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RESEARCH PAPER

The Effect of Anodic Oxidation on the Some Physiomechanical Properties of the Biomedical Commercially Pure Titanium-Strontium Oxide Composite Alloy

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ABSTRACT

Titanium and its alloys are very useful nowadays, due to its sorcerous properties of high strength to weight ratio, high corrosive resistance, and biocompatibility; it is substantive as god metal. In the presented work the anodic film formation and variation in the aesthetic appearance of anodized titanium titanium-strontium oxide composite alloy surface are investigated at certain levels of the applied potential difference across both terminal ends in an electrochemical circuit using sulfuric acid as an electrolyte. This paper focuses on recent developments of new anodized commercially pure titanium -strontium oxide composite alloy, where additions of strontium oxide were prepared specific percentage (6%) by wt.% for biomedical applications. The effect of additions of strontium oxide was investigated using SEM and EDS mapping for elemental analysis, Atomic Force Microscopy(AFM) Analysis before and after anodization, and wettability of the finished surfaces. According to the results, it is found that strontium oxide additives and anodized surfaces results increase roughness. However, in conclusion, the alloy that content at 6 wt.% strontium oxide micro particles additives relatively have high wettability for medical and dental applications. Also, the titanium alloy of 6 wt.% strontium oxide micro particles additives that anodized relatively have high wettability than non-anodized ones. Increased roughness and high wettability showed that the prepared strontium oxide-cp titanium composite alloy has excellent potential for use as biomaterial, mainly in dental applications.

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INTRODUCTION

Titanium (Ti) and its alloys have been widely used as biomedical materials and have found numerous applications as artificial bones and as various types of biocompatible implants due to their highly beneficial chemical, physical, mechanical, and physiological properties. Especially, their chemical resistance upon contact with fluids and tissue encountered in the human body make them suitable implant materials because they meet numerous selection criteria [1-4]. Positive modulation of biological processes is limited because it is unable to induce bone apposition (osteoinduction). [5].

As far as production concerns, metallic commercially pure titanium (cp Ti) , despite its

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high content in the earth's crust, is considered to be rare and expensive material[6]. The reason is that the conventional metallurgical methods are ineffective in case of Ti highly reactive at elevated temperatures, and problematic and ineffective machining, which results in a great loss of expensive material. For these reasons there is growing interest in powder metallurgy (PM) as a cost-effective approach of direct production of complex Ti parts. Moreover, powder metallurgy (PM) approach brings advantage of improved chemical and microstructural homogeneity as well as possibility to produce various composite structures [7] One technique to get porosity at the surface of a component is a powder metallurgy. Using this technique, it is possible to manufacture implants with interconnected pores, allowing bone growth in these areas, thus favoring to osseointegration. Powder metallurgy is a group of techniques used for producing, characterizing of metallic or ceramic powders and consolidation by compacting and sintering. [8].

The topography of a surface has a direct impact on biological reactions at the cellular level, including cell orientation and migration as well as the development of ordered cytoskeletal structures [9]. One of the greatest benefits of PM composite material is increasing the value micro surface roughness. Based on performed mechanical and wettability testing it is anticipated that the bioactive composite alloy can be used as a material to manufacture biomedical implants with improved mechanical compatibility and osseointegration potential for intense load-bearing applications. [10].

various studies have demonstrated surface modification with inorganic metal elements such as magnesium (Mg), zinc (Zn), strontium (Sr) incorporation could achieve rapid osseointegration and promote new bone formation [11]. Also, the bioactive coating calcium phosphate is used to enhance osseointegration and improve the stability of dental implants. [12]. A recently published review article by Marie et al, summarized how strontium oxide affects bone resorption and bone formation by activating pre-osteoblast replication as well as osteoblast differentiation and survival. At the same time SrO was reported to reduce pre-osteoclast differentiation in addition [13]. The success of an implantation of such devices is often limited due to biofilm formation on the surface of the medical implant [14]. To prevent biofilm formation, nanostructures can be applied to the implant surface [15]. In the case of Ti-based materials, biocompatibility improvement can be achieved by applying many different surface treatment techniques. However, electrochemical surface treatment methods offer some advantages, such as relative simplicity and low application cost. For instance, anodization (anodic formation of a surface oxide) is the most common surface treatment technique that forms a protective layer on Al, Ti, and valve metals. Because an anodic oxide layer possesses a low electrical conductivity as compared with that of electrolytes or bulk, metallic Ti, there is a significant potential drop across the oxide layers on the metallic substrate. This potential drop gives rise to a strong electric field across the oxide film, which then drives the migration of Ti^{4+} and O^{2-} ions through the oxide layer. The ions that are driven through the anodic oxide layer by an externally applied electric field promote the growth of anodic oxide. As long as the electric field is strong enough, the ions will be driven through the anodic oxide film and this film will continue to grow during anodization. Many researchers observed that there is usually a linear relationship between the oxide film thickness and the applied voltage. [16]. It also indicated that using the nano TiO_2 material as an antibacterial agent were almost effective. [17]. The biomechanical fixation of implants and the rate of osseointegration have been notice increased when rough surface increased. [18]. Two characteristics required for coatings that will be in contact with blood are homogeneity and a low level of roughness ($Ra \leq 50$ nm) to avoid the promotion of blood clots (thrombosis) [19]. The anodic oxidation technique, with oxidation voltages below the production of spark discharge phenomenon can be used to obtain homogeneous TiO_2 coatings with low roughness [20]. This effect limited the oxidation voltage used in the present work, because (as previously mentioned) homogeneous and low roughness coatings are necessary for hemocompatible applications. [21].

