

Platelet graphite nanofibers synthesized by the pyrolysis of poly(ethylene glycol)/hydrochloric acid mixture and nickel catalyst

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ABSTRACT

The synthesis of platelet graphite nanofibers (PGNFs) by the thermal decomposition of mixture contain poly(ethylene glycol) (PEG) and hydrochloric acid (HCl) at 700 °C using Ni as a catalyst. The mixture was located in the upstream which PEG was serving as carbon source and HCl serving as accelerant for grown nanofiber. The catalysts were prepared by spin coating nickel acetate solution and oxidation at 700 °C. The PGNFs synthesized with mix HCl in carbon source. Electron microscopy revealed the PGNFs is about 60~80 nm in diameter, with a high degree of graphitization, well-ordered graphene layers. The chlorine played an important role in promoted nanofibers grown and formation of PGNFs.

Keywords: platelet, graphite nanofibers, carbon nanofibers, polymer

1 INTRODUCTION

One-dimensional (1D) carbon nanomaterials such as carbon nanotubes (CNTs) and carbon nanofibers (CNFs) have attracted much attention because of their unique crystalline structure as well as excellent physical and chemical properties. 1D carbon nanomaterials are commonly classified into several types based on the stacked orientation of the graphene layers with respect to the fiber axis. Rodriguez et al. [1] classified CNFs into (i) platelet graphite nanofibers (PGNFs) in which the graphene layers are perpendicular to the fiber axis, (ii) herring-bone carbon nanofibers (HBCNFs) in which the graphene layers are inclined at an angle with respect to fiber axis, and (iii) tubular graphite nanofibers (TGNFs, also named multi-walled carbon nanotubes) in which the graphene layers are parallel to the fiber axis.

1D carbon nanomaterials were synthesized mainly by catalytic chemical vapor deposition (catalytic CVD) [1-4]. Catalytic CVD is an efficient method for the production by controlling the synthesis parameters such as reaction temperature, carbon source, and the type of catalyst. 1D carbon nanomaterials can be synthesized by the decomposition of carbon source (CH₄, CO, or H₂/CO) on

catalysts (Fe, Co, Ni, or their alloys) which enable to dissolve carbon to form metal carbides. It has been reported that the morphology of CNFs had a direct relation with the shape of catalysts during the synthesis [2, 5]. The Ni particles treatment by HCl were formed Ni-Cl compound which compared to Ni particle had higher mobility resulted change in Ni particle morphology and feometrical shapes [6].

Polymer pyrolysis in the presence of catalysts has drawn a lot of attention as a method of forming CNTs and carbon nanofibers (CNFs) [7-10]. At the appropriate temperature, this causes polymers to undergo evaporation, dehydrogenation, and decomposition in the presence of the catalyst, resulting in the formation of CNTs and CNFs. Previous studies report several types of CNTs and CNFs formed using the pyrolysis of poly(ethylene glycol), PEG, as a precursor at temperature ranging from 600 °C to 850 °C [8-11].

In this study, the CNFs were synthesized by thermal decomposition of mixture contain poly(ethylene glycol) (PEG) and hydrochloric acid (HCl) using Ni as a catalyst. The Ni-Cl compounds were restructuring the catalyst shape and grown CNFs.

2 EXPERIMENTAL METHODS

Nickel catalyst was prepared by 0.5M Ni(CH₂COO)₂ (purity 99%, Showa), and 0.5M monoethanolamine (MEA, purity 99.7%, J.T. Baker) were dissolved and well mixed in 2-methoxyethanol (purity 99%, Aifa Aesar) at 60 °C until the solution became a translucent green. The catalyst precursor was spin-coated on a 10 x 10 mm² silicon wafer and thermal treatment at 700 °C under air environment. Silicon wafer with nickel catalysts was placed in the downstream of a thermal reactor. The mixture of carbon source was PEG (MW: 200, Sigma) and HCl (37%, Scharlau) was well mixed and placed in a crucible was located in the upstream of the reactor. The tubular furnace was heated at a ramping rate of 15 °C/min and remained at 700 °C for one hour to synthesize the PGNFs. The morphology of PGNFs was observed with a field emission scanning electron microscope (FE-SEM, Hitachi S-4800), while the crystal structure was examined with a high

resolution transmission electron microscope (HR-TEM, Philips Tecnai F20).

3 RESULTS AND DISCUSSION

Thermal decomposition of PEG in the presence of the nickel catalyst on silicon wafer was carried out to produce various carbon nanofibers by controlling the ratio of PEG and HCl mixture at 700 °C. Figure 1(a) shows the SEM image of nickel catalysts after thermal treatment and 1(b)-1(d) were different ratio of PEG/HCl (b) 1/0, (c) 1/0.1, (d) 1/0.2 in wt%.

