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The Effect of Holding Time on the Mechanical and Physical Properties of Titania-Wollastonite-Hydroxyapatite Composites

R. Dinesh, S. Meenaloshini, U. Sankar

Abstract*: The effect sintering parameters and various amounts of additives on the TiO_2-HA-$CaSiO_3$ composites was investigated this study. TiO_2/$CaSiO_3$/HA composites were prepared and characterized by means of physical and mechanical properties. The addition of TiO_2 and HA to wollastonite was studied by means of bulk density and Vickers Hardness. The wollastonite composites containing TiO_2 (10-30 wt%) and HA (20-40 wt%) were sintered between 1230-1270°C, with a ramp rate of 10°C/min and a holding time of 1,1.5 and 2 hours. The results indicate that a higher sintering temperature played a significant part in enhancing the physical and mechanical properties as compared to results shown by pure wollastonite, especially for composites containing higher TiO_2 (25-30wt%) and lower HA (20-25wt%).

Index Terms*: Wollastonite, Mechanical and Physical Properties, Sintering Parameters, Holding time.

I. INTRODUCTION

The regeneration of long load-bearing bones like femur tibia, brings about a major concern in orthopaedics because biomaterials and scaffolds that are unable to take part to the biological processes. These biological processes are responsible of bone formation and remodeling. It is known that bone is a dynamic connective tissue that supports the body [1]. In order to regenerate the bone, different types of biological events by the signalling molecules, growth factors and cells migration onto the site of injury are needed [2]. As there are many accidents and wounding that happens to skeleton due to trauma or fracture, there are many patients who are in need of aid for an internal fixation device or artificial joints to curb the bone problems. Besides that, the bone should contain suitable physical and mechanical properties as they these properties play an important part in the bone's structure and durability. In these recent days, wollastonite has been studied as an implant material as it is bioactive, non-toxic and compatible with hard tissues. The apatite layer that is formed is dense and uniform on flat as well as curved surfaces [3].

Ding and Liu have prepared Titania/wollastonite composites to improve pigments as they studied the bioactivity and compatibility properties of non-heat-treated titania-wollastonite composites. Based on the research, the phases produced were rutile and anatase, besides wollastonite in a lamellar structure [4, 5]. The samples with higher content of wollastonite formed an apatite layer on the substrate, after being immersed in SBF and these samples demonstrated osteoblast proliferation resulting in cytocompatible materials [6-8]. Generally, higher elastic modulus and higher chemical durability than silicates and phosphates are shown on ceramic compounds based on titania. This research aims to improve the physical and mechanical properties by investigating the holding time when preparing the wollastonite composites.

II. MATERIALS AND METHODS

The TiO_2 / HA doped Wollastonite ($CaSiO_3$), with different TiO_2 /HA were synthesized through co-precipitation method. Various weight percentages of TiO_2 and HA were mixed with $CaSiO_3$ by wet milling in ethanol in an ultrasonic machine and milled for 1 hour. The slurry was dried at 60°C in an oven for 12 hours. The mixture was then sieved through a 212µm mesh stainless steel sieve to obtain a ready-to-press TiO_2 / HA powder. The mixed powder was pressed in a hardened steel circular (20 mm in diameter) and rectangular (80 x 50 x 8mm) mold and die set under a hydraulic pressure of 500 MPa.

Pressing was followed by the consolidation of the samples by ambient pressure sintering performed in air using a heating furnace (ModuTemp) between 1230ºC to 1270ºC. The sintering parameters used was by using a ramp-rate of 10ºC/min for both heating and cooling, and holding time of 1, 1.5 and 2 hours prior to cooling to room temperature. All samples were polished using SiC papers (120, 240, 600, 800) from coarse to rough, followed by polishing with a diamond paste to 6µm to obtain an optical reflective surface.

Samples were immersed in distilled water to measure density using the Archimedes' Principle using a Mettler Toledo Balance AG204 densi-meter. Vickers hardness was tested on the polished samples by means of the Vickers indentation method. The load applied to the samples was kept constant at 98.1 N with a loading time of 10s.

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R.Dinesh, Department of Mechanical Engineering, Universiti Tenaga Nasional, Kajang, Malaysia

S.Meenaloshini, Department of Mechanical Engineering, Universiti Tenaga Nasional, Kajang, Malaysia

U. Sankar, Department of Mechanical Engineering, Universiti Tenaga Nasional, Kajang, Malaysia

The compressive strength was determined using rectangular bar samples in order to determine compression at maximum load.

