titanium, the metals have been used for crowns, fixed and removable partial denture frameworks, especially for patients with the allergy to nickel and other specific substances. The distinguished biocompatibility of the titanium is mainly attributed to the surface oxide film that has excellent resistance to corrosion. The physical properties of titanium and its alloys can be greatly varied with the addition of the small traces of other elements such as oxygen, nitrogen and iron. Commercially pure (Cp) titanium is available in four grades (American Society of Testing and Material Grades I-IV), is based on the incorporation of small amounts of oxygen, nitrogen, hydrogen, iron and carbon during the purification process. The mechanical properties of titanium material and its alloys surpass the requirements for an implant material.

Titanium is the most biocompatible and corrosion resistant metallic material for implants in the present use, but paradoxically, the self-protective oxide film on the titanium can be affected by excessive use of the most common preventive agents in the dentistry, prophylactic polishing and topical fluoride applications.<sup>1</sup> The main drawback is poor resistance to wear. Dentifrice includes different abrasives, which are basically used to clean the plaque, pellicle and food debris from the teeth. However, the restorative materials undergo mechanical degradation due to the abrading action of the dentifrice. In the long-term, the abrasion effect causes biological disadvantages, such as the accumulation of the dental plaque and increased element release associated with toxicity. Siirila and Kononen had claimed that the abrasion caused by tooth bristles rather than the effect of the additive fluoride (0.125%), was the main deteriorating factor for titanium. The personal dental hygiene procedure with brush has been reported to produce superficial grooves on the titanium implant abutments.

Abrading and polishing process of the titanium brought about mechanical surface disturbance accompanied by the alteration of the surface composition, such surface disturbance and resultant chemical alteration were also expected to occur during tooth brushing with dentifrice.<sup>2</sup>

Some nickel-based alloys are highly susceptible to passive film breakdown because of pitting and crevice corrosion mechanism. Clinical failures have been associated with nickel based castings used with the titanium root form implants or threaded abutments.<sup>1</sup>

Several *in vivo* studies are performed to examine the positive relationship between the surface roughness and the rate of supragingival bacterial colonization. Rough supra gingival surfaces accumulate and retain more plaque. After several days of undisturbed plaque formation, rough surfaces also harbored a more matured plaque, characterized by a larger proportion of spirochetes and motile organisms. Furthermore, clinically, gingivitis and periodontitis were detected more frequently around crowns and teeth with a rough surface.<sup>3</sup>

Therefore, this study is undertaken in order to draw attention toward the potential effect of prophylactic brushing on cast titanium and nickel-based alloys with a medium abrasive toothbrush and paste in a saline medium.<sup>4,6</sup>

#### **Objectives of the study**

1. To determine the brushing-induced surface roughness of nickel-chromium alloys<sup>7-10</sup>
2. To determine the brushing-induced surface roughness of Grade II Cp titanium<sup>11-19</sup>
3. To compare and evaluate the surface integrity of nickel-based alloys to that of titanium-based alloy after brushing for 48 h in a saline tooth paste medium
4. Evaluation of the difference in the surface roughness

created by prophylactic brushing and dentifrice over the nickel-chromium and titanium samples.<sup>19-21</sup>

#### **Methods**

This study was conducted in Department of prosthodontics, S.D.M College of Dental Sciences and Hospital, Sattur, Dharwad, to evaluate the brushing-induced surface roughness of two nickel-based alloys and Cp Grade II titanium.

The study was divided into three parts: The first part of the study dealt with quantitative analysis of surface roughness of the samples using profilometer (Wyko NT 1100) before surface treatment. The second part of the study dealt with the preparation of the test samples.

The third part of the study dealt with quantitative analysis of surface roughness of the samples using profilometer (Wyko NT 1100) after surface treatment with the brushing induced surface roughness by medium abrasive toothpaste and brush.

#### **Materials**

This study was conducted on 15 CP, ASTM Grade II titanium (Figure 1) test samples and 30 nickel-chromium test samples, 15 samples of two different commercially available, machine-readable cataloging (MAARC) and Wiroloy each sample measuring 10 mm × 10 mm × 2 mm. It was polished on one side and on the other side, it was numbered. 1-15 of MAARC, 16-30 of Wiroloy and 31-45 titanium samples.

#### **Materials used for surface brushing of titanium and nickel-chromium samples**

1. Anchor toothbrushes, two brushes used per sample and changed every 24 h (Figure 2)
2. Anchor toothpaste saline medium prepared by adding 15 g of toothpaste/30 ml of saline (Figure 3).

#### **Equipments used in the study**

1. Tooth brushing machine (Figures 4 and 5)<sup>22</sup>: A Black and Decker 480 w power variable speed pendulum jigsaw machine was modified to a tooth brushing machine set to 90 strokes per min and height adjusted such that the samples are brushed constantly under 200 g pressure
2. Profilometer (Figure 6): Wyko profilometers are noncontact optical profilers that use two technologies, phase shifting interferometry mode and vertical scanning interferometry mode to measure a wide range of surface heights. These compute several surface parameters that provide information about roughness and surface profile
3. Scanning electron microscope (SEM).

#### **Methodology**

##### **Preparation of wax patterns**

The square shaped cast titanium and nickel-chromium samples were prepared using a mold of putty elastomer (Figure 7) having a square shape of 10 mm × 10 mm and a depth of 2 mm.



Figure 1: Grade II titanium ingot.



Figure 4: Tooth brushing machine.



Figure 2: Anchor tooth paste and tooth brush.

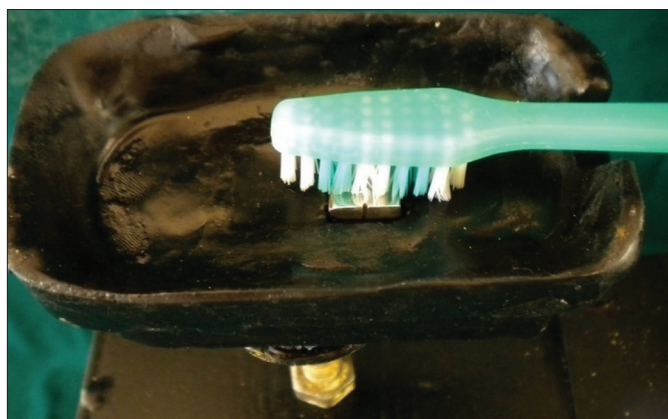


Figure 5: Single sample undergoing tooth brushing test.



