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Processing of Milkfish Bone on Al/Ti/Mg Into Hybrid Composites by Self-Propagating High Temperature Synthesis (SHS)

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***Abstract*—Powder metallurgy is a manufacturing process for a component that shape combine of making powder compaction. Determination final properties on hydroxyapatite (HAp)/Al_2O_3/SiC have an important role as a reinforced to determine the final properties on a biomaterial composite. Using of self-propagating high temperature synthesis (SHS) method can produces the material with high density, strength, ductility, and stiffness. Hybrid composites include the usage of ceramic and metal like aluminum, titanium, magnesium, copper, SiC, and Al_2O_3. By SHS method using the exothermic heat reaction in the solids between the powder that been compacted to produce many kinds of material on high temperature process. Sample on this research consists of HAp, Al, Ti, Mg, Cu, SiC, and Al_2O_3, it was mixed and compacted at 7 tons of pressure. Variable on this research consists of HAp, Al and Ti composition, and holding time on SHS process. SHS process carried out at 600^0C for 2, 3, 4 hours. Highest hardness achieved on usage of composition 2 about 666,52 HVN with using of 20% HAp, 35% Al, 30% Ti and 3-hours of holding time. The lowest hardness about 400.78 HVN achieved on usage of composition 1 with using of 10% HAp, 40% Al, 35% Ti and 3-hours of holding time.**

***Keywords:** hydroxyapatite, aluminum, hybrid composites, self-high temperature synthesis (SHS).*

I. INTRODUCTION

Powder metallurgy (PM) is the most contributed method significantly on developing implant [1]. Used of PM method is flexible to determine the microstructure what we want. Hydroxyapatite (HAp) can be applicated on the orthopedy and dental, because HAp is the most biocompatible and bioactivity material [2]. HAp on medical can be use as coating on a metallic implant and can be use as matrix on composite material, especially hybrid composites [3]. Usage of HAp have disadvantages on properties; brittle, lack of flexibility, cluping, and problems about dispersion [4]. HAp can be source from the fish bone, one of them is chanos chanos forsk (CCF). HAp can be achieved by doing the calcination process. The result of the laboratory research shows, there is have 4.7756% Calcium (Ca), and 1.3125% Phospor (P) that contains in the chanos chanos forsk [5]. To resolve about the problem of hydroxyapatite as a biomaterial, the usage of metallic materials that have better properties can be used. It can be use of hybrid composite method. That is a merging two or more reinforce to achieve the stiffness, strength and ratio between weight to strength. The usage of hybrid composites has a potential, to replace the single reinforced composite to increase few properties that want to be achieve. Metallic metals such as: Al, Ti, Mg can be used as alloy on biomaterial because their compatibility properties. Another metallic metal like Cu can be used as alloy, but in the small amounts. Ceramic reinforce like SiC and Al_2O_3 can be used to improve the structure and the material properties [6]. The usage of SHS on PM can be produce result with high purity, metastable phase, synthesis and densification in all side of the product [5]. SHS process have many advantage if we compare with the conventional method, like in the aspect of production cost [7]. This study used HAp from milkfish bone on calcination temperature of 600^{O}C with the addition on pressing, during the SHS process in order to produce mechanical properties that suit of standart component. Refers to exploring the ideal composition from using HAp/Al_2O_3/SiC for biomaterial applications.

II. Procedures of Experimental

A. *Preparation of calcination on milkfish bones/chanos chanos forsk for hydroxyapatite (HAp)*

Chanos chanos forsk (CCF) soaked in the boiling water to easily the cleaning process and dried under the sun until it dry. After that, the CCF get calcinated in the muffle furnace at 600°C during 5-hours of holding time. Color transform to be white, then the calcinated CCF be mashed till it fine to be powder. As shown in Fig. 1. In the previous experiment [8], HAp was produced with the compound: $Ca_5(PO_4)3(OH)_2$. 5-hours was ideal of time to produce of HAp. If used more than 5 hour and more than 700°C so color will turn black as a result of an oxidation reaction.

