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論文内容の要旨

Nowadays, graphitized carbon including graphene is recently one of the most advanced materials for up-scaling in environment-friendly industrial applications due to its incomparable electronic properties, optical, high thermal conductivity, and excellent mechanical properties. The prerequisite for a broader range of applications is to lower its growth temperature. Thus, its low-temperature growth is one of the challenges in the graphene research field. Many techniques have been utilized to fabricate this two-dimensional material including graphite exfoliation, chemical vapor deposition (CVD), and solid-phase reaction. In the solid-phase reaction method, a solid carbon source, such as a polymer film or an amorphous carbon (a-C) film, is annealed on a metal catalyst substrate, e.g. nickel (Ni) and cobalt (Co). In this dissertation, the synthesis of graphitized nanocarbon at ultra-low temperatures using metallic nanoparticles (NPs) is challenged. Also, the fabrication of Li-embedded carbon nanocomposites (Li-C NC) for easy and safe handling is challenged. The movement of Li atoms during the charge-discharge operation may induce the graphitization of a-C. This will be also the strategy for the low-temperature graphitization.

Chapter 1 includes the introduction to the history, structure, and unique properties of graphene and its potential applications. Graphene synthesis techniques, motivation, and the purpose of the thesis are also included in this chapter.

Chapter 2 discusses the experimental methods (magnetron sputtering and ion beam deposition) and the detailed characterization process used in the present work.

Chapter 3 reports a great decrease in graphitization temperature for well-known catalyst Ni using NP form. Amorphous carbon (a-C) films with Ni NPs were deposited onto microgrids and SiO_2/Si substrate by a simple one-step magnetron sputtering method. The a-C surrounding and in-between the Ni NPs started to be graphitized during the film deposition even at room temperature (RT) and at 50°C. Based on the detailed high resolution TEM (HR-TEM) analyses, the decreased oxidation for NPs and the enhanced solubility of carbon into Ni NPs were believed to be keys for the lower temperature graphitization.

In Chapter 4, Co which possesses higher catalytic activity in graphitization for the CVD method than Ni was attempted. Amorphous-C films containing metallic Co NPs were deposited by a simple one-step sputter-deposition technique at RT. The graphitized layers at around and between Co NPs in the amorphous C matrix were confirmed by HR-TEM with a fast Fourier-transform (FFT). The 2D peak and graphite (002) peak were clearly observed in Raman and x-ray diffraction analyses, respectively. The X-ray photoelectron spectroscopy (XPS) analyses were used to determine the metallic state of Co NPs and sp^2 graphitization in the film. From a comparison in catalytic activity in the RT graphitization between Ni and Co NPs, Co NPs were concluded to be more suitable than Ni NPs for the ultra-low temperature graphitization.

In Chapter 5, encouraged by the finding in the previous chapters that the metallic state is preserved for the NPs dispersed in carbon matrix, Li-embedded carbon was prepared by the ion irradiation method. Vertically aligned conical Li-C NCs, sometimes with a nanofiber on top, grew on a graphite foil. As was expected, metallic Li was preserved inside the carbon matrix. Li-C NCs were found to be highly stable under the ambient conditions, making TEM characterization possible without any sophisticated handling in glovebox. The next step will be the in situ TEM observation of the graphitization during charge-discharge process for this Li-C NCs. This will be helpful also for the development of the novel electrode materials for future Li-air batteries.

Chapter 6 summarizes this work and explores future prospects.