Figure 3: Apparatus used for preparation of tooth paste saline medium.

Forty-five wax patterns were prepared by flowing molten casting inlay wax (Schuller-Dental Ulm-W. Germany) into the square shaped mold space with the help of a thermostat (Dentaurum) (Figure 8). The wax patterns that were retrieved had the dimensions of 10 mm × 10 mm × 2 mm.

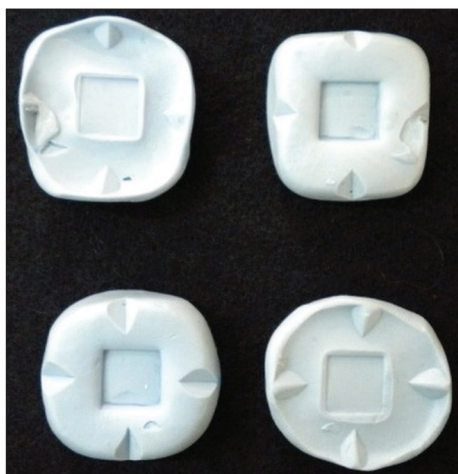


Figure 6: Wyko NT1100 optical Profilometer.

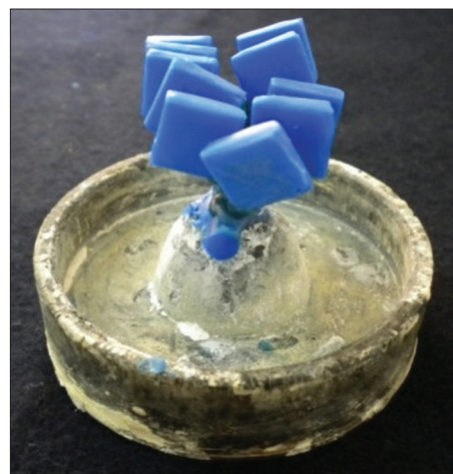
Of the forty-five wax patterns, 15 wax patterns were used for preparing cast titanium samples and 30 wax patterns were used for preparing cast nickel-chromium samples (Figure 9).

**Investing and casting of wax patterns for titanium samples**

Square shaped 10 wax patterns were attached at a time to the runner bar sprue former with sticky and hard



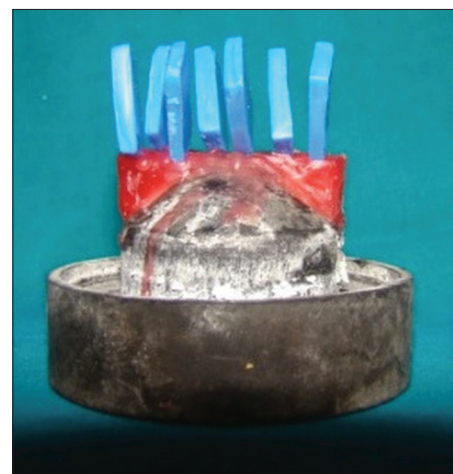
**Figure 7:** Putty index for uniform wax patterns.



**Figure 10:** Wax patterns attached with the sprues on crucible former for nickel-chromium.



**Figure 8:** Materials and armamentarium for wax pattern preparation.



**Figure 11:** Wax patterns attached on crucible former for titanium samples.



**Figure 9:** Wax patterns of the samples.



**Figure 12:** Materials used for investing.

casting wax (Figures 10 and 11). The runner bar was then fixed to a 6 × size crucible former (Titec-Orotig, Verona, and Italy) and sprayed with surface tension-reducing agent (Aurofilm 52019, Bego, Germany) (Figure 12) and allowed to air dry.

A layer of moistened ceramic based liner (Kera-Vlies; Dentaaurum) was placed lining the 6 × investing ring;

550 g of Titec investment material (for the crown and bridge) (Titec; Orotig) was mixed with 75 ml of Titec liquid (Figure 12) in a vacuum mixer (Multivac 4-Degussa, Germany) according to manufacturer's instructions. The setting time of the investment was 40 min, following this burnout procedure (Figure 13) was carried out as per the manufactures instructions. The casting procedure was carried

out in a semiautomatic pressure-type casting machine with one chamber (Titec F210M; Orotig, Figure 14) under argon gas pressure of four bars. After casting, the rings were allowed to cool to room temperature and divested from the investment. The castings were sandblasted with 110µm aluminum oxide powder (Al<sub>2</sub>O<sub>3</sub>). The samples with the individual sprue attached to the runner bar were separated using carborundum disc (Dentorium, New York, USA). In all three samples with internal porosity were discarded and an equal number of samples were fabricated so that the group comprised of 15 samples, each without internal porosities. To ensure the complete removal of the alpha-case layer, surface finishing of all the samples was done using titanium finishing bur (Dentaurum, Germany) with a hand piece (Kavo GmbH, Biberach, Germany), speed ranging from 15,000 to 20,000 rpm. The samples were standardized by holding it to a fixed plane horizontal grinding unit; the movement of the bur was unidirectional with light strokes. For the purpose of standardization, the finishing was done by a single operator (to prevent interoperator error), for a fixed period of time and using light pressure (to minimize intraoperator error) and only three samples were finished and polished per day (to minimize variation due to fatigue). Following this the samples were cleaned in an ultrasonic bath (Figure 15) for 10 min in distilled water and then air dried.

