Heat Treatment of Carbon under Pressure in the Presence of Minerals (Ca-Compounds)

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Good crystals of graphite have often been found in the beds of limestone in the nature. In our laboratory, a remarkable acceleration of graphitization under pressure has been shown. The purpose of the present research is to see the effect of the presence of some minerals on the graphitization under pressure. A part of the work on the effect of limestone was reported previously.

A carbon sample was placed between two tablets made of calcium carbonate, calcium hydroxide or calcium fluoride so as to make a sandwich-type specimens and was heat-treated at various temperatures between 700° and 1400°C under a quasi-hydrostatic pressure of 3 kb in a high pressure apparatus of piston-cylinder type. The carbon sample used was a coke prepared by carbonization of polyvinylchloride and consequent heating to 680°C.

In the presence of calcium carbonate, the carbon specimens heat-treated for 60 min. above 1000°C were obtained as sintered tablets and their profiles of (002) diffraction were composite, a sharp peak at a diffraction angle $2\theta = 26.5°$ ($d = 3.36$ Å) appearing on a broad peak at $2\theta = 25.8 \sim 26.1°$ ($d = 3.48 \sim 3.42$ Å). With the increase in the heat treatment temperature the sharp peak became stronger at the same position and the broad peak decreased its intensity, shifting to a higher angle of diffraction till around 26.1° ($d = 3.42$ Å). The temperature at which the sharp peak began to appear seemed to be higher with the decrease in the residence time, being 1250°C for 20 min. The appearance of the sharp peak at the spacing of 3.36 Å seemed to began at around 1000°C for 60 min. treatment under 3 kb in the presence of calcium carbonate, while the graphitization began to occur at around 1500°C at the same pressure and residence time without calcium carbonate. In the carbon specimens heat-treated above 1000°C calcium

carbonate was detected by X-ray and in those treated above 1200°C detected even visually. The tablets of calcium carbonate heat-treated above 1000°C were obtained as recrystallized ones.

In the presence of calcium hydroxide, the sintered tablets of carbon was obtained above 700°C. On the profile of (002) diffraction of carbon, the sharp peak at the spacing of 3.36 Å appeared on a very broad band even at 800°C. The sharp peak seemed to become stronger with the increase in the temperature and the residence time of the treatment. The heat treatment in the presence of calcium hydroxide could not be carried out above 900°C because of the melting of the hydroxide above that temperature.

In the presence of calcium fluoride, the sintered tablets of carbon were obtained above around 1000°C. The appearance of the sharp peak at the spacing of 3.36 Å was found to begin at around 1300°C.