

**DEPOSITION OF CARBON THIN FILM BY MEANS OF A LOW-FREQUENCY PLASMA SPUTTERING
USING BATTERY CARBON RODS AS A TARGET**

1. Registration (30 Mei 2018)
2. Abstrac submission accepted (30 Mei 2018)
3. Abstrac edited (03 Juni 2018)

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DEPOSITION OF CARBON THIN FILM BY MEANS OF A LOW FREQUENCY PLASMA SPUTTERING USING A TARGET FROM BATTERY CARBON RODS

Authors:
Aladin E. Purkuncoro1, Dionysius J. D. H. Santjojo 2*, Yudy S. Irawan1, Rudy Soenoko1

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Content:
Battery carbon rods were utilized as a target in a plasma sputtering deposition of a carbon thin film. The rods were taken from unused battery waste containing some impurities. This study was intended to investigate the effect of the impurities on the resulted thin film. Furthermore, this work was intended to study the utilization of an unconventional sputtering technique to deposit the carbon film on glass substrates. A low frequency plasma generator of 40 kHz was used to power the sputtering reactor. The plasma was generated from an Argon gas in a medium vacuum pressure. Two deposition parameters studied in this work were

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Keywords:
Carbon thin film, Low frequency plasma, Battery carbon rod, Sputtering

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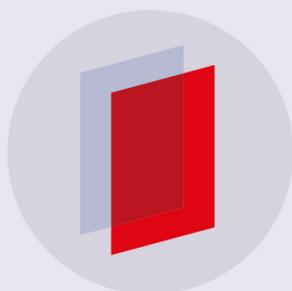
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Abstract. Carbon rods of battery were utilized as a target in a plasma sputtering deposition of a carbon thin film. The rods were taken from unused battery waste containing some impurities. This study was intended to investigate the effect of the impurities on the resulted thin film. Furthermore, this work aimed to study the utilization of an unconventional sputtering technique to deposit the carbon film on glass substrates. A low-frequency plasma generator of 40 kHz was used to power the sputtering reactor. The plasma was generated from an Argon gas in a medium vacuum pressure. Two deposition parameters studied in this work were plasma power varied from 220 watts to 360 watts and substrate temperature varied from 25 °C to 202 °C. The carbon target in this research was also functioned as an electrode in the sputtering system. The deposition process was carried out for 1 hour to produce a reasonably thick carbon thin film. The resulted films were characterized using a Fourier Transform Infrared (FTIR) spectroscopy. The FTIR spectra showed a distinct peak around 1200 cm⁻¹ and 1600-1700cm⁻¹ related to the C-C and C=C vibration respectively. The relatively broad peak consisted of a doublet indicating a complex structure of the carbon film, presumably an amorphous carbon film. It was proven that impure carbon could be used as the target and successfully deposited on the glass substrate.

Keywords: Carbon thin film, battery carbon rod, plasma sputtering, low frequency, impurity

1. Introduction

The amorphous carbon films have attracted interest in both industry and researcher. These films offer a wide range of outstanding physical, mechanical, biomedical, and tribological properties that make them essential for numerous applications [1]. The carbon films have already been synthesized using plasma enhanced chemical vapor deposition, radiofrequency parallel-plate hollow-cathode plasmas and sputtering [2–4]. Among them, sputtering has advantages such as high deposition rate, low substrate temperature, little damage to films, and obtaining high-quality films under lower substrate temperature [5]. Sputtering was initiated due to the bombardment of energetic particles at the target. One of the sources of the energetic particle is plasma. The low-frequency plasma of 40 kHz was used for polyester fabric treatment to reduce the amount of flame retardant agent [6].



Zinc-carbon cell batteries are widely used in many applications, e.g.; toys, remote control, radio, camera. Because the battery waste increases year by year, the disposed batteries on the landfill can change the water's pH and release of metal (Zn, Pb, Hg) into the ambient air [7]. Recycle of the components of battery waste will contribute to the improvement of the environmental quality. Zinc-carbon battery is a disposable battery in which a carbon rod inserted into an electronic conductor with inert properties. The carbon rod is a good electronic conductor with inert properties [8]. Carbon rods can be used as graphene oxide biogas desulfurizer [9–11].

