EFFECT OF HEAT TREATMENT TIME ON GRAPHITIZATION OF KAPTON-DERIVED CARBON FILM AT VARIOUS HEAT TREATMENT TEMPERATURES

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Introduction

Kapton is known as one of aromatic polyimide films from which we can prepare very graphitizable carbon films. We have studied long years on graphitization behavior of Kapton-derived carbon films, especially on that with 25 μm in thickness, with fixed time of heat treatment, 30 min at the top temperature[1]. In this paper, we studied the effect of heat treatment time (HTt) for Kapton-derived carbon film, carbonized at 900°C, by heat treatment at temperatures between 2000 and 2200°C. The effect was investigated by measurements of X-ray diffraction (XRD) and magnetic susceptibility.

Experimental

The starting material used was a Kapton with 25 μm in thickness. It was cut into rectangular specimens of about 10 mm x 20 mm. They were carbonized in a flow of nitrogen gas at 900°C with a residence time at the top temperature of 1 hr by the method described in reference 1. The carbonized films were heat-treated at temperatures of 2000, 2050, 2100 and 2200°C also by the method described in reference 1. Treatment times at each top temperature were 12, 20, 30, 45,75, 90, 120, 150, 180 and 300 min, respectively.

Since carbon films thus heat-treated were well oriented, XRD measurements were carried out for each film specimen in two measurement modes, the reflection and transmission modes, respectively. A cleaved thin HOPG was used as an external standard. The 004 diffraction was measured for each specimen in the reflection mode and the interlayer spacing d_{002} and crystallite thickness L_c were determined. The crystallite diameter L_a was obtained from the 110 diffraction measured in the transmission mode.

Room temperature magnetic susceptibility was measured for each specimen in magnetic fields up to 5 T applied perpendicular and parallel to the specimen surface. The total magnetic susceptibility was determined for each specimen.

Results and Discussion

Figure 1 shows the interlayer spacing d_{002} for the specimens as a function of HTt with heat treatment temperature (HTT) as a parameter. At HTT of 2000°C, d_{002} is 0.3427 nm at HTt of 12 min and decreases very gradually with HTt. At HTT of 2050°C, d_{002} is 0.3417 nm at HTt of 12 min, keeps its value up to HTt of 45 min and then decreases down to 0.3384 nm at HTt of 300 min. Variation of d_{002} at HTT of 2100°C is similar to that at HTT of 2050°C, but the value at HTt of 12 min is 0.3402 nm and d_{002} starts to decrease at HTt around 30 min. The d_{002} value at 300 min is 0.3380 nm. At HTT of 2200°C, d_{002} decreases monotonically from the value of 0.3402 at HTt of 12 min. The d_{002} value at 300 min is 0.3366 nm. It seems that at each HTT the d_{002} value at early stage of HTt is kept in an interval and then graphitization takes place.

The crystallite thickness L_c is shown for the specimens in Figure 2 and the crystallite diameter L_a in Figure 3. At each HTT, both of L_c and L_a increase with HTt, showing a trend of saturation against HTt at higher HTT.

The total magnetic susceptibility (trace of susceptibility tensor) defined by $\chi_T \equiv \chi_\perp + 2\chi_{para}$ is used to investigate the magnetic behavior in the present study. $\chi_\perp$ and χ_{para} are the susceptibilities with the magnetic field perpendicular and parallel to the specimen surface, respectively. All of the specimens show diamagnetic susceptibilities. Figure 4 shows $|\chi_T|$ plotted as a function of HTt with HTT as a parameter. $|\chi_T|$ changes rapidly with HTt at early stage of HTt at HTTs of 2050 and 2100°C. The variation of $|\chi_T|$ against HTt at HTTs of 2000 and 2200°C is weak. $|\chi_T|$ depends on both of d_{002} and L_c as shown in Figures 5 and 6, respectively. With increasing d_{002}, $|\chi_T|$ increases at first, passes through a shallow maximum and then saturates to the value corresponding to that of the single crystal of graphite. $|\chi_T|$ increases at first with increasing L_c, passes through a shallow maximum and then reaches to that of the single crystal of graphite. Similarly, $|\chi_T|$ varies against L_a as varies against L_c, though the variation is not shown as a figure.

References

1. Hishiyama Y, Igarashi K, Kanaoka I, Fujii H, Kaneda T, Koidesawa T, Shimazawa Y, and Yoshida A. Graphitization behavior of Kapton-derived carbon film related to structure, microtexture and transport properties. Carbon 1997; 35 (5): 657-668.

