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Crystal Structures and Thermal Properties of Bamboo Nanofiber Reinforced-Composite Friction Materials of Glass and Metal Wastes

Sutikno Madnasri^{1,a}, Sukiswo Supeni Edi^{2,b} dan Dany Sigit Saputra^{3,c}

^{a,b,c}Department of Physics, Faculty of Mathematics and Natural Sciences,

Semarang State University, D7 Building, 2nd Floor, Sekaran Campus, Unnes, Gunungpati,
Semarang, Republic of Indonesia, 50229

Email: smadnasri@yahoo.com

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Abstract. In Indonesia, a lot of wastes of glasses and metals have potency to be one of brake raw materials. For example, little bottles of used food packages are usually directly thrown into environment. The scraps of metal machining wastes are usually collected to be manufactured into other products. In this research, both wastes are used as fillers for brake friction materials, the effects of them on the thermal properties are studied in details. The glass wastes are crushed, grinded and filtered to simplify the mixing process with other raw materials when the fabrication of brake friction materials done. During fabrication, samples are cured at 190°C for 3 hours. The samples are characterized using x-ray diffractometer and thermogravimetric analysis. Based on these characterizations, the increase of glass powders content plays role in increasing the percentage of crystallinities. This is suspected the glass used as ingredient of friction material has crystalline structure. The glass waste quantity does not influence significantly on the thermal properties. During heating up to 1200°C, the mass loss occurs due to epoxy, bamboo fiber and styrene butadiene rubber decompose. The optimum composition is found at sample B2, a sample with lowest total mass loss (2 mg).

Introduction

At this time, the wastes have become scary monster to big city, and a potential threat to rural communities. Lots of environment damages caused by wastes, such as soil and water pollution, poisoned, stench, and spacial slum. The wastes consist of organic and anorganic materials, in which organic materials include polymers and composites. Each example of them are plastics and woods, respectively. Some of anorganic materials are metals, ceramics and glasses. The wastes originated from different materials are collected in the different drums to simplify their processing. The processing technology for each material is different.

A lot of researchers have given contributions in solving the problems of waste processing technology begin small industry scale activities up to large scale ones. In the small scale, wastes can be formed into creative products, for examples bags, keychains, furnitures, wall hangings, etc. For the large scale, wastes can be processed into electric generator energy source. Except it, wastes also have a potency to be raw materials of industry of motorcycle brake materials [1,2]. This clearly will increase economic value of those waste processing products. The metal fiber wastes can be one alternative for reinforcement of brake friction materials. While organic wastes such as coconut shell can be used as active carbon powders that serves as fillers in the need for fuels of oil and gas. The wastes of metals, glasses and coconut shells can used to fabricate motorcycle brake [3,4], and car [5,6]. The manufacturing technology of brake friction materials are one of advanced technologies that at this time just some modern countries such as China, India, Japan and US have developed it.

The market of brake materials in Indonesia is highly potential, where the population growth of vehicles are very large. The future prestigious projects of Indonesian Government such as development of monorail line and subway also require the support of the domestic brake industry with good product quality and affordable. The development of the aviation manufacture industry

like Dirgantara Indonesia (DI) Co. Ltd., also needs the spare part of brake where disc brake of airplane is a key component in the taking off and landing system [7]. Except it, this product is also employed in the defence industry.

The brake friction materials can be classified into ceramics, non-asbestos organic materials and semimetals [7]. Asbestos materials are proven as a cancer initiator for workers in industry and their consumers [8,9]) and dust which generated of para-aramid fibers can cause lung destroy and respiration system [10], so that many modern countries have banned their use in the production of brake friction materials. Since 1989 US through United States Environment Protection Agency has banned asbestos materials used as raw materials of brake friction materials [11]. Carbon/carbon (C/C) composites have relatively good thermal and mechanical properties and they are light [7]. Carbon materials are the most important of composite brake components. Therefore, brake of organic materials are necessary developed.

The performance of brake friction materials is a complex function of numerical parameters which including formulation, temperature, pressure and environment [12]. The brake composition influences on its thermal stability. The metal powder and glass quantity in the manufacture of brake are the most important aspect. Therefore, the problem in this research is first focused to investigate the crystal structures and the thermal properties of fabricated successfully composite friction materials. Next, those data can be used to determine the optimum composition of them. Specifically, this research also to scrutinize the relationship between quantity of metal and glass waste powders and their crystal structures.

Materials and Method

Raw materials used in this research include SBR-1712 (synthetic rubber), phenolic resin, epoxy, rubber curing agents (sulphur, mercapto and stearic acid), carbon of coconut shell, calcium carbonate (CaCO_3), metal waste powder, glass waste powder, magnesium oxide (MgO), bakelite and bamboo fiber.