In this research, carbon rod from zinc-carbon battery waste with a relatively low purity level of carbon was used as a material target for deposit carbon thin films by plasma sputtering. The argon plasma was generated using LFG 40 KHz. The characterization of the carbon thin film was carried out by Fourier Transform Infrared.

2. Methods

2.1. The sample preparation

The material target used in this research was prepared by carbon rod from zinc-carbon ABC battery waste (ABC industry, Indonesia). The carbon rods were cut into the diameter of 8 mm and length of 55 mm. Then, the carbon rod was cleaned by ultrasonicated in soap, solution for 0neminutes. The cleaned carbon rod was dried in the oven at 300 °C for two hours.

2.2. Deposition thin film

The carbon films were deposited using plasma sputtering. Plasma was generated by low-frequency generator (LFG) 40 KHz. The plasma system had the diameter chamber of 250 mm. The carbon rods as the material target were placed at the electrode. The distance between the material target and the glass substrate was kept at 500 mm. The argon gas was introduced with the flow rate of 10 ml/minute into the chamber after the vacuum process of 1.5 hours. The deposition process was carried out for 1 hour. The beginning temperature process was around 22-28 °C, while the end temperature process was around 125-202 °C. The power of the process varied of 220 watts, 260 watts, 320 watts, and 340 watts. Figure 1 shows the schematic illustration of plasma sputtering used in this experiment.

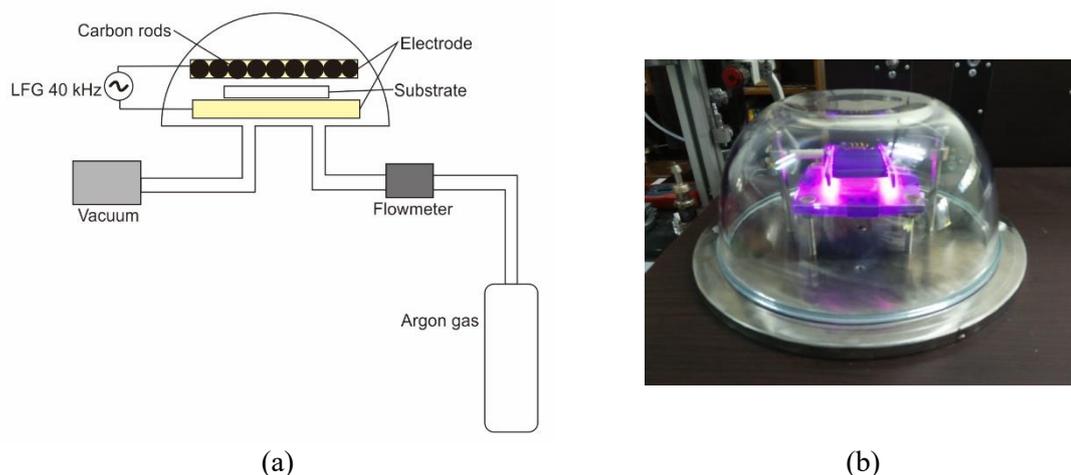


Figure 1. (a) Schematic illustration of plasma sputtering with carbon rods-target (b) Plasma sputtering process

2.3. Characterization

The content of the carbon rod was measured using a scanning electron microscope/energy dispersive using X-ray (SEM-EDX, Fei, Type Inspec-21). The Fourier Transform Infrared (FTIR, 8400S/Shimadzu) was utilized for characterization of the carbon thin film.

3. Results and Discussion

3.1. SEM-EDX results

The carbon rods were observed using SEM-EDX to understand the purity level of carbon. The observation was done at the outer layer of the carbon rods (Figure 2). Table 1 provides the purity level of carbon rods based on SEM-EDX results.