Finally, the purpose of this study is to provide a new type of biomedical ceramic-metal composite, named bio active composite metal, manufactured by effective powder metallurgy PM technique. Hybrid structure of composite alloy comprises bio-inert Ti matrix, which remains permanent after implantation and provides mechanical performance of implant, and biodegradable strontium oxide component, ceramic element in Ti. to obtain smooth and uniform coatings of TiO_2 by anodic oxidation of biomedical ceramic-metal composite alloy in sulfuric acid as electrolyte, to be used in the construction of a dental and orthopedic appliances. This study introduces concept of strontium oxide-cp titanium composite alloy, wherein microstructure and mechanical properties are characterized in as fabricated condition and testing is done.

MATERIALS AND METHODS

Particle size measurements

Powders of (CP Titanium, strontium oxide) were used in this study, CP titanium (China. Particle size 32 µm) and strontium oxide powder (mo.sci, USA. Particle size 3-10 µm) were measured by using particle size analyzer type (Bettersize) for the starting materials' particle size.

Mixing and Ball Milling

A sensitive accurate electronic balance type OHAUS-model 250g-USA with 0.1 mg accuracy was used to weight 50 gm of pureTi powder that was dried at 125 °C by using vacuum dry box (DZF-6020 GERMANY). Milling of Ti powder was carried out using rotating ball mill with AISI 304 SS container. The device of ball milling was fabricated for the present study. Chromium steel balls with 10 mm in diameter were utilized. The mixture charge and balls were introduced with a weight ratio of 1:10, respectively into the container and then the charge was milled for 5 hrs. at 145 rpm.

After that, the strontium oxide powder of was also dried at 125 °C by using vacuum dry box and added to the milled titanium powder and the mixture was blended for 5 hours. The percentages of addition of the strontium oxide powder to the titanium were 6%, as showed in the Table 1. All blending cycles were performed at room temperature and under controlled conditions.

Cold Compaction

Cold compaction was performed in uniaxial way by computed hydraulic press (carver, USA). Pressing mold were used for the production of 12 mm diameter cylindrical samples. Percentages of alloying elements used showed in table 1. the applied pressure 800 MPa was chosen as the best one.

Sintering

The GCs were sintered in electric resistance programmable vacuum furnace type (china) in an argon atmosphere Sintering was performed according by heating the GCs sintering temperature 500, ± 3°C and soaked for 2 hours and the temperature was elevated to 1000± 3°C and also soaked for 2 hours. The heating rate was fixed at 3 °C/min. The furnace was switched off, and the samples were left to cool slowly inside it with a continuous flow of argon gas stream

Table 1. Percentages of alloying elements used in pilot study.

Groups	Pure Ti %	SrO %
Group A	100%	0%
Group D	94%	6%
Group F	90%	10%

Table 2. Chemical composition of simulated body fluid (SBF) solution [25].

S. no.	Reagents	Amount in 1000 ml
1	NaCl	8.035 g
2	$NaHCO_3$	0.355 g
3	KCl	0.225 g
4	K_2HPO_4\$3H_2O	0.231 g
5	$MgCl_2$\$6H_2O	0.311 g
6	1.0 M HCl	39.0 ml
7	$CaCl_2$	0.292 g
8	Na_2SO_4	0.072 g
9	$((HOCH_2)_3CNH_2)$	6.118 g
10	1.0 M HCl	Appropriate amount for adjusting the pH ~ 7.4

under 5 mbar pressure from the commencement of the process until room temperature. The sintering temperature was calibrated by a digital thermometer type DT-830C Aswar/China with aid of K-type thermocouple with measuring range from -20 to 1370 ± 3 °C.

