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Development and Study on Various Properties of Titanium Oxide -Tri Calcium Phosphate Composites through Powder Metallurgy Technique

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ABSTRACT: *In this study, we made an attempt to develop titanium oxide-tricalcium phosphate (TiO_2-TCP) composites due to its excellent biocompatible properties and performance. Pure β-tricalcium phosphate wasprepared via a wet precipitation process and studied its physical and mechanical properties. The synthesized β-tricalcium phosphate powder was analyzed for their phases and functional groups using X-ray diffraction technique (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) respectively. Different quantities (3wt. %, 5 wt.%, and 10 wt. %) of Titanium oxide (TiO_2) were incorporated into β-tricalcium phosphate to fabricate the composites at different sintering temperatures. XRD analysis was also carried out to examine the changes in various phases for the composite products after sintering. The scanning electron microscopy (SEM) was performed to analyze the formation of pore and measure pore size, grain size using perfect screen ruler software. Physical properties like density, porosity, shrinkage and mechanical property like hardness were studied and reported for various sintering temperatures for each sample.*

Keywords—*β TCP, Powder Metallurgy, Sintered structure, Shrinkage, Density, Porosity, Vicker'shardness.*

I. INTRODUCTION

The requirement for new materials are increased increasingly in modern biomedical industry to fulfil the requirement of implants. TCP is well known biocompatible ceramics and considered as the main mineral component for bone substitutes due to their chemical and biological similarity with the constituents of human hard tissue. For this reason, calcium phosphate are widely used as implants in orthopedics and dentistry.

One of the great challenge for biomaterial engineers are to develop the materials having density nearly equal to human bone to avoid stress shrinkage after implantation. Powder metallurgy is one of the reliable technique to develop such materials with exact dimensions at high production rate and low cost [1]. In powder metallurgy products, the second phase is uniformly distributed throughout the matrix to achieve porous structures and this improves structural properties and also reproducibility [2-3]. This porous structure influences for natural bone growth between the implant and natural bone tissue.

In the past research, authors are tried to fabricate doped tri calcium phosphatewith different quantities of MgO, ZnO, CaO and bio glass via chemical route, sol-gel process, or fused deposition modeling process [4-7]. But the development of titanium oxide-tricalcium phosphate (TiO_2-TCP) composites by powder metallurgy technique is rare.

In the present study, first we have synthesis β TCP via wet chemical precipitation synthesis technique and then we have to develop TiO_2-TCP composites through powder metallurgy techniqueat different sintering temperatures.Various physical and mechanical property are observed at the different sintering temperatures for each sample.

II. MATERIALS AND METHODS

Development of TiO_2-TCP composites through Powder Metallurgy Technique

Thewet chemical precipitation methodwas adopted for preparingβ TCP powder using the reactants calcium carbonate and orthophosphoric acid both were purchased from MERCK, INDIA Ltd. and used without any pretreatment. The whole process is reported elsewhere [5, 8] followed by chemical reaction and finally calcination at 800 °C.After that,required amount of TiO_2(produced by MERCK, INDIA Ltd.) was mixed with this β TCP to fabricate the composites followed by pressing and sintering as described in our previous work [9-10].