**Investing and casting of wax patterns for nickel-chromium samples**

Square shaped 8 wax patterns were sprued and invested at a time in a 6 × casting ring (Degussa, Germany) (Figure 16) using Deguvest powder (phosphate carbon free universal investment, Degudent, Germany) and Deguvest investment liquid (Degudent, Germany). Standard procedures were followed for sprue attachment, use of ring liner (Kera-Vlies, Germany), use of Debubbler (Begosol, Bego, Germany), mixing of investment (vacuum mixer, Multivac-4, Degussa, Germany), pouring of investment, wax burnout (Wax elimination furnace, KaVo EWL, Type 5645) and casting was done in an induction casting machine (Dentaurum Megapuls 3000, Germany, Figure 17) using 15 nickel-chromium pellets (MAARC, Figure 18) and (Wirloyloy, Figure 19) each. The castings were retrieved (Figure 20), the sprues were cut, and all samples were finished according to standard procedures (sandblasting-KaVo EWL Type 5417, carborundum discs for cutting sprues, tungsten carbide burs for removing surface irregularities and use of sequential rubber points for finishing and polishing. All the 30 samples were inspected for internal porosity by X-ray. In all, five samples with internal porosity were discarded, and equal numbers of samples were fabricated so that 30 nickel-chromium samples each without internal porosities were prepared. For the purpose of standardization, the finishing was done by a single operator (to prevent interoperator



Figure 13: Burn out procedure.



Figure 14: Titanium casting machine F210.



Figure 15: Ultrasonic cleaner.

error), for a fixed period of time and using light pressure (to minimize intraoperator error) and only three samples were finished and polished per day (to minimize variation due to fatigue). Following this the samples were cleaned in an ultrasonic bath for 10 min in distilled water and then air dried.



Figure 16: Investing of the wax patterns.



Figure 19: Wirrolloy.



Figure 17: Mega plus casting machine.



Figure 20: Cast specimens after sand blasting.

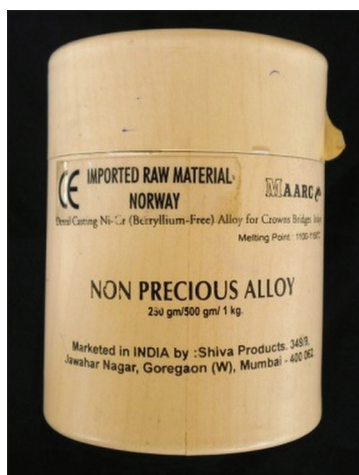


Figure 18: Machine-readable cataloging alloy.

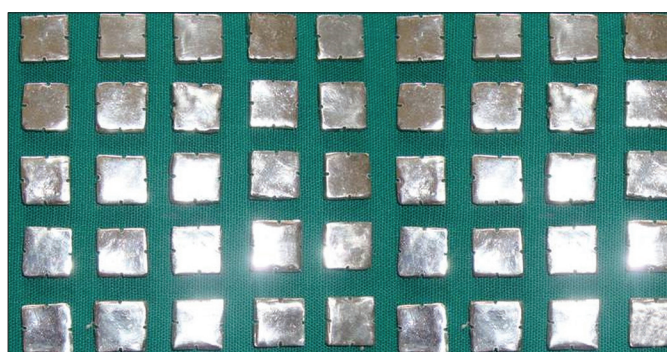


Figure 21: Finished and polished specimens.

### Grouping of the samples

These samples of titanium and nickel-chromium were each divided into three groups of 15 samples each (Figure 21). They are brushed for 48 h clinically simulating 2 years of brushing in a saline toothpaste medium.

### Measurement of surface roughness using profilometer (quantitative)

Profilometer (Wyko NT 1100, USA) (Figure 6) is a measuring instrument used to measure a surface profile, in order to quantify its roughness. Wyko NT 1100 optical profilometer was used for

the study. It had all the advantages of industry-standard Wyko optical profiling, including the full Wyko Vision 32<sup>®</sup> analytical software package, which was mainly used for applications in Micro-electro mechanical systems (MEMS), thick films, optics, ceramics and other advanced materials. This profilometer utilizes white light interferometry for high resolution three-dimensional surface measurements, from sub-nanometer roughness to millimeter-high steps. On super smooth or rough surfaces, the NT 1100 provides repeatable surface measurement for R&D, wear and failure analysis, and process control. This machine had a vertical measurement range of 0.1 nm to 1 mm, vertical resolution of <math><1\text{\AA}</math> Ra, Rms repeatability of 0.01 nm, vertical scan

speed up to 7.2 μm/s (288 μm/s), lateral spatial sampling of 0.08 to 13.1 μm, field-of-view: 8.24-0.05 mm (larger areas with data stitching option) and reflectivity of 1-100%.

The surface roughness Rz, average maximum height of the profile that is, The 10 highest and 10 lowest points were measured.

They were then subjected to SEMs and energy dispersive spectroscopy (EDS) analysis. All the samples from each group that is,<sup>10,13</sup>

- First group of nickel-chromium samples (MAARC) are brushed for 48 h in a saline toothpaste medium.
- Second group nickel-chromium samples (Wiroloy) are brushed for 48 h in a saline tooth paste medium.
- Third group titanium samples are brushed for 48 h in a toothpaste saline medium. Were subjected to profilometric analysis to estimate the roughness of the samples after their respective surface treatments. The results obtained were statistically analyzed using ANOVA (one way, Tukey *post-hoc* comparison  $\alpha = 0.05$ ). The level of significance was set at 5% ( $P < 0.05$ ).<sup>10,13</sup>

**Results**

In the present study, effect of prophylactic brushing on titanium and two nickel-based alloys (MAARC and Wiroloy) were investigated and compared. Surface roughness values (Ra and Rz) of each sample were recorded separately before and after the study.

**Data analysis**

The recorded surface roughness Ra and Rz values are given in the annexure. The values shown are the optical profilometer readings. The mean value and standard deviation (SD) were calculated. The data were then subjected to detailed statistical analysis.

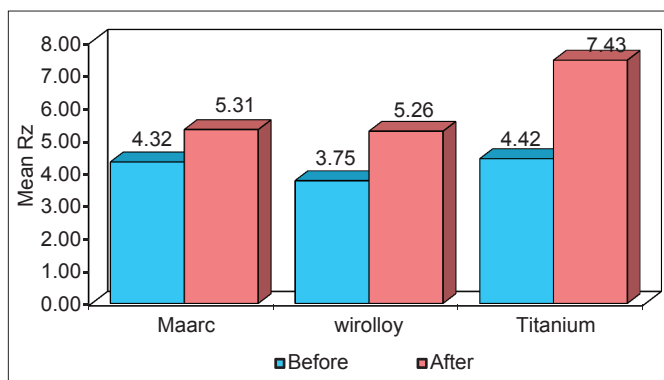
Mean and SD of Rz and Ra were calculated separately for titanium, MAARC and Wiroloy, then the data were subjected to ANOVA test to assess the difference in the surface roughness values of the samples before and after the study.

