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# *In Vitro:* Influence of Zirconium Coatings on Surface Roughness of (Beta-Titanium III and Timolium Titanium) Orthodontic Archwires

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**Abstract** The main purpose of this study was to investigate the significance of Zr Coatings and its appropriate thickness on surface roughness of the beta-titanium orthodontic archwires (β-Ti III) and timolium titanium archwires (TIM), Layers of Zr were deposited on the orthodontic archwires and compared the surface roughness before and after Zr deposition. The surface roughness was examined before and after Zr deposition, using scanning electron microscopy (SEM) and atomic force microscopy (AFM). Two commercially available orthodontic archwires were used in this research, namely, β-Ti III and TIM orthodontic archwires. The archwires were cut to 25 mm length specimens. In this study, the electron beam physical vapor deposition (EB PVD) technique was implemented to coat four various thicknesses ( 5, 10, 25, and 50 nm) of pure Zr on selected archwires and the results thereof were investigated using AFM, SEM. SEM and AFM analysis tests of Zr-coated archwires showed significant improvement in their surface roughness compared with uncoated archwires. (TIM) Zr-coated archwires showed lower surface roughness than (β-Ti III) Zr-coated archwires. The improvement of coatings Zr with 50 nm thick was more reliable than that of coatings Zr with 5, 10, and 25 nm. It is concluded that the most available commercial product for orthodontic archwires could be improved by the surface deposition of Zr. coating 50 nm thick of Zr on β-Ti III and TIM orthodontic archwires is recommended for even sliding mechanics due to the resulting reduced surface roughness.

**Keywords:** Surface roughness, Zirconium (Zr), Beta titanium orthodontic archwires (β-Ti III), Timolium titanium archwires (TIM), Electron beam-physical vapour deposition (EB-PVD).

# تأثير طلاءات الزركونيوم على الخشونة السطحية لأسلاك تقويم الأسنان
# (بيتا تيتانيوم IIIو تيموليوم تيتانيوم)

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**الملخص** الغرض الرئيسي من هذه الدراسة هو التحقق من أهمية طلاءات Zr وسمكها المناسب على خشونة السطح لأسلاك تقويم الأسنان بيتا تيتانيوم (β-Ti III) وأسلاك تيموليوم تيتانيوم (TIM) ، وترسب طبقات Zr على الأسلاك تقييم خشونة السطح قبل وبعد ترسيب .Zr تم فحص خشونة السطح قبل وبعد ترسيب Zr ، باستخدام الفحص المجهري الإلكتروني (SEM) والفحص المجهري للقوة الذرية .(AFM) الطريقة: تم استخدام سلكين لتقويم الأسنان متوافران تجارياً في هذا البحث، وهما β-Ti III و TIM لتقويم الأسنان. تم قطع الأسلاك إلى عينات بطول 25 مم. في هذه الدراسة، تم تنفيذ تقنية ترسيب البخار الفيزيائي للحزمة الإلكترونية (EB PVD) لطلاء بسمك مختلفة (5 و 10 و 25 و 50 نانومتر) من Zr النقي على أسلاك التقويم المختارة وتم التحقيق في نتائجها باستخدام AFM و .SEM النتائج: أظهرت اختبارات تحليل SEM و AFM للأسلاك التقويم المغلفة Zr تحسناً كبيراً في خشونة السطح مقارنة بالأسلاك المقوسة غير المطلية. أظهرت (TIM)الأسلاك المقوسة المغلفة Zr خشونة السطح أقل من (β-Ti III) الأسلاك المقوسة المغلفة .Zr كان تحسين الطلاء Zr بسماكة 50 نانومتر أكثر موثوقية من الطلاء Zr مع 5 و 10 و 25 نانومتر. الاستنتاجات: استنتج أن أكثر المنتجات التجارية المتاحة للأسلاك تقويم الأسنان يمكن تحسينها عن طريق الترسيب السطحي ل .Zr يوصى بطلاء 50 نانومتر من Zr على β-Ti III و TIMحتى تحسن من ميكانيكيه الانزلاق بسبب انخفاض خشونة السطح الناتج من الطلاء ب.Zr .

**الكلمات المفتاحية:** خشونة السطح، الزركونيوم(Zr) ، أسلاك بيتا تيتانيوم لتقويم الأسنان(β-Ti III) ، أسلاك تيموليوم تيتانيوم لتقويم الأسنان (TIM)، ترسيب بخار شعاع الإلكترون المادي (EB-PVD) .