III. RESULTS AND DISCURSION

3.1XRD Analysis

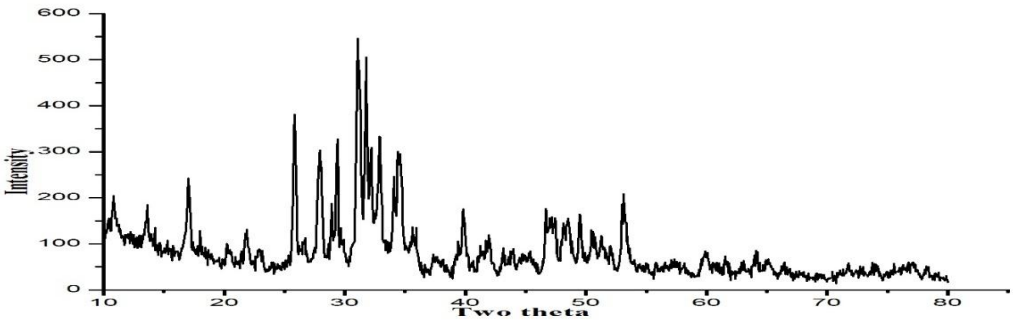


Figure 1: XRD pattern of β TCP

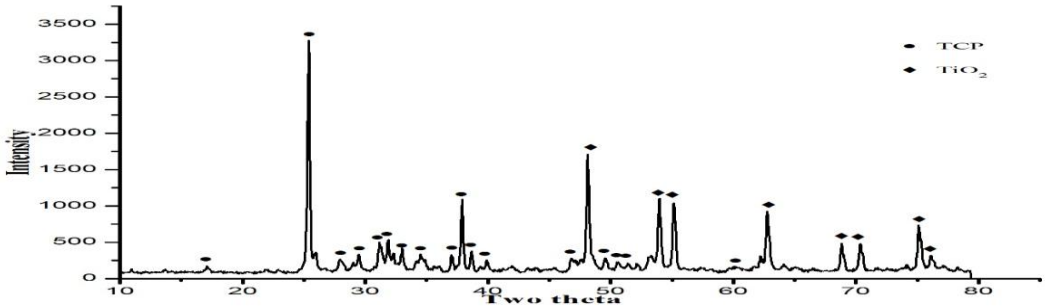


Figure 2: XRD pattern of TiO_2- TCP composite

XRD pattern of synthesizedβ TCP and its composites are reported in fig. 1 and 2 respectively.From fig. 1, pureβTCP shows highest peaks at 2θ angle of ~ 17.08^0 (d= 5.1869), 25.82^0 (d= 3.4345), 27.92^0 (d= 3.1928), 29^0 (d= 3.0763), 29.48^0 (d=3.0273), 31.84^0 (d= 2.8081), 32.24^0 (d= 2.7742), 32.96^0 (d= 2.7152), 34.84^0 (d= 2.5729), 37^0 (d= 2.4275), 38.3^0 (d= 2.3282), 39.88^0 (d=2.2586), 46.76^0 (d= 1.9410), 49.52^0 (d= 1.8391), 52.16^0 (d= 1.7521), 53.12^0 (d= 1.7226) and 62.16^0 (d= 1.4921) as revealed for this experiment, with reference card no. 09-0348 and 09-0169 for tricalcium phosphate. From fig. 2, it is seen that TiO_2-TCP composites show same peak forTCP with the addition of TiO_2 peaks at 2θ angle of ~ 48.12^0 (d= 1.8893), 53.96^0 (d= 1.6978), 55.12^0 (d= 1.6648), 62.76^0 (d= 1.4792), 68.84^0 (d= 1.3627), 70.36^0 (d= 1.3369), 75.12^0 (d= 1.2636) and 76.12^0 (d= 1.2494); analyzed with reference card no. 21-1272. Presence of the individual broad peaks for TiO_2 andTCP imply that no compound are developed other thanTCP and TiO_2, hence no reaction occurs between the matrix and the second phase in this sintering environment.

