

Fabrication of Reduced Graphene Oxide by Electron Irradiation

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1. Introduction

Graphene which is two-dimensional crystalline structure of carbon is promising materials due to its excellent electrical and thermal properties. Graphene have been investigated for electrode materials in energy fields such as supercapacitors, batteries [1]. However, the fabrication method for graphene are time-consuming and expensive thus there are many problems in mass-producing high-quality graphene as an electrode.

Besides, graphene oxide (GO) is one of the graphene base materials and it can be produced with low cost and doesn't require much time. However, the high-resistance of GO reduced the electron transfer. In order to apply the GO to electrode, oxygen reduction process is required. Conventional oxygen reduction process is using harmful chemicals such as hydrazine.

Electron irradiation are eco-friendly and economical process. During electron irradiation on carbon materials, crosslinking is performed and induced the deoxygenation [2]. Therefore, in this study, reduced GO (rGO) are prepared by electron irradiation. GO particles are prepared by anodization in KCl aqueous solution. rGO are produced by electron irradiation on GO particles. The morphologies and chemical properties of rGO are investigated.

2. Methods and Results

2.1 GO particles preparation

High purity graphite rods (1mm dia., 99.9% Good fellow) were used as precursor. Graphite rod was placed in an electrolysis cell filled with 1M KCl electrolyte. Platinum foil (1cm*2cm*0.05cm) was a counter electrode. A constant DC voltage of 10 V was applied to the two electrodes for 5 min. After anodization process, produced GO particles were washed several times to remove the purities and KCl.

2.2 Electron irradiation on GO (E-GO)

In this paper, 50keV electron beam device produced in our lab was used. The GO particles were fixedly placed in the electron beam irradiator, and then the inside of the irradiator was evacuated. At this time, the degree of vacuum was maintained at 10^{-6} Torr by using a rotary pump and a turbo pump. Then, electron beam was irradiated to the GO particles and total fluence was

$1.79 * 10^{16}$ electrons/cm². After irradiation, rGO was characterized by a scanning electron microscopy (SEM, hitachi-4800) and X-ray photoelectron spectroscopy (XPS).

2.3 Characterization of E-GO.

During anodization process, GO particles are detached from the graphite rod and GO particles were suspended in the electrolyte. GO particles are dried and irradiated with electrons. The SEM image of GO particles and E-GO particles are shown in Fig.1. After the electron irradiation, shape and size of E-GO particles are not highly changed.

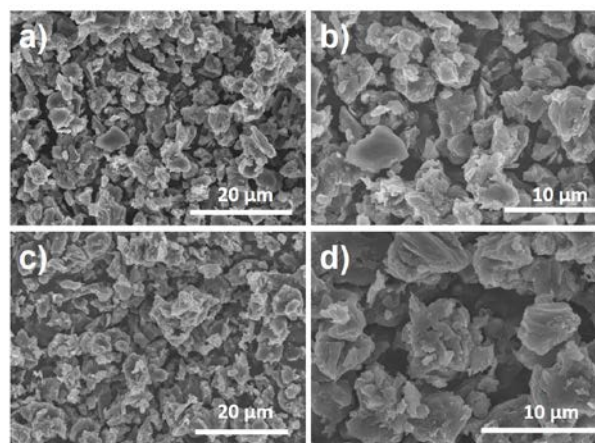


Fig. 1. (a),(b) SEM images of GO particles anodized at 1M KCl aqueous solution. (c),(d) TEM images of electron irradiated GO particles.

Table I: Atomic ratio of graphite powder, GO particles and E-GO particles

(at.%)	Graphite powder	GO particles	E-GO particles
C	96.48	76.5	80.56
O	3.52	23.5	19.44

However, atomic ratio of E-GO was changed slightly. Table 1 is the atomic ratio from the XPS survey spectrum of graphite powder, GO particles and E-GO particles. After the anodization, oxygen content in GO increased rapidly to 23.5 at%. After the electron irradiation, oxygen content of E-GO slightly reduced and it is induced by crosslinking of GO by electron

beam. Crosslinking process promotes the recombination of C=C bonds and deoxygenation. Thus, rGO can be produced by electron irradiation.

3. Conclusions

rGO particles are fabricated by anodization and electron irradiation. GO particles are prepared by anodization of graphite rod in KCl aqueous solution. Dried GO particles are irradiated with electron in vacuum. Morphologies and size of rGO are not changed by electron beam, however oxygen contents of rGO are slightly reduced. It is expected that fabricated GO can be used at electrode materials for energy storage fields.

REFERENCES

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