Table 1. The compositions of battery carbon rods based on SEM-EDX results

| Element | Wt% |
|---------|-------|
| C | 72.81 |
| O | 16.54 |
| Al | 1.95 |
| Si | 2.41 |
| Cl | 1.42 |
| K | 0.99 |
| Ca | 1.29 |
| Fe | 2.59 |

Based on the SEM-EDX results, the carbon purity level of carbon rods was relatively small compared to carbon commonly used in sputtering; it was more than 90%. However, it still could be used as the target for plasma sputtering. This phenomenon was proven by FTIR results. The impure carbon will form amorphous carbon thin film. There are 3 different characteristics formed at the substrate namely a diamond with configurations sp^3 , a graphite with configuration sp^2 , and a small amount of sp configuration. The temperature of substrate and power of plasma sputtering will affect the ratio of those configurations. Then, the different configurations will form a different structure of the thin film.

The SEM observation was carried out on the outermost part of carbon rods. In the sputtering process, the outer part of the battery will be eroded first. Therefore, SEM observations were carried out on the outer side of carbon rods. The carbon content increased proportionally to carbon rods depth. The higher level of carbon resulted in more sp^3 formed.

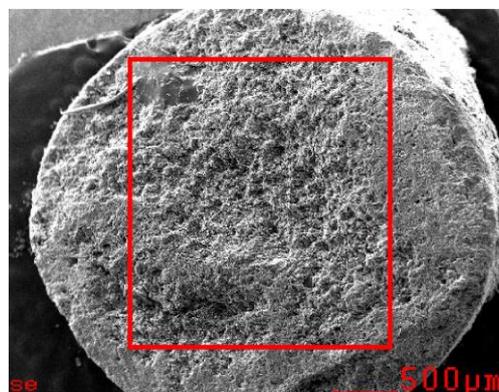


Figure 2. SEM imaging of carbon rod outer layer

3.2. FTIR results

The carbon was successfully deposited onto a glass substrate, At Figure 3, it was shown by the formation of C-C and C=C functional group around 1200 cm^{-1} and 1600-1700 cm^{-1} respectively [12]. The various power used affected the number of carbons deposited on the glass substrate, indicated by the absorption intensity of functional groups. At higher power, the absorption of C=C bonds relatively increased. This possibly showed that there were more carbons deposited onto the glass substrate. Table 2 shows the information about the wavenumber of C-C and C=C formed at different powers.

