



ISSN (Paper) 1994-697X

Online 2706 -722X

<https://doi.org/10.54633/2333-022-047-028>

## The microhardness Property of the Biomedical commercially pure titanium-strontium oxide composite alloy after anodic oxidation

Soodad A. Muhammed<sup>1</sup> · Aseel Mohammed Al-Khafaji<sup>2</sup>Haydar H. Jamal Al-Deen<sup>3</sup>.<sup>1</sup>Department of Prosthodontics, College of Dentistry, University of kufa, Alnajaf, Iraq<sup>2</sup>Department of Prosthodontics, College of Dentistry, University of Baghdad, Baghdad, Iraq<sup>3</sup>Department of Metallurgical Engineering, Materials Engineering Faculty, University of Babylon, Hillah, Iraq<https://orcid.org/0009-0001-6776-2697>[soodada.alhiloh@uokufa.edu.iq](mailto:soodada.alhiloh@uokufa.edu.iq)

### Abstract

high level of corrosion resistance, a high strength to weight ratio, and bio-compatibility, titanium as well as its alloys are extremely valuable today because of its enchanted qualities. With the use of sulfuric acid as electrolyte in the electrochemical circuit, the formation of anodic films and variations in aesthetic appearance regarding anodized titanium titanium-strontium oxide composite alloy surface are examined in the work that is currently being presented. This article concentrates on the most recent advances of a novel, commercially pure titanium-strontium oxide composite alloy that has been anodized. With regard to biomedical applications, strontium oxide was added in a particular quantity (6%) by wt.%. With the use of microhardness testing device, the impact of strontium oxide additions and anodic oxidation were examined. The findings show that strontium oxide additions increase microhardness. Additionally, titanium alloys with 6 wt.% strontium oxide microparticle additions that have been anodized exhibit no difference than those that have not been anodized. The produced strontium oxide-cp titanium composite alloy offers significant potential for use as a biomaterial, particularly in dental applications, as evidenced by high wettability and increased roughness.

Corresponding Author

**Keywords** commercially pure Titanium · strontium oxide· Powder metallurgy· microhardness ·

## 1 Introduction

Because of their highly advantageous physical, chemical, physiological, and mechanical qualities, titanium (Ti) and its alloys were extensively employed as bio-medical materials and have found multiple uses as artificial bones and as other forms of bio-compatible implants. They meet several selection criteria, but in particular, their chemical resistance when in contact with the fluids and tissue that are found in the human body makes them appropriate materials for the implants [Kim et al 1997, Ducheyne et al 1986]. Numerous works have shown that modifying surfaces with inorganic metal ions including zinc (Zn), magnesium (Mg), and strontium (Sr) insertion can achieve rapid osseointegration and encourage the production of new bone [Martin et al 2017, Safi et al 2019]. Marie et al.'s newly released review study detailed how SrO influences bone formation and resorption by promoting osteoblast differentiation and survival and pre-osteoblast replication. Pre-osteoclast differentiation was also observed to be decreased concurrently by SrO [Marie et al 2011, Tukmachi et al 2023].

To create homogenous TiO<sub>2</sub> coatings with a low level of roughness, anodic oxidation approach could be utilized with oxidation voltage values that are below those that cause the phenomenon of the spark discharge [Vera 2013]. This impact restricted the oxidation voltage utilized in the current study since, as was already indicated, hemocompatible applications call for homogeneous and low-roughness coatings [Vera et al 2017, Al-Khafaji et al 2021].

For example, the most widely-used surface treatment method that creates protective layer on Al, Ti, and valve metals is anodization (anodic creation of a surface oxide). There's a considerable potential drop between oxide layers on metallic substrate since anodic oxide layers have a low electrical conductivity in comparison with electrolytes or bulk, metallic Ti. The O<sup>2-</sup> and Ti<sup>4+</sup> ions subsequently migrate across oxide layer due to the strong electric field created by this potential decrease across the oxide film. An electric field provided from outside the anodic oxide layer causes ions to be driven through it, which encourages anodic oxide growth. The ions will be driven through anodic oxide film, which will keep expanding throughout anodization as long as electric field is sufficiently strong. Numerous works have noted that oxide film thickness and the applied voltage typically have a linear relationship [Ohtsuka and Nomura 1997].

