The effect of milling time on the structure properties and morphology of aluminium powder from construction industry waste milled using high energy milling

Ratu Inayah Ilahi, Fitri Suryani Arsyad*, Dwika Sari, and Miftahul Jannah
Departemen of Physic FMIPA, Sriwijaya University, Ogan Ilir, South Sumatera 30662, Indonesia

*Corresponding author: fsarsyad90@gmail.com

Abstrak. The effect of milling time on the structure and morphology of aluminum frame powder has been investigated. In this study, aluminum was made in aluminum powder, as a raw material for making bipolar plates in the Proton Exchange Membrane Fuel Cell (PEMFC) technology. Aluminum powder is synthesized using high-energy milling (HEM) with variations in milling time for 10, 20, 30, 40, and 50 minutes. Aluminum powder that has been milled, is characterized using X-Ray Diffraction (XRD) and Scanning Electron Microscope (SEM). From the results of XRD characterization, an aluminum frame that is milled for 10-20 minutes has an aluminum phosphide phase. Whereas for an aluminum frame that is milled for 30, 40 and 50 minutes has an aluminum phase Rudashevskyite. From the results of XRD also obtained, as aluminum frame has a cubic crystal structure with lattice parameters α = β = γ = 90°, and aluminum powder r/n each has a crystal size of 258.31, 865, 218.2, 560, and 255.9 Å for 10 milling times, 20, 30, 40, and 50 minutes, so it can be concluded that the size of the aluminum frame powder crystal fluctuates. While from the results of SEM, aluminum nanoparticle powder obtained from the nanosheet structure, where the gray one is aluminum particles and the black one shows the presence of other elements. The composition of order 94% aluminum nanoparticles is pure aluminum and 6% other elements, this is reinforced from the SEM image which looks more dominant gray.

1. Introduction
The Industrial Revolution became the beginning of increasingly rapid technological development. Technological developments make energy consumption increase sharply, but energy reserves derived from fossil fuels will not be enough to meet energy needs because fossil fuels are natural resources that cannot be renewed. Therefore, a study of various renewable and renewable energy sources is needed. One alternative energy source currently being developed is fuel cell technology. Fuel cell is an electrochemical energy conversion tool that can convert hydrogen and oxygen into water simultaneously producing electricity and heat [3]. This fuel cell technology is more efficient, because it does not cause pollution as well as petroleum energy. Therefore, fuel cell technology is known as an environmentally friendly technology because it can minimize emissions produced so that it can reduce the effects of global warming.

One type of fuel cell that is being developed in Indonesia is Polymer Electrolyte Membrane Fuel Cell (PEMFC). PEMFC is the simplest type of fuel cell and is a promising candidate as a zero-emission power source because it works at low operating temperatures of around 60-80 °C, has high...
efficiency, high power density, fast startup, and good resilience system [5]. One of the main components of the constituent PEMFC is the bipolar plate which functions as a current collector, electrical connections between individual cell stacks, gas separation between adjacent cells, and heat conditioning [9]. Because of its function as a structural support for fuel cells, bipolar plates must also have a sturdy and lightweight physical structure [7]. With this function, a bipolar plate is needed which has the optimum conductivity value. Aluminum is one of the materials that can increase the conductivity value of composite graphite bipolar plates. Therefore in this study Al-powder will be made by optimizing its physical properties and structure to have good conductive values for bipolar plate applications in PEMFC. Making aluminum powder will be carried out using the high energy milling (HEM) method by utilizing Al waste which is widely spread in the environment such as drinking cans (coca cola) or the rest of the window display, home frames or roofs. Although the purity level of aluminum waste is lower than commercial pure aluminum, but with various optimizations to be carried out such as optimization of milling time in HEM, it is expected to obtain nano-sized, homogeneous and strong aluminum powder with good conductivity values for bipolar plate applications.

Based on the description, the study will focus on the following problems: The threat of an energy crisis because of the availability of conventional energy sources which are decreasing over time. The absence of industrial-produced aluminum waste treatment in the environment as a component of PEMFC component bipolar plate making for alternative energy sources, the expensive production price of PEMFC manufacture.

