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Effect of Titania Addition on Mechanical Properties and Wear Behavior of Alumina-10 wt.% Tricalcium Phosphate Ceramics as Coating for Orthopedic Implant

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Abstract

The aim of this study is to determine the effect of Titania on mechanical properties and wear behavior of Alumina-10 wt.% TCP ceramics and to evaluate the performance of Titania in improving their resistance to these effects. Al_2O_3 -10 wt.% β -TCP mingled with TiO_2 to obtain a mixture which is considered as a bioactive coating that may be used in orthopedic implants. Representative bioceramic samples of such blends were prepared with different percentages of Titania and then tested using different methods and techniques. Mechanical properties, fracture toughness were evaluated using the modified Brazilian, semi-circular bending specimens. A pin-on-disk tribometer was retained to study the wear behavior. Based on the obtained results, it was found that the best mechanical properties and wear resistance was displayed for Alumina-10 wt.% TCP-5 wt.% Titania composite. This composite presents a good combination of flexural strength ($\sigma_f \approx 98$ MPa), compressive strength ($\sigma_c \approx 352$ MPa), fracture toughness ($K_{IC} \approx 13$ MPa $\text{m}^{1/2}$) and micro-hardness ($H_v \approx 8.4$ GPa). In terms of tribological properties, the lowest wear volume and wear resistance was recorded for Al_2O_3 -10 wt.% TCP – 5 wt.% TiO_2 composition.

Keywords: Titania, mechanical properties, wear behavior, fracture, biomaterial

1. Introduction

Biomaterial research is described through the introduction of biotechnology and advances in the comprehension of the biocompatibility of human tissues [1]. In this context, Tissue engineering applies several methods from materials engineering and life sciences in order to create artificial constructs and to achieve better and faster biological healing outcomes [2]. Bone tissue engineering researchers are interested to develop new synthetic biomaterials with similar properties to native bone [2]. Among the biomaterials, bioceramics which are widely used in medical applications and more precisely for implants in orthopedics [2].

Special attention has been given to β -tricalcium phosphate (β -Ca₃(PO₄)₂) (β -TCP) due to their bone-like chemical composition as well as excellent biological properties and its outstanding biological responses to physiological environments. The use of TCP has been limited in the human body due to its weak rupture resistance [3]. Therefore, much research has been interested in enhancing the mechanical resistance of β -TCP by the inclusion of several additives [4].

Alumina (Al₂O₃) have been widely studied due to their bioinert with human tissues, high wear resistance, fracture toughness and high strength [5]. The study by Sakka et al. [6] has recently been concerned with alumina/ β - tricalcium phosphate system with different percentages of β -TCP. These Al₂O₃/ β -TCP composites have showed a good combination of tensile strength (26.69 MPa), compressive strength (173,468 MPa) and fracture toughness (8,762 MPa m^{1/2}).

The functions of additives for alumina have been often aimed to lower the sintering temperature, customize the microstructure as well as improve the product properties. In order to improve the mechanical and tribological resistance of these composites, it is essential to introduce a reinforcing agent: metallic dispersion or ceramic oxide. In this case, among the ceramic oxide agents, the addition of Titania (TiO₂) has been reported to promote the sintering. TiO₂ addition not only reduces the sintering temperature of alumina, but also influences the mechanical properties [7]. Due to its excellent wear resistance, its biocompatibility, its chemical inertness and its chemical stability in aqueous environments, we have chosen titania as the agent of reinforcement to be added to the Al₂O₃-10 wt.% TCP composites [8, 9]. The amounts of Titania were varied from 0 wt.% to 10 wt.%. Hence, this study aims to investigate the effects of TiO₂ addition to the Alumina-10 wt.% TCP composite mechanical and tribological properties and evolution of microstructure.

Firstly, after implantation, the bone substitute suffers from several mechanical stresses notably bending and compressive stresses. Fracture, compression and bending tests are essential to ensure adequate resistance and compatibility with bone resistance and to evaluate the fracture behavior of the substitute used under tensile-shear loading [10–12].

Secondly, cracks and flaws which certainly exist in the sample reduce in a significant way the load-bearing capacity and then cause the substitute to break [13, 14]. The fracture toughness and stress intensity factor have been proposed to express the critical stress states in the vicinity of the crack tip, in the aim to analyze crack initiation and propagation [14].

Thirdly, the problem of wear due to friction in the prosthesis for the substitution of knee joints and hip has been addressed by many authors [15, 16]. This problem induces inflammatory responses in the tissues surrounding the joint which leading to a surgical Intervention. In the same vein, in artificial joints, the surfaces must be biocompatible and resisted to wear to reduce debris generation [17]. For that, the surface of the prosthesis must have sufficient mechanical and tribological stability when subjected to stresses associated with moving to avoid detachment of the surface of the implant.