## Introduction

Biomaterials based on metals and metals alloys are widely used in both medical and dental applications [1]. several metals passively used as substitutes for hard tissues as dental implants and also can be used as fracture healing aids (screws, bone plates, orthodontic archwires, and brackets) [2,3 and 4]. The most extensively used metallic biomaterials are pure Ti and Ti alloys, stainless steel (SS), and chromium-cobalt (Cr-Co) alloys [5-6]. The β-Ti III archwires have a composition of 78% Ti, 11.5% Mo, 6.0% Zr, and 4.5% Sn. Al, V, Mn, and Cr are four elements which are common to many α and α+β alloys, Timolium (TIM) is an advanced technology titanium archwire. It contains Ti (89.9%), Al (6.1%), and V (3.2%), which confirms that this product is a mixture of α and β phases [7]. Orthodontic archwires are made from different alloys. It is presently possible to level phases of treatment with orthodontic archwires according to its mechanical characteristics. On this foundation, the titanium molybdenum alloys (TMA) beta phase produces an attractive combination of flexibility and strength when used as orthodontic archwires to apply biomechanical forces that affect tooth movement [8]. Recently it has gained extended popularity in orthodontic treatment. However, there are disadvantages associated with the use of orthodontic archwires, such as high surface roughness, which increases friction at the archwire-brackets interface during the sliding process [9-10]. The surface roughness of dental materials is of utmost importance and one of the characteristics of orthodontic wires, which affects various factors such as friction, the amount of microbial plaque accumulation and corrosion of wires, and each of the above-mentioned variables, in turn, has a role in orthodontic treatments. Friction affects the sliding movements of the bracket and wire [11]. Characteristics such as good load deflection, tensile strengths, hardness, and low modulus of elasticity and resistance against corrosion & wear determine the area of the contact surface, thereby influencing the friction. Saliva, as it contains bacteria, viruses, yeast, fungi, and their products [12-13], may cause corrosion of orthodontic appliances [14]. The formatted of alloys which used in orthodontic appliances are a passive oxide film so it can resist corrosion but this layer can be disrupted by mechanical attacks and chemical since it is not ideal. [14-15]. Many approaches have been used to overcome the aforementioned disadvantages. However, some various materials and techniques can be used for surface coating of orthodontic archwires, as well as modifying the surface of archwires and brackets, which are among those strategies developed to enhance both the mechanical and biological properties of metals used in orthodontics [1],[16]. The main objective of this study was to improve the surface roughness by Deposit four different layer thicknesses (5, 10, 25 and 50 nm) of Zr on the two types of Ti-alloyed archwires and Investigate the structure of their composition and the surface roughness, before and after Zr deposition, using scanning electron microscopy (SEM) and atomic force microscopy (AFM).

## Materials and Methods

### 1. Materials

Two orthodontic archwires were procured. Rectangular β-Ti III orthodontic archwires 0.48×0.64 mm were purchased from 3M Unitek® (USA) and TIM orthodontic archwires 3M (0.40×0.55 mm) were purchased from TP Orthodontics® (USA). Pure Zr was obtained from Good Fellow® of the United Kingdom through local agencies in Cape Town, South Africa.

**2. Electron beam–physical vapour deposition (EB-PVD)** The β-Ti III and TIM orthodontic archwires were cut into 25-mm-long from the straight ends of the archwire. The EB-PVD technique was applied in this study to coat orthodontic archwires with pure Zr in different thickness 5, 10, 25 and 50 nm on selected archwires. Coatings produced by the Electron beam-physical vapor deposition (EB-PVD) process usually have a good surface finish and a uniform microstructure [17]. An EB-PVD instrument made in Pennsylvania, USA was used. β-Ti III and TIM orthodontic archwires were substrates, the target material in this case was pure Zr. The Zr was placed into the crucible and the β-Ti orthodontic archwires substrates were mounted into the e-beam evaporator. The machine was closed and evacuated for 24hr. Thereafter, the e-beam deposition of Zr onto the β-Ti orthodontic archwires substrates was completed. One Zr deposition of the same duration was done on β-Ti III and TIM orthodontic archwire substrates. This experiment was repeated under different deposition rate of 5, 10, 25 and 50 nm Zr, within the ranges from 0.6 Å/sec to 1.2 Å/sec. The vacuum pressure was approximately 2×10-6 mbar and the current 180 mA. In order to better simulate the oral cavity environment, samples were be immersed in artificial saliva (pH 5) at 37 °C for 28 days [18].