Based on these problems, the specific objectives in this study were: Making waste aluminum nanopowder using the HEM method with variations in the time of milling and making nanopowder Al based bipolar plates as PEMFC components

2. Research Methodology
This research was conducted as a preliminary study for the development of the manufacture of composite bipolar plates made from aluminum frames as a result of construction industry waste. In this study, aluminum frame was made in the form of powder by grinding using a file. Waste aluminum powder that has been crushed, then ground using high energy milling (HEM). The milling process is done by optimizing the milling time with variations in milling time for 10, 20, 30, 40 and 50 minutes. Waste aluminum powder that has been milled, then characterized using X-Ray Diffraction (XRD) to determine its structural properties and Scanning Electron Microscope (SEM) to determine surface morphology[1][14][15]. Figure 1. is a flow diagram for making bipolar plates from aluminium frame waste powder

![Figure 1. Flowchart for making bipolar plates from aluminium frame waste powder](image-url)
3. Result and Discussion
Aluminium have been the grinding process can reduce particle size, size reduction causes a significant change in the structure and morphology of aluminium frame powder, as a result of severe plastic deformation of particles in the grinding process which can produce better characteristics. The grinding process results in changes in the structure and morphology of the aluminium powder of the frame as a result of plastic deformation. The results of the measurement of the frame aluminium diffraction pattern are shown in Figure 1. XRD diffraction patterns of nanocomposite aluminium powders frame milled for different times[5][11].

![Figure 2. XRD diffraction patterns of powders nanocomposite aluminium frame waste milled for different times](image)

Figure 2 shows the suitability of the aluminium frame ICDD data at the high intensity peaks appearing at an angle of 2θ (hkl field) as follows:38.595° (111), 44.836° (200), 47.385° (202), 65.163° (311), from these peaks, aluminium frames have a face center cube crystal structure (FCC) with \(a=b=c=4.0475\) Å, \(\alpha = \beta = \gamma = 90°\), and space group Fm-3m [4][17].

From Figure 2 Get crystal structure, phase name, sample composition, crystal size can be known from the characterization data that has been processed, to get the crystal size calculated using the scherrer method:

\[
D = \frac{K\lambda}{B \cos \theta}
\]

with \(k\) is constant the scherrer 0.9 and \(\lambda\) Cu = 1.540562 Å, B is the FWHM value, with an angle 2θ : 28.51, 38.595, 44.836, 47.385, 65.163, 78.38, obtained by the crystals size of aluminium frame 258.31, 865, 218.2, 560, and 255.9 Å each of which was milled for 10, 20, 30, 40 and 50 minutes using HEM[9][12][6]. From XRD data, it is also seen that aluminium which is dimmed 10 and 20 minutes has a phase aluminium phosphide. Whereas aluminium is milled with variations of 30, 40, and 50 times having phase aluminium rudashevskyite. From the crystal size data obtained by the scherrer method, it is obtained Figure 2. Graph of the effect of milling time on the size of the aluminium frame crystal.
From Figure 3 shows that the aluminum frame nanocomposite powder has a fluctuating crystal size, this can be caused by washing when the jar is not clean, so the other elements left in the jar are mixed, and agglomerated with aluminum frame, can also be caused when digging the tool suddenly stopped because of a technical error, which caused the engine to stop for a long time, causing the particles that had been reduced to become agglomerated again[13].

From the XRD data it is known that the composition of the sample is 94.4% Aluminum and 5.6% other elements, meaning that the aluminum content is very high in aluminum frame, by using aluminum waste we can save costs for making bipolar plate as a PEMFC compound, this is reinforced by SEM characterization namely the characterization of skeletal aluminum powder morphology shown in Figure 3.
Collision between aluminum particles with milling balls and aluminum frame particles causes reduced aluminum particle size. Figure 4 shows non-uniform particle size distribution, which is grey is aluminum powder particles while black is another element. From the graph, it can be seen that the majority of the samples are grey, meaning 94% of the sample composition is pure aluminum frame. Nanoparticle aluminum frame have a nanosheet structure.

4. Conclusion
The effect of milling time on the structure and morphology of aluminum frame powder has been investigated. In this study, aluminum was made in aluminum powder, as a raw material for making bipolar plates in the Proton Exchange Membrane Fuel Cell (PEMFC) technology. Aluminum powder is synthesized using high-energy milling (HEM) with variations in milling time for 10, 20, 30, 40, and 50 minutes. Aluminum powder that has been milled, is characterized using X-Ray Diffraction (XRD) and Scanning Electron Microscope (SEM). Aluminum frame powder has a Crystal Center Cubic (FCC) structure located at high intensity peaks appearing at an angle of 2θ (hkl plane) as follows: 38.595° (111), 44.836° (200), 47.385° (202), 65.163° (311) with a=b=c=4.0475 Å, α = β = γ = 90°, and space group Fm-3m. The size of aluminum frame powder crystals is obtained using the Scherrer method, which is milled using High Energy Milling with varying grinding times for 10, 20, 30, 40 and 50 Minutes respectively 258.31, 865, 218.2, 560, and 255.9 Å, so it can be concluded that the size of the aluminum frame powder crystal fluctuates. While from the results of SEM, aluminum nanoparticle powder obtained from the nanosheet structure, where the grey one is aluminum particles and the black one shows the presence of other elements.

