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by Budiarto Budiarto

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Study of the effect of supercapacitors types on crystal structure and microstructure of supercapacitor electrode materials

Edward B.O. Sihite¹, Stepanus¹, and Budiarto²

¹Departement of Electrical Engineering, Faculty of Engineering, Universitas Kristen Indonesia

²Departement of Mechanical Engineering, Faculty of Engineering, Universitas Kristen Indonesia, Jl. MayjenSutoyo no.2, Cawang, Jakarta

¹edwardsihite@uki.ac.id, stepanus@uki.ac.id, ²budidamz@uki.ac.id

Abstract. Research on the effect of supercapacitor types on crystal structure and microstructure on the electrode material of supercapacitors has been carried out. Supercapacitor is a new technology developed from conventional capacitors for storing the energy needed. Supercapacitors have energy densities far greater than conventional capacitors and have a much larger power density than batteries. This research was carried out to determine the crystal structure, crystal lattice diameter size, microstructure and elemental composition of the different types of electrode materials of supercapacitors namely 4.700 μF , 6.800 μF , and 15.000 μF . Tests carried out included the determination of the crystal structure and size of the crystal lattice using X-ray diffractometers and observations of surface morphology and elemental composition by Scanning Electron Microscopy and X-ray Dispersive Energy. The XRD test results from the three supercapacitor electrode materials have the form of a Cubic central body or Cubic Body Center structure, with a crystal lattice diameter between 25 - 428 nm. The results of observations with SEM-EDXS show that the microstructure of phase particles is scattered in the boundary area of the main phase (matrix). And the chemical composition is C: 6-7% by weight, O: 34-36% by weight, Al: 53-58% by weight, and P: 0.23-0.60% by weight.

1. Introduction

In today's modern life, electricity is a major necessity that cannot be avoided. Various technologies exist today, most of which require electrical energy storage devices. For example cellular phones and laptops need batteries as energy storage devices. However, the problem is that the battery has a small enough power density besides that it also takes a long time to charge (store) the electrical energy into the device. Therefore, technology is needed that has greater energy density and power density and shorter charging times to meet future technological needs. So far there has been great interest among researchers to develop and refine more efficient energy storage devices. One of these devices is a supercapacitor.

Supercapacitors have been used extensively in the fields of electronics and transportation, such as digital telecommunications systems, computer and pulse laser systems, hybrid electrical vehicles, and so on. Supercapacitors have several advantages compared to conventional batteries and capacitors, including a longer life time, principles and simpler models, shorter charging times, safety and high power density of 10-100 times greater.

Supercapacitor or Electrochemical Double Layer Capacitor (EDLC) has high power density, high energy density and has a long life cycle. This tool has several components



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including electrodes, electrolytes and separators. The electrode consists of semiconductor materials such as carbon. Electrolytes can be either liquid or non-liquid depending on the manufacture of the supercapacitor. While this separator is made from a membrane that serves to pass ions that exchange from positive and negative electrodes.[2,3]

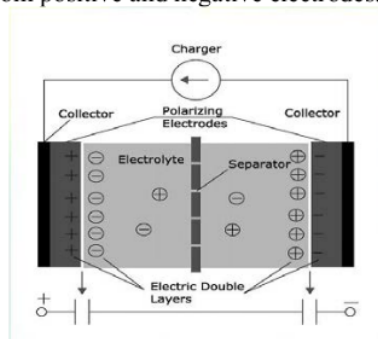


Figure 1. Type of configuration of supercapacitors

The working principle of a supercapacitor as shown in Figure 1 is that when charging, the ions from the two electrodes exchange through the separator. The negative ion from the positive electrode will move towards the negative electrode through the separator membrane and vice versa. When the ion exchange is stored in the supercapacitor, when discharge, the ions from the two electrodes that exchange the charge process return to their original position. The negative ion returns to the positive electrode so vice versa. The load in this process is removed from the supercapacitor and can be used for various purposes.[2]

2. Methodology

Materials and tools

The commercial material used in this study is τ_7 supercapacitor electrode material. The material is cut into a laboratory scale into 21 parts with a size of 10 mm x 10 mm x 0.05 mm. Equipments are used in this research is X-ray diffractometer with Cu K- α monochromatic radiation source (1.54056 Å), Miniflex 600, Rigaku brand, and SEM-EDXS brand ZEISS.

Ways of working.

- X-ray diffractometer Source of Cu K- α monochromatic radiation (1.54056 Å).