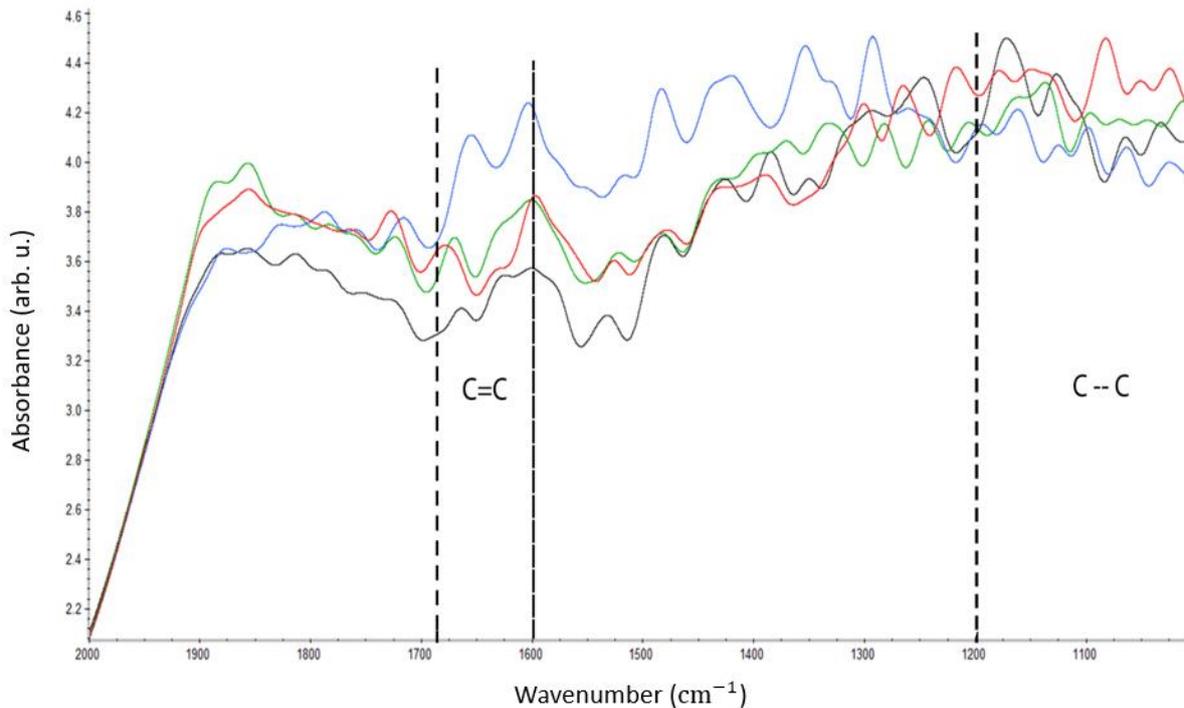


Figure 3. FTIR spectra of carbon sputtering with the various power

Table 2. C-C and C=C bonds formed at a glass substrate obtained by FTIR spectroscopy

| Power | Wavenumber (cm^{-1}) | |
|-------|---------------------------------------------|------------------|
| | C-C | C=C |
| 220 W | 1032.77; 1064.14; 1127.20; 1172.92 | 1601.03; 1664.05 |
| 260 W | 1024.26; 1050.95; 1081.79; 1150.21; 1178.25 | 1679.27 |
| 320 W | 1012.14; 1096.82; 1135.85 | 1600.56; 1670.05 |
| 340 W | 1024.96; 1062.96; 1098.02; 1124.49; 1161.34 | 1603.50; 1655.44 |

The plasma argon bombarded the carbon rods; consequently, the carbon ions would be formed. Then, the ions moved towards the glass substrate. To achieve a stable state, the ion tried to attract the ion around it and formed covalent bonds namely C-C and/or C=C bonds. Thus, the carbon thin films were deposited onto the glass substrate. The relatively broad peak consisted of a doublet (C=C) indicating a complex structure of the carbon film, presumably an amorphous carbon film. The formation of C-C and C=C bands indicating different electron configurations presence are sp^3 , sp^2 [13,14], and a small amount of sp .

4. Conclusion

Battery carbon rods were observed using SEM-EDX to obtain their components namely C (72.81%), O (16.54%), Fe (2.59%), and the other elements (8.06%). This impure carbon was used as target sputtering. Based on FTIR results, C-C bonds and C=C were formed at 1200 cm^{-1} and $1600\text{-}1700\text{ cm}^{-1}$. At a higher power, the intensity was increased. It indicated that there were more carbons deposited onto the substrate. Thus, it can be concluded that impure carbon of carbon rods can be deposited using plasma sputtering on the glass surface and resulted in amorphous carbon films.

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2.2. Deposition thin film

The carbon films were deposited using plasma sputtering. Plasma was generated by low-frequency generator (LFG) 40 KHz. The plasma system had the diameter chamber of 250 mm. The carbon rods as the material target were placed at the electrode. The distance between the material target and the glass substrate was kept at 500 mm. The argon gas was introduced with the flow rate of 10 ml/minute into the chamber after the vacuum process of 1.5 hours. The deposition process was carried out for 1 hour. The beginning temperature process was around 22-28 °C, while the end temperature process was around 125-202 °C. The power of the process varied of 220 watts, 260 watts, 320 watts, and 340 watts. Figure 1 shows the schematic illustration of plasma sputtering used in this experiment.