In this context, our research was undertaken to discuss the influence of Titania on the densification, the mechanical and tribological behavior and microstructures of those composites as a coating for orthopedic implant. Within this context, we are interested in examining the effect of TiO₂ (1 wt.%, 2.5 wt.%; 3 wt.%; 4 wt.%; 5 wt.%; 7.5 wt.% and 10 wt.%) on the Al₂O₃-10 wt.% TCP composites sintered at 1600°C for 60 min. To reach this purpose, after sintering, flattened Brazilian discs (FBD) were used to determine the tensile strength (σ_t) and the elastic modulus (E). The semi-circular bending test was realized to study the of and the mode I K_{IC} and the mode I stress intensity factor K_I was determined using the CSTBD specimen. Compression tests were conducted to determine the compressive strength (σ_c). Finally, the

specimens were tested in sliding experiments to measure wear volume and friction coefficient. The characteristics were examined by X-ray diffraction and scanning electron microscopy. All those parameters are used to compare various formulations and then to retain the best formulation for testing samples.

2. Materials and methods

2.1 Preparation of ceramics

To produce biocomposites, the synthesis of β -tricalcium phosphate powder was conducted by solid-state reaction from calcium carbonate (CaCO_3) and calcium phosphate dibasic anhydrous (CaHPO_4). Stoichiometric amounts of high purity powders such as CaCO_3 (Fluka, purity $\geq 98.5\%$) and CaHPO_4 (Fluka, purity $\geq 99\%$), were sintered at 1000°C for 20 h according to the reaction reported in Ref [18]. While Alumina and Titania used are of commercial origin. They are high purity powders (Riedel-de Haien, purity $\geq 99\%$).

It is worthwhile to note that the size of particles of the powder was ($2.53 \pm 0.2 \mu\text{m}$ for Al_2O_3 , $2.79 \pm 0.2 \mu\text{m}$ for β -TCP and $0.11 \mu\text{m}$ for TiO_2).

The initial mixture was 90 wt% Al_2O_3 and 10 wt% TCP and was mixed with different amounts of TiO_2 (1 wt.%, 2.5 wt.%, 3 wt.%, 4 wt.%, 5 wt.%, 7.5 wt.% and 10 wt.%). The mixtures were homogeneously mixed in an agate mortar and were milled in ethanol utilizing an ultrasound machine then dried for 24 h at 80°C to eliminate the ethanol and generate a finely divided powder. The dried powder was pressed in metallic mold depending on the geometry of the specimens and uniaxially pressed using LLOYD model test (machine LR50K) at 67 MPa.

Subsequently, those compacted samples of all the compositions were sintered at 1600°C for 60 min within a programmable muffle furnace (Vecstar furnace model XF5) and in normal atmospheric conditions. All specimens were heated at a rate of $10^\circ\text{C min}^{-1}$ and cooled at a rate $20^\circ\text{C min}^{-1}$.

The densification of the sintered samples was evaluated from measurements of the dimensions of the samples and the relative error of the densification value was about 1%.

2.2 Mechanical tests

At least six specimens were tested under each condition and then average values (E , σ_t , σ_f , σ_c and K_{IC}) were considered.

2.2.1 Modified Brazilian test

For the Flattened Brazilian Disc, the specimen would be separated into two halves. In fact, the crack initiation point is at the center of the specimen surface, the resulting crack would propagate in a plane normal to the loading direction. The tensile stress meets the maximum tensile strength criterion.

The FBD is more favorable to measure the tensile strength σ_t [19].

For FB test, the form of specimens is a cylindrical with a thickness of 5 mm, a diameter of 30 mm and the width of the flat $2b$ of 5 mm ($2\alpha = 20^\circ$) (**Figure 1**).

Using the Griffith strength criterion, for a loading angle $2\alpha = 20^\circ$, the tensile strength (σ_t) was determined by this equation [20]:

$$\sigma_t = 0.9644 \frac{2P_c}{\pi Dt} \quad (1)$$

Figure 1.
Flattened Brazilian disc.

Where D is the specimen diameter, P_c is the tensile load, t is the specimen thickness.

2.2.2 Hardness measurements

In order to exclude the effect of Titania, the samples are evaluated mechanically by NanoIndenter (INNOVASTEST). The nanoindentation experiments were performed using the instrumented indentation techniques. The indented area was measured by optical microscopy for hardness calculation.

2.2.3 Bending test

The mechanical properties of the samples were assessed using Semi Circular Bending tests to determine the flexural strength σ_f and the fracture toughness K_{IC} . The samples were positioned on the loading platform by 3-point compressive loading, at an uniform loading speed of 0.075 mm/min. The SCB specimen diameter is equal to 30 mm and 5 mm for thickness (**Figure 2a**).

The flexural strength σ_f is given according to Refs. [21–23]:

$$\sigma_f = \frac{P_{mac}}{\pi R t} Q \quad (2)$$

In which P_{max} is the maximum load, t is the specimen thickness and R is the specimen radius. Q is the corresponding components of the dimensionless stress tensor, for the isotropic case $Q = 5.132$ [24].

