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The effect of the ball size on the product characteristics of shaker HEM to produce nano particle from bamboo charcoal

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Abstract. The aim of this research is to study the effect of the ball size on characteristics of the product of high energy milling (HEM) of shaker type to produce nano particle from bamboo charcoal. A new shaker mechanism is developed. The characteristics of the particle are represented by particle size, surface morphology, and the substances presence in the product. Particle size analyzer (PSA) is conducted to have the particle sizes, whereas SEM and EDX are used to have surface morphology and substances presence in the products respectively. The mixtures of 11 gr of bamboo charcoal powder of 200 mesh and about 299 gr of steel ball are placed in vials. The vial is from stainless steel cylinder with 11/4 inch diameter and 120 mm length. The stroke of the shaker is 54 mm at 233 rpm. The number of cycle is 4 million cycles. The steel ball sizes are 1/8, 5/32, 3/16 and 1/4 inch. PSA results show that there is no certain correlation between the steel ball size and particle size. The final shape of the particles is determined by fracture mechanism. The highest substance presence in the result is C.

1. Introduction

High energy milling (HEM) is a production method where a blend of powder put in the ball mill and is exposed to high impact energy of collision from the balls. HEM often called as mechanical alloying is one of the most broadly method used to produce ultrafine materials. Mechanical alloying was established in the mid 1960 by John Benjamin to have nickel-based super alloys of oxide dispersion strengthened (ODS) for the applications of gas turbine. HEM process is commonly employed in the industries of ceramic and metal processing [1]. The process in HEM comprises of repetitive fracture, mixing, and cold welding of a fine blend powder (alloy particles) causing in size reduction of the particles and sometimes in chemical reactions. Currently, the HEM is used to make nanostructured materials which are intensively researched. HEM equipment are available in different types both for mechanical alloying and nanoparticle formation. They are different in their size and or milling efficiency.

Examples of research in preparing nanostructured material using HEM can be seen in [2-6]. Li and Ramanujan [2], studied the microstructure evolution and formation of nanocrystal line FeCo based alloys. Mishra *et al.* [3] conducted a research to study the magnetic characteristics of nano-composite materials of iron nitride–alumina made by HEM. Hamzaoui *et al.* [4] studied the magnetic properties and structure of nanocrystalline of mixture of Fe-10wt % Ni and Fe-20 wt% Ni alloys. Pilarczyk *et al.* [5] did a research to examine the structure and characteristics of Fe-Co-Ni-B-Si-Nb alloy. El-

Eskandarany *et al.* [6] conducted a research to study the effect of milling speed in ball milling process. They have a conclusion that by increasing the speed of rotation, it will decrease the time necessary for the development of an amorphous phase in the Co-Ti system.

Nanotubes, nanowires and nanorods are one-dimensional (1D) nano materials. It has been showed by many researchers that nanomaterials have many advantages compared to the bulk material in terms of properties, functionalities and a wide range of promising applications. However, for the future industrial usages there is a major obstacle for the large-quantity production of the nanomaterials. Chen *et al.* [7] reported that there is potential process to achieve mass production of nanomaterials by having ball milling and annealing process. To show such capability, several examples are presented in their work. The examples included C, BN nanotubes and SiC, Zn nanowires. Vertical rotating ball mill and a planetary ball mill are used in this Study. From this research, it can be stated that the formation of the nanotubes and nanowires is determined by the prior HEM process. The nanotubes and nanowires cannot be produced by annealing process without the prior ball-milling process.

In this research, shaker type of HEM is used to produce nano particle from bamboo charcoal. A new shaker mechanism is developed. This research is part of the examination of the new mechanism to examine some variables which influence the nano particle results. This paper reports the effect of the ball size to the characteristics of the results.

On the other hand, the demand for nano particle of pure carbon in large quantities and low costs is a lot. Charcoal is one of the alternative of carbon sources. In Indonesia the used of bamboo is very much which results in a lots of unused material. Bamboo has carbon content more than 90% of its weight [8]. It is a potential carbon source. The unused material of bamboo can be made to become charcoal. As far as, author's knowledge, there is very limited research on the bamboo as carbon source for nano-carbon particle. This research tries to have carbon nano-particle at large quantity and low cost production from bamboo.

2. Experiments

Different types of high-energy mills (HEM) have been created. In this research, a new shaker mechanism is developed (see figure 1(a)). The shaker mechanism is obtained from rotational motion changed to translational motion by connecting rod. The length of the stroke is determined by the length of the connecting rod. The speed of the motor can be controlled and the ration of the motor pulley to the connecting rod pulley is 1:3.

(a) (b)

Figure 1. Materials used in this research (a) shaker type of HEBM (b) vials.