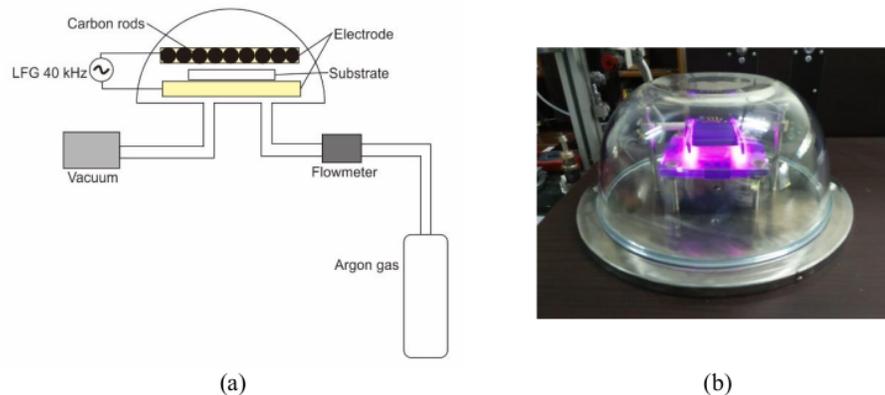


Figure 1. (a) Schematic illustration of plasma sputtering with carbon rods-target (b) Plasma sputtering process

2.3. Characterization

The content of the carbon rod was measured using a scanning electron microscope/energy dispersive using X-ray (SEM-EDX, Fei, Type Inspec-21). The Fourier Transform Infrared (FTIR, 8400S/Shimadzu) was utilized for characterization of the carbon thin film.

3. Results and Discussion

3.1. SEM-EDX results

The carbon rods were observed using SEM-EDX to understand the purity level of carbon. The observation was done at the outer layer of the carbon rods (Figure 2). Table 1 provides the purity level of carbon rods based on SEM-EDX results.

Table 1. The compositions of battery carbon rods based on SEM-EDX results

| Element | Wt% |
|---------|-------|
| C | 72.81 |
| O | 16.54 |
| Al | 1.95 |
| Si | 2.41 |
| Cl | 1.42 |
| K | 0.99 |
| Ca | 1.29 |
| Fe | 2.59 |

Based on the SEM-EDX results, the carbon purity level of carbon rods was relatively small compared to carbon commonly used in sputtering; it was more than 90%. However, it still could be used as the target for plasma sputtering. This phenomenon was proven by FTIR results. The impure carbon will form amorphous carbon thin film. There are 3 different characteristics formed at the substrate namely a diamond with configurations sp^3 , a graphite with configuration sp^2 , and a small amount of sp configuration. The temperature of substrate and power of plasma sputtering will affect the ratio of those configurations. Then, the different configurations will form a different structure of the thin film.

The SEM observation was carried out on the outermost part of carbon rods. In the sputtering process, the outer part of the battery will be eroded first. Therefore, SEM observations were carried out on the outer side of carbon rods. The carbon content increased proportionally to carbon rods depth. The higher level of carbon resulted in more sp^3 formed.

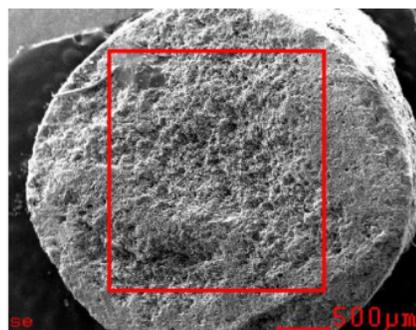


Figure 2. SEM imaging of carbon rod outer layer

3.2. FTIR results

The carbon was successfully deposited onto a glass substrate, At Figure 3, it was shown by the formation of C-C and C=C functional group around 1200 cm^{-1} and 1600-1700 cm^{-1} respectively [12]. The various power used affected the number of carbons deposited on the glass substrate, indicated by the absorption intensity of functional groups. At higher power, the absorption of C=C bonds relatively increased. This possibly showed that there were more carbons deposited onto the glass substrate. Table 2 shows the information about the wavenumber of C-C and C=C formed at different powers.