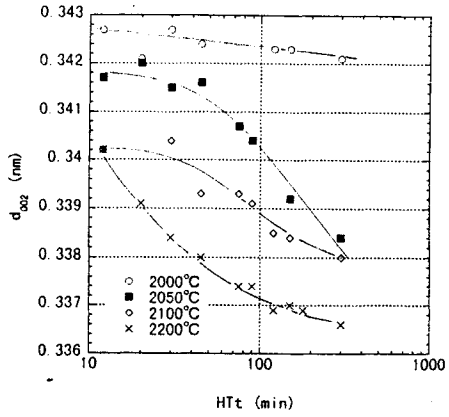

Figure 1. Interlayer spacing d_{002} for specimens plotted as a function of HTt with HTT as a parameter.

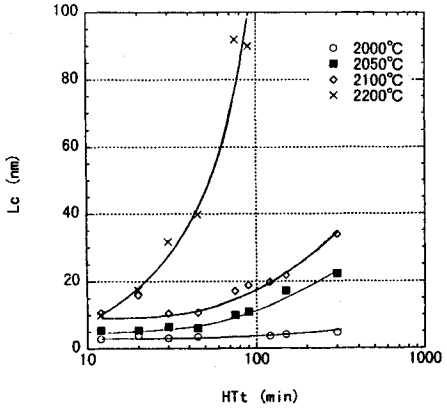

Figure 2. Crystallite thickness L_c for specimens plotted as a function of HTt with HTT as a parameter.

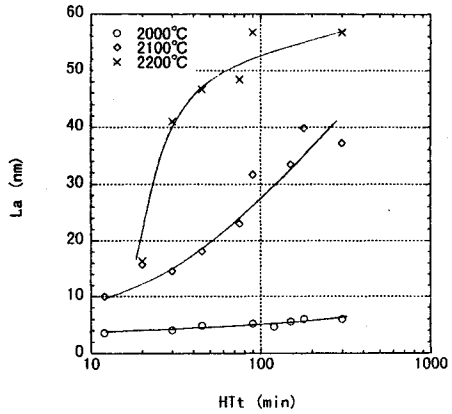

Figure 3. Crystallite diameter L_a for specimens plotted as a function of HTt with HTT as a parameter.

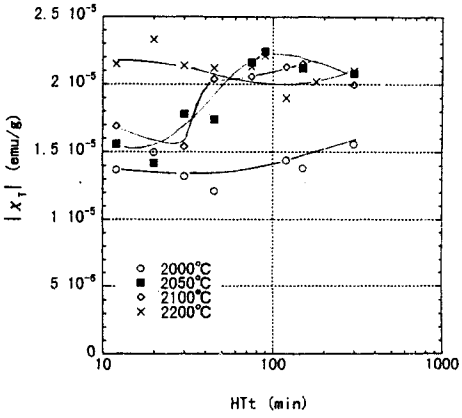

Figure 4. $|\chi_T|$ plotted as a function of HTt with HTT as a parameter.

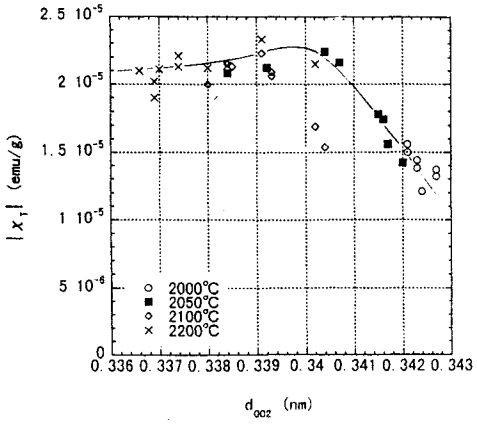

Figure 5. $|\chi_T|$ for specimens plotted as a function of d_{002}.

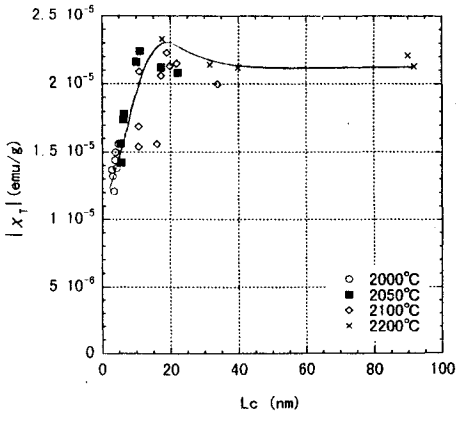

Figure 6. $|\chi_T|$ for specimens lotted as a function of L_c