In terms of fracture toughness K_{IC} . The same specimen dimension was used by adding a crack of 4 mm in the semi disc, as shown in (**Figure 2b**). The crack-length-to diameter ratio S/D was 0.13.

Using the SCB specimen with straight crack, the fracture toughness K_{IC} was calculated with the following formula [21]:

$$K_{IC} = \frac{P_{max} \sqrt{\pi a}}{2 R t} Y_I(a/R, S/R) \quad (3)$$

Figure 2.
Semi-circular bending Disc: (a) uncracked SCB specimen and (b) cracked SCB specimen.

Where a is the crack length, $P_{\max}$ is the maximum load, D is the cylindrical block diameter and Y_I is the geometry factor. The latter is a function of the ratio of the crack length (a) over the semi-disc radius (R) and the ratio of the half-distance between the two bottom supports (S) over the semi-disc radius (R) (**Figure 2b**). The geometry factor Y_I is expressed as follows [21]:

$$Y_I(a/R, S/R) = \frac{S}{R} \left(2.91 + 54.39 \frac{a}{R} + 391.4 \left(\frac{a}{R} \right)^2 + 1210.6 \left(\frac{a}{R} \right)^3 - 1650 \left(\frac{a}{R} \right)^4 + 875 \left(\frac{a}{R} \right)^5 \right) \quad (4)$$

2.2.4 Compression test

For the compressive test, $D = 9 \text{ mm}$ and $l = 18 \text{ mm}$ where D is the diameter and l is the length of the cylindrical specimen (**Figure 3**), as specified in ASTM standards [24, 25]. During the compressive test, the samples are positioned between compressive plates parallel and then compressed at a loading rate of 1 mm/min .

Figure 3.
Specimen for compressive test.

Figure 4. Schematic representation of sliding (pion-on-disk configuration) test, showing experimental sliding parameters for Alumina- 10 wt.% TCP-TiO₂ samples (pressureless sintering at 1600°C for 1h) against ZrO₂ ball. The Sliding distance length is 377 m.

In terms of compressive properties, the compressive strength σ_c is given as follows:

$$\sigma_c = \frac{4P_{\max}}{\pi D^2} \quad (5)$$

Where D is the cylindrical block diameter and $P_{\max}$ is the maximum load.

2.3 Tribological tests

In order to evaluate the tribological properties of the Alumina-10 wt.% TCP-TiO₂ composite, sliding wear tests were carried out against sintered composites under 9 N normal load [26, 27]. Wear tests were performed using a rotating pion-on-disk tribometer, **Figure 4**, that was developed in the LGME lab [28]. A Zirconia ball of 10 mm diameter and 11 GPa hardness is fixed using a suitable device used to solicit our samples in sliding. All specimens are polished cylindrical discs of 30 mm diameter and 5 mm thickness. The parameters set for the sliding tests are a sliding velocity of 0.1 m/s. All the tests were achieved out without lubricated environment for the normal test duration of 3600 s, at room temperature (20°C) and with a relative humidity of $35 \pm 5\%$. For each test, the friction coefficient and the wear rate are measured.

The coefficient of friction is determined from the tangential effort and the normal force by the following formula (6) [29]:

$$COF = \frac{F_t}{F_n} \quad (6)$$

Where COF is the coefficient of friction (dimensionless), F_n is the normal applied force (N) and F_t is the tangential effort (N).

The wear rate of all compositions is deduced from the measurement of mass loss. It is determined using this following equation (Eq. (7)) [30]:

$$W_r = \frac{(M_0 - M_t)}{M_0} * 100 \tag{7}$$

in which W_r is the wear rate (%), M_0 is the initial mass and M_t the mass of the specimen after sliding test.

In order to control the wear of Zirconia ball, the ball was weighed before and after each sliding test.

The wear volume of all compositions is determined from the measurement of worn surfaces of tested samples. The wear tracks were determined using 2D-profilometer.

To determine the wear volume, a profilogram (2D-profiles) is drawn in the perpendicular direction of the wear scars over a length of 8 mm [31, 32] with which we can determine the area (S_-) of the wear track (**Figure 5**). The wear volume is determined using by the following formula (8):

$$W_v = 2 \pi r_t S_- \tag{8}$$

Where is the wear volume (mm^3), S_- is the area of the wear track and r_t is the radius of the wear track.

2.4 Physico-chemical characterization

The characterization of the samples after the tests is carried out using several techniques.

X-ray powder diffraction analysis (DIFFRAC SUITE, Brucker, Germany) was conducted in order to analyze the phase transformation in the different structures of each composites before and after the sintering process. The Xray radiance was created by using $\text{CuK}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$) in the 2θ range $5\text{--}60^\circ$ at a current of 40 mA, a voltage of 40 kv, and a scanning rate of $1.2^\circ/\text{min}$. The identification phase was identified out by comparing the experimental XRD-patterns with the standard files assembled by ICDD (the International Center for Diffraction Data).