### 3. Analytical techniques

**3.1 Atomic force microscopy (AFM)** The AFM was obtained from Surface imaging Systems, Veeco, Germany. The AFM determines the surface topography of a specimen with a sharp probe, while observing the interaction forces acting between the specimen surface and probe .The specimen do not require any treatment, the AFM can provide quantitative values for the investigated parameters. AFM was studied further to see the shape of the crystals and determine the influence of deposited Zr on the selected archwires surfaces. AFM was used to study the surface morphology of the layers on the β-Ti/TIM treated and non-treated archwires. It was at into 14 mm lengths with a probe from the instrument in order to provide the required data. Use was made of the east-AFM software to translate the data of the high resolution 3-D surface images of the archwires.

**3.2 Scanning electron microscopy (SEM)** A FEI Nova Nano SEM 230 instrument, (Eindhoven, The Netherlands), was used to analyses samples. The images were recorded at a 34° angle and the working height 5 mm. An area with a positive radius of curvature increases the chances of a secondary electron escaping and vice versa, while regions with negative radius of curvature reduces the secondary electron current. Since the intensity of the image depends upon the number of secondary electrons reaching the detector, secondary electron imaging provides topographic images of rough surfaces [19].

## Results

Topographic irregularities were observed in all the archwires tested Fig 1 and 2 show three-dimensional AFM topography images (5×5 nm) of all archwires analyzed. All archwires show significant changes in roughness between the as-received and the Zr coated state.

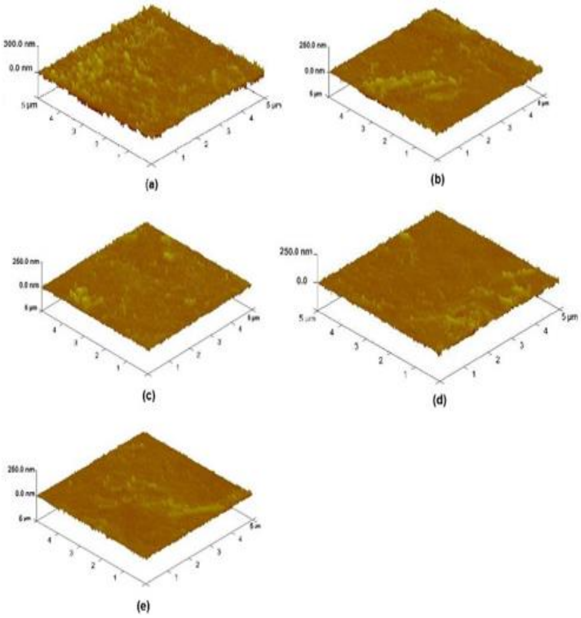

**Figure 1:** Results of AFM shown surfaces roughness of (a) Uncoated β-Ti III archwire (b) Coated β-Ti III archwire with 5nm of Zr (c) Coated β-Ti III archwire with 10 nm of Zr (d) Coated β-Ti III archwire with 25 nm of Zr (e) Coated β-Ti III archwire with 50 nm of Zr.

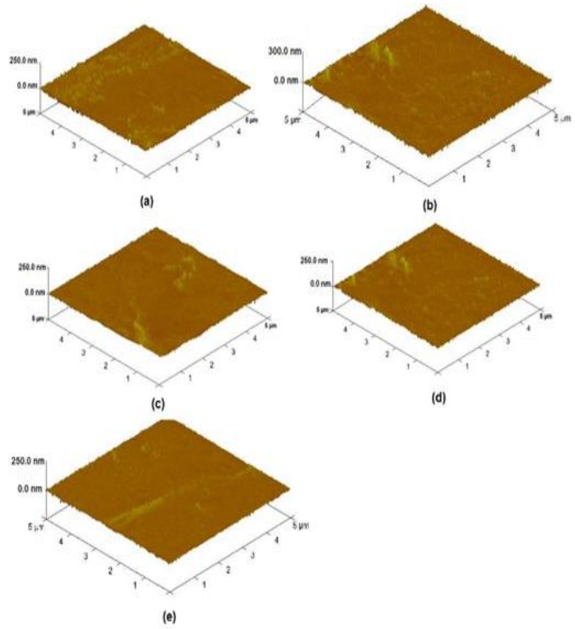

**Figure 2:** Results of AFM shown surfaces roughness of (a) Uncoated TIM archwire (b) Coated TIM archwire with 5nm of Zr (c) Coated TIM archwire with 10 nm of Zr (d) ) Coated TIM archwire with 25 nm of Zr (e) Coated TIM archwire with 50 nm of Zr.