3.2 FTIR Analysis

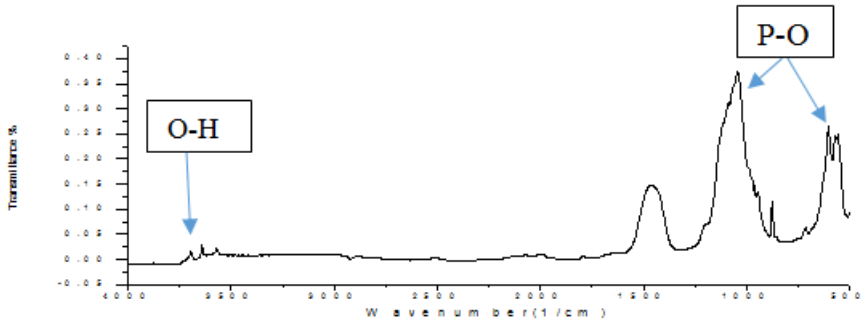
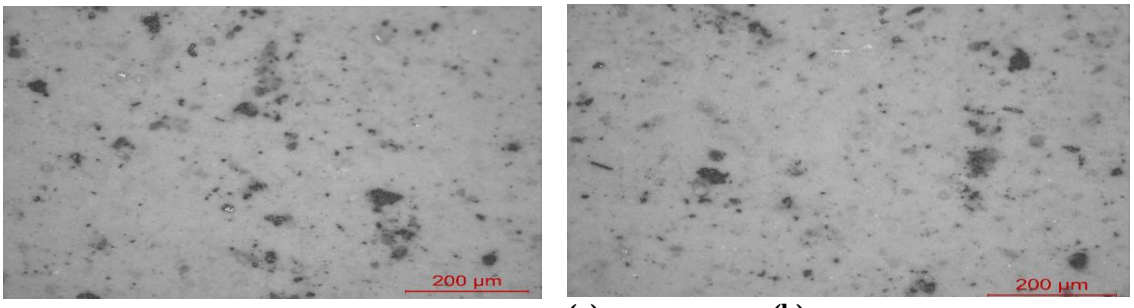


Figure 3: FTIR analysis for β TCP

All most all molecules absorb light in the IR area of the electromagnetic spectrum according to their bonds present in that molecule. In the fig. 3, intensity of absorption bonds are around 565 to 1046 cm^{-1},

associated with P-O bonding [11- 15]. This fig. also shows the presence of carbonate group around 1500 cm^{-1} and hydroxyl group around 3500 to 3800 cm^{-1}.

3.3 Microstructure



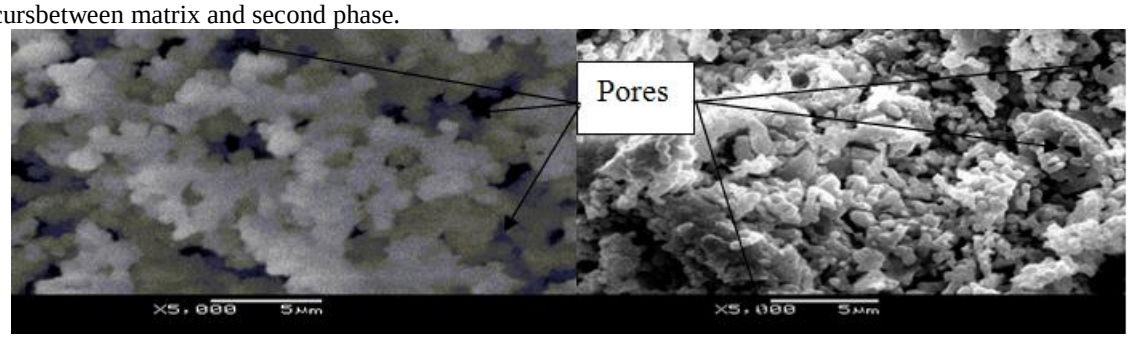
(a) (b)

Figure 4: Microstructure by optical microscopy for (a) 3 Wt. % and (b) 5 Wt. %TiO_2- TCP composite

Microstructure image was taken to observe surface structure at 100 X magnification of the developed composites as shown in fig. 4 (a) and (b). Figure shows that fine grain structure is developed and the pores are randomly oriented through out its surface. The number of pores are reduced with TiO_2 contain.

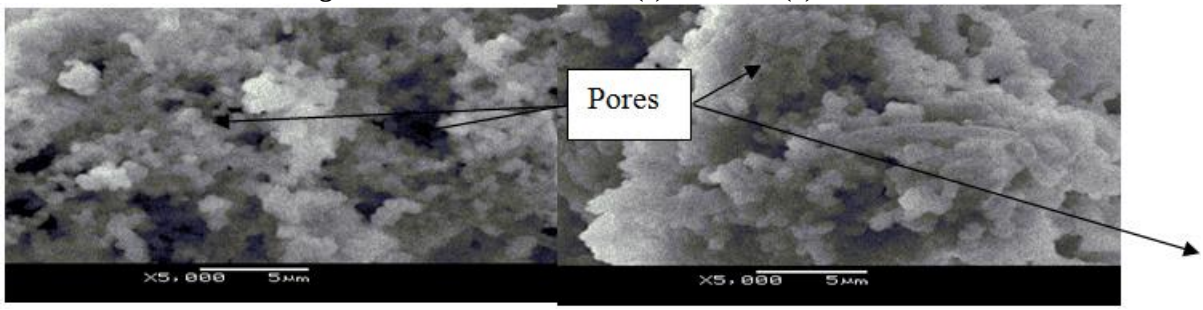
3.4 SEM Analysis

Fig.5- 8 shows the SEM image for sinteredTCP and its composites. Fig.6- 8 implies that better adhesion occursbetween matrix and second phase.