However, the embedded materials must be solid and resilient enough to survive the physiological loads applied and required to operate for a much longer duration or until a lifetime without loss or revision surgery. An important equilibrium between strength and rigidity is also necessary to match that of the bone. [Al-Murshdy et al 2021].

The final goal of this research is presenting a novel bio-medical ceramic-metal composite type, which is called bio active composite metal, produced using a successful PM process. For obtaining uniform and smooth coatings of TiO<sub>2</sub> through anodic oxidation regarding biomedical ceramic-metal composite alloy in the sulfuric acid as electrolyte, for use in the construction of orthopedic and dental appliances.

## 2 Experimental Procedure

### 2.1 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 particle size of the starting materials.

### 2.2 Preparation of Sample

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. By using a calculated hydraulic press (carver, USA), cold compaction was carried out in a uniaxial manner. Samples with a 12mm diameter were created using a pressing mold.

The samples have been sintered in an argon environment in electric resistance programmable vacuum furnace (China). samples were heated to a sintering temperature of 500,  $\pm 3^{\circ}\text{C}$ , and then the temperature was raised to 1000,  $\pm 3^{\circ}\text{C}$ , and the process was repeated for 2 hrs. The rate of heating was set at 3  $^{\circ}\text{C}/\text{min}$ . When the process was complete, the furnace has been turned off, and samples have been allowed to progressively cool inside of it while being continuously surrounded by an argon gas stream at a pressure of 5 mbar.

On a grinding machine (mekton/UK), the samples of manufactured sintered compacts have been wet ground with silicon carbide emery papers of (400, 800, 1500, 2000, and 3000  $\mu\text{m}$ ) grit, respectively. Afterwards, the samples have been organized into categories that were ready for examinations and tests [Bahador et al 2018].

### 2.3 Anodic Oxidation of Titanium and titanium-strontium oxide samples

DC power supply, a reaction beaker, a special sample holder, a magnetic stirrer, an optional current sensing circuit, a silver counter electrode, and a computer to record the results make up the experimental apparatus. We employed sulfuric acid ( $\text{H}_2\text{SO}_4$ ) as an electrolyte. To eliminate the naturally produced oxide layer on Ti and Ti-SrO disks (12 mm  $\times$  12 mm  $\times$  8 mm), 10% aqueous hydrogen peroxide was applied for 10 minutes [Sawada et al 2019]. Applying a DC electric current between the Ti-alloy anode and Pt cathode, separated from one another by 6 mm in a beaker glass that contains the electrolyte, for 6-7 minutes at room temperature anodized Ti and Ti-SrO disks. The 400-rpm suggested speed for magnetic stirrers. Sulfuric acid ( $\text{H}_2\text{SO}_4$ ) solution served as the electrolyte, and its concentration was 1 M. The oxidized samples have been cleaned with de-mineralized water and dried with hot air immediately [Napoli et al 2018].

### 3 Sampling and groups distribution

The groups of the prepared specimen were split into four main groups (Groups A, B, C, and D), and each main group was further divided into three subgroups for each test. The microhardness measurements have been made with the use of digital micro vicker hardness tester (UH 250, BUEHLER, GERMANY).

### 4 Analysis of Variance

A statistical technique for identifying changes between experimental groups is ANOVA. In the case when there are multiple experimental groups within at least one independent (categorical) variable, an ANOVA is appropriate. Levels are collections of independent variables within every factor in ANOVA. Also, ANOVA is a computer technique which examines each parameter change's relative contribution to the overall response variation in experiment. There is a possibility to evaluate effects of every input parameter on the machining process using ANOVA [Kumanan et al 2014, Atiyah et al 2014]. Multiple comparison test (Games-Howell) that compared between each two groups in the same period. Significant difference between each two groups shows  $P \leq 0.01$ .