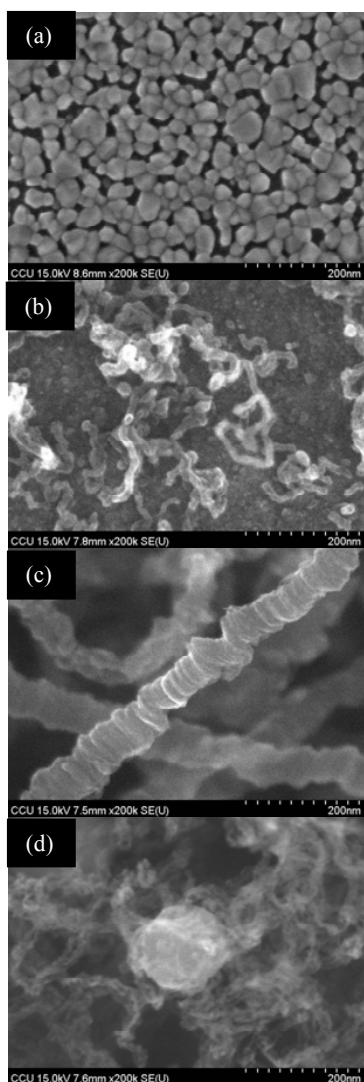
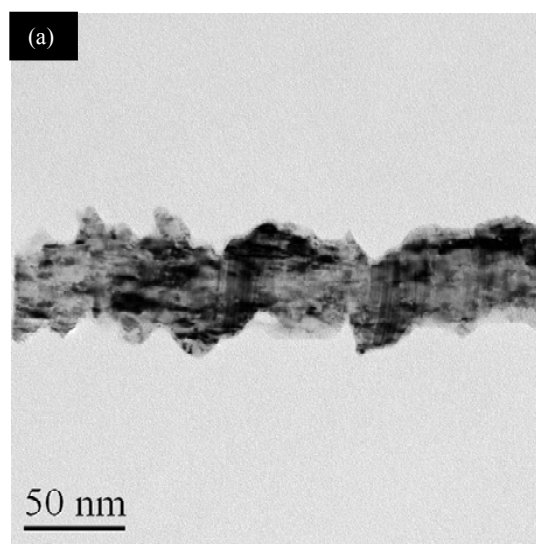


Figure 1: SEM images of (a) NiO, (b) 1/0, (c) 1/0.1, (d) 1/0.2 in wt% of PEG/HCl mixture.

The carbon nanofibers were 40-60 nm in diameter and few μm in length. The HCl amount in carbon source

mixture was the important factor in growth PGNFs. The PGNFs yield was higher when HCl contain 10 wt%. However, carbon particles and CNFs with random morphology were appeared in Figure 1(b) synthesis by only PEG as carbon source. The carbon materials can be thought as transition state of PGNFs and CNFs. Figure 1(d) shows the turbostratic CNFs and catalyst which diameter was about 150-200 nm and larger than CNFs. The morphology of CNFs was effected by their catalyst shape[10]. The PGNF was grown layer by layer perpendicular to the axis direction while the catalyst had a smooth surface. Turbostratic CNF was similar to PGNF, the reason of morphology changed was due to the rough catalyst surface which made the graphene layer couldn't grown along the axis easily. The morphology and shape of nickel catalyst was changed by add HCl in carbon source mixture. During the thermal treatment the HCl was vaporized at first, then, the chlorine was built into the Ni sites to formed Ni-Cl compound[6]. The chlorine was effected the melting points and mobility of nickel resulted in a complete change in nickel particle morphology and geometrical shapes. Figure 2(a) shows the TEM image of PGNF, the side of the PGNF was rough and consist of lots of notches. Figure 2(b) shows the HR-TEM image of PGNF which had a good arrangement of the graphene layers. The graphene layers were grown perpendicular to the nanofiber axis layer by layer. According to SAED pattern image the PGNF a high graphitization degree.



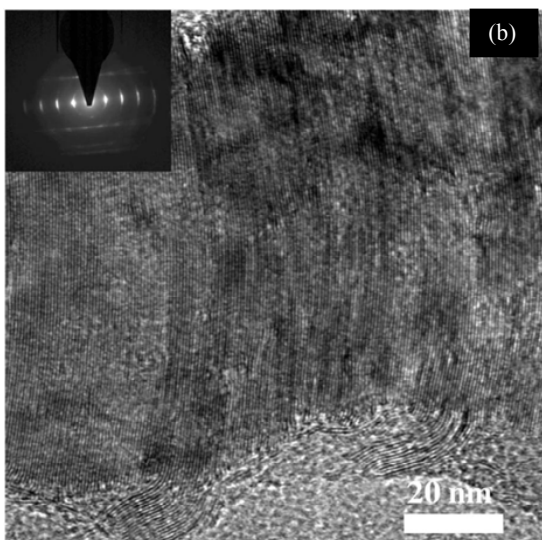


Figure 2: (a) TEM and (b) HR-TEM image of PGNF, inset image is SAED pattern.

4 CONCLUSION

PGNFs were synthesized by the pyrolysis of PEG/HCl mixture and nickel catalyst. The chlorine effect the catalyst shape and CNFs morphology. During the thermal treatment process the more chlorine consisted the more Ni-Cl compounds formed. Ni-Cl compounds were restructuring the catalyst until CNF growth. PGNFs were fabricated when HCl contain 10 wt% in PEG which had 60-80 nm in diameter and good graphitization degree. Turbastratic CNFs were synthesis from the large catalyst particle under the HCl amount was 20 wt%.

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