The production equipments used consisted of autoclave, mixer, mould (dies), and hot isostatic pressing machine. The specimens were characterized using x-ray diffractometer (XRD) and thermogravimetry analyzer (TGA) and tested using brinnel hardness testing machine (Karl Frank GmbH).

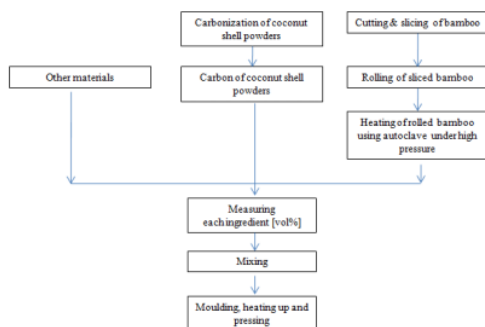


Figure 1. Flowchart of fabrication process of nanofibers reinforced-brake friction materials.

The fabrication process of nanofibers reinforced-brake friction materials is graphically shown in Figure 1. The fresh bamboo betung was cut along 1 m, the hulled and halved. The bamboo strips were flattened using the bamboo crusher 10 times, then cut along 5 cm. A total of 250 g (oven dry weight) of bamboo particles were cooked using solution of NaOH 2,5% (1:10) for 10 hours. After that, the softened already bamboo was filtered of its cooking liquid, and washed up to neutral PH achieved. The resulted pulp was stored in the freezer. The bambo fibers were dispersed for 2 hours at 240 rpm to achieve nanosize. Five grams pulp which processed by using stone refiner were dipped into 300 ml aquadest and processed using ultra torax [13].

The specimens were prepared based on the volume percent of the total volume of mold (ml), through hot pressing and curing processes at 190°C for 3 hours using hot isostatic pressing machine. For testing, the specimens were cut in the suitable dimension [14]. The fabrication technique developed by Cho *et al.* (2007), before moulding the specimens were pre-formed at room temperature [15].

To determine crystal structure, phase, and phase fraction of brake friction materials, the specimens were characterized using XRD. The thermal properties were characterized using TGA and their hardness were tested using Brinell hardness testing machine (Karl Frank GmbH) with load 153,2 N.

In this research, the contents of glass and metal fibers were fixed as independent variable, and the thermal properties of friction materials were fixed as dependent variable. The other fabrication parameters, such as pressing temperature and duration, pressure, and heat treatment temperature and duration of heat treatment, were assumed to be constant and fixed as controlled variables.

First, the content of glass and metal fibers were varied between 11,73%-24,05% of total volume of mold, other materials including bamboo fibers were also measured at constant quantity. The relationship between the thermal properties with glass and metal quantities are studied.

Results and Discussion

Five specimens varied were tested successively. The specimens were varied based on the sums of metal and glass powders as tabulated in Table 1. The brake materials were manufactured under pressure of 300 kg/cm² at 190°C for 3 hours. The bamboo fibers with 5 cm in size were mixed with other raw materials randomly based on the flowchart as appear in Figure 1.

Table 1: The composition of specimens.

Ingredients	Ingredient volume [ml]				
	B1	B2	B3	B4	B5
Glass powder	11,43	14,43	17,43	20,43	23,43
Metal powder	17,14	14,14	11,14	8,14	5,14
Epoxy	6	6	6	6	6
Bamboo fiber	11,43	11,43	11,43	11,43	11,43
Styrene butadiene rubber (SBR-1712)	12,85	12,85	12,85	12,85	12,85
Magnesium oxide	7,86	7,86	7,86	7,86	7,86
Zinc oxide	2,14	2,14	2,14	2,14	2,14
Stearic acid	2,14	2,14	2,14	2,14	2,14
Powder of Coconut shell carbon	7,14	7,14	7,14	7,14	7,14
Bakelite	9,29	9,29	9,29	9,29	9,29
Sulphur	2,14	2,14	2,14	2,14	2,14
Calciumcarbonate	7,86	7,86	7,86	7,86	7,86

The phases present were analyzed using (XRD, PANALYTICAL, X'PERT, Netherlands). The gravimetric analysis was used to determine the quantities of calcium carbonate (CaCO₃), zinc oxide (ZnO), iron (Fe) and carbon (C).

In the characterization using XRD, specimens were scanned between 2θ angle of 10 up to 90 with a scan step size of 0,02 and time scan step of 0,05. The generator was set up at 40 kV and 30 mA. The anode material used is copper (Cu). The wavelengths generated are K-Alpha1 (1.54060Å), K-Alpha2 (1.54443Å) dan K-Alpha3 (1.39225 Å). Whereas K-Beta is 1.39225 Å. The ratio of K-Alpha1 and K-Alpha2 is 0,5.