### 5 results and discussion of microhardness

In this study, vicker microhardness was measured for cp Ti alloy and Ti-SrO specimen's composite alloys. shown in Tables (1,2). In the hardness ratings for the cp Ti and Ti-SrO alloys may be seen in the function of the strontium oxides content. Multiple comparison test (Games-Howell) that compared between each two groups in the same period was shown in the table (3). The table shown that there was highly significant difference between each two groups  $P \leq 0.01$ , except group A and group B, and group C and D that showed no significant difference. The effect of reinforcing the solid solution and hardening of the fine increases the strontium oxides content. The hardness caused by a martensitic process occurrence in the cp Ti

and Ti–SrO alloys systems has highly risen. group A and group B, and group C and D showed no significant difference before and after anodic oxidation, due to Nanostructures could be formed to the implant surface, so that, this Nano-scaled layer cannot have measured by digital micro vicker hardness tester. [Kumanan et al 2014].

**Table 1** Descriptive statistics of vicker microhardness t for all groups

Descriptive statistics								
Groups	N	Mean	S.D.	S.E.	5% Confidence Interval for Mean		Min.	Max.
					Lower Bound	Upper Bound		
					<b>A</b>	10		
<b>B</b>	10	238.400	7.560	2.391	232.992	243.808	227	250
<b>C</b>	10	436.500	3.951	1.249	433.674	439.326	431	443
<b>D</b>	10	443.300	7.558	2.390	437.893	448.707	432	454

**Table 2** ANOVA analysis of vicker microhardness of all groups

One way ANOVA					
	Sum of Squares	d.f.	Mean Square	F-test	p-value
<b>Between Groups</b>	416725.275	3	138908.425	3408.563	<b>0.000</b>
<b>Within Groups</b>	1467.100	36	40.753		
<b>Total</b>	418192.375	39			

**Table 3** Games-Howell analysis of vicker microhardness of all groups

Games-Howell test						
Groups		Mean Difference	S.E.	p-value	95% CI	
					Upper Bound	Lower Bound
<b>A</b>	B	-5.100	3.005	0.356	-13.651	3.451
	C	-203.200	2.208	<b>0.000</b>	-209.518	-196.882
	D	-210.000	3.004	<b>0.000</b>	-218.549	-201.451
<b>B</b>	C	-198.100	2.698	<b>0.000</b>	-205.972	-190.228
	D	-204.900	3.380	<b>0.000</b>	-214.454	-195.346
<b>C</b>	D	-6.800	2.697	0.101	-14.670	1.070

## 6 Conclusions

A novel and extremely significant composite Ti alloy (anodized Ti-SrO alloy) has been created with the use of PM, in accordance with prior findings. Preparing was done on samples of the Ti-SrO alloy that had 6% by wt.% additions. For the orthopedic and dental implants, such new biomedical alloys will be very conductive bio-composite.