Scanning electron microscopy (JEOL JSM-5400) was used to observe the surfaces of the fractured samples after the sintering process. It was equally used

Figure 5.
Example of profilogram.

for the assessment of wear mechanisms, the microstructure of the sliding zone of the tested sample.

3. Results and discussion

In this section, we studied the effect of Titania on the mechanical behavior of Alumina-10 wt.% TCP composite with different additive amounts (1 wt.%; 2.5 wt.%; 3 wt.%; 4 wt.%; 5 wt.%; 7.5 wt.% and 10 wt.%).

The particle size analysis of the Al_2O_3 -10 wt.% TCP- TiO_2 composites illustrated in **Figure 6** show that the particle distribution and the average grain size of the Al_2O_3 -10 wt.% TCP- TiO_2 mixtures vary a function of the amount of Titania additive.

The particle size distribution of composites without titania is narrow, while mixtures containing TiO_2 exhibit a wide particle size dispersion. The median diameter (D_{50}) is around 0.34 μm .

3.1 Sintering behavior and densification effect

Figure 7 shows the densification behavior of various Al_2O_3 -10 wt.% TCP- TiO_2 samples that contain different amounts of Titania.

It is noted that the addition of Titania has been effective in improving the densification and lowering the porosity. In fact, the addition of TiO_2 improves the density (by 30%) compared to that recorded without TiO_2 . The maximum recorded densification rate is around 90% following an addition of 5% TiO_2 which is suitable for an optimum in the porosity of the order of 10%. Beyond this percentage, the density of these samples decreases slightly. So, a higher densification and a lower porosity for this composition indicates its good resistance.

3.2 Mechanical proprieties

In order to evaluate the material stiffness, ultrasound technique, is retained to estimate the elastic modulus E for different percentages of TiO_2 . The evolution of elastic modulus of Al_2O_3 -10 wt.% TCP- TiO_2 is illustrated in **Figure 8**. As can be seen, the elastic modulus increases with the amount of Titania additive until 194.96 GPa for 5 wt.% TiO_2 . Beyond the 5 wt.% TiO_2 , the overall stiffness falls

Figure 6.
Average particle size distribution plot composition for different amounts of TiO_2 .

Figure 7.
Relative density and porosity of specimens added with different amounts of Titania.

Figure 8.
Elastic modulus and hardness of Alumina-10 wt.% TCP-TiO₂ versus percentage of Titania sintered at 1600 °C for 1 h.

gradually. The Vickers indenter was applied to determine the hardness. On the other hand, at the same filler content of 5 wt % Titania, it was found that this composite provides the highest hardness values than those given by Alumina-10 wt% TCP (**Figure 8**).

Regarding the mechanical stresses, these were determined via bending, compression and Flattened Brazilian tests. The results are presented in **Figure 9**.

The tensile, flexural and compressive strength appears as being dependent on the amount of Titania additive.

It is observed that the tensile strength σ_t switches from 26.68 MPa for Alumina-10 wt.% TCP to 86.65 MPa with the addition of 5 wt.% TiO₂. For the results of the bending test, the flexural strength σ_f relative to the Alumina- 10 wt.%TCP-TiO₂ composite reaches 98 MPa.

On the other hand, and for the variation in the compressive strength σ_c of the elaborated specimens with different percentages of the Titania, one notices an

Figure 9. Tensile strength, flexural strength and compressive strength Alumina-10 wt.% TCP-TiO₂ versus percentage of Titania sintered at 1600 °C for 1 h.

improvement in the compressive resistance σ_c whose maximum value reached is of the order of 352 MPa in the presence of 5 wt.% Titania (**Figure 9**).

Figure 10 shows the fracture toughness of different amounts of Titania added to Alumina-10 wt% TCP composite. As sintered at 1600°C/1 h, the toughness is improved to 13 MPa m^{1/2} after 5 wt% TiO₂ adding.

3.3 Friction and wear behavior results

Likewise, the impact of adding Titania on the tribological properties of alumina-10 wt% TCP was explored with various TiO₂ percentages.

Representative plots the evolution of friction coefficients (COF) as a function of the time for different percentages of Titania tested under normal loading of 9 N at a sliding speed of 200 rpm are displayed in **Figure 11a**.

All friction curves display similar tendency for the different samples.

COF was found to increase abruptly at the beginning of the sliding test. This can be attributed to the topography of the initial surface roughness or fresh surface that has an influence on the contact intensity. Then, the variations in the curves become approximately constant and the COF values stabilized. This constant values

Figure 10. Fracture toughness of different amounts of Titania added to alumina-10 wt.% TCP composite.

Figure 11.
(a) Plots of coefficient of friction (COF) versus time for Alumina-10 wt.% TCP-Titania composites in dry ambient condition. (b) The average steady COF as a function of % Titania.

corresponds to the average steady state value of the COF [33]. This step is named: stable wear stage.

