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Original Article

Dentistry Section

Assessment of Surface Roughness and Wettability of Hafnium Oxide Coated Titanium Discs: An In-vitro Analysis

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ABSTRACT

Introduction: Surface modifications of titanium implants play a crucial role in enhancing their performance, particularly in terms of surface roughness and wettability, which directly influence osseointegration and biological responses. Hafnium Oxide (HfO_2) coatings are emerging as potential surface modifiers due to their unique physicochemical properties.

Aim: The aim of the present study was to evaluate and compare the surface roughness and wettability of uncoated titanium discs and hafnium oxide coated titanium discs using Atomic Force Microscopy (AFM) and contact angle goniometry.

Materials and Methods: The present in-vitro study was conducted in the Department of Prosthodontics, Saveetha Dental College and Hospitals, Chennai, India, between March 2024 and June 2024. Twenty titanium discs were divided into two groups: uncoated Ti (Group A) and hafnium oxide coated Ti (Group B). Surface roughness parameters (Ra, Rq, Rm) were evaluated using AFM and wettability was assessed by static contact angle goniometry. Data were analysed with the Shapiro-Wilk test for normality and independent samples t-tests for intergroup comparison.

Results: AFM analysis revealed that the hafnium oxide coated titanium discs (group B) exhibited significantly higher surface roughness (Ra: 62.19 nm; Rq: 78.123 nm) compared to the uncoated titanium discs (group A) (Ra: 37.762 nm; Rq: 49.261 nm) ($p<0.05$). Contact angle measurements indicated that group B had substantially higher contact angles (average: 86.56°) compared to group A (average: 41.42°) ($p=0.018$), suggesting reduced surface wettability following HfO_2 coating.

Conclusion: Hafnium oxide coating on titanium discs significantly increases surface roughness while decreasing surface wettability. These surface modifications could potentially influence the biological behaviour of titanium-based implants.

Keywords: Biomaterials, Dip coating, Surface modifications, Atomic force microscopy

INTRODUCTION

The materials and technologies used for dental implants, restorations, and prosthetics have advanced significantly in dentistry due to the pursuit of better treatment outcomes and patient satisfaction. One important part of these developments is the creation of different surface treatments intended to improve the qualities of dental materials [1]. The difficulties in attaining the best possible mechanical stability, biocompatibility, and aesthetic appeal have led to the development of these coatings, which have improved the integration of dental devices with surrounding tissues [2]. The ability of conventional dental materials, like metals and ceramics, to interact well with biological systems has historically been restricted. Research on altering surface properties to improve tissue integration, decrease microbial colonisation and improve adhesion started as dental professionals realised how crucial the interface is between dental materials and oral tissues [3,4]. This resulted in the creation of coatings with enhanced wettability, surface roughness, and bioactivity. Progress in materials science and a better comprehension of biological processes have had a major impact on the development of dental surface coatings.

These methods of surface modification consist of plasma spraying, acid etching, and sandblasting. The downside of this is that contamination particles may form on Ti implant surfaces due to the unpredictability and imprecise nature of mechanical methods [5]. In a similar direction, acid etching cannot create regulated surface topographies and may leave behind surface acids that are harmful to the development of new bone [6]. Moreover, the implant surface created by these methods has non-uniform pore sizes at both the macro and microscales. Osteoblasts, however, might be more acclimated to a nanoscale environment than a microscale one [7]. Therefore, in order to encourage bone growth, more appealing techniques for altering Ti surfaces are required. Modifying the surface of metallic materials using a variety of techniques, such as dip coating, is one way to improve their properties. This method makes it possible to apply a thin layer to a variety of materials, including polymers, metal, and glass.

The physical and chemical characteristics of semiconductor/dielectric thin discs that are metal doped, metal oxide, metal sulfide, metal nitride, and nanostructured have been extensively studied for many years in an effort to understand their underlying mechanisms and explore potential technological uses [8,9]. Researchers have been particularly interested in thin films of Titanium (Ti) and its alloys because of their special optical, wettable, mechanical, thermal, electrical, magnetic, and chemical properties [10,11]. Excellent characteristics of HfO_2 were discovered, including low density, strong mechanical strength, high resistance to corrosion, low electrical resistivity, good thermal and chemical stability, high melting point, biocompatibility, high transparency and good optical absorption [12].

