Testing on porosity of composite material composed by
ultrafine amorphous silica (UFAS) from rice husk using X-ray
micro-computed tomography

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Abstract. In general, this study was aimed to test the porosity of composite materials. The test was using X-ray micro-computed tomography (micro-CT) system developed at the Department of Physics, Universitas Gadjah Mada, Indonesia. Testing was done for three composite materials comprised by ultra fine amorphous silica (UFAS) from rice husk. Samples were made in three variations of compositions M1, M2, M3 namely a) UFAS1 plus rice husk powder, resin and catalyst, b) UFAS2 plus rice husk powder, resins, and catalysts, and c) UFAS3 plus rice husk, resins, and catalysts. Rice husks powder has an average fineness of 150 meshes. The testing procedure included preparation, process of scanning, data processing, and data analysis. The results showed that the linear attenuation coefficient of materials M1, M2, and M3 were \((9.2 \pm 0.5) \times 10^{-3}\) cm\(^{-1}\), \((9.7 \pm 0.1) \times 10^{-3}\) cm\(^{-1}\), and \((10.0 \pm 0.1) \times 10^{-3}\) cm\(^{-1}\) respectively. The porosity of materials M1, M2, and M3 were 13.74%, 11.31%, and 7.22% respectively.

1. Introduction
Composite material is a mixture of two or more distinct constituents or phases [1]. One kind of particle composite was made from rice husk [2]. As a new material that will be used as a substitute material for the manufacture of wood carving craft, it is necessary to test the feasibility of it so that the quality of products made from these materials is assured. Therefore, it is necessary to test for internal quality composite material with nondestructive testing (NDT), should had no internal structure damage. One of NDT techniques that can be used to inspect the internal parts of a material is a computed tomography (CT) [3]. Tomography is an imaging technique that presented a cross-section of an object without going through surgery [4]. Tomography was first used to diagnose women who have brain tumors [5], then it used in variety test such as examine the structure of polymers, steel, and ceramics [6], capacitors [7], frozen biomaterial [8], pore structure of material [9].

A set of micro-CT system has been developed at the Department of Physics, Universitas Gadjah Mada, Indonesia. Micro-CT is operated using a computer device, from the process of scanning until the process of reconstructed images to produce cross-sectional images of objects. Testing of the internal structure of material would provide the results of the internal structure of materials that can be used as basic analysis of the quality of material. The quality of the composite material is determined by several things such as stiffness, strength of materials, density comprising homogeneity and porosity of materials [1]. Porosity is closely related to the mechanical properties of composite materials such as...
stiffness and strength of the material [10]. Porosity testing aims to determine the ratio between the total volumes of pores to the amount of the overall volume of materials.

The micro-CT system that has been developed in the Department of Physics, Universitas Gadjah Mada, Indonesia, was adopted the first generation CT. First generation CT is called a translation-rotation tomography that has a single X-ray source and a single detector [5]. There are interaction between X-ray and object when the X-ray passes through the object. According to the Beer-Lambert law, the intensity of X-rays that pass through the object to be diminishing exponentially can be expressed in Eq. (1)

\[
I = I_o \exp \left(-\int_{I_o(x_r)} \mu(x_r, y_r) dy_r \right),
\]

with \(I\) is X-ray intensity after its interaction with an object, \(I_o\) is initial intensity of X-ray, and \(\mu(x_r, y_r)\) is linear attenuation coefficient of the object.

Projections from different angle inspection were arranged into a set of sinogram. Projection is a set of ray-sum in translation trajectory from \(-R\) to \(R\) in Radon space. The ray-sum \(p_p(x_r)\) is obtained by line integral of linear attenuation coefficient of ray-path across the object along the trajectory. In other to produce good quality of reconstructed image, so it used the Summation Convolved Back Projection (SCBP) as reconstruction method on the operation. Thus, the distribution of linear attenuation coefficient was expressed by

\[
\mu(x, y) = \int_0^\pi p'(x_r, \phi) d\phi
\]

with \(p'(x_r, \phi)\) is convolved ray-sum. The reconstruction method gives an accurate reconstructed image. The reconstructed images that have been obtained from the reconstruction method represent the distribution of linear attenuation coefficient of the certain cross-sectional slice according to Eq. (2).

The basic compositions of composite material are UFAS, rice husk powder, resin and catalyst. These composite materials are categorized as particle-reinforced composites. There were three kinds of UFAS namely UFAS1, UFAS2, and UFAS3. The UFAS successively obtained from the combustion at a temperature of 400\(^\circ\)C, 600\(^\circ\)C and 800\(^\circ\)C. Porosity associated with the permeability of material. Surely, porosity of material affects the quality of the material, especially the mechanical properties of composite materials. One method for measuring the porosity of a material is through tomography image analysis. The results will display the cross-sectional slices of the material, so that the internal part of the material is clearly visible between the pores and solid parts. Based on the image of the cross section can be calculated pore volume and the overall volume of material. Porosity measurements with CT method can be done by calculating the total black area of the entire slice on each material compared with the total volume of material. This statement can be formulated as

\[
P\% = \frac{\sum_{n=1}^{n} A_{Bl_n}}{\sum_{n=1}^{n} A_{Tn}} \times 100\%,
\]

with \(P\) is material porosity, \(n\) is the number of cross-sectional slice of each material, \(A_{Bl_n}\) is total black area of the \(n\)-sliced, and \(A_{Tn}\) is total area of the \(n\)-sliced. In addition, by calculating comparing total black area with total area of certain slice, we can measure the area porosity of each slice.
One method of testing porosity is measurement by CT method [9][11]. Each slice cross-sectional tomography shows the internal structure of a material that cannot be seen directly from the outside of the material. Pore structure in the material will affect the value of the material porosity. This paper presents our study of micro-CT system that has been developed expected to be used for testing composite materials. The results will be able to provide information on the quality of the internal structure of material that can be proposed to be a measure of quality composite material. Linear attenuation coefficient, porosity, and profile of reconstructed image were observed and compared for each composite material.