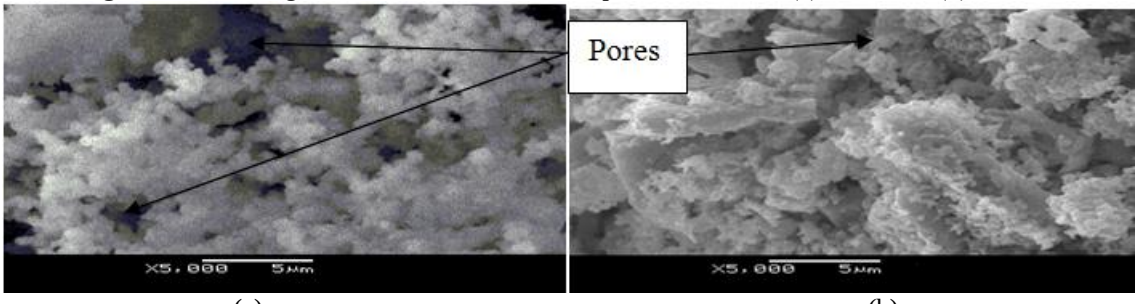
(a) (b)

Figure 5: Pure TCP sintered at (a)900^0C and (b) 1000^0C



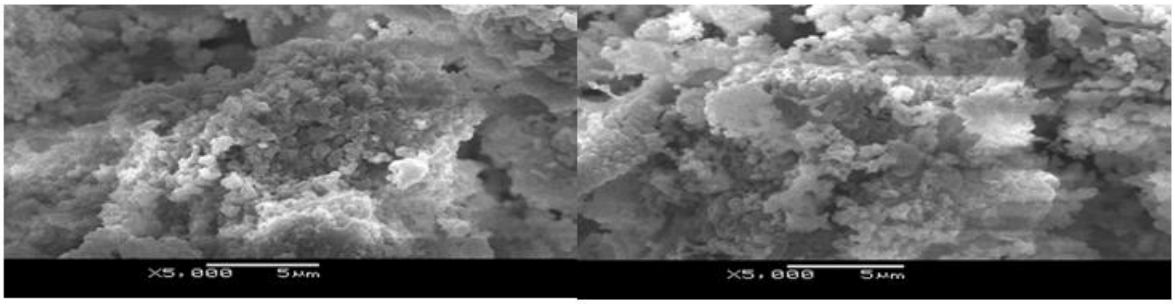
(a) (b)

Figure 6:SEM image of 3Wt. % TiO_2- TCP composite sintered at (a)900^0C and (b) 1000^0C



(a) (b)

Figure 7: SEM image of 5 Wt. % TiO_2- TCP composite sintered at (a)900^0C and (b) 1000^0C



(a) (b)

Figure 8: SEM image of 10 Wt. % TiO_2- TCP Composite sintered at (a)900^0C and (b) 1000^0C

Theformation of grain and pores of the developed composites were visualized and measured by using perfect screen ruler software imaged in SEM.The average grain size and pore size are reported in table 1 and 2respectively.