Recent research has demonstrated that the mechanical, optical, electrical, and thermal properties of Ti thin films are significantly influenced by their microstructure, which includes surface morphology, grain size, porosity, and film thickness [13,14]. Numerous studies have been published thus far on the growth of Ti thin films through various deposition methods [15,16]. Because of its versatility and reliable reproducibility, in the present study, titanium in the form of discs which are then coated with HfO_2 was used. Wettability and surface roughness is the test performed that is critical for biological applications as it optimises biological cell adhesion, spreading, and proliferation [17,18].

There is a scarcity of literature specifically evaluating the surface property alterations of titanium discs following hafnium oxide nanoparticle coating, particularly with regard to surface roughness and wettability. While the influence of various surface modifications on titanium has been extensively studied [19,20], limited data is available on the use of hafnium oxide as a coating material in this context [21,22]. The current manuscript is a part of a larger ongoing in-vitro research project focused on enhancing the biological performance of titanium-based implant materials through surface modification strategies. While a previous publication from the authors investigated antimicrobial and antioxidant properties of hafnium oxide coated titanium surfaces, the present study specifically focuses on evaluating surface roughness and wettability characteristics using AFM and contact angle goniometry.

MATERIALS AND METHODS

The present in-vitro study conducted in the Department of Prosthodontics, Saveetha Dental College and Hospitals, Chennai, India, between March 2024 and June 2024. The study was initiated after obtaining approval from the Institutional Ethical Committee (SRB/SDC/PROSTHO-2303/24/268). Commercially pure grade 2 titanium discs (15 mm diameter, 2 mm thickness, gross weight 0.2 kg) were procured from Tiaano® (Ti Anode Fabricators Pvt., Ltd., Chennai, India).

Inclusion and Exclusion criteria: Only commercially pure grade 2 titanium discs with dimensions of 15 mm diameter and 2 mm thickness, free from surface defects, deformities, irregular thickness, or scratches, were included in the study. Discs that failed to meet dimensional accuracy or had visible manufacturing flaws were excluded. For the coating group, only discs that underwent uniform polishing with silicon carbide emery sheets (400, 600, 800, and 1000 grit) were considered. Any discs damaged during polishing, coating, or handling were excluded from analysis.

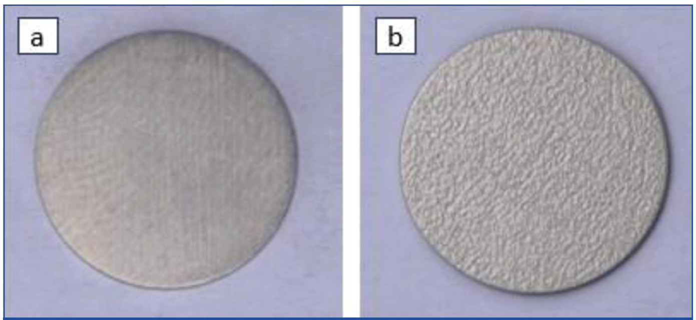
Sample size selection: A total of 20 titanium discs were included, with 10 discs allocated to each group. Group A comprised uncoated titanium discs, while group B comprised hafnium oxide nanoparticle-coated titanium discs (HfO_2-Ti). The sample size was determined based on feasibility for an in-vitro pilot analysis, ensuring adequate replicates for surface wettability and surface roughness evaluation under standardised testing conditions.