Grinding and Polishing

The fabricated sintered compacts samples were wet ground with (400, 800, 1500, 2000 and 3000) grit silicon carbide emery papers, respectively on a grinding machine (type: mekton / UK). Afterwards, the overall grinding period for each sample surface preparation for the required test and examinations is fixed at constant period equal to 15 min. Then, they were washed with distilled water and dried in the electric vacuum oven at 125 °C for 30 minutes. Thereafter, the samples were become ready for the tests and examinations categorized as followed paragraphs. [22].

Anodic Oxidation of Titanium and titanium-strontium oxide samples

The experimental setup consists of a reaction beaker, a DC-power supply, magnetic stirrer, a special sample holder, a counter electrode made of silver, an optional current sensing circuit and a PC to record the data. As electrolyte we used sulfuric acid (H_2SO_4). Titanium and titanium-strontium oxide disks (12 mm × 12 mm × 8 mm) was washed with 10% aqueous hydrogen peroxide for 10 min to remove its naturally formed oxide layer. [23] The traditional anodizing technique employed a lab DC power supply to apply a constant voltage/current directly and the anodizing processed for a period of time. [24] Titanium and titanium-strontium oxide disks was anodized with direct current (3 A, 30 V) for 6-7 min, under the carried out at room temperature (25 ◦C) applying a DC electric current between the Pt cathode and Ti-alloy anode, separated from each other by 6 mm in a beaker glass containing the electrolyte. recommended speed for magnetic stirrer was 400 rpm. The electrolyte was sulfuric acid (H_2SO_4) solution, and the concentration was 1 M. Immediately after oxidation, the oxidized samples were rinsed with demineralized water and dried with hot air [21].

Sampling and groups distribution

The prepared specimen's groups were divided to four main groups (Group A, Group B, Group C and Group D) and each main group was subdivided into five subgroups according to each test. SEM type (Axia chemi sem. Thermo scientific company. Holland) was used. Vicker hardness device types (Digitals Micro Vicker Hardness Tester UH 250, BUEHLER, GERMANY) using a load of 1.96 N for 25 s according to ASTM (E10-15). It is necessary to

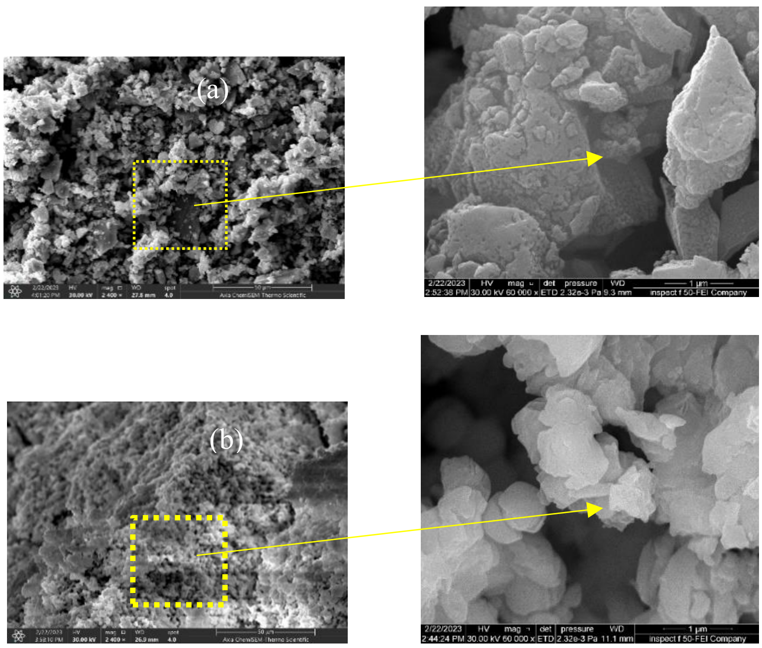

Fig. 1. Images for a SEM for pure titanium powder, b SEM for strontium oxides powder.

study the behavior of Ti–15Mo–xIn as implants in the human body, so the corrosion test is used for pre-prepared specimens in SBF solution with PH 7.4 at 37±1 °C, its chemical composition is illustrated in Table 2 [24-25]. Atomic force microscopy (Model TT-2 ,2023 AFM Workshop, USA) is used to test the topographic roughness of the surface of prepared alloys. Water contact angle measurements were performed by the sessile drop method on a contact angle goniometer (an instrument generally called a contact angle goniometer or tensiometer is used to measure the static contact angle.

