FURTHER CARBONIZATION OF ANISOTROPIC AND ISOTROPIC PITCH BASED CARBONS BY MICROWAVE IRRADIATION

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Introduction

It has long been known that the thermal treatment of the carbon materials significantly influences various thermal, electrical and mechanical properties. Microwave provides a new source of energy to process a wide variety of materials including polymers, ceramics, chemicals (organic and inorganic) and etc. Microwave energy is derived directly to materials through molecular interactions with the electromagnetic waves. The uniqueness of the microwave is to process materials with selective energy, higher rate, uniform heating and energy conservation [1].

Experimental

Naphthalene derived mesophase pitch (NMP, Mitsubishi Gas Chemical Co., Japan) and pyrolysis fuel oil derived isotropic pitch (PFO, Samsung Chemical Co., Korea) were used for precursors for carbonization. Graphite powder was also used for a reference.

The thermal carbonizations were proceeded in an electrical furnace up to 600 at a heating rate of 5 /min for 1 hour under N_2 flow. The bulk samples carbonized at 600 were granulized in an attrition mill. The further carbonizations were followed at 700, 1300 and 1500 in a conventional electric furnace for 1 hour at a given temperature under Ar flow.

Microwave irradiation was performed in a modified cooking oven. Enough amount (12 g) of the granulated sample for covering the sensing part of thermocouple was deposited in a 30 mm diameter quartz tube and followed by irradiation of microwave (frequency: 2.45 GHz; wavelength: 12.24 cm) under flow of inert gas of Ar or N_2. Irradiation time was accumulated by 7.5 min interval to avoid overloading of the oven. An identification of the sample NMP/M-700-Ar-30 represents NMP thermally carbonized at 700 and followed by microwave irradiation for 30 min under Ar flow. Inconel sheltered thermocouple was used for sensing the temperature of samples in the sample holder.

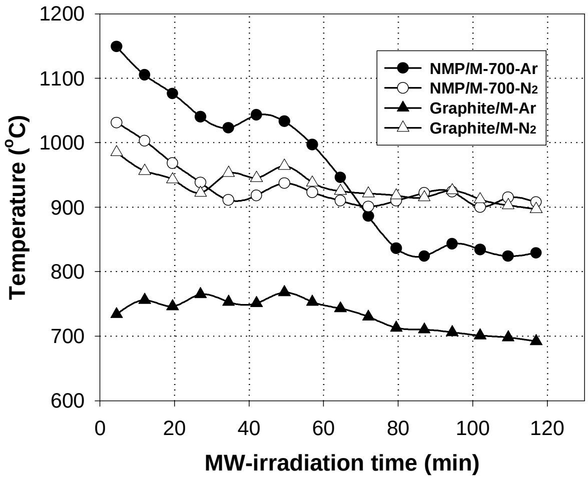
The morphological structures of carbonized samples at various conditions were characterized by X-ray diffraction, and electric conductivity was measured also to illuminate the effect of the morphological structure.

Results and discussion

Only the samples thermally carbonized at above 600 showed heating accompanying emissions of ruddy and blue colored light with the microwave irradiation under flow of N_2 or Ar respectively. Fig. 1 shows the temperature dependence of NMP and graphite on irradiation time. The peak temperature observations with a lapse of irradiation time would be related with the variations of polarizability on the process of structural development upon microwave irradiations. Fig. 2 shows the stack height dependence on the irradiation time. In general, the microwave irradiation increased the stack height with irradiation time. The microwave heating was more effective to increase $Lc_{(002)}$ value than thermal heating in a reduced treatment time. Fig. 3 shows the electric conductivity dependence on the irradiation time. The conductivity of the sample carbonized at 1300 showed higher values than those microwave irradiated, which were opposite results in Lc(002) values (Fig. 2). The results would be from the lower contact resistance related with wider surface area of the particles thermally heat treated in comparison with the particles carbonized by microwave irradiations.

Reference

1. Menendez JA, Menendez EM, Garcia A, Parra JB, Pis JJ. Journal of Microwave Power and Electromagnetic Energy 1999;34(3):137-143.



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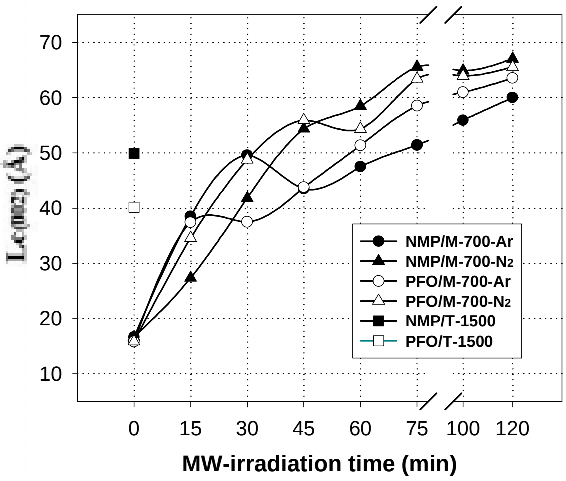

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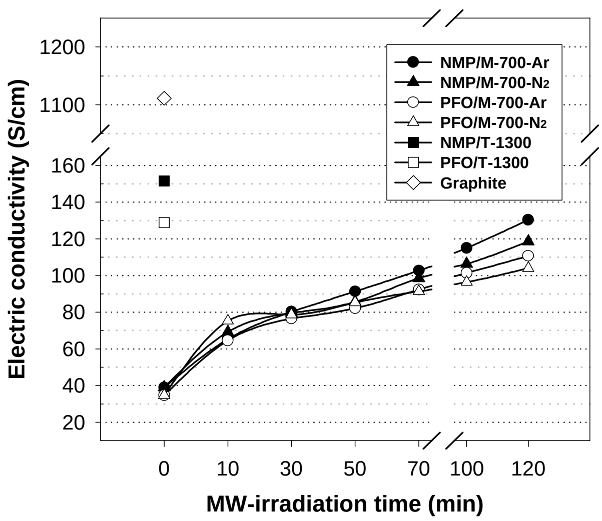

Fig. 3. Variations of electric conductivity as a function of irradiation time.