Subsequently pairwise comparisons were performed between the test groups by using the Tukey *post-hoc* procedure at level of significance ( $P < 0.05$ ).

Then the Rz and Ra values were subjected to Student's *t*-test separately for MAARC, Wiroloy and titanium samples for the readings of before and after the study.

**Evaluation of titanium samples**

The mean and SD of the surface roughness values obtained for each titanium specimen of the three groups have been given in the Table 1 and Graph 1. The mean and SD values of surface roughness Rz values of titanium samples before and after



**Graph 1:** Comparison of three groups with Rz values.

Group	Before		After		Difference	
	Means	SD	Means	SD	Means	SD
MAARC	4.32	0.88	5.31	0.99	0.98	0.95
Wiroloy	3.75	0.94	5.26	1.48	1.51	1.86
Titanium	4.42	1.01	7.43	1.94	3.01	2.33

SD: Standard deviation, MAARC: Machine-readable cataloguing. Ra: Represents the roughness average, the arithmetic mean of the absolute values of the surface departures from the mean plane. Rz: Average maximum height of the profile, it is the average of ten highest and ten lowest points on the data set

surface treatment were 4.42 ± 1.01 μm to 7.43 ± 1.94 μm with a difference of 3.01 ± 2.33 μm. The results are then compared group wise by ANOVA test. From the results of the Table 2, a significant difference in the Rz values of all the samples was seen after the test. Since the *F* value is significant, 9.9098 and  $P = 0.0003$ , to know the significant difference before and after of all groups, pairwise comparison analysis was done by Tukey *post-hoc* procedure.<sup>23-27</sup>

From the results of the Table 3, a significant difference is present in the titanium samples compared to that of the MAARC and Wiroloy samples. There was no significant difference in the values of the surface roughness between the groups before the study. However, after the study, there was a significant difference in the Rz values of titanium samples when compared to that of nickel chromium alloy samples. To know the difference in the values of the titanium samples before and after Student's paired *t*-test was carried out. Table 4 presents the comparison of before and after Rz values in three groups by paired *t*-test. Results show that there is a significant change in the Rz values of titanium samples. To further evaluate the surface roughness, Ra values were also statistically analyzed.

The mean and standard deviation of the surface roughness Ra values obtained for each titanium specimen of the three groups have been given in the Table 5 and Graph 2. The mean and SD values of surface roughness Ra values of titanium samples before and after surface treatment were 526.07 nm ± 169.45 nm to 824.05 nm ± 221.46 nm with a difference of 297.97 ± 278.44 nm. The results are then compared group wise by ANOVA test. From the results of the Table 6, a significant

**Table 2: Comparison of three groups with Rz values by ANOVA test.**

Variable	Source of variation	Degrees of freedom	Sum of squares	Mean sum of squares	F value	P value
Before	Between groups	2	3.87	1.94	2.1672	0.1271
	Within groups	42	37.53	0.89		
	Total	44	41.40			
After	Between groups	2	45.94	22.97	9.9098	0.0003*
	Within groups	42	97.35	2.32		
	Total	44	143.29			
Difference	Between groups	2	33.28	16.64	5.1032	0.0104*
	Within groups	42	136.97	3.26		
	Total	44	170.26			

\*Significant at 5% level of significance (P<0.05). P value: Probability, t: Absolute t-value, Ra: Represents the roughness average, the arithmetic mean of the absolute values of the surface departures from the mean plane. Rz: Average maximum height of the profile, it is the average of ten highest and ten lowest points on the data set

**Table 3: Pair wise comparison by Tukeys post-hoc procedure of Rz values.**

	Group	MAARC	Wiroloy	Titanium
Before	Mean	4.3247	3.7527	4.4153
	MAARC	-	-	-
	Wiroloy	0.2336	-	-
	Titanium	0.9628	0.1457	-
After	Mean	5.3093	5.2647	7.4300
	MAARC	-	-	-
	Wiroloy	0.9965	-	-
	Titanium	0.0014*	0.0011*	-
Difference	Mean	0.9847	1.5120	3.0147
	MAARC	-	-	-
	Wiroloy	0.7053	-	-
	Titanium	0.0101*	0.0700	-

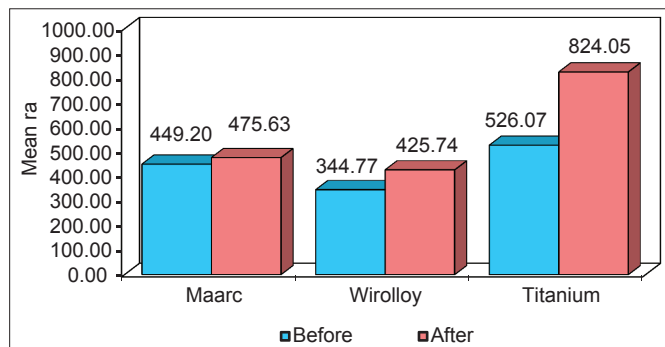
\*Significant at 5% level of significance (P<0.05). MAARC: Machine-readable cataloging. Ra: Represents the roughness average, the arithmetic mean of the absolute values of the surface departures from the mean plane. Rz: Average maximum height of the profile, it is the average of ten highest and ten lowest points on the data set

difference in the Ra values of all the samples was seen after the test. Since the F value is significant, 28.6088 and P value 0.0000 to know the significant difference before and after of all groups, pairwise comparison analysis was done by Tukey post-hoc procedure. From the results of the Table 7, a significant difference is present in the titanium samples compared to that of the MAARC and Wiroloy samples. There was statistically significant difference in the values between titanium samples and Wiroloy samples before the study. But, after the study there was a significant difference in the Ra values compared to that of both groups of nickel chromium alloy samples.

To know the difference in the values of the titanium samples before and after Student's paired t-test was carried out. Table 8 presents the comparison of before and after Ra values in three groups by paired t-test. Results show that there is a significant change in the Ra values of titanium samples with P value 0.0010.