The vial is from stainless steel cylinder with 1¼ inch diameter and 120 mm length (see figure 1(b)). The contents of the vial are 11 gram of bamboo charcoal powder of 200 meshes and about 299 gram of steel ball with diameter of 1/8, 5/32, 3/16 and 1/4 inch. A total of four vials can be run at the same time to produce large quantities of materials. The stroke of the shaker is 54 mm at 233 rpm. Characterization of the particle is conducted by Particle Size Analyzer (PSA), Scanning Electron Microscope (SEM) and Energy Dispersive X-Ray (EDX). The characteristics of the particle are represented by particle size, surface morphology, and the substances presence in the particle.

3. Results and discussion

The results of particle size analyzer are shown in table 1. It can be seen that the increase in diameter of the steel ball does not have any certain correlation to the decrease of the particle size. The smallest particle having average size of 273.8 nm is produced by the ¼ inch ball diameter. The sizes of 490.1 nm, 573.3 nm and 515.2 nm are produced by the 1/8, 5/32, and 3/16 inch ball diameter respectively. The size reduction process is explained as follows: at the time of the milling process, materials are repetitively crushed, broken and welded [7]. Collisions happen in between the balls or the balls and the vial wall. The collisions setup some particles between their surfaces. The high impacts energy from the collision severely deform the particles and create atomically fresh, new surfaces, as well as a high density of dislocations and other structural defects. Moreover, the deformation and breaking of particles make the continuous size reduction.

Table 1. Particle size analyzer results.

Steel ball diameter (inch)	Average Particle Size (nm)
1/8	490.1
5/32	575.3
3/16	515.2
1/4	273.8

The increase in diameter means the increase in mass. At the same speed, it also means that the impact energy is increased. If the impact energy is higher, it should reduce the particle size smaller. In this case, the 5/32 inch and 3/16 inch steel ball diameter have higher impact energy than 1/8 inch. They should produce smaller particle than 1/8 inch. However, it does not happen. According to [9] and [10] too long milling time cause agglomeration. Under SEM examination, the morphology of the agglomerated particles seems bigger than un-agglomerated particles. In this research, the agglomeration of the particles occurred for the ball sizes of 1/8, 5/32, and 3/16 inch. It can be seen in figure 2 (a), (b), and (c). The particles produced by the three balls appear in sheet due to the agglomeration. Whereas, the particle resulted from the ¼ inch ball did not agglomerate. It can be seen on figure 2(a).

If the impact energy is enough to prevent agglomeration, according a research conducted by [11], there will be grinding limit. Grinding limit is condition at which the increase number of milling time has no effect on the particle size. Kim reported that after HEM process for 30 hours of Fe-Co powders, the milling process attained a steady state. In this condition, the particles have become homogenized in size and shape. Other research, showing the grinding limit is done by [12]. After milling of Ni–Al powder for 100 hours, the particle size and shape become homogenous. Umemoto *et al.* [13] conducted research on milling of Fe-C. After 500 hours milling time the grinding limit is reached and the particle size is 4.7 nm with homogenous shape.

The agglomeration did not happen for the ¼ ball after 4 million cycles. But grinding limit is not reached yet. SEM morphology of the particles as in figure 2(a) shows this fact. It can be seen that the particle size is not homogenous yet. Even though the average size of the particle is 273.8 nm (as in PSA result), it can be seen that there are still particles with more than 1 µm. In this research, 4 million cycles are equivalent to 288 milling hours. The particles have fractional shape resulted from fracture mechanism.