III. RESULTS

A. Bulk Density

The bulk density of the $TiO_2/CaSiO_3$/HA composites sintered with 3 different holding times (i.e. 1, 1.5, 2 hours) was investigated and observed for various sintering temperatures as shown in Figures 1, 2 and 3. Almost all samples of all holding times showed an increasing trend in density at a low sintering temperature (1230-1240), with results varying between 2.7 to 3.2g/cm^3. Between this temperature range, the achieved the highest bulk density values were obtained for 30wt% TiO_2/50wt% $CaSiO_3$/20wt% HA samples with a holding time of 1.5 hours, approximately 3.15 g/cm^3. The value obtained proved to be a significant increase from the theoretical value of density for wollastonite (2.90 g/cm^3) [9], approximately 8.6% higher. Sintering at 1240°C with a holding time of 1.5 hours showed increasing densification for all compositions except for the 15wt% TiO_2/50wt% $CaSiO_3$/35wt% HA samples. Hence it could be concluded that the optimum sintering temperature and holding time was found to be 1240°C and 1.5 hours respectively. Both the sintering temperature and holding times play a pivotal part in increasing the bulk density. During the sintering process, the porosity of the sample may have been reduced, resulting in higher bulk density.

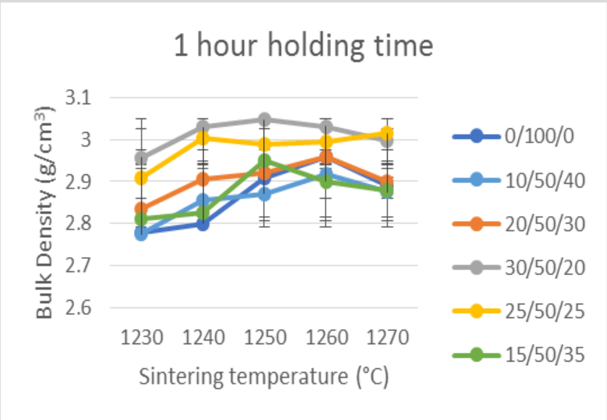

Fig. 1 sintering temperature

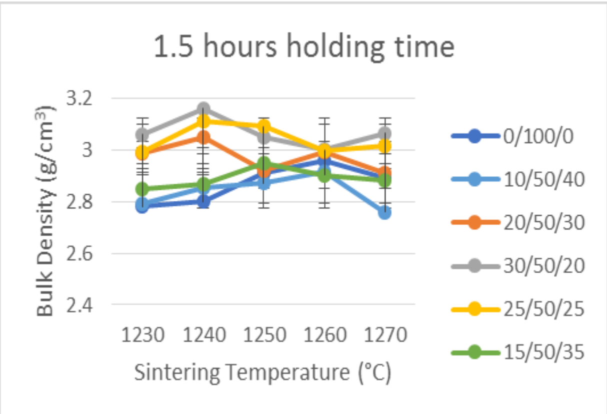

Fig. 2 sintering temperature

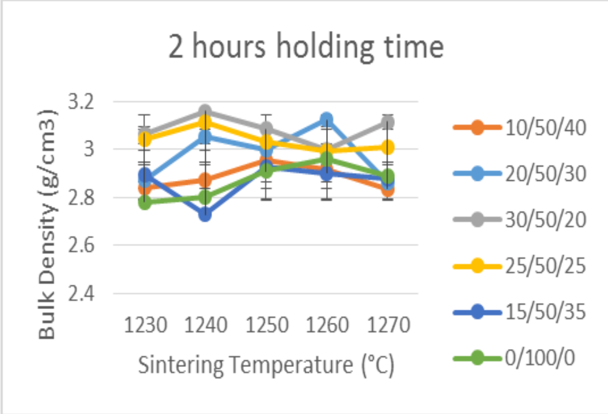

Fig. 3 sintering temperature

B. Hardness

The mechanical properties of the sintered composites was investigated based on Vickers hardness, relative to the sintering parameters such as the holding times, additive amount and sintering temperatures. The effect of TiO_2 and HA additions on the Vickers hardness of wollastonite sintered from 1230ºC to 1270ºC with various holding times is shown in Figures 4,5 and 6. The Vickers hardness values of the composites varied in a more even manner as compared to that of bulk density. All samples displayed an increasing trend at low sintering temperatures (1230 – 1250ºC), followed by a slight decline as the temperature increased from 1250 - 1270ºC onwards. Pure wollastonite showed the lowest value of hardness obtained, about 3.00 GPa at the initial sintering temperature (1230°C), followed by a very minor increase with increasing temperature, and the highest hardness achieved was 3.1 GPa. The highest hardness achieved was approximately 4.4 GPa at 1240ºC, for the samples composition of 30wt% TiO_2/ 50wt% $CaSiO_3$/ 20wt% HA. The hardness obtained was seen to be slightly higher than the theoretical hardness of wollastonite (~4.09 GPa). The results show that the holding times and additives enhanced the hardness of wollastonite sintered at lower temperatures (between 1240 - 1250°C). Theoretically, the density and hardness of the wollastonite composites are directly proportional to each other (seen between 1230- 1240°C). This was proven as the results in Figure 1 and Figure 2 showed a similar increasing trend, especially for sample compositions 30wt% TiO_2/ 50wt% $CaSiO_3$/ 20wt% HA and 25wt% TiO_2/ 50wt% $CaSiO_3$/ 25wt% HA. The increase in density in the composites exhibit stronger bonding between the grains in the sintered composites, which suggests that greater hardness and strength can be obtained. Liu et. al [10] made an observation stating that increase of hardness with sintering temperature due to high degree of crystallization resulted in more wollastonite existing within the matrix of the composite material.