Figure 11b presents the evolution of the friction coefficient versus the different compositions of samples during one hour (1 h) of sliding.

It is observed that a significant difference was noted between Alumina-10 wt.% TCP and Alumina-10 wt.% TCP- TiO₂ composites. Actually, the specimens were found to behave distinguishable depending on the percentage of titania under the explored conditions. In fact, the specimen of Alumina-10 wt.% TCP recorded maximum COF-values around 0.49, while Al₂O₃-10 wt.% TCP-TiO₂ specimens showed lowest COF-values about 0.325.

After each test, wear resistance and wear volume were estimated for different specimens.

The evolution of the wear rate and wear volume of Alumina-10 wt.% TCP-TiO₂ as a function of different Titania percentages is presented in **Figure 12**. According to this figure, it is noticed that the increasing of Titania content can lead to an increase in the wear resistance. The significant increase of this resistance is observed for 5 wt.% of Alumina- 10 wt.% TCP-TiO₂.

For the optimum composition, the wear volume decreases four times compared to Alumina- 10 wt.% TCP. Likewise, the lowest wear volume (0.26) was recorded for the Al₂O₃-10 wt.% TCP-5 wt.% TiO₂ composite.

Figure 12.

Wear volume and wear rate of different samples (Alumina- 10 wt.% TCP- TiO_2) composites.

These low wear volumes are expected by the significantly higher values of the micro-hardness of the composites which go from 3800 MPa for Al_2O_3 -10 wt.% TCP, to reach 9000 MPa, in the presence of 5% TiO_2 with a tenacity of the order of $13 \text{ MPa m}^{1/2}$. Indeed, the good densification of Al_2O_3 -10% TCP-5% TiO_2 composite is due to mass transfers in liquid phase sintering. In this study, the liquid phase corresponds to an eutectic transformation between the initial powder (TCP) and the additive (TiO_2).

The tribological behavior of the biomaterials and specially the biocoating depends on the geometry of the contact, the tensile strength and also the surface roughness. The topography and surface roughness is an essential parameter during sliding contact which influences factors that govern sliding and wear behavior, the contact mode and the behavior of the interfacial medium.

The presence of pores in materials decreases the actual contact area between the two materials in contact, thus appears the cracks between the pores, causing wear debris to form. The wear resistance is thus reduced. The increase of the wear of these composites was probably due to the poor porosity of the Al_2O_3 -10 wt.% TCP-5 wt.% TiO_2 (10%) (**Figure 7**).

To provide more information on wear mechanisms, microscopic observations of the worn surfaces are retained.

The specimens were analyzed by SEM after a duration of 1 hour and at a sliding speed of 200 rpm under a load of 9 N. **Figure 13** presents typical damages such as wear scars, micro-cracking, mechanical fracture and abrasion marks on the surfaces of all samples.

By comparing the **Figure 13a-c** and **e**, it is observed that the area of wear scar was smaller on the Al_2O_3 -10 wt.%TCP- TiO_2 composite surface than Al_2O_3 -10 wt.% TCP composite. This confirms that the addition of Titania to Al_2O_3 -10 wt.%TCP enhances the wear resistance and tribological behavior [34]. For Al_2O_3 -10 wt.% TCP-Titania composites, the debris became less deep and often compacted (**Figure 13b, d and f**).

3.4 Physicochemical characterization

The mechanical and tribological behavior confirms that a significant improvement in mechanical and tribological properties in the presence of 5 wt.% titania.

Figure 13. SEM images of worn surfaces after testing against zirconia ball under 9N of (a) and (b) Al_2O_3 -10 wt.% TCP (c) and (d) Al_2O_3 -10 wt.% TCP-5 wt.% TiO_2 , (e) and (f) Al_2O_3 -10 wt.% TCP-10 wt.% TiO_2 .

In order to find an explanation for this improvement, an in-depth physico-chemical study was carried out to obtain more information, in particular, the microstructural changes of the samples.

Figure 14 exhibits XRD patterns of Al_2O_3 -10 wt.% TCP, and Alumina-10 wt.% TCP-5 wt.% Titania composites sintered for 1 h at 1600°C. A new information was added about solid-state reactivity in the ternary system Al_2O_3 -TCP- TiO_2 . In fact, the spectra indicate the presence of traces of β -TCP, α - Al_2O_3 , and new phases relative to β - Al_2TiO_5 ; aluminum titanate (**Figure 14**). This is an intermetallic compound relating to (β - Al_2TiO_5) in the binary system Al_2O_3 - TiO_2 . It is a thermodynamically stable compound above 1280°C, formed as a result of a reaction between α - Al_2O_3 and TiO_2 in an oxidizing atmosphere. Note also that the intensity of the peak increases with the TiO_2 content in the Al_2O_3 composite – 10 wt.% TCP.