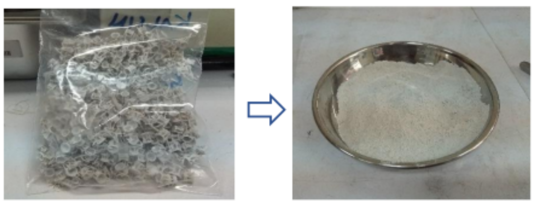


Fig. 1. HAp made from calcination. (*for matrix of hybrid composites*)

B. *Fabrication of hybrid composites*

The calcination process of CCF is able to convert banding bone into HAp, then mixed with metal powder and other ceramics such as Al, Ti, Mg, Cu, SiC, Al_2O_3. This study uses 3 types of composition, variations of Al, Ti, and HAp. The composition of the study is presented in table 1. After that, the mixed powder was weighed 1 gram for each sample. Then compacted using a mold at compaction pressure of 7 tons.

TABLE I. Composition of Hybrid Composites

Element of Materials	Element of Hybrid Composites		
	C1	*C2*	*C3*
HAp	10%	20%	30%
Al	40%	35%	35%
Ti	35%	30%	20%
Mg	10%	10%	10%
Cu	1%	1%	1%
SiC	2%	2%	2%
Al_2O_3	2%	2%	2%

C1, C2, C3: Composition 1, 2, 3

C. *Self-propagating high temperature synthesis (SHS) proces*

The SHS process for all hybrid composite samples with different percentage compositions, as shown in table 1. Hybrid composite samples were placed in a refractory with a closed position by the SHS tube. Then put into a muffle furnace. The process is carried out using a temperature of 600°C and holding time varies, between: 2, 3, and 4 hours for each sample composition. The scheme of the SHS process is shown in figure 2

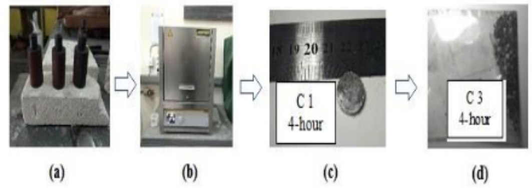


Fig. 2. a). SHS Preparation b). Muffle Furnace c). Sample Result d). Broken Sample

III. Result and Discussion

The research led to variations in three different compositions and different times of detention. From the results of the use of different compositions, there were 3 damaged samples destroyed as presented in figure 2. The phenomenon of damage to composition samples 2: when the sample temperature reached room temperature, after the SHS process was carried out. Because of this failure, the sample cannot be tested.

The characterization used consisted of hardness, X-ray fluorescence, chemdraw analysis and scanning electron microscope. Hardness testing using a tool: microhardness Vickers tester MITUTOYO HM-200 type, to determine resistance to deformation at each point. while the x-ray aims to determine the composition of elements formed after the SHS process, using INNOVX type OLYMPUS. To determine the reaction of compounds formed, Chemdraw analysis simulation was used as a tool. Scanning Electron Microscope (SEM) FEI QUANTA 650 type was used to determine the grain morphology distribution.

A. *Microhardness Vickers (VHN)*

Microhardness vickers testing have been done on the sample of hybrid composites from SHS process shown the result on the table 2 and fig. 3. As shown in the hardness table varies from various compositions. The best hardness was achieved in composition 2 which consists of: 76% metal (Al/Ti/Mg/Cu) and 24% was ceramic (HAp/Al_2O_3 and SiC). In composition 2 the hardness uses a percentage of 86% metal composition accompanied by 14% ceramics.