*Evaluation of nickel-chromium samples*

The mean and SD of the surface roughness values obtained for each nickel-chromium samples of the three groups have been given in the Table 1 and Graph 1. The mean and SD values of surface roughness Rz values of MAARC samples before and after surface treatment were 4.32 ± 0.88 um to 5.31 um ± 0.99 um



**Graph 2: Comparison of three groups with Ra values.**

with a difference of 0.98 um ± 0.95 um, and of Wiroloy samples 3.75 ± 0.94 um to 5.26 um ± 1.48 um with a difference of 1.51 um ± 1.86 um the results are then compared group wise by ANOVA test. From the results of the Table 2, a significant difference in the Rz values of all the samples was seen after the test.

Since the F value is significant, 9.9098 and P value 0.0003 to know the significant difference before and after of all groups, pairwise comparison analysis was done by Tukey post-hoc procedure.

From the results of the Table 3, a significant difference is present in the titanium samples compared to that of the MAARC and Wiroloy samples. There was no significant difference in the values between the groups before the study. Even after the study, there was no significant difference in the Rz values between MAARC and Wiroloy samples.

To know the difference in the values of the nickel-chromium samples before and after Student's paired t-test was carried out. Table 4 presents the comparison of before and after Rz values in three groups by paired t-test. Results show that there is a significant change in the Rz values of these samples. To further evaluate the surface roughness, Ra values were also statistically analyzed.

The mean and SD of the surface roughness values obtained for nickel-chromium alloys of the three groups have been given in the Table 5. The mean and SD values of surface roughness Ra



Table 4: Comparison of before and after Rz values in three groups by Student's paired t-test.

Group	Time	Mean	SD	Mean difference	SD difference	Paired t-value	P-value
MAARC	Before	4.3247	0.8758	-0.98	0.95	-4.0348	0.0012*
	After	5.3093	0.9894				
Wiroloy	Before	3.7527	0.9425	-1.51	1.86	-3.1458	0.0072*
	After	5.2647	1.4813				
Titanium	Before	4.4153	1.0124	-3.01	2.33	-5.0128	0.0001*
	After	7.4300	1.9444				

\*Significant at 5% level of significance (P<0.05). MAARC: Machine-readable cataloging, P-value: Probability, t: Absolute t-value, Ra: Represents the roughness average, the arithmetic mean of the absolute values of the surface departures from the mean plane. Rz: Average maximum height of the profile, it is the average of ten highest and ten lowest points on the data set

Table 5: Mean and SD of Ra according to groups.

Group	Before		After		Difference	
	Means	SD	Means	SD	Means	SD
MAARC	449.20	168.31	475.63	117.45	26.44	149.00
Wiroloy	344.77	114.22	425.74	105.97	80.97	132.64
Titanium	526.07	169.45	824.05	221.46	297.97	278.44

SD: Standard deviation, MAARC: Machine-readable cataloging, Ra: Represents the roughness average, the arithmetic mean of the absolute values of the surface departures from the mean plane. Rz: Average maximum height of the profile, it is the average of ten highest and ten lowest points on the data set

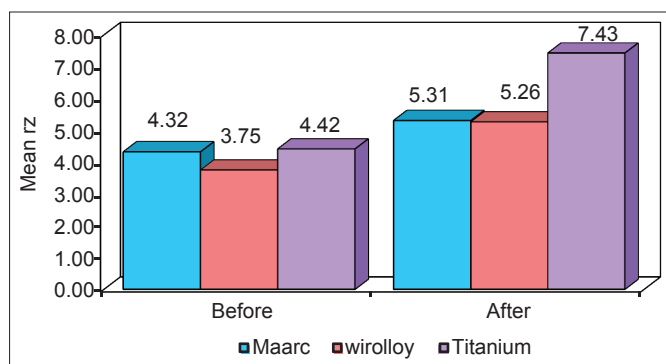
values of MAARC samples before and after surface treatment were 449.20 ± 168.31 nm to 475.63 nm ± 117.45 nm with a difference of 26.44 nm ± 149.00 nm, and of Wiroloy samples 344.77 nm ± 114.22 nm to 425.74 nm ± 105.97 nm with a difference of 80.97 nm ± 132 nm as given in the Graph 2. The results are then compared group wise by ANOVA test. From the results of the Table 6, a significant difference in the Ra values of all the samples was seen before and after the test. Since the F value is significant, 28.6088 and P = 0.0000 to know the significant difference before and after of all groups, pairwise comparison analysis was done by Tukey post-hoc procedure.

From the results of the Table 7, a significant difference is present in the titanium samples compared to that of the MAARC and Wiroloy samples. There was statistically insignificant difference in the values between MAARC samples and Wiroloy samples both before and after the study. But, after the study there was a significant difference in the Ra values of titanium samples compared to that of both groups of nickel-chromium alloy samples.

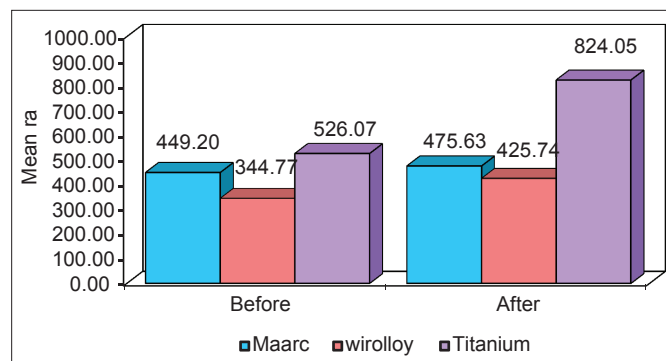
To know the difference in the values of the nickel-chromium samples before and after the study Student's paired t - test was carried out. Table 8 presents the comparison of before and after Ra values in three groups by paired t-test.

Comparison between titanium and nickel-chromium samples

The difference in mean surface roughness scores Rz and Ra values of titanium and nickel-chromium before and after the study is presented in the Tables 1 and 5, Graphs 3 and 4. The results showed that difference in mean surface roughness before and after treatment of titanium samples



Graph 3: Comparison of three groups with Rz values.



