PREPARATION OF GRAPHENE LIKE SHEETS OBTAIN THROUGH ELECTROCHEMICAL PROCESSING

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Introduction

Remarkable properties of graphene (Graphene composed from a hexagonal carbon layer) have been reported up to present time include high values of Yong's modulus (-1100 GPa) [1], fracture strength (- 125 GPa) [1], charge carriers's mobility (2 x 10^5 cm^2 V^{-1} S^{-1}) [2] and thermal conductivity (-5000 W m^{-1} K^{-1}) [3]. Graphene and chemically modified graphene are promising candidates as components in applications such as energy storage materials [4] and polymer composites [5,6]. Graphene has been prepared from various methods such as micromechanical exfoliation, chemical vapor deposition (CVD) or reduction of graphite oxide. First CVD with epitaxial growth deposited on Ni metal surface through ethylene decomposition [7]. Micromechanical exfoliation of graphite is also known as the "Scoth tape" or peel-off method followed on micromechanical exfoliation from Kish graphite [8]. It is the reduction of graphite oxide generated through exfoliation process of graphite particles [9]. In comparison of their methods, preparation of substrate free graphene by using CVD is difficult. On the other hand, micromechanical exfoliation could be yielded small size graphene. Preparation of graphene and graphene like sheets having large area through intercalation compounds and graphite oxide by using electrochemical processing such as exfoliation of carbon fibers [10 - 11] is discussed in this presentation.

Experimental

Graphene and graphene like sheets were prepared from exfoliated graphite sheet with different thickness in this study. Exfoliated graphite sheet was prepared sheet like morphology by compressing of the exfoliated graphite particles. Preparation of graphene and graphene like sheets were successfully carried out via intercalation compounds. Intercalation compounds of it were formed by anodic polarization in acid electrolyte such as nitric acid, sulfuric acid, formic acid and other acid solutions. Graphite sheet with 5cm x 3 cm x 0.6 cm was used and it was put between platinum plate and platinum mesh to ensure the electrical contact. Its sheet was anodically polarized in their acid solution at constant current by using a galvanostat. A silver-silver chloride electrode was employed as reference electrode. After electrolysis of its graphite sheet, its sheet was rinsed with distilled water and then dried it under ambient atmosphere for 24 h. Intercalation compounds and preparation of graphite oxide were confirmed by XRD for electrolyzed exfoliated graphite sheet. Its intercalation behavior suggested that intercalation into graphite sheet proceeds from the free end of sheet to towards the center of exfoliated graphite sheet with increasing electric charges. By rapid heating to 800 to 1000 ^oC for 5 to 15 sec, marked morphological changes were confirmed like the exfoliated graphite particles. Graphene or graphene like sheet was separated by irradiation of ultra-sonication with agitation to the exfoliated graphite particles obtained in the distilled water or alcohol. The evaluation of the sample obtained was carried out by X-ray diffraction (XRD), scanning electron microscope (SEM) observation, transmission electron microscope (TEM) observation.

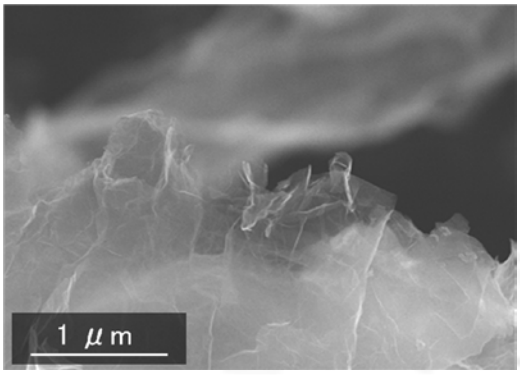

Fig. 1 Exfoliated graphite particles

Results and Discussions

An additional peak around $2\theta = 11^o$ is observed after electrolysis which corresponds to an interlayer spacing of about 0.80 nm. This new peak was reasonably supposed to be due to the preparation of graphite oxide (GO). Morphological changes of the exfoliated graphite sheet are shown by SEM images in Fig. 1, in which marked exfoliation of it after rapid heating to 1000 ^oC. From its observation, it is possible to thinly exfoliate to the degree in which the opposite side is transparent.

Graphene like thin layers obtained from exfoliated graphite sheet isolated by ultra-sonication with agitation. Results by TEM observation of its thin layers are shown in Fig. 2 (a) and (b). It was indicated that dimension of its thin layers is around 5 mm in length, and it was exfoliated until few to few decade hexagonal carbon layers, because the electron beam sufficiently penetrates. *002* lattice image of thin layers is shown at right upper part in the Fig. 2(a). All thin layers were found to be composed well-oriented hexagonal carbon layers, which by the spot state in the electron beam diffraction pattern [at right upper part Fig. 2 (b)]. From these results, it became clear that the graphene thin layers prepared by this procedure had layered structure with the regularity. It became possible that nano sheet with the thickness of the nanometer size was easily prepared in comparison with CVD methods or micromechanical exfoliation method.

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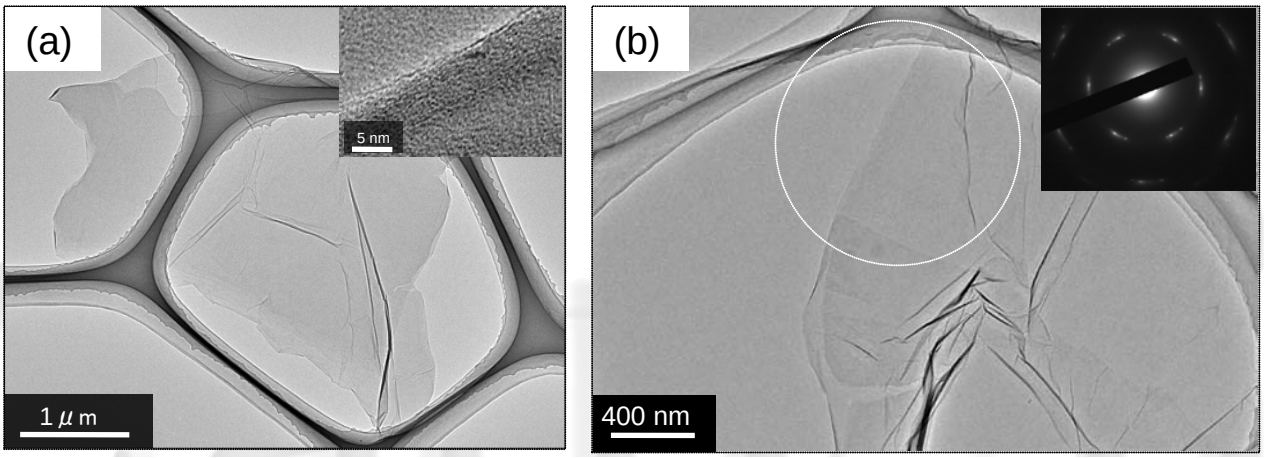

Fig. 2 TEM micrographs of (a) graphene-like carbon thin layers (b) SAED pattern on its carbon thin layers.