EVOLUTION ON THE STRUCTURE OF PI FILM-DERIVED CARBON PRODUCTS DURING THE CARBONIZATION BY WAXD METHOD

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Introduction

During recent years there have been many studies on the carbonization and graphitization of polyimide film "Kapton"[1-5]. In this study,by X-ray diffraction technique we were determined the structure of the carbon products derived from PI(Polyimide)film made in our country .this material at high temperature have attracted much attention as accessible synthetic metals. The article authors have obtained the informations and parameters of relatively detail during the carbonization of sample .These very significative results provided the scientific bases for understanding of the carbon functional key.

Experimental

The sample used in this study was prepared from aromatic dianhydride and diamino polycondensation,again imidized by the temperature around 300 ℃,original film can be transferred a white into a brown-yellow,obtaining transparent no-pinprick brown-yellow thin film carbonized to 1000 ℃ under nitrogen atmosphere according to a heating rates. The investigative samples at different temperature for PI film obtained.An analysis of X-ray diffraction for sample were proceeded on a Rigaku Denki model D/max-rA rotating anode X-ray diffractometer with CuK_α(1.5418Å)incident radiation.The rotating anode X-ray generator operating at 40 KV and 80mA, CuK_α beam diffracted by the specimen was selected through a monochromator of graphite whiched to the detector.Diffraction patterns were measured in reflection mode.Divergence slit of 1°,scattering slit of 2° and receiving slit of 0.5mm were used for all specimens.The diffraction angle 2θ was scanned between 3 and 32°.The scanning of 2θ was repeated many times under a scanning speed(1/4)°/min and diffraction intensity was numbers.Diffraction patterns were collected ,stored and processed by a data system based on an apple II microcomputer.

Results and discussion

Fig.1 shows the patterns of X-ray diffraction for a variation of PI film with pyrolytic temperature under nitrogen atmosphere.As can be seen from Fig.1, two equatorial diffraction peaks at room temperature appeared (One at 2θ =5.85° is a weak peak,and the other at at 2θ =18° is a strong peak),their interplanar spacings(d)were calculated approximately.The spacing d ≈ 0.151nm,corresponding to the (002) reffection,may be attributed to the periodic repeating stucture within the polymer chain.The spacing d ≈ 0.493nm,corresponding to the (010) reflection,may be attributed to the spacing between adjacent parallel chains.These results are in agreement with the experimental data by M.Kochi[6].When the temperatures raising to 600℃,the diffraction peak at 2θ =5.85° is disappeared, the diffraction peak at 2θ =18° is transferred the sharp peak into like-steamed bread peak,this is due to an arrangement of the molecular chains exhibited a disordering state.The diffraction peak at 2θ =18° disappeared,and new weakling peak at 2θ =22° appeared and grew gradually with increase in the pyrolytic carbonization temperature, implying the formation and growth of the ladder polymer structures of hexagonal layer planes (like (002) planes).

As can be seen from fig.2, in the temperature range of 700-1000 ℃ 2θ values are ascended slowly in alignment.The half high widths are decreased suddenly before 800℃, after are decreased gradually.This is due to the forming hexagonal carbon layer crystallite close up to the graphite structure with increase in the pyrolytic carbonization temperature.

Fig.3 shows the c-axis interplanar spacing and the microcrystallite size of sample calculated from the angle of the (002) reflection peak as a function of pyrolytic carbonization temperature.As the pyrolytic temperaturs raised the interplanar spacing is decreased, but the microcrystallite size is increased.These results offers evidence for the grow of like graphite structure of the solid products.

Conclusions

The structural evolution of the pyrolytic products of PI film during the carbonization have been measured by using X-ray diffraction technique. From the variations of the diffraction peaks with the pyrolytic carbonization temperature it was found that the pyrolysis of the molecule chain for sample occurred and the lamellar supermolecular structure to transition the randon state.when the temperature raising to 700 ℃ the (002) diffraction peak of six-angular network plane of a graphite -like structure was observed,the diffraction intensity increases with raising pyrolysis temperature.On the basis of Bragg formula and Scherrer experience expression the relationships between the microcrystalline sizes and the spacing of the c-axis interplanar versue pyrolytic temperatures were discussed.

References

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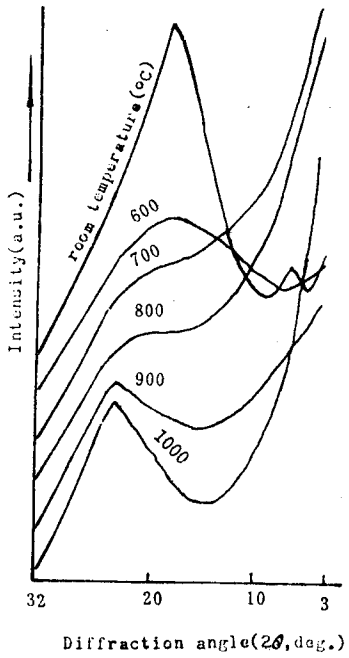

Fig.1 Patterns of X-ray diffraction for a variation of PI-film with pyrolytic temperature under nitrogen atmosphere

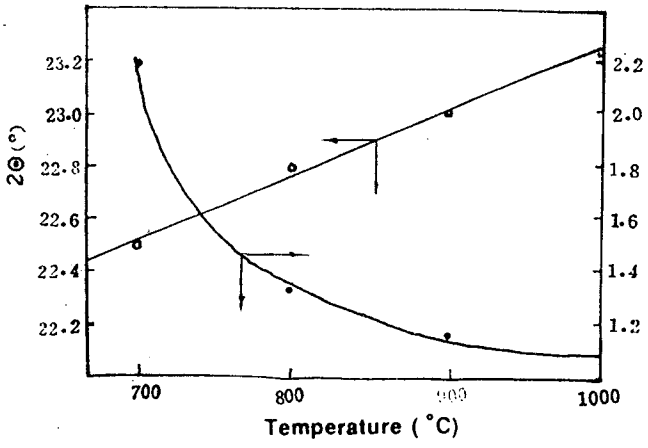

Fig.2. Relationships between 2 θ values and the half high widths VS.pyrolytic temperatures.

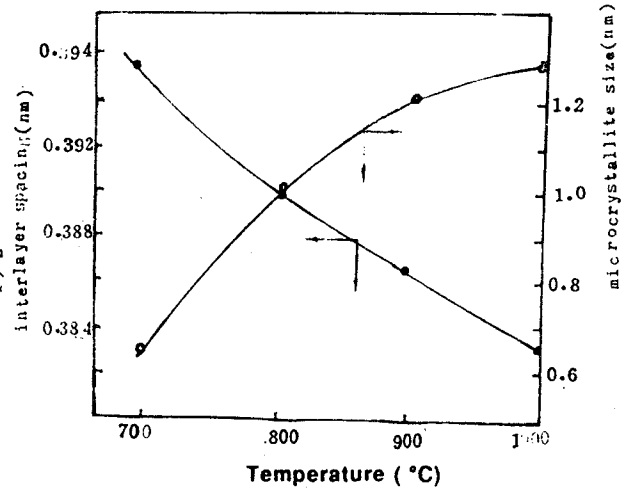

Fig.3. Dependence of the Spacing of Lattice and the Microcrystalline Sizes on Pyrolysising Temperatures.