Graph 4: Comparison of before and after Ra values in three groups.

was significantly higher as compared to the nickel-chromium samples.

From the results of the Tables 3 and 7, a significant difference of surface roughness of both Ra and Rz values is present in the titanium samples compared to that of the MAARC and Wiroloy samples.

The results thus indicated that titanium is more susceptible to the surface roughness when subjected to the clinically simulated 2 years of prophylactic brushing as compared to nickel-chromium.

Discussion

The bristle tooth brush appeared about the year 1600 in china, was first patented in America in the year 1857, and has since undergone little change. Generally, toothbrushes vary in size and design, as well as in the length, hardness, and arrangement of the bristles. The American dental association

**Table 6: Comparison of three groups with Ra values by ANOVA test.**

Variable	Source of variation	Degrees of freedom	Sum of squares	Mean sum of squares	F-value	P-value
Before	Between groups	2	248446.0	124223.0	5.3172	0.0087*
	Within groups	42	981232.1	23362.7		
	Total	44	1229678.1			
After	Between groups	2	1412663.9	706332.0	28.6088	0.0000*
	Within groups	42	1036950.9	24689.3		
	Total	44	2449614.8			
Difference	Between groups	2	618980.3	309490.2	7.9139	0.0012*
	Within groups	42	1642505.7	39107.3		
	Total	44	2261486.0			

\*Significant at 5% level of significance (P<0.05). P-value: Probability, t: Absolute t-value, Ra: Represents the roughness average, the arithmetic mean of the absolute values of the surface departures from the mean plane. Rz: Average maximum height of the profile, it is the average of ten highest and ten lowest points on the data set

**Table 7: Pair wise comparison by Tukeys post-hoc procedure of Ra values.**

	Group	MAARC	Wiroloy	Titanium
Before	Mean	449.20	344.77	526.07
	MAARC	-	-	-
	Wiroloy	0.1598	-	-
	Titanium	0.3618	0.0064*	-
After	Mean	475.63	425.74	824.05
	MAARC	-	-	-
	Wiroloy	0.6623	-	-
	Titanium	0.0001*	0.0001*	-
Difference	Mean	26.44	80.98	297.97
	MAARC	-	-	-
	Wiroloy	0.7322	-	-
	Titanium	0.0016*	0.0123*	-

\*Significant at 5% level of significance (P<0.05). SD: Standard deviation, MAARC: Machine-readable cataloging, Ra: Represents the roughness average, the arithmetic mean of the absolute values of the surface departures from the mean plane. Rz: Average maximum height of the profile, it is the average of ten highest and ten lowest points on the data set

has described the range of the dimensions of acceptable brushes; these have a brushing surface of 25.4-31.8 mm long and 7.9-9.5 mm wide, two to four rows of bristles and 5-12 tufts per row. Here, for the study a tooth brush (anchor) whose bristle transplanted portion was 20 mm in length and 6 mm in width was automatically reciprocated over a stroke of 10 mm and at 90 strokes/min for 48 h. Multi tufted toothbrushes contain more bristles and may clean more efficiently than the skimpier brushes. So for the study multi tufted toothbrush with contra angled shanks are used with a constant force of 200 g over the sample. Diameters of commonly used bristles range from 0.007 inch (0.2 mm) for soft brushes to 0.012 inch (0.3 mm) for medium brushes and 0.014 inch (0.4 mm) for hard brushes. Opinions regarding the merits of the hard and soft brushes are based on the studies carried out under different conditions, these studies are often inconclusive and often contradict each other. Soft brushes are more flexible, clean beneath the gingival margin (sulcus brushing), and reach further into the proximal tooth surfaces. Use of the hard bristle tooth brushes is associated with gingival recession than frequent brushers who use soft toothbrushes. However, the manner in which a brush is used, and the abrasiveness of the dentifrice affect the action of the abrasion to a greater degree than the bristle hardness itself.

Brush hardness does not significantly affect the wear on the enamel surfaces.<sup>28</sup> So for this study to minimize the effects of the both a toothbrush of medium abrasive quality is used.

A dentifrice is usually used in combination with tooth brushing with the purpose of facilitating the plaque removal and applying agents to the tooth surfaces for therapeutic or preventive reasons. The addition of the abrasives facilitates plaque and stain removal. Fluoride is usually omnipresent in commercially available toothpastes.<sup>29</sup> Dentifrice used in the study is anchor white toothpaste, which is of medium abrasivity. However, restorative materials undergo mechanical degradation due to the abrading action of dentifrice. In the long-term, the abrading effect causes biological disadvantages, such as accumulation of dental plaque and increased elemental release associated with the toxicity.<sup>2</sup>

In dentistry, alloys with nickel contents of ≥50% are commonly used for definitive prosthodontic restorations because of their high strength, high modulus of elasticity and hardness. These alloys are less expensive than gold or palladium based alternatives, and increased costs of palladium and gold in the recent years have amplified this cost advantage and promoted an increased use of nickel-based alloys. In spite of excellent mechanical properties, the biocompatibility of nickel-based alloys is perennially questioned because nickel alloys corrode significantly and affect biological systems. Because corrosion results in elemental release, nickel-based alloys release nickel ions *in vitro* and *in vivo*, and some evidence supports the theory that nickel is preferentially released from many nickel-based alloys. Dental restorations comprised of these alloys are often in intimate contact with the oral tissues. Tissue concentrations of nickel (normally <0.01 μm), have been estimated to reach 20-2000 μm, depending on the *in vivo* model. The biological effects of released nickel ions at these concentrations include hypersensitivity, inflammation and necrosis. The susceptibility of some nickel-based alloys to chemical and mechanical stresses suggest that common intraoral stresses such as tooth brushing may adversely affect the surfaces of these alloys.<sup>22</sup> Hence, the purpose of this current study was to evaluate the brushing induced surface

Table 8: Comparison of before and after Ra values in three groups by Student's paired t-test.