Study Procedure

Sample preparation: All titanium discs were first polished using a polishing machine with silicon carbide emery sheets of progressively finer grits (400, 600, 800, and 1000). For group B discs, coating was performed using a 2% hafnium (Nano Research Elements, Haryana, India) sol treatment [23]. The discs were washed with deionised water in a bath sonicator, rinsed 2-3 times, and dried in a hot air oven at 50°C. A coating solution was prepared by dispersing 200 mg of hafnium oxide nanoparticle powder (Nano Research Elements, Haryana, India) in double-distilled water, followed by sonication for uniform dispersion [24]. The solution was subjected to a direct current power supply set-up, with titanium as the anode and platinum as the cathode, at an applied voltage of 20 V. Discs were immersed in the solution for two hours to facilitate uniform nanoparticle deposition via electron transfer from the electrolyte to the metal surface [24,25]. The samples of both groups are shown in [Table/Fig-1].

Parameters studied: The prepared titanium discs were subjected to two primary analyses:

- Surface Wettability: Static contact angles were measured using a contact angle goniometer (Kernco Model No: G II, Kernco Instrument Co., Texas, USA). Goniometer tests are widely used in various fields, including materials science, coatings, and surface engineering, to evaluate and optimise the surface properties of materials for applications such as adhesives, paints, and biomedical implants [26-28]. A 5 µL droplet of deionised water was gently placed on the specimen surface, and images were captured immediately. The contact angle was recorded on both the left-side and right-side of the droplet using image analysis software, providing two independent measurements for each specimen in each group. The static left/right values were averaged to represent the mean contact angle for each group [29,30].
- Surface roughness: AFM was used to obtain 2D and 3D images and quantify nanoscale surface topography [31]. Surface roughness parameters studied were average roughness (Ra), root mean square roughness (Rq), and maximum profile valley depth (Rm). AFM is extremely flexible and useful in materials science, nanotechnology, and biology.



[Table/Fig-1]: The samples of both the groups involved: (a) Pure titanium discs of group A; (b) HfO_2-Ti coated discs of group B.

All analyses were performed under standardised laboratory conditions, and measurements were recorded using calibrated instruments.

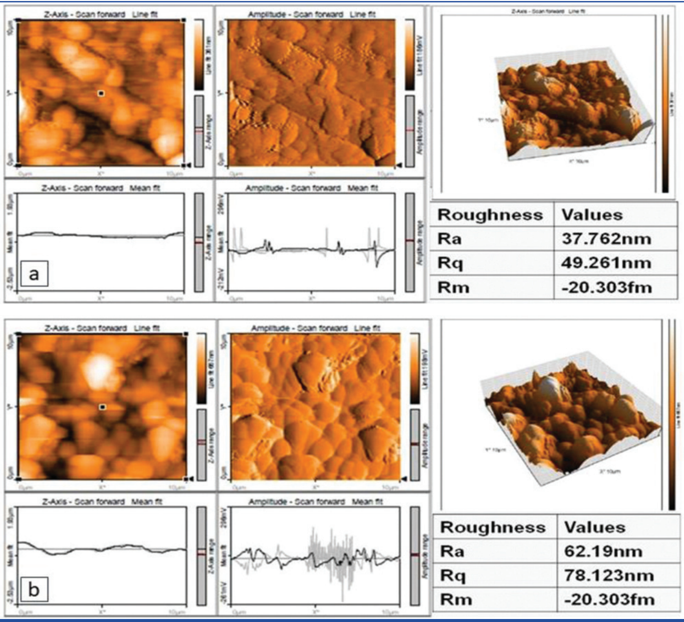
STATISTICAL ANALYSIS

The data obtained for surface roughness parameters (Ra, Rq, and Rm) and contact angle measurements were tabulated and analysed using IBM Statistical Package for Social Sciences (SPSS) Statistics (version XX, IBM Corp., Armonk, NY, USA). Normality of data distribution was assessed using the Shapiro–Wilk test. Since the data followed a normal distribution, intergroup comparisons between titanium discs (group A) and hafnium oxide coated titanium discs (group B) were performed using the independent samples t-test for both surface roughness and wettability parameters. For each comparison, 95% Confidence Intervals (CI) of the mean difference were calculated. A p-value <0.05 was considered statistically significant. All values are expressed as mean±Standard Deviation (SD).