Figure 2. X-Ray Diffractometer (XRD) Miniflex 600

The purpose of testing using XRD Miniflex 600 is to determine the size of the crystal lattice diameter, the degree of crystallinity, space group. Based on the position of the peaks

generated from XRD experimental data with a large angle of 2θ and comparing it with the database obtained from COD (Crystallography Open Database) Inorg REV 198327 2017.07.03 can get the phase that matches the tested sample. To find out the size of the crystal lattice diameter and the shape of the crystal can be known by using the D.Scherrer equation [1]:

$$t = k\lambda / (\beta \cos\theta) \quad \dots 1$$

Information :

t = Size of crystallite diameter

k = Scherrer Provisions (0.89)

λ = Wavelength of X-ray diffraction (1.54046Å)

β = Overall width of maximum diffraction peak (FWHM)

θ = Bragg angle read by XRD engine

The XRD test data obtained were used to estimate crystallite size through analysis of the widening values of each crystal sample. Of the highest peaks generated from the tested samples shown from the image and obtained FWHM values.

7 b. Scanning Electron Microscopy Energy Dispersive X-Ray Spectroscopy (SEM-EDXS)

The microstructure, surface morphology and particle size of solid samples were analyzed using SEM-EDXS. EDXS can make elemental mapping by giving different colors to each element on the surface of the material being observed. It can also be used to analyze quantitatively and qualitatively.

The principle is the wave nature of electrons which is diffraction at a very small angle. Electrons are scattered by charged samples (due to their electrical properties). The formed image shows the structure of the sample being tested.



Figure 3. Scanning Electron Microscopy (SEM) ZEIS

3. ResultAnd Discussion

a. Crystal structure analysis

In this study, the samples tested were supercapacitor electrode material to identify the crystal shape, space group, X-ray diffraction intensity, and crystal lattice diameter size. The relationship between the crystal grain size and the effect of the magnitude of the capacitance is 4.700 μ F, 6.800 μ F, and 15.000 μ F to be analyzed. The grain size of the crystal is influenced by the width of the XRD spectrum, which is the FWHM value and the angle 2θ . If the value of FWHM and angle 2θ are small then the size of the grain of a large crystal is also the opposite.

The XRD test results of the three supercapacitor material samples in the form of X-ray diffractogram can be seen in figures 4, 5 and 6 and tables 1, 2, and 3, below.

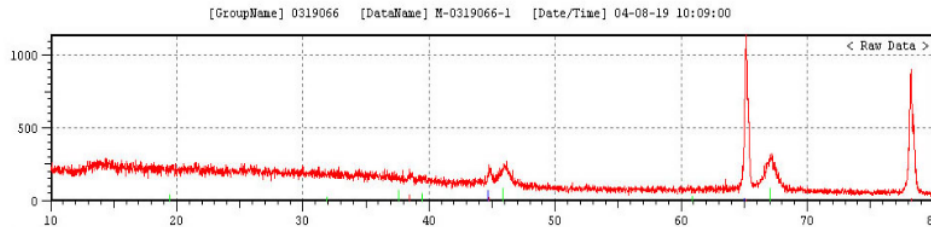


Figure 4. X-ray diffractogram pattern from a sample of 4.700 μF supercapacitors

Table 1. Data on XRD test results for supercapacitors 4.700 μF

Posisi 2 θ ($^{\circ}$)	Intensity (cts)	FWHM ($^{\circ}$)	Space group	Crystal form	Chemical Elements	Size of dia. crystal (nm)
65.18	542.44	0.3811	Fm-3m	Cubic	Fe	428,312
78.24	688.29	0.5778	Im-3m	Cubic	Al	311,500

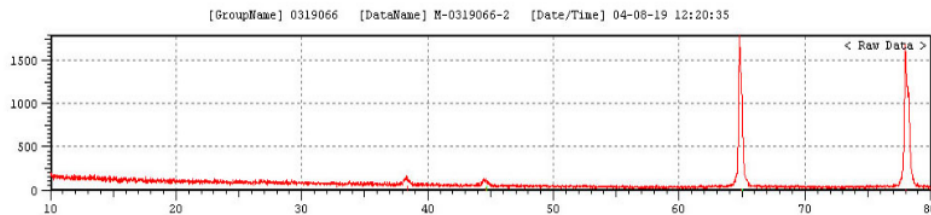


Figure 5. X-ray diffractogram pattern from a sample of 6.800 μF supercapacitors

Table 2. Data on XRD test results for supercapacitors 6.800 μF

Posisi 2 θ ($^{\circ}$)	Intensity (cts)	FWHM ($^{\circ}$)	Space group	Crystal form	Chemical Elements	Size of dia. crystal (nm)
64.81	1080	0.1088	Fm-3m	Cubic	Al	149,302
77.96	956	0.956	Im-3m	Cubic	Fe	185,216