TABLE II. Hardness of Hybrid Composites

Sample	Holding Time		
	2 Hour (HVN)	**3 Hour (HVN)**	**4 Hour (HVN)**
Composition 1	561.03	400.78	514
Composition 2	609.73	666.52	611.03
Composition 3	*Unidentified*	*Unidentified*	*Unidentified*

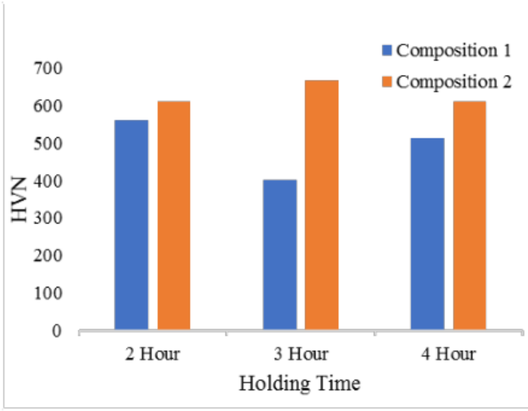


Fig. 3. Result of Vickers-Hardness Testing

Hardness shows the ability of the metal to withstand the load against plastic deformation, so that by measurement of microhardness it will reveal the mechanical properties of the material measured [9]. In the results of this study the use of 3 hours of holding time for the SHS process produces the best mechanical properties of 666 VH, compared to 1-hour/4-hour, with an average hardness achievement of only 609-611 VH. 3-hour temperature holding in the SHS process is the best time to produce mechanical properties. This indicates that the 3-hour time is the best time to diffuse between the constituent elements (HAp, Al, Ti, Mg, SiC and Al_2O_3) to produce the complex compound shown in the chem draw analysis illustrated in Figure x

B. X-ray fluorescene (XRF) characterization

XRF testing is performed to determine the composition of elements formed after the SHS process. The XRF test results are shown in tables 3 - 5 below. Based on the results of the mechanical properties shown in table 2 and fig. 3, when compared with the XRF results, showed the highest and lowest mechanical properties, as well as the hybrid composite fracture failure. hybrid composites with the highest mechanical properties contain 35% Al, but samples with the lowest mechanical properties contain 17.06% Al and failure samples have the lowest percent Al. Aluminum is the non-ferrous metals cluster, by very wide applications. It has specials properties such as being light weight, ductile [10]. The most striking characteristic of aluminum is its melting point which is suitable for producing elemental reactions with nonmetallic material elements such as ceramics [11].

TABLE III. XRF COMPOSITION 1 OF HYBRID COMPOSITES

Material	**Holding Time**		
	2-hour (%)	**3-hour (%)**	**4-hour (%)**
Mg	30.38	65.24	31.86
Al	34.47	17.06	31.69
Si	0.79	0.2067	0.59
Ti	29.14	15.44	31.63
Cu	2.41	1.2199	1.5624
HAp	*remains*	*remains*	*remains*

TABLE IV. XRF COMPOSITION 2 OF HYBRID COMPOSITES

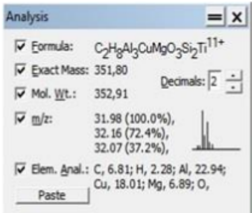
Material	**Holding Time**		
	2-hour (%)	**3-hour (%)**	**4-hour (%)**
Mg	23.05	26.66	21.98
Al	34.51	35.46	35.34
Si	1.53	1.24	1.13
Ti	33.7	30	36.52
Cu	3.44	2.74	2.53
HAp	*remains*	*remains*	*remains*

TABLE V. XRF COMPOSITION 3 OF HYBRID COMPOSITES

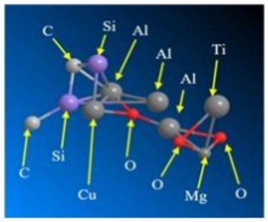
Material	Holding Time		
	2-hour (%)	3-hour (%)	4-hour (%)
Mg	-	-	-
Al	9.02	10.65	8.47
Si	2	2.42	1.71
Ti	61.23	59.48	65.87
Cu	14.14	13.9	12.48
HAp	*remains*	*remains*	*remains*