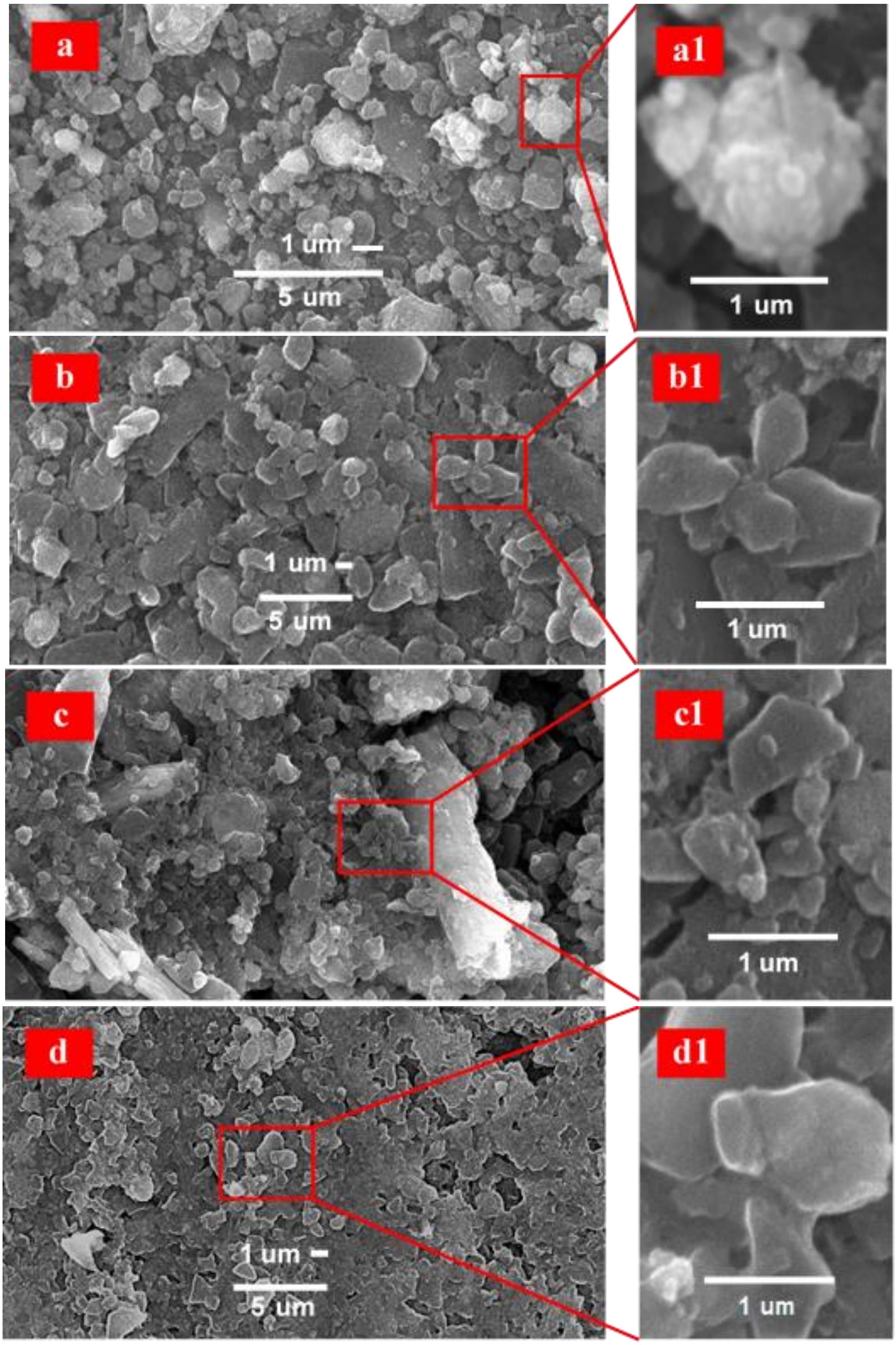

Figure 2. SEM morphology of 4 million cycles, (a) is for ¼ inch, (b) is for 3/16 inch, (c) is for 5/32 inch and (d) is for 1/8 inch).

Table 2 is the substances presence in the particles as a result of EDX. It can be seen that the highest component is carbon. This result is not in line with the result of [8]. They reported that the carbon content of bamboo is more than 90% of its weight. In this research the only one having carbon more than 90% is the result of ¼ inch ball diameter which is 93.03%. The results of 1/8 inch, 5/32 inch, and 3/16 inch are 80.27%, 59.94% and 66.39% respectively.

K_2O, SiO_2 and P_2O_5 are the dominant components after carbon. K_2O has percentage of 0.55%, 8.07%, 14.47%, and 20.41% respectively for ¼ inch, 1/8 inch, 3/16 inch, and 5/32 inch. P_2O_5 has percentage of 0%, 1.37%, 2.64%, and 6.19% respectively for ¼ inch, 1/8 inch, 3/16 inch, and 5/32 inch. It can be seen that the increase percentage of carbon will have the decrease percentage of K_2O and P_2O_5. This finding needs to be studied more to have the answer. However, it can be said that bamboo is the right alternative carbon producer because it is a renewable natural resource. Bamboo is a kind of biomass that has been widely planted. Currently, bamboo resources are plentiful and the area of bamboo is about five million hectares. Similar to wood, bamboo is composed of hemicellulose, cellulose and lignin. It has great potential as a future bio-energy resource.

Table 2. Particle composition.

Element	Composition (% weight)			
	Steel ball 1/8 inch	Steel ball 5/32 inch	Steel ball 3/16 inch	Steel ball 1/4 inch
C	80.27	59.94	66.39	93.03
K_2O	8.07	20.41	14.47	0.55
SiO_2	5.66	7.90	10.99	4.19
P_2O_5	1.37	6.19	2.64	0.00
Na_2O	0.26	1.02	0.44	0.00
SO_3	0.40	0.00	2.64	0.00
Cl	1.07	3.28	0.93	0.00
FeO	0.76	0.87	0.89	0.57
CuO	0.68	0.00	0.59	0.95
ZnO	0.62	0.00	0.00	0.55
ZrO_2	0.84	0.00	0.00	0.00
MgO	0.00	0.20	0.00	0.00
Al_2O_3	0.00	0.20	0.00	0.16

4. Conclusion

HEM can be used to prepare nano particle from bamboo charcoal. The size reduction depends on the impact energy. The final shape of the particle is determined by fracture mechanism. The most dominant element presence is carbon. It seems that bamboo charcoal is a potential source to produce nano carbon.

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