Chemical composition of powders

The purity of cp titanium was 99.971 according to analysis of the chemical composition of titanium powder (wt.% analysis). while the purity of strontium oxide was 99.9% based on trace metals analysis.

Analysis of Variance

Analysis of variance (ANOVA) is a statistical method for detecting variations between experimental groups. ANOVA is justified when there are several experimental groups within one or more independent (categorical) variables. In analysis of variance (ANOVA), independent variables are called factors, and levels are groupings within each factor. ANOVA is a computer method that quantitatively evaluates the relative contribution of each parameter change to the total response variation in a given experiment. Through ANOVA, it is possible to assess the impact of each given input parameter on the machining process [26]. Multiple comparison test (Games-Howell) that compared between each two groups in the same period. Significant difference between each two groups shows P≤ 0.01.

RESULTS AND DISCUSSIONS

Microstructure of the Prepared Specimens

The microstructure and quantitative determination of microstructural data were obtained using a scan electron microscope (SEM) and an energy-dispersive X-ray analyzer (EDS) on % of weight-graded specimens. The surface morphological analyses of CP Ti and SrO powders were seen in Fig. 1 a and b. The shapes of CP Ti and SrO powders with different sizes are irregular and also rounded particles can be seen. The specimens have been etched to reveal the grain boundaries in the microstructure. Fig. 2 shows photographs of non-oxidized samples. As noticed from Fig.2, SEM revealed characteristic differences at the micro

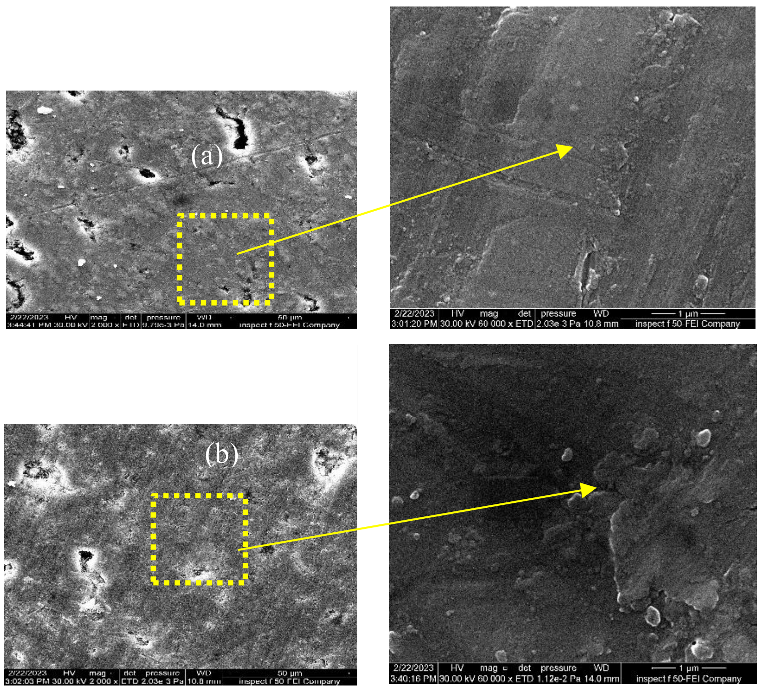

Fig. 2. Images for a SEM for pure titanium alloy, b SEM for pure titanium -strontium oxides alloy.

level. photographs showed that the surfaces of the titanium-strontium oxide composite alloy were contain ceramic compounds (strontium oxides) that are known for their more porous features characteristics than the pure titanium-alloy. [27]. The metallography can give a simplified idea about the relationships between the microstructure of the material and the microscopic properties because the size and the grains directly affect the material's behavior. While Fig. 3 shows photographs of oxidized samples at 30 voltages and the corresponding Scanning Electron Micrographs. The overall analysis of the surface morphology of samples oxidized in H_2SO_4 indicates the formation of a mesoporous oxide layer. A compact anodic film structure is formed at 30 V in the sulfuric acid and the anodic film blisters are formed due to the bursting of oxygen bubbles within the film formed (Fig. 3a). The ruptures of the films induce nanocracks, which are evident after anodization. the ruptures of the film are initiated within the blisters due to the bursting of oxygen bubbles. [28]. SEM images of the oxide layer exhibit that pores are distributed on the entire surface which has crater type morphology, characteristics of pores formed i.e. shape, diameter, depth was greatly different. [29].