C. Chemdraw Analysis

To predict the compounds formed in hybrid composites made from HAp, Al, Ti, Mg as well as Sic and Al_2O_3, the analysis used "*Chemdraw Ultra*". Besides that, the analysis is to find out the molecular bonds and formulas formed from the use of HAp composition with metals and ceramics. The formula that is formed is $C_2H_8Al_3CuMgO_3Si_2Ti^{11+}$. Fig. 4 a, was shown a compound formed from the results of the SHS process. The compounds formed are chain bonds with different processes. This was a bond of produces a hybrid composite base material. Among the bonds, in fig. 4 b, the oxide (O) is in the position of the bond lattice between Al-Mg-Ti, this happens because the affinity of the oxygen value is closer to the metal element than to ceramics. This phenomenon results in increased of hardness, but decreased of elongation, so the value of formability will low. Composition 3 in table 1-C3, there was no Mg content, referring to XRF data on the table 5. According to [12] Mg acts as a wetting agent, so that if there is no mg content there is no bonding contact area between metal particles and ceramics, so that the elements in the hybrid composite do not bind.



(a)



(b)

Fig. 4 a). The result of chemdraw analysis b). Molecular Bond of hybrid composites

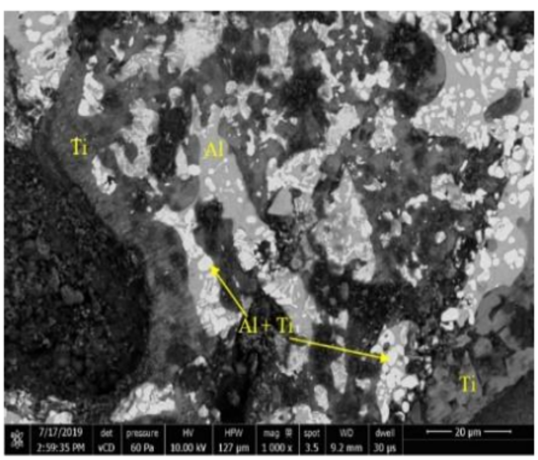
Based on the results of the reaction between the constituent elements of hybrid composites, such as HAp from the banding bone with metal and ceramic elements, the product compounds are obtained as follows:

$$HAp+Al+Ti+Mg+Cu+SiC+Al_2O_3$$

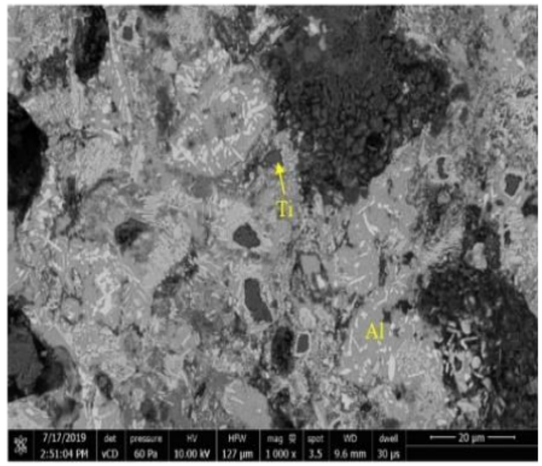
$$\Rightarrow C_2H_8Al_3CuMgO_3Si_2Ti^{11+} \quad (1)$$

D. Scanning Electron Microscope (SEM)

SEM observation has done with the sample that have the highest hardness and the lowest hardness that achieved in this research. This testing was done by using the magnification 1000x on the surface of the sample. The fig. 5 a. shows the SEM result from the highest hardness sample and fig. 5 b. shows the SEM result from the lowest hardness sample. Based on the SEM result, the white color of that figure shows the combination of HAp+Al+Mg+Ti+Cu phase. Then, the grey color is the phase of Al and the dark color that pointed on the figure is Ti. From the both of figure 5, the lowest hardness was achieved because the Al phase didn't perfectly forms. Meanwhile on the sample with the highest hardness was achieved because there is have many forms of Al/Mg/Ti phase if we compare with the lowest hardness sample. So that with the Al and Ti that forms on the sample, it cause the sample to achieved highest hardness.