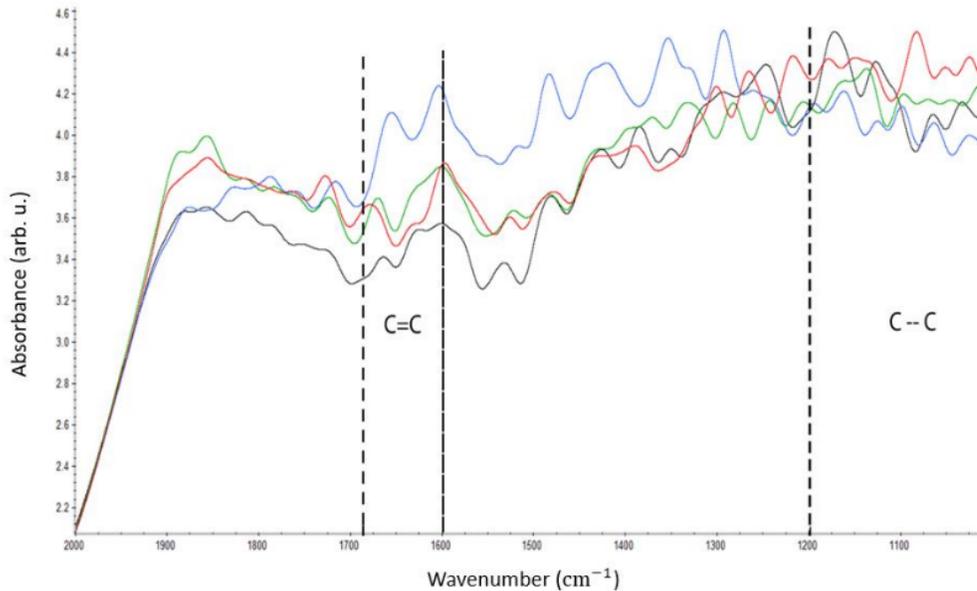


Figure 3. FTIR spectra of carbon sputtering with the various power

Table 2. C-C and C=C bonds formed at a glass substrate obtained by FTIR spectroscopy

| Power | Wavenumber (cm^{-1}) | |
|-------|---------------------------------------------|------------------|
| | C-C | C=C |
| 220 W | 1032.77; 1064.14; 1127.20; 1172.92 | 1601.03; 1664.05 |
| 260 W | 1024.26; 1050.95; 1081.79; 1150.21; 1178.25 | 1679.27 |
| 320 W | 1012.14; 1096.82; 1135.85 | 1600.56; 1670.05 |
| 340 W | 1024.96; 1062.96; 1098.02; 1124.49; 1161.34 | 1603.50; 1655.44 |

The plasma argon bombarded the carbon rods; consequently, the carbon ions would be formed. Then, the ions moved towards the glass substrate. To achieve a stable state, the ion tried to attract the ion around it and formed covalent bonds namely C-C and/or C=C bonds. Thus, the carbon thin films were deposited onto the glass substrate. The relatively broad peak consisted of a doublet (C=C) indicating a complex structure of the carbon film, presumably an amorphous carbon film. The formation of C-C and C=C bands indicating different electron configurations presence are sp^3 , sp^2 [13,14], and a small amount of sp .

4. Conclusion

Battery carbon rods were observed using SEM-EDX to obtain their components namely C (72.81%), O (16.54%), Fe (2.59%), and the other elements (8.06%). This impure carbon was used as target sputtering. Based on FTIR results, C-C bonds and C=C were formed at 1200 cm^{-1} and $1600\text{-}1700\text{ cm}^{-1}$. At a higher power, the intensity was increased. It indicated that there were more carbons deposited onto the substrate. Thus, it can be concluded that impure carbon of carbon rods can be deposited using plasma sputtering on the glass surface and resulted in amorphous carbon films.

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