The effect of Zr coating on Surface Roughness of (β-Ti III and TIM) is shown in Fig 3, 4. From the figures, it is clear that the coating with Zr decreases the Surface Roughness. As the thicknesses of Zr were increased the Surface Roughness decreased hence the archwires coated with 50 nm Zr showed lowest Surface Roughness, The p-value was significant, p=0.00. Therefore, the effect of Zr layer thicknesses on the Surface Roughness of (β-Ti III and TIM) was significant. Fig 3, 4 also shows uncoated archwires gives higher Surface Roughness compared to coated archwires. In Fig 4, TIM coated with 50nm showed the lowest Surface Roughness than any of the other specimens.

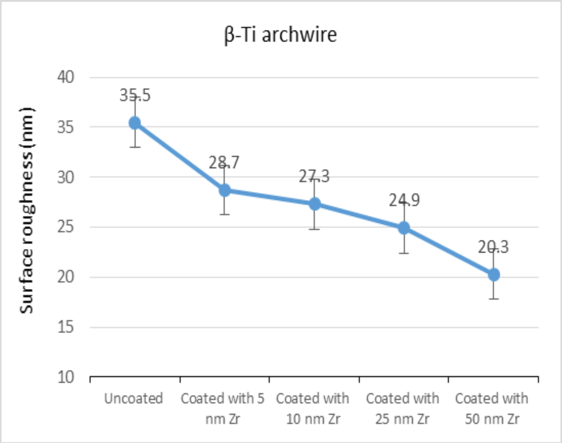

**Figure 3:** Mean of AFM measurements of surfaces roughness of uncoated β-Ti archwire and coated with 5, 10, 25 and 50 nm Zr.

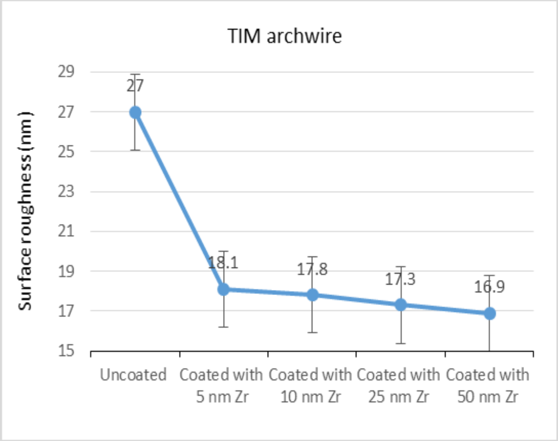

**Figure 4:** Mean of AFM measurements of surfaces roughness of uncoated TIM archwire and coated with 5, 10, 25 and 50 nm Zr.

The surface topography of each archwire, as observed by SEM, is summarized in Fig 5, 6. Each specimen had its characteristic surface structure. The scanning electron micrographs of the β-Ti and TIM archwire without Zr coating showed more lines and grooves (parallel to the long axis of the archwire) than the samples of the other archwires in Fig 5, 6 a . Wherefore uncoated archwires had the highest surface roughness of all the tested archwires. β-Ti and TIM coated with 5, 10, 25, and 50 nm Zr had significantly reduced surface roughness p=0.05 (Fig 5,6 b, c, d, and e).

The results of the analysis using SEM and AFM showed that the TIM archwires exhibited less roughness than the β-Ti archwires. Deposition of 5, 10, 25, and 50 nm Zr on both β-Ti III and TIM archwires led to reduced surface roughness of these archwires. Uncoated β-Ti III archwires showed the greatest surface roughness of the tested archwires which led the archwires undergoing stress making them more brittle. Also, corroborate that TIM coated with 50nm showed the lowest surface roughness than any of the other specimens.