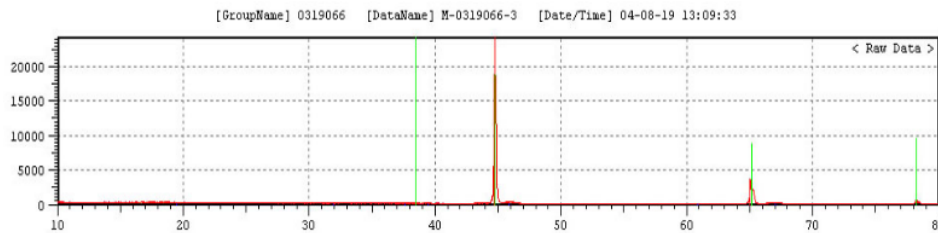


Figure 6. X-ray diffractogram pattern from a sample of 15.000 μF supercapacitors

Table 3. Data on XRD test results for supercapacitors 15.000 μF

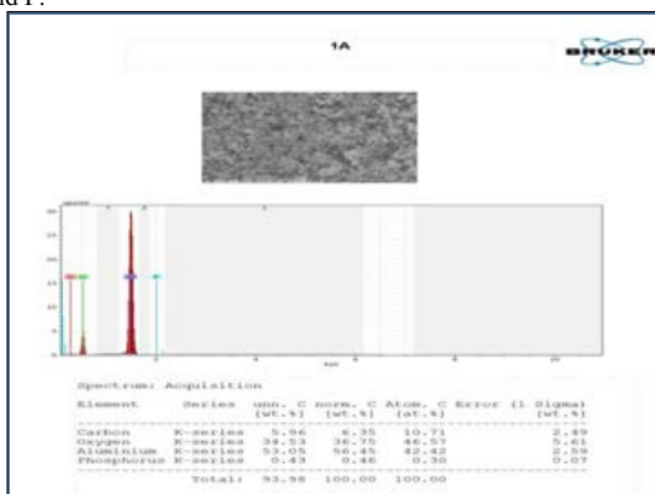
Posisi 2θ ($^{\circ}$)	Intensity (cts)	FWHM ($^{\circ}$)	Space group	Crystal form	Chemical Elements	Size of dia. crystal (nm)
44.78	22060	0.5778	Im-3m	Cubic	Fe	25,649
65.16	14600	0.1468	Fm-3m	Cubic	Al	110,809

In figure 4, the diffractogram of a sample of 4.700 μF supercapacitors is seen at $2\theta = 65,18^{\circ}$ with an intensity of 542 cps, space group Im-3m, cubic crystal shape, chemical element iron (Fe) and crystal diameter size 428,312 nm and aluminum chemical elements (Al) and the size of the crystal diameter is 311,500 nm. Compared to figure 6, a sample of 15.000 μF supercapacitors was seen in $2\theta = 44,78^{\circ}$ with an intensity of 22060 cps, Im-3m space group, cubic crystal form, iron (Fe) chemical element and 25,649 nm crystal diameter size and 14600 cps intensity, aluminum chemical elements (Al) and the size of the crystal diameter of 110.809 nm.

From the X-ray diffraction pattern shows the most dominant peaks from images 4, 5 and 6, are at $2\theta = 44,78^{\circ}$ (figure 6) with an intensity of 22060 cps and a crystal diameter size of 25,649 nm. The higher the intensity of X-ray diffraction shows the stronger the bond and the smaller the grain boundary between the sample particles. In addition, the stronger the bond and the smaller the grain boundaries have implications for the hardness of the material and the ability of the material to conduct electricity and heat. From the above analysis showed that there is a relationship between the value capacitance of the three supercapacitor electrode material samples to the crystal lattice diameter size. Where was explained above.

b. Microstructure analysis and composition

The SEM-EDX spectrum was taken on the surface of the electrode material samples of supercapacitors 4.700 μF , 6.800 μF , and 15.000 μF shown in Figures 7, 8, and 9 and the chemical composition and quantification of the spectrum were presented in percent weight. The analysis results obtained information that the chemical composition of the three samples had C, O, Al and P.

