POSTER

ESR BEHAVIOR OF PITCH CARBON

Kazuro Kawamura

Dept of Materials Science and Engineering The National Defense Academy 1-10-20 Hashirimizu, Yokosuka 239, Japan

INTRODUCTION

ESR study has been done for carbon materials from various origins. When pitch giving soft carbon is heat-treated at the temperature below 1000 °C, radicals are produced and stabilized in carbonized structure. As the result, ESR absorption spectrum is observed. The intensity of ESR absorption spectrum shows the maximum at heat treatment temperature (HTT) about 600°C and it decreases as HTT rises. At HTT 1200-1400°C ESR spectrum usually disappears, and appeared again at HTT above 1500 °C. The disappearance of the ESR absorption spectrum in this region is wellknown, but a clear explanation has not been made regardless of number of reports [1-4]. There are two possible explanations: (1) The line width is too broad to be caught. (2) The radical concentration decreases so largely that it is impossible to measure. In this report, this disappearance was discussed from the heat treatment time (HTt) dependence measurement.

EXPERIMENTAL

Coal tar pitch (chemical compositon (wt%), C:91.9, H:4.84, N: 1.29, S:0.5, ash:0.02) was carbonized at 1000°C in argon atmosphere. The obtained pitch carbon was heat treating at 1100-2250 °C for HTt 1-25 hr in argon. The ESR absorption spectra were measured on the powdered sample in air in a high vacuum by using JEOL Xband spectrometer. The radical concentration was determined by the comparison of the area of the spectrum of sample with that of DPPH.

RESULTS AND DISCUSSION

Measurement in air

On the ESR absorption spectra at HTTs 1100 °C and 1500 °C there was a very much change in intensity with HTt. The intensity at HTT 1100 °C decreased as HTt became longer. While, the intensity at HTT 1500 °C increases as shown in Fig. 1. At HTT 1250 °C the ESR absorption spectrum could not be measured. The changes of line width of spectra at HTT 1100-2250°C are shown in Fig. 2. The line width decreases largely at HTT 1100-1500 °C.

Measurement in a high vacuum There is a possibility to get a fine spectrum in a high vacuum because adsorbed air on the sample surface is removed. Figure 3 shows the ESR absorption spectra at HTT 1100-1250°C. Two components are present in the spectrum at HTT 1100 °C. The line width of the broad line is about 100G and the narrow one is 17-20G. The intensity of the both lines decreased as HTt became longer, and only a weak, narrow line was observed at HTT 1250 °C. That is, an ESR absorption spectrum was detected at HTT 1250 °C.

The HTt dependence measurement supports that the disappearance of the ESR absorption spectrum seen in the pitch carbon at HTT near 1250 °C is caused by a large decrease of radical concentration. At the measurement in a high vacuum, a duplicate ESR absorption spectrum composed of a broad and narrow line was observed at HTT 1100 °C, and a weak, narrow line was detected at HTT 1250 °C. If the broad and narrow lines are attributed to the localized and delocalized unpaired electrons respectively, the HTT 1250 °C seems to be a noticeable temperature on the process of graphitization of the pitch carbon.



Fig. 1 Changes in intensity of ESR spectra in air.

REFERENCES

- 1. J.G. Castle, Jr., Phys.Rev. 94, 1410 (1954).
- 2. G.R. Henning, B. Smaller and E.L. Yasaitis, Phys.Rev. 95, 1088 (1954).
- S. Mrozowski, Carbon 17, 227 (1963).
- 4. S. Orzeszko and K.T. Yang, Carbon, 12, 493 (1974).



Fig. 2 Changes in line with (Δ H) of ESR spectra in the HTT of 1100 - 2250 °C in air.



Fig. 3 ESR spectra in the HTT of 1100-1250 °C in a high vacuum.