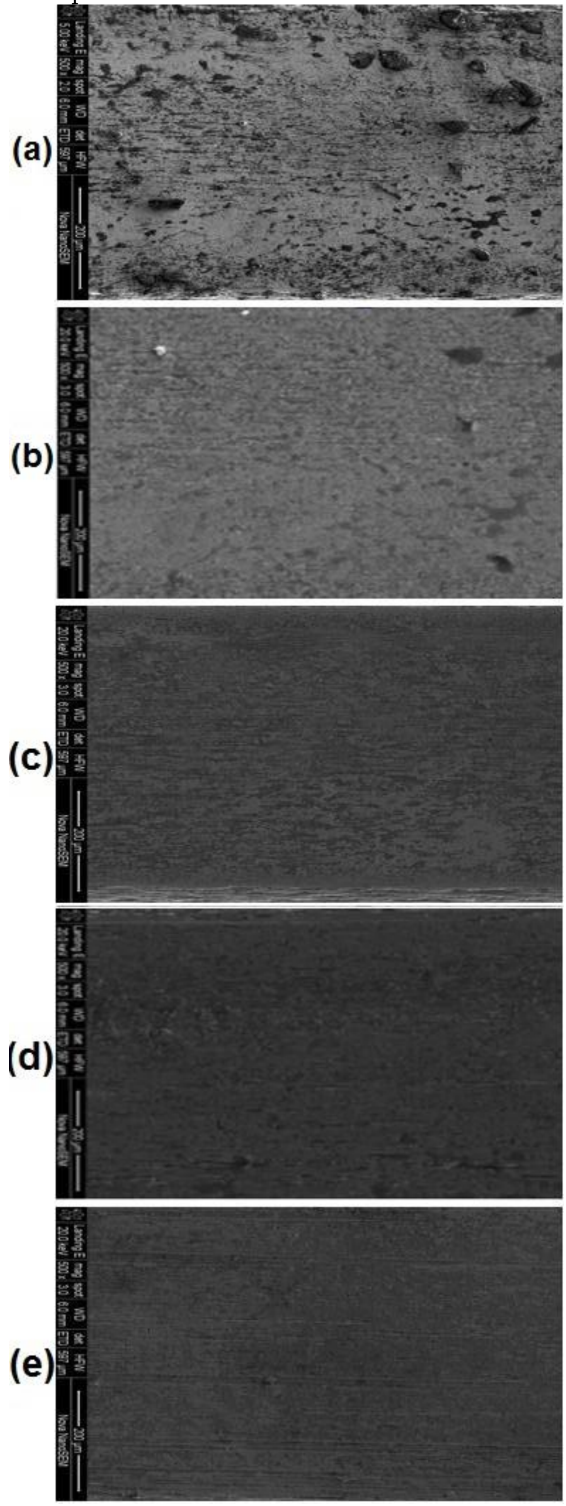

**Figure 5:** Surfaces analysis by SEM for (a) Uncoated β-Ti III archwire (b) Coated β-Ti III archwire with 5nm of Zr (c) Coated β-Ti III archwire with 10 nm of Zr (d) ) Coated β-Ti III archwire with 25 nm of Zr (e) ) Coated β-Ti III archwire with 50 nm of Zr at (500 ×) magnification