RESULTS

The AFM images and roughness data provide a comparative analysis of the surface topography between samples of both groups. The Two-dimensional (2D) and Three-dimensional (3D) surface images revealed distinct differences in surface morphology [Table/Fig-2]. The group A samples showed a relatively smoother and less textured surface. In contrast, the group B samples with HfO_2-Ti coating, exhibited a significantly rougher surface (p<0.001) suggesting an increased surface irregularity and roughness [Table/Fig-3].

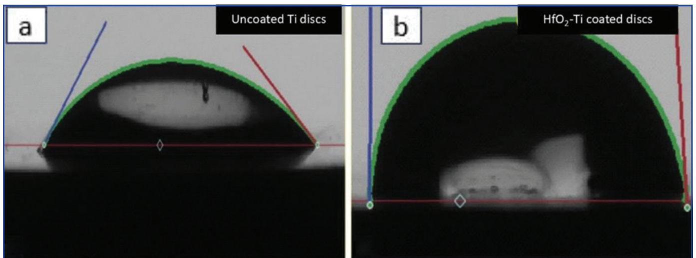
The contact angle measurements depicted in the images provide a comparison of the wettability between the two surfaces: Ti and HfO_2-Ti [Table/Fig-4]. Contact angle goniometry showed average angles increasing from 41.42° (group A-Ti) to 86.56° (group B-HfO_2-Ti), confirming a shift towards hydrophobic surface behaviour [Table/Fig-5]. Statistical analysis using independent t-test showed this difference to be significant (p<0.05).



[Table/Fig-2]: 2D and 3D topography and roughness profile of polished surface of: (a) pure titanium discs of group A; (b) HfO_2-Ti coated discs of group B.

Surface roughness	Group A	Group B	t value	p-value
Ra	37.76±1.89 nm	62.19±3.11 nm	-97.43	<0.001
Rq	49.26±2.46 nm	78.123±3.91 nm	-156.36	0.001
Rm	-20.30±1.02 fm	-20.30±1.02 fm		---

[Table/Fig-3]: Surface roughness parameters (Ra: average roughness, Rq: root mean square roughness, Rm: maximum profile valley depth) of group A (Ti) and group B (HfO_2-Ti) discs.



[Table/Fig-4]: Contact angle measurement of: (a) Group A (Ti discs); (b) Group B (HfO_2-Ti).

Contact angle parameters	Left angle	Left RMS error	Right angle	Right RMS error	Average angle	t value	p-value
Group A (Ti discs)	47.01°	0.67	35.84°	0.42	41.42°	-7.37	0.018
Group B (HfO2-Ti discs)	89.08°	0.45	84.04°	0.70	86.56°		

[Table/Fig-5]: Static contact angle measurements (left and right-sides of droplet) for group A (Ti discs) and group B (HfO_2-Ti discs) with mean±SD, their respective root mean square errors, and the average contact angle measurements.

DISCUSSION

The results of the present research showed elevation in Ra and Rq values observed in the HfO_2-Ti group reflects the effective deposition and adhesion of the HfO_2 coating onto the titanium substrate. The Ti sample exhibits a less textured surface, in contrast, the HfO_2-Ti sample, modified with a doped coating, presents a markedly rougher surface. The line scan profiles further emphasise this difference, with the HfO_2-Ti sample displaying greater surface variations and amplitude deviations compared to the Ti sample. The similarity in maximum peak-to-valley roughness (Rm) values are comparable for both samples, the distinct differences in overall roughness highlight the potential for the HfO_2-Ti sample to facilitate better biological responses. Consequently, the modified surface characteristics of the TiHf sample may lead to improved performance in implant applications, making it a promising candidate for further investigation in the development of advanced biomaterials aimed at enhancing osseointegration and long-term stability in clinical settings [32,33].

For the Ti sample, the left and right contact angles were relatively low suggesting that the surface of Ti is hydrophilic. Vergani LM et al., has emphasised the same and proved that promotes better cell adhesion, spreading, and bioactivity characteristics which are important in biomedical implant materials [34]. In contrast, the Ti-Hf sample exhibits significantly higher contact angles. This indicated a reduced hydrophilic surface, where the liquid drop forms a more spherical shape, suggesting that the addition of hafnium (Hf) to the titanium surface substantially reduces the surface wettability [35,36].