(a)



(b)

Fig. 5. a) SEM result of hybrid composites highest hardness: 666.52HVN b) SEM result of hybrid composites lowest hardness: 400.78HVN

IV. CONCLUSSION

Based on the research have been done to produce the hybrid composite with the usage of CCF waste as the source of hydroxyapatite, with using the self-propagating high temperature synthesis (SHS) method, can be concluded the effect of holding time and composition variation of Al-Ti to the microstructure that produce on the sample is:

1. Highest hardness was achieved by using the composition 2 and using the 3 hours of holding time, about 666,52 HVN. Meanwhile the lowest hardness result was achieved by using the composition 1 and 3-hour of holding time, about 400.78 HVN.

2. Sample with the highest hardness was achieved because there is have many forms of Al+Ti phase if we compare with the lowest hardness sample. So that with the Al and Ti that forms on the sample.

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REFERENCES

[1] Dewidar Montaser. Influence of Pprocessing Parameters and Sintering Atmosphere on the Mechanical Properties and Microstructure of Porous 316L Stainless Steel for Possible Hard-tissue Applications. International Journal of Mechanical & Mechatronics Engineering IJMME-IJENS Vol: 12 No: 01 (2012).

[2] Mucalo Michael. Hydroxyapatite (HAp) for Biomedical Applications. Woodhead Publishing, Cambridge. ISBN 978-1-78242-041-5. (2015).

[3] Gary E Wnek, Gary L Bowlin, (2008). Encyclopedia of Biomaterials and Biomedical engineering - second edition, volume 1. Informa Healthcare, Newyork. ISBN: 978-142007956-2.

[4] Zindani, Divya Kumar, Kaushik Davim, J Paulo, (2019). Mechanical Behavior of Biomaterials. Elsevier Ltd. Doi: https://doi.org/10.1016/B978-0-08-102174-3.00004-8.

[5] Pramono, A. Kommel L. Kollo, L and Veinthal, R (2016). The Aluminum Based Composite Produced By Self Propagating High Temperature Synthesis, Medziagotyra, 22 (1), pp. 41–43. doi: 10.5755/j01.ms.22.1.7500.

[6] Chabri, Sumit Chatterjee, Subhranshu Pattanayak, Santanu Chakraborty, Himel Bhowmik, Nandagopal Sinha, Arijit, (2013). Development and Characterization of Al2O3 Dispersed Al/Mg/Cu/Ti Matrix Composite. Journal of Materials Science & Technology. Vol. 3. Elsevier Ltd. Doi: 10.1016/j.jmst.2013.07.004.

[7] Xanthopoulou, G. and Vekinis, G. (2001). An overview of some environmental applications of self-propagating high-temperature synthesis, Advances in Environmental Research, 5(2), pp. 117–128. doi: 10.1016/S1093-0191(00)00048-4.

[8] Pramono, A., Sulaiman, F., Suryana. Milandia. A., Calcination Process of Milkfish Bone - Chanos Chanos Forsk (CCF) for Hybrid Composites Application. IOP Conf. Series: Materials Science and Engineering 532 (2019) 012004, pp. 1-8: IOP Publishing doi:10.1088/1757-899X/532/1/012004.

[9] Pramono A. Dhoska K. Markja I. Kommel L (2019) Impact Pressure on Mechanical Propereties of Aluminum Based Composites by ECAP-Parallel Channel. Pollack Periodica-An International Journal for Engineering and Information Sciences, 14. 1 pp. 67–74.

[10] Pramono, A. Kollo, L. Kallip, K. Veinthal, R and Gomon, JK. Heat Treatment of Ultrafine Grained High-Strength Aluminum Alloy. Key Engineering Materials Vol. 604 (2014) pp 273-276.

[11] Pramono, A. Jamil, AM and Milandia, A. Aluminum based composites by severe plastic deformation process as new methods of manufacturing technology. MATEC Web of Conferences 2018; 218, 04011, pp. 1-9: //doi.org/10.1051/matecconf/201821804011.

[12] Pramono, A. Milandia, A. Dhoska, K and Kommel, L. Properties of Metal Matrix Composites by Pressureless Infiltration (PRIMEXX). Proceedings Book The 8th of Annual BASIC Science International Conferences.March 6-7, 2018.Pp. 272-279

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