The micrograph (**Figure 15a**) displays a continuous phase and the spherical pores in the Al_2O_3 -10 wt.% TCP composites. The microstructure of the Al_2O_3 -10 wt.% TCP composites sintered with Titania show a high densification (**Figure 15b-d**).

Figure 14.

XRD patterns of different composites Alumina-10 wt.% TCP-Titania versus percentage of Titania sintered at 1600 °C for 1 h.

For Al_2O_3 -10 wt.% TCP-2.5 wt.% TiO_2 composite, we notice a high intergranular porosity (**Figure 15b**).

An important improvement of the characteristic performances of the Al_2O_3 -10 wt.% TCP-5 wt.% TiO_2 composite was obtained by the addition of titania.

The microstructural analysis of the Al_2O_3 -10 wt.% TCP-5 wt.% TiO_2 composite reveal high densification (90%) of the specimens. The creation of a liquid phase (between TCP and TiO_2) (**Figure 15c**) that can induce a higher wear and mechanical strength through the transition from solid sintering to liquid sintering. In summary, a small amount of liquid phase due to the presence of a TCP- TiO_2 eutectic caused the densification of the composites and the improvement of the mechanical properties of the Al_2O_3 -10 wt.% TCP-5 wt.% TiO_2 composite.

On the other hand, the addition of high percentages of TiO_2 hinders densification was found that the intragranular pores and the growth of the grains are more important, consequently decreases in mechanical properties. For the composite Al_2O_3 -10 wt.% TCP-10 wt.% TiO_2 composite (**Figure 15d**), it was found that there are intragranular pores and grain growth.

When increasing TiO_2 content in Al_2O_3 -10 wt.%TCP composites, the mechanical and wear resistance of the Alumina-10 wt.% TCP composites were enhanced. In fact, the wear volume and the mechanical stresses of the Al_2O_3 -10 wt.% TCP composites decreased to a minimum value with 5 wt.% Titania. In our research, the mechanical and tribological behavior of Alumina-TCP- TiO_2 bioceramics is

Figure 15.
SEM micrographs of the Alumina-10 wt.% TCP-Titania composites sintered at 1600 °C for 1 h: (a) Al_2O_3 -10 wt.% TCP, (b) Al_2O_3 -10 wt.% TCP-2.5 wt.% TiO_2 (c) Al_2O_3 -10 wt.% TCP-5 wt.% TiO_2 , (d) and (e) Al_2O_3 -10 wt.% TCP-10 wt.% TiO_2 .

enhanced by incorporating TiO_2 , that's agrees well with other similar works [34, 35].

An important improvement of the characteristic performances of the Al_2O_3 -10 wt.% TCP-5 wt.% TiO_2 composites were successfully obtained by the addition of titania oxide.

This important improvement can be explained by the high densification of the samples (90%), the creation of a liquid phase (between TCP and TiO_2) (**Figure 15c**).

4. Conclusion

In this chapter, we have focused on the effect of Titania on mechanical and tribological characterizations of Alumina-10 wt.% TCP biomaterials manufactured as coating for orthopedic implant.

After the sintering process, Alumina-10 wt.% TCP-Titania composites have been characterized by using X-ray diffraction and SEM analysis. The mechanical properties have been investigated by the Flattened Brazilian test, compression test, semi-circular bending test and NanoIndenter. A pin-on-disk tribometer was used to ensure sliding and wear experiments under dry condition and against zirconia ball. A 2D profilometer was retained to measure the wear volume.

The main results are summarized as follows:

The produced Al_2O_3 -10 wt.% TCP- TiO_2 composites with different percentages of Titania (1 wt.%; 2.5 wt.%; 3 wt.%, 4 wt.%, 5 wt.%, 7.5 wt.% and 10 wt.%) exhibited much better mechanical properties than the reported values of Alumina-TCP without titania.

After the sintering process at 1600°C for 1 hour, the Al_2O_3 -10 wt.% TCP composites showed a higher elastic modulus, compressive strength, flexural strength, and fracture toughness which certainly increased with the Titania content and reached the optimum value with 5 wt.%. However, no cracks were observed in the microstructure of this composition.

In terms of tribological properties, the composite Alumina-10 wt.% TCP-5 wt.% Titania presents excellent wear rate and wear volume. Accordingly, the lowest wear volume (0.003%) was recorded for this composite. The mechanical properties and wear behavior of Alumina-TCP- TiO_2 biomaterial is enhanced by incorporating Titania. This important improvement can be explained by the high densification of the composite (90%) and the creation of a liquid phase (between Tricalcium Phosphate and Titania).