Fig. 4a shows photographs of EDS elemental chemical analysis of control specimen showed the presence of titanium element. Anodization of specimens in 1M sulfuric acid solution for 6 min showed an increase in oxygen percentage. Fig. 4b shows photographs of EDS elemental chemical analysis of control specimen showed the presence of titanium and titanium oxide elements. it also showed trace elements of nickel, carbon, iron and sulfur. Fig. 4c illustrates photographs of EDS elemental chemical analysis of titanium-strontium oxide composite alloy specimen showed the presence of titanium and strontium oxide elements. Anodization of titanium-strontium oxide composite alloy specimens in 1M sulfuric acid solution for 6 min showed an increase in oxygen percentage as showed in the Fig. 4 d.

The formation of the oxide layer is quite complex because some basic concepts of chemistry are involved in this process. The anodic layer generally called as an outside layer which is exposed to the

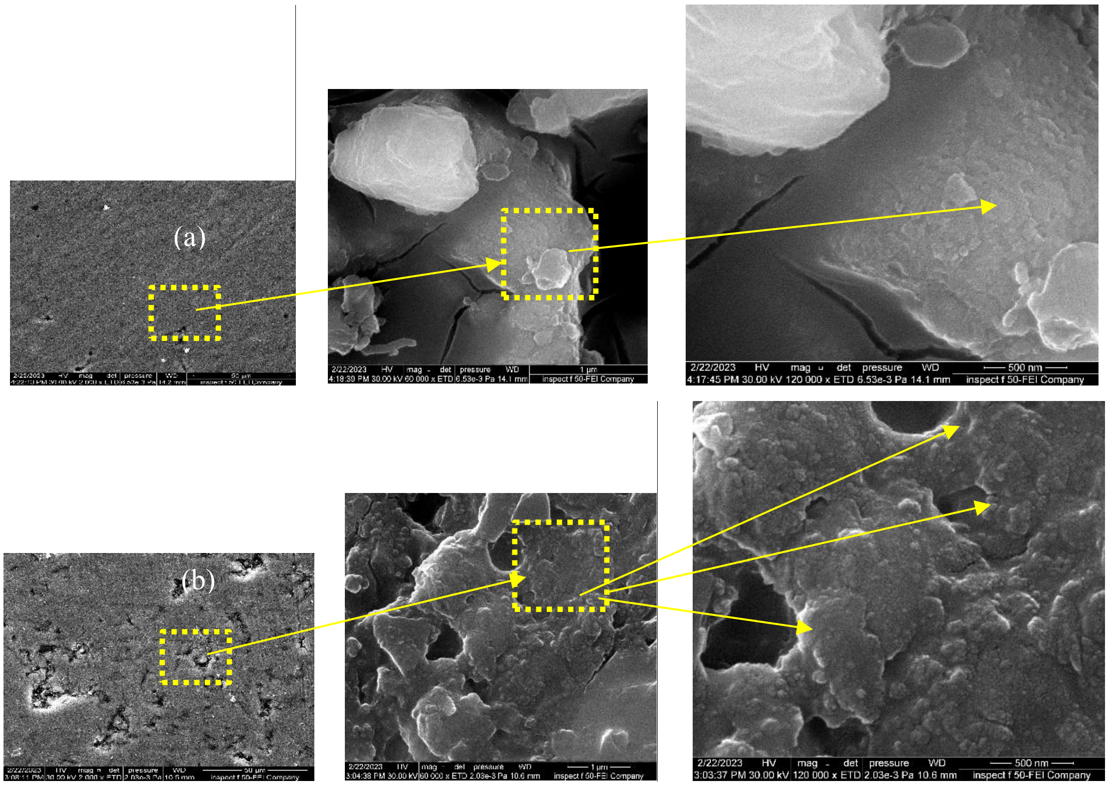

Fig. 3. Images for a SEM for anodized pure titanium alloy, b SEM for anodized pure titanium -strontium oxides alloy.

electrolyte (H_2SO_4) has a large amount of OH-ions as compared to the inner layer and is considered to be Ti(OH)4. The inner layer, where the removing of hydrogen occurred, is represented as titanium oxide. Actually, there is variation in electrolyte concentration across the film, which can be written as $TiO_2.xH_2O$, to comprise the inner and outer anodic oxide layer. [29]. The metallography can give a simplified idea about the relationships between the microstructure of the material and the microscopic properties because the size and the grains directly affect the material's behavior [30]. The roughness positively interferes with osteoblastic cells' development during cell recognition and adhesion processes, ensuring better stability [31].