References
[1] Baig M, Ammar H and Seikh AH 2015 Thermo-Mechanical Respons of Nanocrystalline Al-Fe Alloy Processed Using Mechanical Alloying and High Frequency Heat Induction Sintering Material Science & Engineering Vol A 655 (2016) : 132-141
[2] Mahmoud M R I and Tash MM 2016 Characterization of Aluminum-Based-Surface Matrix Composites with Iron and Iron Oxide Fabricated by Friction Stir Processing. Materials Vol 9 (2016) 505 : doi:10.3390/ma9070505
[3] Heydarzadeh Sohi M, Hojatzadeh S M H, Moosavifar ShS, Heshmati-Manesh S 2014 Liquid phase surface melting of AA8011 aluminum alloy by additionof Al/Al2O3 nano-composite powders synthesized by high-energy milling Appl. Surf. Sci. 313, 76–84
[4] Giannini C, Ladisa M, Altamura D, Siliqui D, Sibillano T, and Caro LD 2016 X-ray Diffraction: A Powerful Technique for the Multiple-Length-Scale Structural Analysis of Nanomaterials: A Review. Crystals 6, 87; doi:10.3390/crystals6080087
[5] Ahamed H, Senthilkumar V 2010 Role of nano-size reinforcement and milling on the synthesis of nano-crystalline aluminium alloy composites by mechanical alloying J. Alloys Compd 505, 772–782
[6] Razavi Hesabi, Z Simchi A Seyed Reihani SM 2006 Structural evolution during mechanical milling of nanometric and micrometric Al2O3 reinforced Al matrix composites Mater. Sci. Eng. A 428, 159–168
[7] Zearjad SM, Sajjadi SA 2007 Dependency of physical and mechanical properties of mechanical alloyed Al–Al2O3 composite on milling time Mater. Des. 28, 2113–2120.
[8] Khaerudini DS, Prakoso GB, Insyandya DR, Widodo H, and Indiyaningish N 2017 Effect of graphite addition into mill scale waste as potential bipolar plates material of proton exchange membrane fuel cells Journal of Physics. Conf. Series 985 (2018) 012050
[9] Suryanarayana C 2001 Mechanical Alloying and Milling Handbook Department of Metallurgical and Materials Engineering, Colorado School of Mines, Golden, CO 80401-1887, USA. Progress in Materials Science 46 (2001) 1-184
[10] Mawdsley JR, Carter JD, Wang X, Niyogi S, Fan CQ, Koc, R, and Osterhout G 2013 Composite-coated aluminium bipolar plates for PEM fuel cells. Journal of Power Sources Vol 231 (2013) : 106-112
[11] Hossein-Zadeh M, Razavi M, Mirzaee O, Ghaderi R 2013 Characterization of properties of Al–Al2O3 nano-composite synthesized via milling and subsequent casting. J. King Saud Univ Eng. Sci. 25, 75–80

[12] Prabhu B, Suryanarayana C, An L, Vaidyanathan R 2006 Synthesis and characterization of high volume fraction Al–Al2O3 nanocomposite powders by high-energy milling Mater. Sci. Eng. A 425, 192–200

[13] Antunes, RA, Oliveira M C L, Ett G, and Ett V 2010 Corrosion of Metal Bipolar Plates for PEM Fuel Cells: A Review International Journal of Hydrogen Energy. Vol 35 (2010) : 363with2-3647

[14] Razavi Tousi SS, Yazdani Rad R, Salahi E, Mobasherpoor I, Razavi M 2009 Production of Al–20 wt % Al2O3 composite powder using high energy milling Powder Technol 192, 346–351

[15] Ostovan, F, Matori KA, Toozandehjani M, Oskoueian A, Yusoff HM, Yunus R, Ariff AHM 2016 Nanomechanical behavior of multi-walled carbon nanotubes particulate reinforced aluminum nanocomposites prepared by ball milling. Materials 9, 140

[16] Wang Y and Northwood D O 2007 Effects of O2 and H2 on the corrosion of SS316L metallic bipolar plate materials in simulated anode and cathode environments of PEM fuel cells Electrochimica Acta Vol. 52 (24) : 6793–6798

[17] Karimi S, Fraser N, Roberts B, and Foulkes FR 2012 A Review of Metallic Bipolar Plates for Exchange Membrane Fuel Cells: Materials and Fabrication Methods. Advances in Materials Science and Engineering Vol 1 (2012) p 22