2. Methods

Figure 1 shows the procedure of experiment. The micro-CT system that has been developed at the Department of Physics, Universitas Gadjah Mada, Indonesia comprises an X-ray generator of a Molybdenum anode target, rotation system of object stage, detector with fluorescence screen, and computer unit. The fluorescence screen was coupled with CCD camera to capture the radiograph of object. The object stage is rotated with a controller motor. The X-ray generator was operated on 40 kV and current 30 mA. The objects of this study are three kinds of composite materials with diameter 1 cm and height 2 cm. By rotation of the object, radiographs of the composite material on different angles can be obtained along the scanning process.

A set of 2D projections was collected for each angle from 0° until 360° with interval 1°. A set of 3 radiographs was taken for every 1° angle projections. The 360 radiographs were collected from the system. Then, the data row from a certain height of radiograph was considered as a projection of cross-sectional image of the object at that height. This data row was called a sinogram that has 360 projections. Then, a set of sinogram can be reconstructed to a set of axial cross-sectional slice of the object. The cross-sectional slice of the object is called tomography image.

Figure 1. Scheme of experiment procedure consists of a composite material, set of micro-CT system, radiograph, sinogram, and cross-sectional slice or original CT image.

The internal structure of object can be observed from the cross-sectional slice for each composite material. The cross-sectional slice represented linear attenuation coefficient distribution along the area of image. Thus, we can measure the linear attenuation coefficient of each material. Figure 2 shows the original cross-sectional slice that represented the pore structure of material. We assumed that the darkness area is pore of material. In order to make a simple analysis, the original CT image was changed into binary particle image by selecting the threshold value of it. Black area indicates the pores. Then, the analysis of porosity was done by calculating the total black area.
3. Results and Discussion

The result of reconstruction process was produce 540 cross-sectional slices for each material. The cross-sectional slice or reconstructed image obtained has good quality. The images clearly showed the contrast of pores on the each slice area as is shown in Figure 3. The X-ray energy of 40 kV and 30 mA was able to provide good quality reconstructed image and to distinguish between pores and total area of each slice.

![Figure 2. The binary particle image (pore is black) from original CT image in order to porosity analysis](image)

![Figure 3. Result of reconstructed image of composite material from inspection with X-ray on 40 kV and 30 mA.](image)

The result of each material linear attenuation coefficient ($\mu$) and each material porosity analysis is shown in Table 1 and Table 2.
Table 1. Linear attenuation coefficient of each material

| Materials | Linear attenuation coefficient ($\times 10^3$ cm$^{-1}$) |
|-----------|------------------------------------------------------|
| M1        | 9.2 ± 0.5                                            |
| M2        | 9.7 ± 0.1                                            |
| M3        | 10.0 ± 0.1                                           |

Table 2. Porosity of each material

| Materials | Porosity (%) |
|-----------|--------------|
| M1        | 13.74        |
| M2        | 11.31        |
| M3        | 7.22         |

The result indicated that $\mu$ value of composite materials M1, M2, and M3 were respectively greater, but porosity value of composite materials M1, M2, and M3 were respectively smaller. The difference of material linear attenuation coefficient and porosity value on each composite material is influenced by the different temperature combustion and different composition of it.

The total pore structure in the material affects the density of material indirectly. If the material porosity is being greater, then the material density is being smaller. Material density has inverse effect to the linear attenuation coefficient. This theory is suitable to the experiment results that have been obtained. In addition, based on the result in Table 1 and Table 2, there is an inverse relationship between the values of material porosity with linear attenuation coefficient of material. The analysis result showed that the greater of material porosity, so the material linear attenuation coefficient gets smaller.

In general, the material is required to have porosity value between 10% until 20% for commercial needs. The result of porosity testing by X-ray micro-CT is consistent with the standard method. The advantages of using it for the porosity testing are 1) the testing is one kinds of non-destructive testing because the testing object is not damaging and changing its integrity, 2) the internal structure of object was displayed more clearly by the cross-sectional slices that have been obtained, 3) we can distinguish the pores and the total area of each slice clearly, and 4) we can see the internal condition of the object.

4. Conclusion
The micro-CT system that was developed at Department of Physics, Universitas Gadjah Mada, Indonesia can be used for porosity testing of composite material. The X-ray generator was operated on 40 kV and 30 mA. It was able to provide good cross-sectional slice of each composite material and to distinguish pores of each area slice. The results showed that the differences of linear attenuation coefficient and material porosity were affected by the different composition of each composite material. The higher temperature combustion of material gives effect the greater value of material porosity and the smaller value of linear attenuation coefficient. Besides that, we can estimate that M1 and M2 have a requisite porosity value to be a good material for commercial needs. Therefore, M1 and M2 can be used as a substitute material for the manufacture of wood carving craft. We suggested testing other properties of the composite material in other to get a clearly quality of it.

Acknowledgement
The authors would like to express gratitude to Mr. I Wayan Karyasa for his sharing during the experiment. Thank you also to Gede Arya Wiguna, and I. B. G. Putra Pratama for their assistance during the experiment.
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