Table 1: Average Grain size Measurement

Samples after sintering	Sintering temperature900°C	Sintering temperature 1000°C
3 Wt. % TiO_2- TCP composite	501 nm	513 nm
5 Wt. % TiO_2- TCP composite	535 nm	541 nm
10Wt.% TiO_2-TCP composite	554 nm	560 nm

Table 2: Average Pore size Measurement

Samples after sintering	Sintering temperature900°C	Sintering temperature1000°C
3 Wt. % TiO_2- TCP composite	170 nm	142 nm
5 Wt. % TiO_2- TCP composite	162 nm	132 nm
10 Wt.% TiO_2-TCP composite	143 nm	112 nm

3.5 Shrinkage Measurement

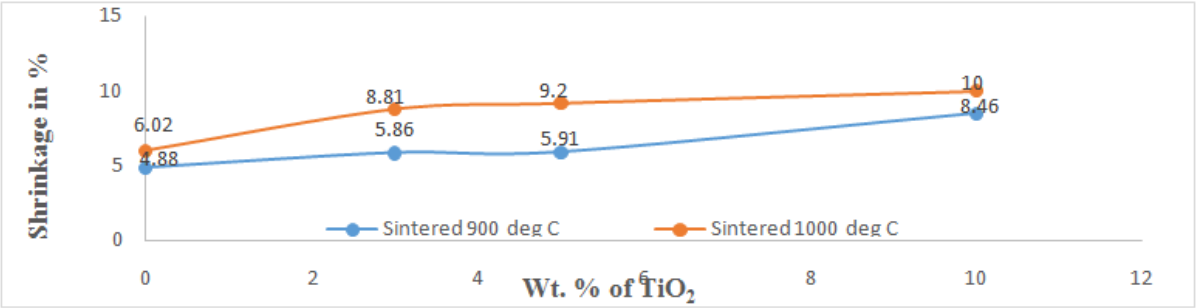


Figure9: Shrinkage for TiO_2- TCP composites sintered at different temperatures

From the shrinkage data obtained (shown in fig.9), it is found that shrinkage value increases with both temperature and TiO_2 contents. Hence, maximum shrinkageis observed for 10 % TiO_2-TCP composite sintered at temperature 1000^0C for 2 hours.

3.6 Density Measurement

Density, hence weight of the material is increased significantly after sintering. Table 3 represents the green density of the materials before sintering. Fig. 10 shows the variation of density for the developed samples, sintered at different temperatures. After sintering, density affects little bit with both sintering temperature and TiO_2 content. Table 3:Green Density

Compositions	Green density(g/cc)
Pureβ TCP	1.50
3 Wt. % TiO_2-TCP	1.54
5 Wt. % TiO_2-TCP	1.59
10 Wt. % TiO_2-TCP	1.76