Atomic Force Microscopy (AFM) Analysis before and after anodization

In this work, the surface analysis of specimens was performed with atomic force microscopy. One of the advantages of AFM is the possibility to

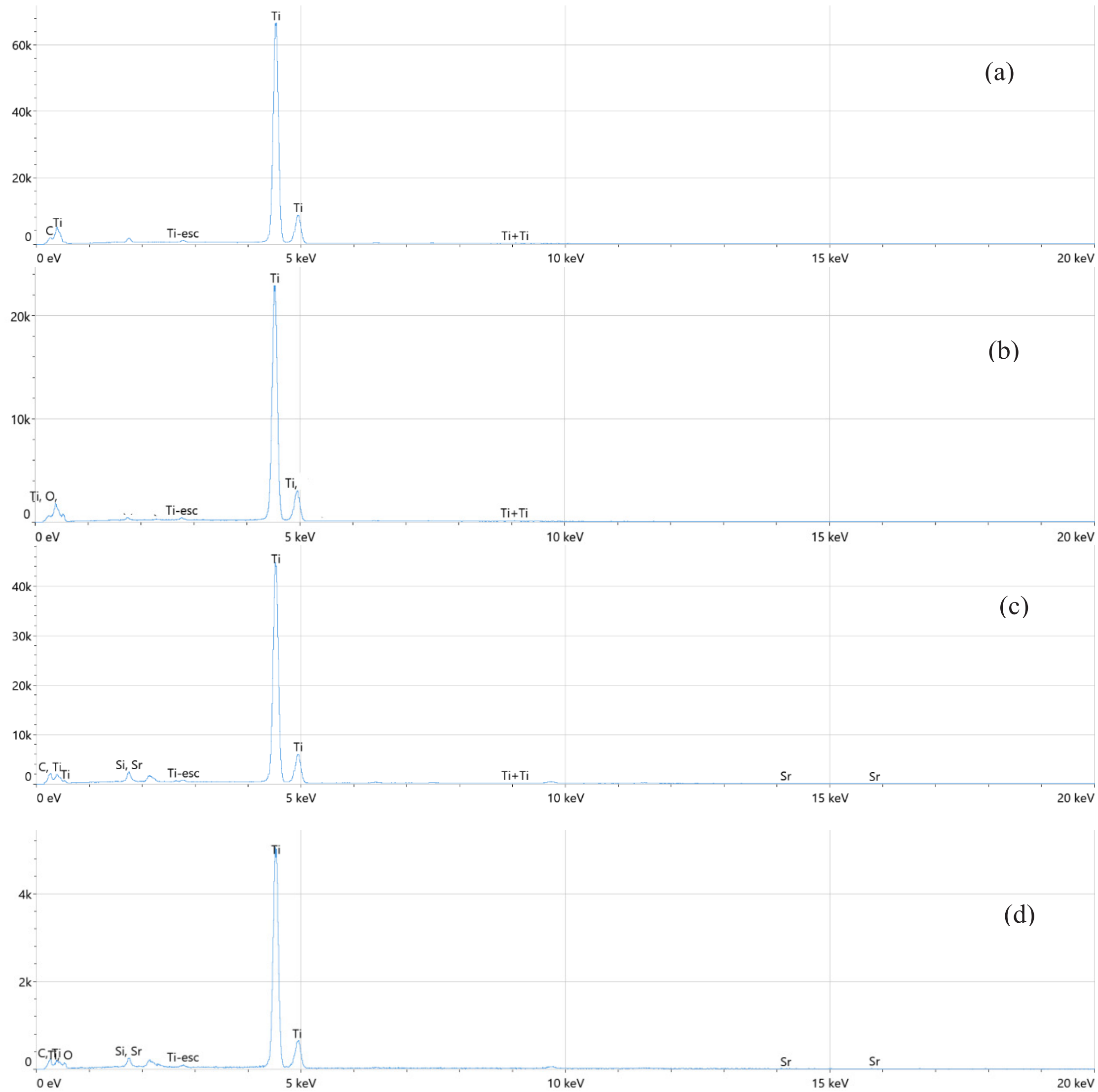

Fig. 4. EDX images of specimens a control specimen (pure Ti), b specimen anodized in 1M sulfuric acid for 6 min, c specimen titanium-strontium oxide. composite alloy, and d specimen anodized titanium-strontium oxide composite alloy in 1M sulfuric acid for 6 min.