## Eferences

- Kim. H.M., Miyaji, F. Kokubo, T. and Nakamura, T.: Effect of heat treatment on apatite-forming ability of Ti metal induced by alkali treatment. *J. Mater. Sci.: Mater. Med.* 8, 341 (1997). DOI: [10.1023/a:1018524731409](https://doi.org/10.1023/a:1018524731409)
- Ducheyne, P., W.V. Raemdonck, J.C. Heughebaert, and M. Heughebaert: Structural analysis of hydroxyapatite coatings on titanium. *Biomaterials* 7, 97 (1986). [doi.org/10.1016/0142-9612\(86\)90063-3](https://doi.org/10.1016/0142-9612(86)90063-3)
- Martin Balog, Mateja Snajdar, Peter Krizik, Zdravko Schauer, Zlatko Stanec, Amir Catic, (2017) "Titanium-magnesium composite for dental implants (BIACOM)." 2 Faculty of Mechanical Engineering and Naval Architecture, University of Zagreb, Ivana Lucica 5, 10000 Zagreb, Croatia. DOI: [10.1007/978-3-319-51493-2\\_26](https://doi.org/10.1007/978-3-319-51493-2_26)
- Safi I. Nabeel, Ali Hussein B. Mohammed, Al Shammari A. Majeed, Tawfiq T. (2019) Abid "Implementation and characterization of coating pure titanium dental implant with sintered b-TCP by using Nd:YAG laser" *Saudi Dental Journal* 31, 242–250. [/doi.org/10.1016/j.sdentj.2018.12.004](https://doi.org/10.1016/j.sdentj.2018.12.004)
- Marie PJ, Felsenberg D, Brandi ML (2011). "How strontium ranelate, via opposite effects on bone resorption and formation, prevents osteoporosis". *Osteoporos Int.* 2011;22:1659–67. DOI: [10.1007/s00198-010-1369-0](https://doi.org/10.1007/s00198-010-1369-0)
- Tukmachi, Mustafa S.; Abdul-Baqi, Hikmat J.; and Hussein, Falah H. (2023) "Biofunctionalization of polyetheretherketone implant material by bone-forming peptide-2 immobilization," *Karbala International Journal of Modern Science: Vol. 9 : Iss. 1 , Article 9.* Available at: <https://doi.org/10.33640/2405-609X.3283>
- Vera, M.L. (2013). *Obtención y Caracterización de Películas Hemocompatibles de TiO<sub>2</sub>.* Ph.D. Thesis, Universidad Nacional de General San Martín, Buenos Aires, Argentina, March. (In Spanish) <https://ri.conicet.gov.ar/handle/11336/84026>
- Vera ML, Colaccio Á, Rosenberger MR, Schvezov CE, Ares AE. (2017) Influence of the Electrolyte Concentration on the Smooth TiO<sub>2</sub> Anodic Coatings on Ti-6Al-4V. *Coatings*; 7(3):39. <https://doi.org/10.3390/coatings7030039>
- Al-Khafaji Aseel M. S. and Hamad I. Thekra (2021), "Surface Analysis of the PEKK Coating on the CP Ti Implant Using Laser Technique" *International Medical Journal* Vol. 28, Supplement No. 1, pp. 29 - 32, June. [https://www.researchgate.net/publication/352002071\\_Surface\\_Analysis\\_of\\_the\\_PEKK\\_Coating\\_on\\_the\\_CP\\_Ti\\_Implant](https://www.researchgate.net/publication/352002071_Surface_Analysis_of_the_PEKK_Coating_on_the_CP_Ti_Implant)
- Ohtsuka T. and Nomura N.: The dependence of the optical property of Ti anodic oxide film on its growth rate by ellipsometry. *Corros. Sci.* 39, 1253 (1997). [doi.org/10.1016/S0010-938X\(97\)00025-5](https://doi.org/10.1016/S0010-938X(97)00025-5)
- Al-Murshdy J. M. S · Jamal Al-Deen H. H. · Hussein Sh. R. (2021). "Investigation of the Effect of Indium Addition on the Mechanical and Electrochemical Properties of the Ti-15Mo Biomedical Alloy". *Journal of Bio- and Tribo-Corrosion* (2021) 7:148 <https://doi.org/10.1007/s40735-021-00581-w>
- Bahador A, Hamzah E, Kondoh K, Abubakar TA, Yusof F, Umeda J, Ibrahim MK (2018) "Microstructure and superelastic properties of free forged Ti-Ni shape-memory alloy". *Trans Nonferrous Met Soc China* 28(3):502–514 [doi.org/10.3390/cryst12020145](https://doi.org/10.3390/cryst12020145)
- Sawada R, Katou Y, Shibata H, Katayama M, Nonami T (2019). "Evaluation of Photocatalytic and Protein Adsorption Properties of Anodized Titanium Plate Immersed in Simulated Body Fluid" *Hindawi International Journal of Biomaterials* Volume 2019, Article ID 7826373, 8 pages <https://doi.org/10.1155/2019/7826373>
- NAPOLI, G., PAURA, M., VELA, T., DI SCHINO, A. (2018). "COLOURING TITANIUM ALLOYS BY ANODIC OXIDATION" ISSN 0543-5846 METABK 57(1-2) 111-113 UDC – UDK 669.295.7.018:621.35.6676:541.451=111 <https://hrcak.srce.hr/file/278980>

- Kumanan S, Manikandan N, Narayanan CS (2014) Investigations of process parameters on electrochemical machining of titanium Ti6Al4V alloy using grey relational analysis. International colloquium on materials, manufacturing and metrology, ICMMM 2014 August 8–9, 2014, IIT Madras, Chennai, India doi:10.1088/1757-899X/653/1/012049
- Atiyah, A. H.; Baban, L. M.( 2014), " Fracture resistance of endodontically treated premolars with extensive MOD cavities restored with different composite restorations: an in vitro study". Journal of Baghdad College of Dentistry. DOI 10.12816/0015139