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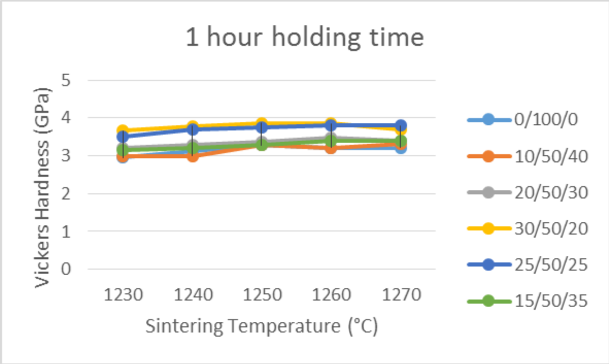

Fig. 4

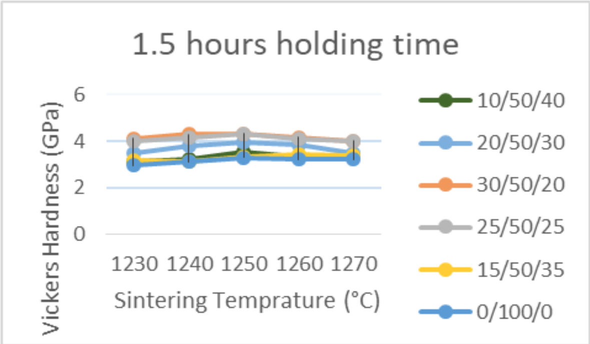

Fig. 5

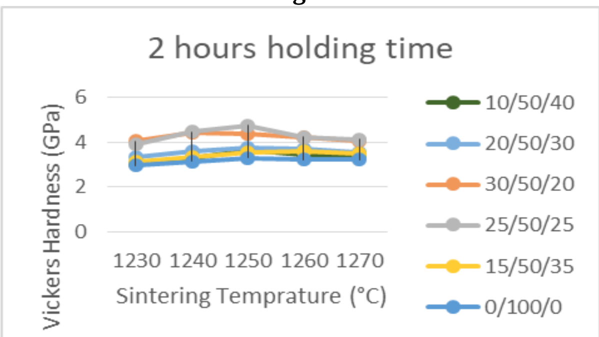

Fig. 6

IV. CONCLUSION

The present study investigated the effect of holding times on the TiO_2 and HA additions to improve the physical and mechanical properties of wollastonite. The results show that 1.5 hours holding time the properties significantly enhanced the mechanical properties of wollastonite. The highest value of density obtained was about 3.15 g/cm^3, approximately 8.6% higher than the theoretical density value (2.90 g/cm^3). The mechanical properties of wollastonite also showed an increase above. Sintering at low temperatures (1230-1240ºC) with a holding time of 1.5 hours was seen to be the optimum temperature and holding time to achieve the best properties. Sintering above 1250ºC was found to cause detrimental changes to both physical and mechanical properties.

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AUTHORS PROFILE

Dinesh Ragurajan obtained his Bachelor's Degree as well as Master's Degree in Mechanical Engineering at Universiti Tenaga Nasional. He is currently pursuing his PhD in Mechanical Engineering at Universiti Tenaga Nasional. He specialized in Advanced Ceramics, Biomaterials, Nanomaterials as well as Material Characterization. He has published about 25 journal papers thus far. He has also participated in several exhibitions such as ITEX and MTE and won Gold, Silver and Bronze medals. He is currently an active researcher and a teacher at Regent International School.

Dr Meenaloshini Satgunam obtained her first degree at Western Michigan University, and then pursued her Master's degree in Universiti Putra Malaysia and finally her Ph.D degree in Universiti Tenaga Nasional(UNITEN). She had successfully completed 2 e-Sciencefund grant and 1 FRGS grant. She specializes in the area of Biomaterial, Nanomaterial and Material Characterization. She has published over 40 journal papers . She has also participated in several exhibitions such as PECIPTA, ITEX and MTE and won several Gold and Silver medals. She currently holds the position of Head of Unit, Student-Staff Affairs in the Department of Mechanical Engineering in UNITEN.

Umma Sankar Gunasegaran obtained his first degree at Universiti Tenaga Nasional (UNITEN), and then pursued his Master's degree in UNITEN and now pursing PhD in UNITEN. He specializes in the area of Biomaterial, Nanomaterial and Material Characterization. He has published about 3 journal papers . He has also participated in several exhibitions such as PECIPTA and ITEX and won several Gold and Silver medals. He currently holds the position of Programme Leader in the Department of Mechanical Engineering in Tunku Abdul Rahman University College.