Group	Time	Mean	SD	Mean difference	SD difference	Paired t-value	P-value
MAARC	Before	449.1967	168.3081	-26.44	149.00	-0.6872	0.5032
	After	475.6327	117.4462				
Wiroloy	Before	344.7653	114.2177	-80.97	132.64	-2.3645	0.0330*
	After	425.7400	105.9700				
Titanium	Before	526.0747	169.4541	-297.97	278.44	-4.1447	0.0010*
	After	824.0487	221.4603				

\*Significant at 5% level of significance ( $P < 0.05$ ). SD: Standard deviation, MAARC: Machine-readable cataloging, P value: Probability, t: Absolute t-value, Ra: Represents the roughness average, the arithmetic mean of the absolute values of the surface departures from the mean plane. Rz: Average maximum height of the profile, it is the average of ten highest and ten lowest points on the data set

roughness of two commonly used commercially available MAARC and Wiroloy nickel-chromium alloys. The study further involves Cp titanium as it is more biocompatible compared with-chromium alloys.

Clinical applications of titanium in medical and dental practices have often been documented with a great attention on its excellent biocompatibility. With the development in dental casting for titanium, the metal has been used for crowns, fixed and removable partial denture frameworks. Orthodontic brackets made up of pure titanium have been well-accepted, especially for patients with an allergy to nickel and other specific substances. The distinguished biocompatibility of titanium is mainly attributed to the surface oxide film that has excellent resistance to corrosion. The high corrosion resistance of titanium is due to the formation of a dense and stable layer of titanium oxide on its surface. Titanium oxide is responsible for chemical stability in the human body. This layer is formed quickly because of the reactivity of the titanium with oxygen, which originates several oxides, with  $TiO_2$  being the major oxide formed. The thickness of this layer ranges between 10 and 20 nm, and must not be disrupted under any condition. Oxidative processes can thicken and condense the titanium oxide layer on the surface, improving the corrosion stability of the underlying titanium. On the other hand, reducing agents, such as fluoride ( $F^-$ ), may have the opposite effect and attack this layer.<sup>30,31</sup> Strietzel *et al.*<sup>32</sup> demonstrated that titanium ion release was enhanced in the presence of  $F^-$  and this effect was even further accelerated at low pH. High fluoride ion concentrations and an acidic pH are known to impair the corrosion resistance of titanium, and as a result crevice and pitting corrosion occur. However, the main drawback is poor resistance to wear. Most of the studies relating to the dentifrice effects on titanium surface has been involved in the corrosive action of the fluoride added to toothpaste, taking the corrosion aspect into account, Siirila and Kononen had claimed that the abrasion caused by tooth bristles rather than the effect of the additive fluoride (0.125%), was the main deteriorating factor for titanium. The personal dental hygiene procedure with brush has been reported to produce superficial grooves on the titanium implant abutments.

On the other hand, the abrading and polishing process of titanium brought about mechanical surface disturbance

accompanied by alteration of the surface composition. Such surface disturbance and resultant chemical alterations were also expected to occur during tooth brushing with dentifrice. The chemically altered surface may have biological responses rather than that have been delivered for titanium with spontaneous oxide film. Therefore, brushed titanium surface needs to be chemically characterized from the view point of long-term biocompatibility.

Therefore, this study is undertaken in order to draw attention toward the potential effect of prophylactic brushing with a medium abrasive toothbrush and paste in a saline medium.

This was felt essential because the surface defects caused, may lead to increased plaque, calculus and microbial retention on their surfaces. Studies on the corrosion resistance of titanium are generally performed by incubating titanium in saline or artificial saliva. In such solutions, titanium exhibits a superior corrosion resistance due to the presence of a stable and dense titanium oxide film that spontaneously covers the metal surface. However, despite its excellent corrosion resistance when tested *in vitro*, titanium has been reported to be sensitive to prophylactic brushing. Hence, this study was conducted *in vitro*.

In the present conducted study, specimens in equal dimensions of  $10 \times 10 \times 2$  mm were cast in titanium Cp Grade II and nickel-chromium. All the cast specimens were subjected to radiographic examination to detect internal porosities with a simple routine dental X-ray as proposed by Wang and Boyle.<sup>33</sup> Samples with obvious porosities were eliminated, and equal numbers were added. After sandblasting all the samples were surface grounded with titanium finishing bur in one direction with light strokes to remove the alpha-case layer.<sup>34</sup> Following this the samples were cleaned in an ultrasonic bath for 10 min in distilled water and then air dried.<sup>35</sup>

Based on its usage in 2 years, the specimens of titanium and nickel-chromium samples were brushed for 48 h in a saline toothpaste medium simulating 2 years of brushing, 2 min per session under 200 g load clinically simulating 2 years of brushing.

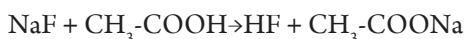
The surface roughness on the samples was quantitatively analyzed with an optical profilometer (Wyko surface profiler,

USA) as it has the following advantages over a contact profilometer:

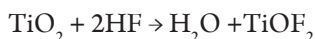
1. Speed: Because the non-contact profilometer does not touch the surface, the scan speeds are dictated by the light reflected from the surface and the speed of the acquisition electronics
2. Reliability: Optical profilometer does not touch the surface and therefore cannot be damaged by surface wear or careless operators
3. Spot size: The spot size, or lateral resolution, of optical methods ranges from a few micrometers down to submicrometer.

The specimens were then further analyzed qualitatively with SEM and EDS analysis.

From the observations, it is clear that the titanium samples have undergone a significant change in the surface morphology. This may be attributed to the mechanical action of the tooth bristles and the fluoride in the toothpaste as there is a reaction of the fluoride ions with the surface oxides on the titanium. This phenomenon is interpreted as being the result of incorporation of fluoride ions in the oxide layer, whose protective properties are considerably reduced. A low quantity of fluoride, in the presence of acidic medium, induces the following chemical reaction:



The fluorhydric acid molecules can react on titanium oxide:

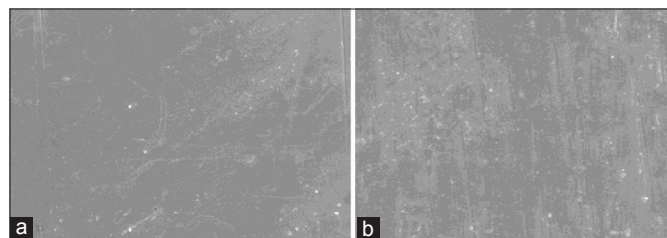


to give rise to titanium oxyfluorides in solid state. The lattice parameter of these compounds is such that they induce many structural defects in the oxide coating, whose protection is considerably loosened.<sup>36</sup>