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References

- [1] Anderson J.M, Biological responses to materials. Annual review of materials research.2001;31(1),81-110. doi.org/10.1146/annurev.matsci.31.1.81
- [2] EBRAHIMI Mehdi, BOTELHO, Michael G., et DOROZHUKIN, Sergey V. Biphasic calcium phosphates bioceramics (HA/TCP): Concept, physicochemical properties and the impact of standardization of study protocols in biomaterials research. Materials Science and Engineering: C.2017;71, 1293-1312. doi:10.1016/j.msec.2016.11.039. Epub 2016 Nov 12
- [3] D Williams, An introduction to medical and dental materials, in: D. Williams (Ed.), Concise Encyclopedia of Medical & Dental Materials, Pergamon Press and The MIT Press. 1990; pp. xvii–xx
- [4] Sprion S, Guicciardi S, Dapporto M, Melandri C, Tampieri A. Synthesis and mechanical behavior of β -tricalcium phosphate/titania composites addressed to regeneration of long bone segments. J MechBehav Biomed Mater. 2013;17:1–10. doi.org/10.1016/j.jmbb.2012.07.013
- [5] Barker LM. A simplified method for measuring plane strain fracture toughness. EngFract Mech. 1977;9(2): 361–364. doi.org/ 10.1016/0013-7944(77)90028-5
- [6] Sakka S, Bouaziz J, Ben Ayed F. Sintering and mechanical properties of the alumina-tricalcium phosphate-titania composites. Mater. Sci. Eng. C. 2014;40:92–101. doi.org/10.1016/j.msec.2014. 03.036
- [7] Chih-Jen Wang, Chi-Yuen Huang. Effect of TiO_2 addition on the sintering behavior, hardness and fracture toughness of an ultrafine alumina. Materials Science and Engineering: A.2008: 492(1-2), 06-310. doi.org/10.1016/j.msea.2008.04.048
- [8] KIM, Hyun-Min, MIYAJI, Fumiaki, KOKUBO, Tadashi, et al. Preparation of bioactive Ti and its alloys via simple chemical surface treatment. Journal of Biomedical Materials Research: An Official Journal of the Society for Biomaterials and The Japanese Society for Biomaterials. 1996;32(3),409-417. doi: 10.1002/(SICI)1097-4636(199611)32:3<409::AID-JBM14>3.0.CO;2-B.
- [9] KANEKO Hideki, UCHIDA, Masaki, KIM, Hyun Min, et al. Process of apatite formation induced by anatase on titanium metal in simulated body fluid. In: Key Engineering Materials. Trans Tech Publications Ltd. 2002; p. 649-652. doi:10.1016/j.biomaterials.2005.01.048
- [10] MEHMANPARAST, H. N., MAC-THIONG, J. M., et PETIT, Yvan. Compressive properties of a synthetic bone substitute for vertebral cancellous bone. International Journal of Biomedical and Biological Engineering. 2012;6(5),144-147. doi.org/10.5281/zenodo.1077803
- [11] LIU, Chen, WAN, Peng, TAN, Li Li, et al. Preclinical investigation of an innovative magnesium-based bone graft substitute for potential orthopaedic applications. Journal of Orthopaedic Translation, 2014; 2(3)139-148. https://doi.org/10.1016/j.jot.2014.06.002
- [12] Liu H, Li H, Cheng W, Yang Y, Zhu M, Zhou C. Novel injectable calcium phosphate/chitosan composites for bone substitute materials. Acta Biomater. 2006;2(5):557–565. DOI: 10.1016/j.actbio.2006.03.007
- [13] Ayatollahi MR, Aliha MRM, Saghaei H. An improved semicircular bend specimen for investigating mixed mode brittle fracture. EngFract Mech. 2011;78(1):110–123. doi.org/10.1016/j.engfracmech.2010.10.001 11.