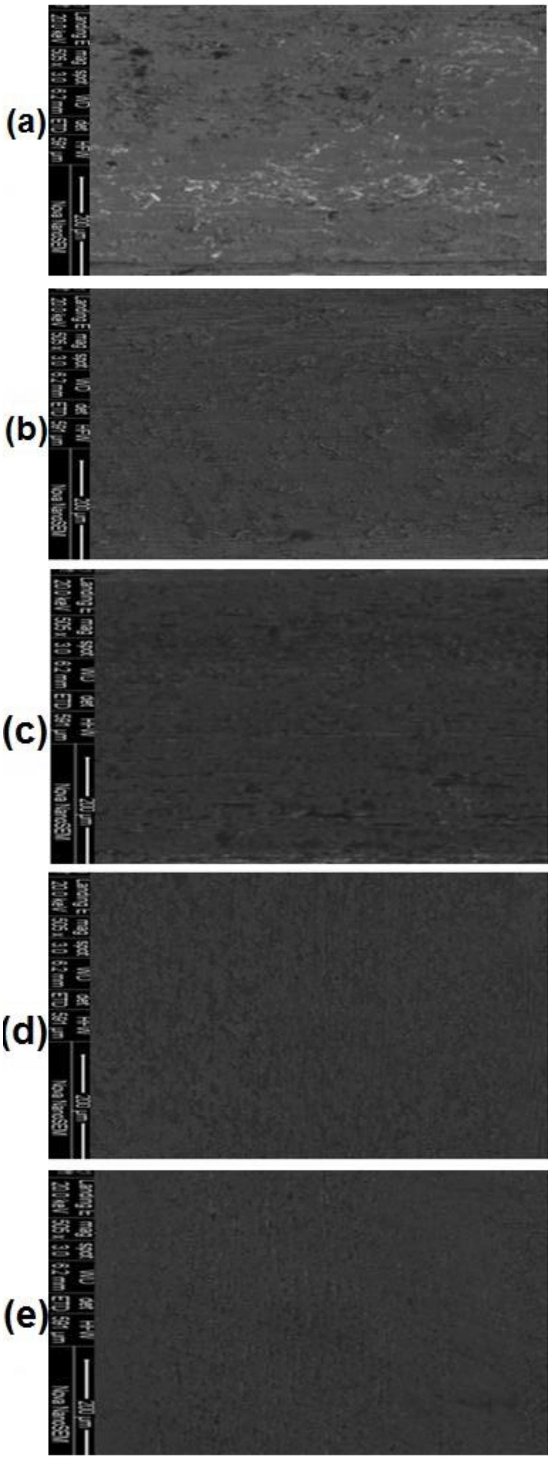

**Figure 6:** Surfaces analysis by SEM for (a) Uncoated TIM archwire (b) Coated TIM archwire with 5nm of Zr (c) Coated TIM archwire with 10 nm of Zr (d) Coated TIM archwire with 25 nm of Zr (e) Coated TIM archwire with 50 nm of Zr at (500 ×) magnification

## Discussion

This study showed that the surface roughness of two types of β-Ti archwires varied considerably, irrespective of the reputation of their brands .It has shown from results in this study that the surface Zr coating of archwires does improve surface roughness. Previous study observed that the frictional properties were improved if a surface treatment was applied, for example, polyethylene or ion implantation [20-21], the β-Ti archwires treated with ion implantation showed low surface roughness [22], which supports findings in this study. In contrast, surface roughness of orthodontic archwires may be measured using several methods, including laser spectroscopy, contact-surface profilometry, and atomic force microscopy [23-24] in this study topographic surface characteristics of orthodontic archwires were evaluated by AFM. From the results of AFM measurements, coated β-Ti III archwires had more smooth surfaces than uncoated β-Ti III archwires of approximately 25 nm, whereas coated TIM archwires had more smooth surfaces than uncoated TIM archwires of approximately 17 nm. It seemed that although the uncoated β-Ti III archwires experienced higher frictional resistance than in the uncoated TIM archwires experienced. Coated β-Ti III archwires and coated TIM archwires experienced showed significant decrease in surface roughness. In this study, the results showed that the least rough archwire was the TIM archwire, this is consistent with previous studies that demonstrate that TIM has a smoother surface than other β-Ti wires [25-26]. The β-Ti III archwires were the roughest, which could be associated with the great friction generated by this material. Like other studies on surface roughness of uncoated wires [27] or coated wires [28], the present study on coated wires as well showed differences between brands. The surface structure depends on the complex manufacturing processes, the surface finish treatments, and the alloy used [27], [29- 30]. SEM showed differences in the patterns of irregularities on the β-Ti III archwires and TIM archwires produced by the different manufacturers. However, the patterns observed on the β-Ti III archwires and TIM made in USA were different to each other, with deep scratches parallel to the long axis of the archwires. In contrast, the β-Ti III archwire lacked such clear horizontal and vertical grooves and had shallow dimples. Consideration in this status is that the high magnification of SEM limited the interpretation of the surface roughness of a smaller area of the orthodontic archwires [31]. SEM micrographs obtained in this study showed more porosities, scratches and defects on the β-Ti III archwires than on the TIM archwires and the difference was significant. Although results in this study indicate the overall superior smoothness of the TIM archwires, the difference observed was significant. This can be attributed to the increase in the differences between the surface roughness of β-Ti III archwires wires and TIM archwires because of the very high variation observed between the different samples of the different types of β-Ti archwire [32]. Coating of Zr, 5,10, 25 and 50 nm thick, on β-Ti orthodontic archwires using EB-PVD should lead to a decrease in the surface roughness of the archwires and improve the sliding between orthodontic archwires and brackets with no effect on the archwires strength [33]. We noticed that as the layer thickness of Zr increases the surface roughness decreases this is, in agreement with previous study, which says that, friction increased with increasing surface roughness, and decreased with increasing coating thickness. Therefore, in present study optimum layer thicknesses of coating Zr seems to be that of 50 nm for the both archwires [34].

## Conclusion

Surface characteristics revealed a smooth surface with little surface irregularity for uncoated TIM archwires. Coated TIM with 50 nm Zr showed the smoothest surface in all tested archwires. Uncoated β-Ti III archwires exhibited oriented cracks, vertical wire drawing lines, and a rough surface. It also clearly showed that the treatment with a Zr coating on archwires improved their quality of surface roughness. Coated archwires are much smoother than conventional ones, regardless of the brand tested. Future studies should assess other features of these archwires such as frictional resistance.

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