evaluate the area of implants that will effectively be in contact with the biofluid during the bone integration of implants. [32]. Anodization in 1M sulfuric acid solution for 6 min increased the surface roughness with more than two folds, also the surface area increased linearly with increasing

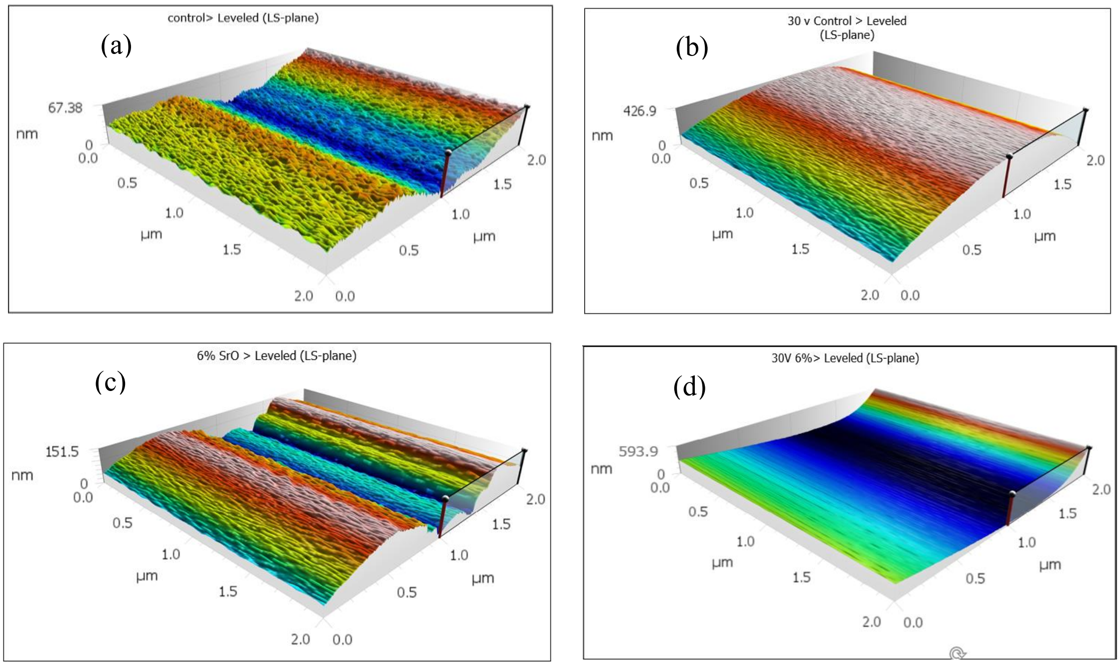

Fig. 5. AFM images of specimens a control specimen (pure Ti), b specimen anodized in 1M sulfuric acid for 6 min, c specimen titanium-strontium oxide composite alloy, and d specimen anodized titanium-strontium oxide composite alloy in 1M sulfuric acid for 6 min.

Table 3. Descriptive statistics of Atomic Force Microscopy for all groups.

Groups	N	Mean	S.D.	S.E.	Min.	Max.
A	10	8.094	0.135	0.043	7.790	8.300
B	10	48.453	0.921	0.291	46.950	49.560
C	10	26.665	0.903	0.285	24.860	27.820
D	10	58.035	1.137	0.359	55.950	59.280

Table 4. ANOVA analysis of Atomic Force Microscopy of all groups.

	Sum of Squares	d.f.	Mean Square	F-test	p-value
Between Groups	15045.999	3	5015.333		
Within Groups	26.754	36	0.743	6748.584	0.000
Total	15072.753	39			

Table 5. Games-Hawell analysis of porosity of all groups.

Groups		Mean Difference	S.E.	p-value	95% CI	
					Upper Bound	Lower Bound
A	B	-40.359	0.294	0.000	-41.270	-39.448
	C	-18.571	0.289	0.000	-19.464	-17.678
	D	-49.941	0.362	0.000	-51.065	-48.817
B	C	21.788	0.408	0.000	20.636	22.940
	D	-9.582	0.463	0.000	-10.895	-8.269
C	D	-31.370	0.459	0.000	-32.674	-30.066

time.

(Tables 3-5) shows that the surface roughness of the cp Ti and Ti-SrO alloys has a gradual increase according to the gradually progression of anodization process. this can be attributed to the homogenous increase of the TiO_2 film thickness at the initial stage of anodization Spark discharge did not occur thus porosity was not observed [33].