**Figure 7.** Microgram of the electrode material of the supercapacitor 4.700 μF , magnification of 5000x

From figures 7, 8 and 9, the results of observations with SEM-EDXS show that the microstructure of phase particles is scattered in the boundary area of the main phase (matrix). And the chemical composition is C: 6-7% by weight, O: 34-36% by weight, Al: 53-58% by weight, and P: 0.23-0.60% by weight.

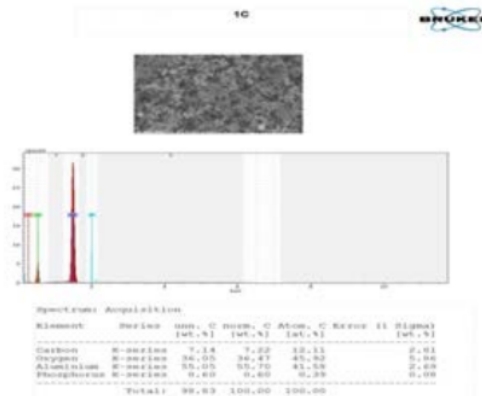


Figure 8. Microgram of the electrode material of the supercapacitor 6.800 μF , magnification of 5000x

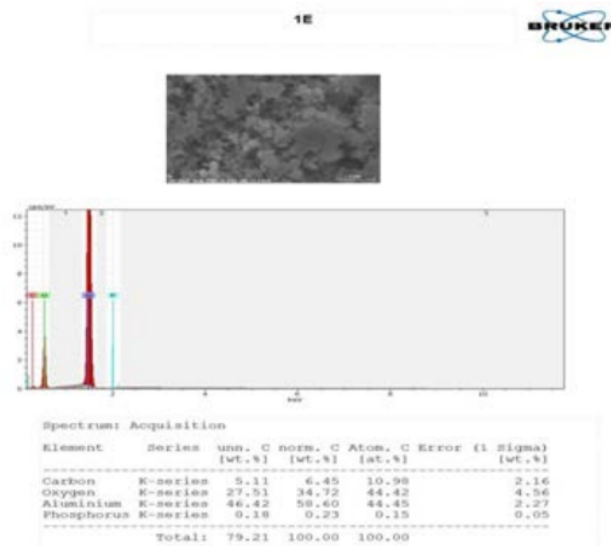


Figure 9. Microgram of the electrode material of the supercapacitor 15.000 μF , magnification of 5000x

Figure 7 shows the surface morphology in the form of pores of carbon, aluminum and phosphorus powder with a magnification of 5000 times. The results of observations with SEM-EDXS showed that the microstructure of phase particles spread over the boundary area of the main phase (matrix). And its chemical composition is C: 6% by weight, O: 34% by

weight, Al: 53% by weight, and P: 0.23% by weight. The results of testing the crystal structure and microstructure and chemical element composition showed the presence of the most dominant element of aluminum (Al) from the three supercapacitor electrode material samples. And there is a relationship between the magnitude of the capacitance value of the Crystal lattice diameter size, the greater the capacitance value, the smaller the crystal lattice diameter size in the three superkasitor electrode material samples. The results of this SEM test were strengthened by Stepanus researchers who said that it included the pseudocapacitor because it had particles that were white and crystalline. As well as the superiority of energy storage supercapacitors are located in two layers coated with activated carbon in the absence of chemical reactions. This is what causes supercapacitors to be able to store more energy.[5]

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4. Conclusion

The results of the characterization and analysis of the crystal structure and microstructure test and chemical element composition in the commercial supercapacitor materials and the development of the PSMM-FT-UKI using SEM and XRD can be concluded that: - The three supercapacitor materials have a structure namely cubic crystal center or Cubic Body Center, with the diameter of the crystal lattice between 25 - 428 nm. - Microstructure of supercapacitor material has phase particles scattered in the boundary area of the main phase (matrix). And the chemical composition is C: 6-7% by weight, O: 34-36% by weight, Al: 53-58% by weight, and P: 0.23-0.60% by weight. There is a relationship between the magnitude of the capacitance value of the crystal lattice diameter size, the greater the value of the capacitance, the smaller the crystal lattice diameter size in the three samples of the superkasitor electrode material.

Acknowledgement

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