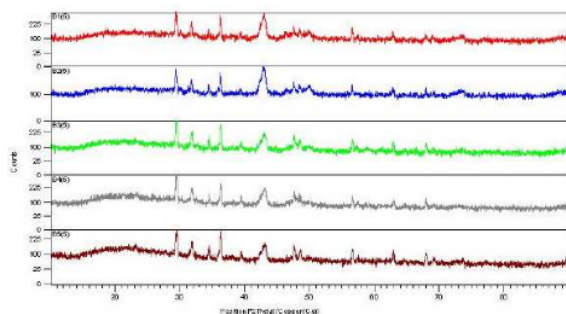


Figure 2. XRD curves of specimens.

In the x-ray scanning of B1(5), B2(5), B3(5), B4(5) up to B5(5), some compounds were found in the dominant peaks as shown in Figure 2. In general, they those are found in the dominant peaks are zinc oxide (ZnO), austenite iron ((Fe- γ), and *calcite* (CaCO₃). The other compounds also appear in the peaks, nevertheless they do not so dominant. There are three compounds only relatively dominant found in the graphs due to their distribution in the specimens are inhomogenous and the specimens taken for XRD scanning represent just selected area.

The calcium carbonate (CaCO₃) or calcite (mineral name) has crystallographic parameters as follow: crystal system of rhombohedral, space group R-3c, and space group amount of 167. Additionally, values of each lattice constant, a, b, and c, respectively are 4,9803Å, 4,9803Å and 17,0187Å with beta angle of 90° and gamma of 120°.

The crystallographic parameter of austenite iron (Fe- γ) is space group Fm-3m amount of 255. This crystal system has lattice constants of a, b and c, namely 3,6599Å, 3,6599Å dan 3,6599Å, respectively. The calculated cubic density is 7,57 g.cm⁻³. The crystallography parameter of zinc oxide are hexagonal crystal system, and 163 space groups of P63mc. The hexagonal system has lattice constants of a (3,2465Å), b (3,2465Å), and c (5,2030Å), three angles of alpha (90°), beta (90°) and gamma (120°).

The carbon element and its compound do not appear as a dominant peaks, due to carbon is not crystalline but amorphous. The area of the amorphous phase is calculated based on the area of sample amorphous reduced instrument area. The area of the crystal phase is difference between the total area and an area of amorphous samples. While the percentage of crystal calculated in the following way.

$$\% \text{ crystalline} = \text{Crystalline area} / (\text{amorphous phase area} + \text{crystalline}) * 100 \% \quad (1)$$

From Table 2 and Figure 3 it is known that the amorphous phase in each sample representing the dominant presence of carbon elements. This is consistent with testing results using EDAX conducted last year for determination of the presence of the carbon element. Crystalline phases here are contributed by metal and glass.

Table 2: The phase area and the percentage of crystallinity.

Sample	Phase area		% Crystallinity
	Crystalline	Amorphous	
B1(5)	1160.87	1688.54	40.74
B2(5)	1345.26	1688.54	44.34
B3(5)	1423.173	769.16	64.91
B4(5)	1600.663	769.16	67.54
B5(5)	758.54	1415.10	34.89

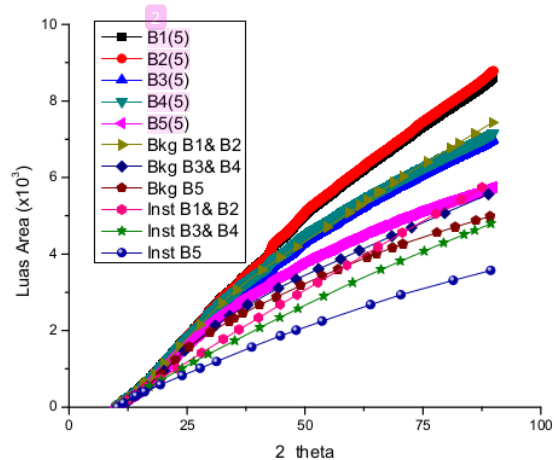
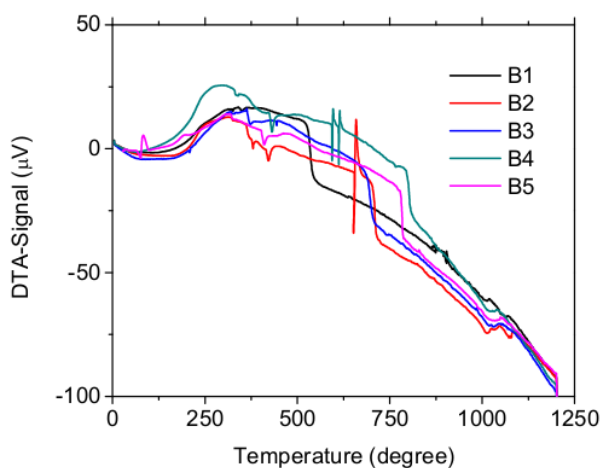