Wettability test (contact angle test) before and after anodization

Contact angle goniometer was used to make water contact angle measurements were performed by the sessile drop method on a (an instrument generally called a contact angle goniometer or tensiometer is used to measure the static contact angle,) at room temperature and ten measurements were conducted and averaged for each specimen, then compared with samples of cp Ti alloy as a control (group A). Water contact angle images were taken for all study groups as shown in Fig. 6, the control group A shown higher water contact angle (87.842)0, while the group B shown decrease in the water contact angle (35.912) 0 after anodization. The group C shown higher water contact angle (56.219.) 0 while the group D which shown the lowest water contact angle (24.704) 0. (Tables 6-8) shows that the contact angle test of the cp Ti and Ti-SrO alloys has a gradual decrease according to anodization process. In Fig. 5 represents an oxide layer deposited on titanium substrate after anodization. The decrease in contact angle on group C was attributed to surface composition alteration and the degree of roughness due to laser surface structuring that enhancing the cell attachment on the surface. These results were confirmed with the results of Majumdar et al. in 2015 [34]. While the decrease in contact angle on group D was attributed to oxide layer deposition on titanium substrate after anodization, with applied voltage, the oxide layer of the film was observed become porous, rougher

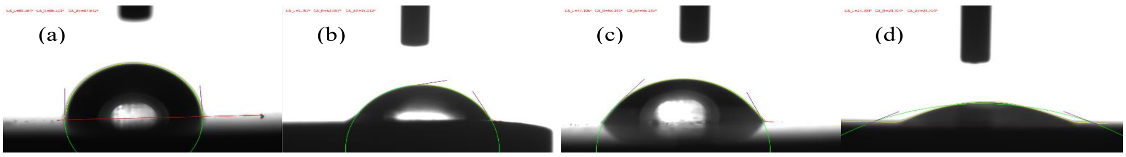

Fig. 6. contact angle test images of specimens a control specimen (pure Ti), b specimen anodized in 1M sulfuric acid for 6 min, c specimen titanium-strontium oxide composite alloy, and d specimen anodized titanium-strontium oxide composite alloy in 1M sulfuric acid for 6 min.

Table. 6. a Descriptive statistics of contact angle values for all groups.

Groups	N	Mean	S.D.	S.E.	Min.	Max.
A	10	86.455	1.170	0.370	84.324	88.002
B	10	34.845	1.031	0.326	32.911	36.091
C	10	55.719	1.389	0.439	53.987	58.022
D	10	24.626	1.391	0.440	22.895	27.136

Table. 7. ANOVA analysis of contact angle values for all groups.

	Sum of Squares	d.f.	Mean Square	F-test	p-value
Between Groups	22344.939	3	7448.313		
Within Groups	56.675	36	1.574	4731.183	0.000
Total	22401.614	39			

Table 8. Games-Howell analysis of contact angle values of all groups.

Groups		Mean Difference	S.E.	p-value	95% CI	
					Upper Bound	Lower Bound
A	B	51.610	0.561	0.000	50.099	53.121
	C	30.736	0.561	0.000	29.225	32.247
	D	61.829	0.561	0.000	60.317	63.340
B	C	-20.874	0.561	0.000	-22.385	-19.363
	D	10.219	0.561	0.000	8.707	11.730
C	D	31.093	0.561	0.000	29.581	32.604

and thicker. [35].

Numerous previous studies had claimed that the highest surface roughness displayed the lowest water titanium contact angle, and subsequently more osteoblasts adhere and proliferate on titanium surface [30].

CONCLUSION

According to the previous results, a new and very promising Nobel composite titanium alloy (anodized Ti–SrO alloy) have been prepared by using powder metallurgy technique. Samples of the Ti–SrO alloy with additions of 6% by wt.% were pre pared. They were characterized by SEM and EDS map, AFM, and contact angle measurements. There was a reduction in the contact angle measurements compared of anodized alloy to cp-Ti alloys; therefore, the applied voltage has a significant influence on the properties of anodised Ti such as colour, surface morphology, mineralogy, microstructure, and thickness. Therefore, it is expected that this surface treatment method will be efficiently used for bio activation of Ti and its alloys. Furthermore, based on the results obtained in this work, the Ti–SrO alloy obtained the most significant biomedical application potential. For further work, a model will be designed to apply this alloy in the organism's body, especially the leg region, to determine the concentration of stresses and weaknesses of this alloy. And finally, these biomedical new alloys will be very conducting bio-composite for the dental and orthopedic implants.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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