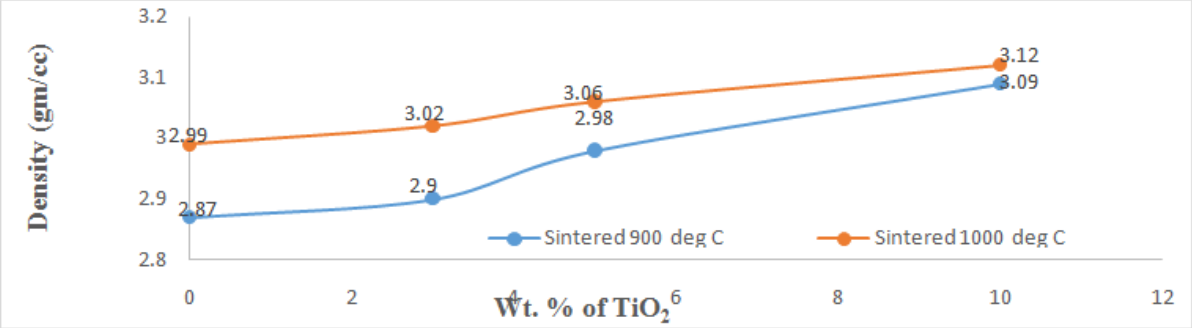


Figure 10: Density for TiO_2- TCP composites sintered at different temperatures

4.7 Apparent Porosity

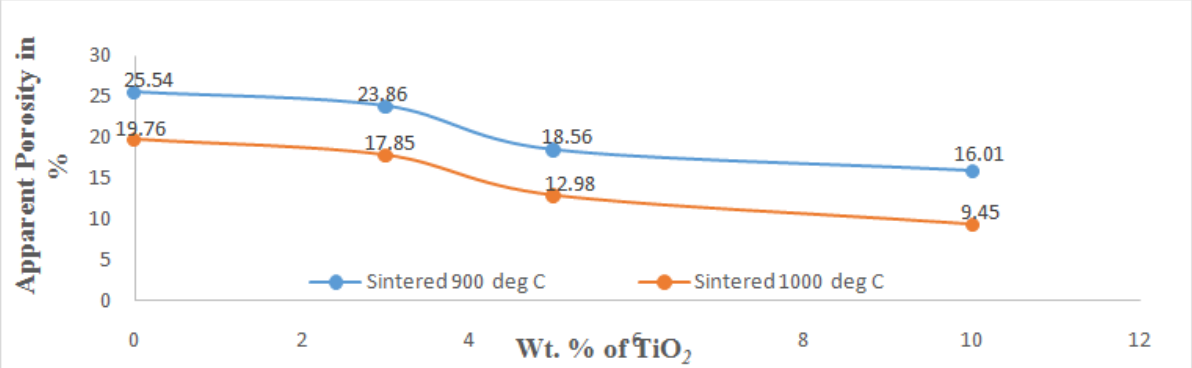


Figure 11: Apparent porosity for TiO_2- TCP composites sintered at different temperatures

Figure 11 shows the variation of apparent porosity due to increment of both sintering temperature and TiO_2 content into TCP matrix. This curve is decreasing in nature indicating that porosity, hence formation of pores is reduced with sintering temperature and TiO_2 content.

4.8 Hardness

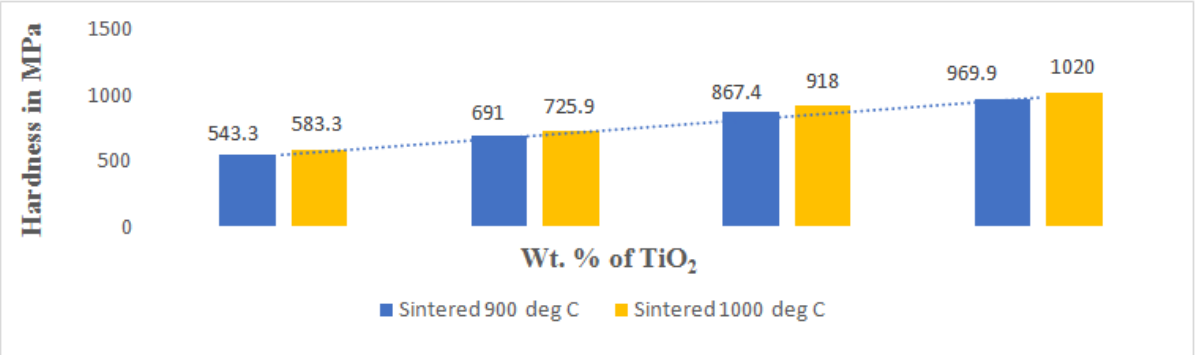


Figure12: Variation of hardness due to increment of TiO_2 into TCP matrix

Fig. 12 shows that Vicker'shardness were increased due to increment of TiO_2 into TCP matrix. Hardness value is also improved with sintering temperatures as shown in fig. 12. Hence, maximum hardness value was observed 1020MPa for 10 Wt.% of TiO_2- TCP composite,which is more than 1.7 times hardness than pure sintered TCP.

IV. CONCLUSIONS

The significant conclusions of the studies on TiO_2- TCP composites are as follows:

- TiO_2- TCP composites were developed successfully by powder metallurgy technique with tolerable porosity for use as implants.
- Density is improved after sintering, indicates the formation of bonding between matrix and second phase. After sintering, density of the composite products are slightly affected with temperature and TiO_2 content.

- Shrinkage is increased with sintering temperature and TiO_2 content. Hence maximum shrinkage is observed for 10 Wt. % TiO_2- TCP composite sintered at 1000^0 C for 2 hours.
- Hardness is increased with both sintering temperature and with incorporating TiO_2 into TCP matrix. Hardness is increased significantly up to 5 Wt. % TiO_2 content and after that improvement of hardness is not so fast with increase the percentage of TiO_2 content into TCP matrix.

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