The differences in surface roughness are important for biomedical applications, as increased roughness can enhance cell adhesion and promote osseointegration, making the HfO_2-Ti sample potentially more suitable for applications like titanium implant coatings where surface interaction with biological tissues is critical [37]. The AFM analysis provides valuable insights into the surface topography of the samples, highlighting significant morphological differences that are critical for their potential applications. This increased roughness indicates a greater degree of surface irregularity, which is essential for promoting cell adhesion and osseointegration, thereby enhancing the material's suitability for applications such as titanium implant coatings [38].

The contact angle is an indicator of surface hydrophilicity or hydrophobicity, where lower contact angles represent higher wettability (more hydrophilic surfaces) and higher contact angles indicate lower wettability (more hydrophobic surfaces) [39]. The hydrophobic nature could affect the material's interactions with biological fluids and cells, possibly hindering cell attachment or promoting a different type of interaction, such as antibacterial activity, depending on the application [40]. While preparing Ti plates, initially surface treatments was carried out; hence, it is well known that the porosity was created on the pure plates, which initiates the impregnation of HfO_2 while coating. Therefore, it was expected that hydrophilicity was decreased with the coating of material. The significant difference in contact angles between the two samples highlights the influence of surface chemistry or morphology on their wettability. The lower contact angle of Ti suggests it may be more suited for applications requiring higher surface energy and better integration with biological tissues, while the higher contact angle of HfO2-Ti may be beneficial in scenarios where reduced wettability is advantageous, such as in antifouling or antibacterial applications [40-42].

Hafnium based coatings have been researched upon in past few years. The biocompatibility and antibacterial properties of medical stainless steel and titanium modified by alumina and hafnia films prepared by atomic layer deposition are potentially applicable in biomedical applications [43]. Hafnium dioxide obtained by atomic layer deposition possesses the ability to induce the formation of biological apatite [44]. In-vitro experiments showed that the HfN coating with a thickness of 50 nm, N_2 flow rate of 2.5 sccm, and Ar flow rate of 80 sccm had a notable antibacterial effect against *S. aureus* and was not toxic to Bone Marrow Steomal Cells (BMSCs) [45].

Limitation(s)

The present study provides critical insights into the surface roughness and wettability of hafnium oxide coated titanium discs however, certain limitations must be acknowledged. Being an in-vitro investigation, the results may not fully replicate clinical conditions, where variables such as saliva, temperature fluctuations, and masticatory forces can influence surface roughness and wettability. Only a single HfO_2 coating protocol and thickness were tested, and variations in deposition technique or layer thickness might yield different outcomes. The study employed a relatively small sample size (n=20 per group), which, while statistically adequate, may limit the generalisability of the results. Furthermore, only commercially pure titanium discs were examined, and the findings may not be applicable to other titanium alloys or dental materials. Surface characterisation was limited to AFM-based parameters (Ra, Rq, Rm), without complementary analyses such as Scanning Electron Microscopy (SEM) or X-ray Photoelectron Spectroscopy (XPS) to provide detailed morphological and elemental insights.

CONCLUSION(S)

In conclusion, the incorporation of Hafnium oxide into titanium surfaces significantly alters the surface topography and wettability, as demonstrated by AFM and goniometer analyses. The coated samples, with their increased roughness and irregularity, show promising potential for promoting cell adhesion and osseointegration, which are critical for implant integration. However, the goniometer results indicated increased hydrophobicity in HfO_2-coated discs compared to uncoated titanium, suggesting that while roughness may favour biological attachment, surface wettability may require further optimisation. Overall, these findings underscore the potential of HfO_2-coated titanium surfaces in biomedical applications, while highlighting the need for tailored modifications to balance both surface roughness and wettability for enhanced implant performance. Future research should focus on optimising coating deposition techniques, assessing long-term degradation behaviour, and correlating these surface characteristics with in-vitro and in-vivo biological responses, and on optimising coating parameters to balance mechanical stability with bioactivity.

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