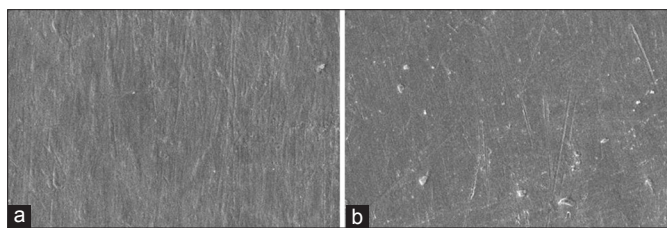
Thus, the above discussed studies and the present study have the concurrent finding of titanium being susceptible to corrosion and surface roughness when subjected to the prophylactic toothpaste containing fluoride. Whereas there was no statistical significant change in the Ra values of the MAARC alloy test samples before and after the study. This can be justified by the fact that chromium in nickel-chromium alloys hinders the active dissolution of the nickel matrix, by the formation of an adherent passivating surface film.<sup>37,38</sup>

#### Scanning electron microscope observations

Titanium and nickel-chromium specimens were randomly selected from each group and analyzed by SEM examination (Figures 22 and 23). Specimens of titanium and nickel-chromium before the study showed surface irregularities and scratches produced during finishing and polishing procedures.



**Figure 22:** Scanning electron microscope (SEM) photomicrograph (a and b -  $\times 500$ ) of titanium and nickel-chromium specimen before the study was conducted. (a) Titanium, (b) Nickel-chromium: Surface irregularities and scratches produced during finishing and polishing are observed in these photomicrographs.



**Figure 23:** Scanning electron microscope photomicrograph (a and b -  $\times 500$ ) of titanium and nickel-chromium specimen after the study was conducted these represent the morphology images of the corresponding brushed surfaces. (a) Titanium: Brushed surface appears to be more roughened compared to that of (b) Nickel-chromium alloy.

The titanium specimen after the study showed irregular surface. The irregularities on the surface roughness may have been produced due to the prophylactic brushing and dentifrice.

On the other hand, the nickel-chromium specimen showed only surface irregularities and scratches produced during finishing and polishing without any much changes after the study, which indicates when compared to the titanium nickel-chromium is more wear resistance.

#### Energy dispersive spectrometer observations

The EDS is an analytical technique used for the elemental analysis or chemical characterization of a sample. Its characterization capabilities in large part are due to the fundamental principle that each element has a unique atomic structure allowing X-rays that are characteristic of an element's atomic structure to be identified from each other. However, the EDS analysis done in the present study can only detect elements having atomic number equal to or more than 11. Thus, detection of elements by the EDS analysis conducted in the present study starts from sodium in the periodic table. Fluoride, which has an atomic number of nine, is therefore not detected.

Energy dispersive spectroscopy analysis of both titanium specimen and nickel-chromium alloys show that there is no

significant change in the surface composition of elements before and after the study.

Limitations of the study are as follows,

- During the preparation of the test specimens, routine procedures such as removal of alpha layer, sandblasting, finishing and polishing might have caused little variation in the surface roughness
- Limited numbers of samples were used with each group consisting of 15 cast titanium and two different commercial nickel-chromium specimens each
- In clinical situations, the actual duration and pressure of brushing will be different as it differs from patients. It differs from right handed persons to left handed
- The results may vary with usage of different designs of tooth brushes and dentifrices
- The EDS analysis used was incapable of detecting fluoride due to its lower atomic number.

Further scope of the study is as follows,

- Effect of different dentifrices such as ayurvedic toothpastes can be evaluated on the nickel-chromium and titanium alloys
- Effect of brushing with dentifrices can be evaluated on other restorative materials such as cobalt chromium, amalgam, composites and ceramic restorations glazed and unglazed
- Surface roughness and corrosion caused on these restorations may lead to increased adherence of microbial flora which can be further evaluated
- Effect of fluoridated and non-fluoridated toothpastes can be evaluated on titanium as it is very vulnerable to fluorides
- Effect of brushing in various acidic pH conditions on titanium and nickel-chromium alloys can be evaluated
- Corrosion behavior between titanium implant and nickel-chromium abutments can be further evaluated, if nickel-chromium alloys are planned for implant abutments.

Implications of the results of the study are as follows,

- This study highlighted the detrimental effect of using fluoridated toothpaste prophylactic brushing on the surface on titanium and also to a certain extent, on nickel-chromium. Based on the findings of this *in vitro* study, the following considerations need be noted by the clinician
- The surface roughness caused by brushing may lead to increased plaque and calculus retention and may distract from aesthetics
- The alloys used for implant abutments should be carefully selected as the surface roughness over the abutments may lead to periimplantitis and thereby failure of the same
- Patients who have titanium and nickel-chromium restorations should be properly educated about the type of brush, dentifrice, duration and technique of brushing. This will become more important as a growing number of patients are treated with titanium implants and dental restorations
- Precision attachments and fastening screws may be damaged due to surface abrasion with faulty tooth brushing habits

- Dentifrices with lower abrasivity may be advisable for patients with the dental titanium device.

### Conclusion

Within the limitations of the present study and based on the results obtained, the following conclusions may be drawn:

- Titanium surface was abraded and roughened more compared to that of nickel-chromium alloys when subjected to the clinically simulated 2 years of prophylactic brushing under 200 g of standard load
- The surface composition of all the samples was altered probably because of reactions with the abrasive material
- There was no statistical difference in the surface roughness Ra value of MAARC alloy before and after this *in vitro* study
- Comparison between the surface roughness of titanium and nickel-chromium revealed that titanium is more susceptible to corrosion and abrasion than nickel-chromium.

Therefore, within the limitations of the study it is concluded that prophylactic brushing with the fluoridated toothpaste have an effect on the surface roughness of titanium and also to a certain extent, on nickel-chromium. Therefore, careful consideration must be given to the selection of the toothbrushes and toothpastes with the medium abrasivity in patients with these restorations.

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