- [14] XIE, Yousheng, CAO, Ping, JIN, Jin, et al. Mixed mode fracture analysis of semi-circular bend (SCB) specimen: a numerical study based on extended finite element method. *Computers and Geotechnics*. 2017;82:157–172. doi.org/10.1016/j.compgeo.2016.10.012
- [15] KUMAR, Alok, BISWAS, Krishanu, et BASU, Bikramjit. Fretting wear behaviour of hydroxyapatite–titanium composites in simulated body fluid, supplemented with 5 g l⁻¹ bovine serum albumin. *Journal of Physics D: Applied Physics*. 2013;46(40),404004. doi:10.1088/0022-3727/46/40/404004
- [16] FELLAH, Mamoun, SAMAD, Mohammed Abdul, LABAIZ, Mohamed, et al. Sliding friction and wear performance of the nano-bioceramic- α - Al_2O_3 prepared by high energy milling. *Tribology international*. 2015; 91,151-159. doi.org/10.1016/j.triboint.2015.07.006
- [17] Nine MJ, Choudhury D, Hee AC, Mootanah R, Osman NAA. Wear debris characterization and corresponding biological response: artificial hip and knee joints. *Materials*. 2014;7(2),980–1016. doi: 10.3390/ma7020980
- [18] SAKKA, S., AYED, F. Ben, et BOUAZIZ, J. Mechanical properties of tricalcium phosphate–alumina composites. In: *IOP Conference Series: Materials Science and Engineering*. IOP Publishing, 2012;012028. DOI: 10.1088/1757-899X/28/1/012028
- [19] MAZEL, Vincent, GUERARD, Sandra, CROQUELOIS, Benjamin, et al. Reevaluation of the diametral compression test for tablets using the flattened disc geometry. *International journal of pharmaceutics*, 2016; 513,669-677. DOI : 10.1016/j.ijpharm.2016.09.088
- [20] WANG, Q. Z., JIA, X. M., KOU, S. Q., et al. The flattened Brazilian disc specimen used for testing elastic modulus, tensile strength and fracture toughness of brittle rocks: analytical and numerical results. *International Journal of Rock Mechanics and Mining Sciences*. 2004;41(2) 245-253. doi.org/10.1016/S1365-1609(03)00093-5
- [21] KURUPPU, Mahinda D. et CHONG, Ken P. Fracture toughness testing of brittle materials using semi-circular bend (SCB) specimen. *Engineering Fracture Mechanics*. 2012; 91,133-150. doi.org/10.1016/j.engfracmech.2012.01.013
- [22] DAI, F., XIA, K., ZUO, J. P., et al. Static and dynamic flexural strength anisotropy of Barre granite. *Rock Mechanics and Rock Engineering*. 2013; 46(6),1589-1602. DOI: 10.1007/s00603-013-0390-y
- [23] LIM, I. L., JOHNSTON, I. W., CHOI, S. K., et al. Fracture testing of a soft rock with semi-circular specimens under three-point bending. Part 1—mode I. In: *International journal of rock mechanics and mining sciences & geomechanics abstracts*. Pergamon. 1994;185-197. DOI: 10.1016/0148-9062(94)90463-4
- [24] LIU, Hua, LI, Hong, CHENG, Wenjun, et al. Novel injectable calcium phosphate/chitosan composites for bone substitute materials. *Acta Biomaterialia*. 2006;2(5)557-565. doi.org/10.1016/j.actbio.2006.03.007
- [25] ASTM C1424-15, Standard Test Method for Monotonic Compressive Strength of Advanced Ceramics at Ambient Temperature, ASTM International, West Conshohocken, PA, 2015C.
- [26] RAHMATI, Maryam et MOZAFARI, Masoud. Biocompatibility of alumina-based biomaterials—A review. *Journal of cellular physiology*. 2019;234(4)3321-3335. doi.org/10.1002/jcp.27292

- [27] NATH, Shekhar, UMMETHALA, Raghunandan, et BASU, Bikramjit. Fretting wear behavior of calcium phosphate-mullite composites in dry and albumin-containing simulated body fluid conditions. *Journal of Materials Science: Materials in Medicine*. 2010;21(4)1151-1161. doi.org/10.1007/s10856-009-3983-y
- [28] KHALLADI, A. et ELLEUCH, K. Tribological behavior of wheel-rail contact under different contaminants using pin-on-disk methodology. *Journal of Tribology*, 2017;139(1). doi.org/10.1115/1.4033051
- [29] JENG, Yeau-Ren, LIN, Tsung-Ting, HSU, Hsiu-Ming, et al. Human enamel rod presents anisotropic nanotribological properties. *Journal of the mechanical behavior of biomedical materials*. 2011;4(4)515-522.DOI: 10.1016/j.jmbbm.2010.12.002
- [30] KARAVAEV, M. G. et KUKAREKO, V. A. Automated tribometer with reciprocating. *Nadezhnostmashin-itekhnikheskikhsistem-Reliability of machines and technical systems*. 2001(1),37-39.
- [31] Sharma S K, Kumar B V M, Kim Y W. Effect of WC addition on sliding wear behavior of SiC ceramics. *Ceram Int*. 2015;41(3),3427-3437. doi.org/10.1016/j.ceramint.2014.10.144
- [32] Guerrazi N, Elleuch K, Ayedi HF, Fridrici V. Kapsa P, Tribological behaviour of pipe coating in dry sliding contact with steel. *Mater Des*. 2009;30(8):3094-104. doi:10.1016/j.matdes.2008.12.003
- [33] KALIN, Mitjan, JAHANMIR, Said, et IVES, Lewis K. Effect of counterface roughness on abrasive wear of hydroxyapatite. *Wear*. 2002;252(9-10) 679-685. DOI: 10.1016/S0043-1648(02) 00028-5
- [34] SUN, X. J. LI, J. G. Friction and wear properties of electrodeposited nickel-titania nanocomposite coatings. *Tribology Letters*. 2007;28(3)223-228. DOI: 10.1007/s11249-007-9254-5
- [35] Miao. X, Sun. D, Hoo. P. W, Liu. J, Hu. Y, Chen. Y, Effect of titania addition on yttria-stabilised tetragonal zirconia ceramics sintered at high temperatures. *Ceramics international*. 2004;30(6)1041-1047. DOI: 10.1016/j.ceramint.2003.10.025