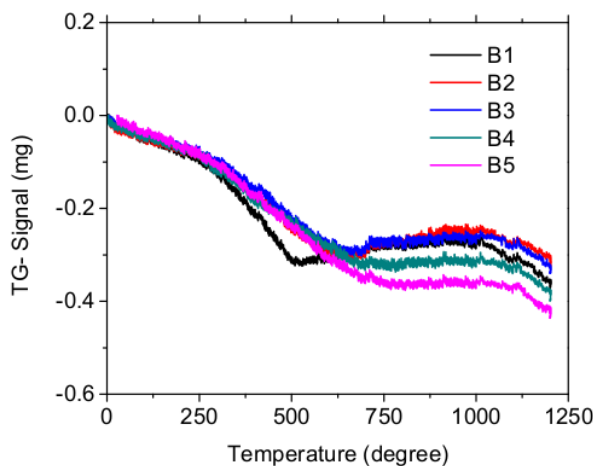
Figure 3. Comparison of specimen phase areas.

TGA used is Merk linseiss. Data is analyzed in three steps. In Figure 4, blue color indicate DTA curve (μV), whereas red and green one each shows operation temperature and mass change in milligram. In this characterization, each sample is characterized for 550 minutes.

Figure 4 shows that total mass losses of each specimens (B1, B2, B3, B4 and B5) at temperature 1200°C are 0,35 mg, 0,2 mg, 0,3 mg, 0,4 mg, and 0,4 mg. Those phenomenon are indicated by green color in each graph. The total mass loss correspond to some mass losses and mass gain that accompanied by exothermic peaks of specimens B1, B2, B3, B4 and B5, each at $200\text{-}500^\circ\text{C}$, $100\text{-}400^\circ\text{C}$, $250\text{-}300^\circ\text{C}$, $100\text{-}400^\circ\text{C}$, and $100\text{-}400^\circ\text{C}$. The mass loss that occur in the specimen of B1 appears different compared with others, where at temperatures under 100°C for B1 specimen experienced highest mass loss. It is supposed that the volatile materials have evaporated. Specimen B1 has experienced endothermic reaction at 100°C up to 200°C .



(a)



(b)

Figure 4. TGA and DTA curves for specimens: B1, B2, B3, B4, and B5.

A mass gain accompanied by isothermic peak indicates that a oxidation process has occurred. The decomposition of binders is detected as mass loss. Therefore, two mass losses accompanied by exothermic peak occurred due to there are carbon combustion and binder decomposition. Mass gain can correspond to iron oxidation or other metals. The carbon combustion should correspond to its carbon percentage in the specimen. The binder decomposition system can not be seen clearly, and this needs testing using other equipments. The carbon combustion caused the formed gases release and accompanied by mass loss.

Table 3. Brinell hardness number (BHN) of specimens.

Specimens	BHN [kg.mm^{-2}]
B1(5)	30
B2(5)	30
B3(5)	22
B4(5)	28
B5(5)	36

The highest hardness number is found in the specimen B5(5) as shown in Table 3. This value is contributed significantly by the glass powders. As we know, the glass has high hardness. The increase of the glass powders content in the specimen is not consistently improving their hardness numbers due to their distribution are inhomogenous. Nevertheless, these hardness data have been still in agreement with data of crystallinity percentages in the Table 2 in which the glass powders are amorphous.

Conclusion

The increase of glass powder content in the specimen contributes consistently to the percentage of crystallinity. It is be fathomed that the glass used as ingredient of brake friction material has crystalline structure. The quantity of glass powder does not correlate directly on the thermal properties of brake friction materials. Based on the above discussion, it is known that the mass loss due to heating effect up to 1200°C, where epoxy, bamboo fiber and *styrene butadiene rubber* decompose. The optimum composition of the fabricated brake material is found for the specimen B2, it is proven with the total mass loss as much as 2 mg which caused by the heating up to 1200°C.

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PAGE 2

PAGE 3

PAGE 4

PAGE 5

PAGE 6

PAGE 7

PAGE 8
