ACTIVATION OF MESOPHASE PITCH BASED CHOPPED CARBON FIBER USING POTASSIUM COMPOUNDS

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Introduction

A number of porous carbons have been produced from isotropic or non-graphitizeble precursors through the variety of activation procedures. Recent study reveled variety of porous carbons are ascribed not only to porosity but also to be surface chemistry such as functional groups and surface carbon structure, hexagonal size and stacking. Hence it is one of interests to propose a porous carbon from anisotropic precursor, which carries higher stacks of cluster and larger domains of the texture. In the present study activation of mesophase pitch based carbon was activated by KOH [1] since KOH has been reported to provide very large surface at moderate yield from any carbon material [2-3]

Experiments

The sample was mesophase pitch based chopped fiber (650℃, carbonized) 0.6g of CF and KOH mixed weight by ratio of 4, 3 or 1, were placed in the bottom of metal tube. The other CF is placed in the top of the reactor as a trap. The activation was performed by heating up to 600-900℃ of final temperature in the N_2 flow holding 1-7h、 at the final. After activation, CF was washed by water and dried under vacuum for 12h. The surface area of activated CF was measured using sorptmatic 1990. The morphology of ACF was observed with high resolution SEM.

Results and Discussion

Tables 1 and 2 summarize the surface area and elemental compositions of ACFs prepared from mesoporus pitch through the activation with K_2CO_3 and KOH (the weight ratio was 4 in both cases), respectively at 600-700℃ for 1h. K_2CO_3 gasified CF but no marked increase of surface area was obtained, the area being 140m^2/g by activation at 800℃. In contrast KOH provided very large surface area, which did depended very much on the activation temperature. Activation at 800℃ appears optimum giving surface area as large as 2300m^2/g. The yield of ACF was 65%. Fairly large yield regardless of the surface area was noted. Table 3 summarizes the influence of holding time 1h appears sufficient to activated CF with the present condition. Longer reaction time tends to reduce the surface area. Influence of KOH/CF ratio is also includes in Table 3. By the present study, the procedure appears to require large amount of KOH to obtain the large surface area. Figures 1and 2 illustrate morphology of ACF in long and short filaments. There were observed to elarctension feature. One is definite crack running along with the fiber axis. It is more marked with the longer filament. The edge of the filament showed wide open of the crack. The shorter filament showed much narrower crack. Second feature is that a number of shallow spurs run also along the fiber axis in both long and short filaments.

The present paper report that mesophase pitch based carbon fiber can be activated with KOH into ACF of very large surface area at excellent yield, which is over 2000m^2/g. In contrast, K_2CO_3 was founded to give much smaller surface area although similar amount of CF was gasified. Different surface chemistry of mesophase pitch based carbon fiber may develop unique functions of the graphitizable ACF. The mechanism for the activation by KOH appears simple in chemistry where KOH oxidized the carbon fiber, producing K, H_2O and CO_2metallic may vaporizes K out from the system to be trapped by any means. K adheres to CF in form of K_2CO_3. Small grains of K_2O may gasify selectively the carbon, digging pores of rather uniform and small diameter. From the morphologies of activated fiber, there are two mechanisms of KOH (K)-CF interaction. The surface of CF is activated at the interfaces of fibrils which run parallel to the fiber axis, inducing a number of spurs , micro pores are believed to develop along the spurs. Another interest produces a crack with in the filament, running along the fiber axis. The gasification may start from the cross-section of the filament and shrinkage along the periphery of the filament may induce the crack, which is the largest at the cross sectional surface.

Conclusions

1.KOH provided large surface area to mesophase pitch-based Carbon Fiber.
2.Maximum surface area 2255 m^2/g was achieved at reaction condition of KOH/CF=4, reaction Temp. =800℃, 1h hold.

References

1. Arnold N. et al., US Patent 3,817,874, 1974

2. Chan B K, Thomas K M, Marsh H. Carbon 1993; 31:1071.

3. Kasuh T, Scott DA, Mori M. Extended Abstracts, Carbon'88,Newcatsle upon Tyne U.K., 1988:146

Table 1 Elemental analysis of K_2CO_3 treated fibers

HTT(℃)	Surface area[m^2/g]	H	C	N	O(diff.)	Ash
As-received	-	2.57	94.45	0	2.98	-
600	47	2.18	88.67	0.13	8.97	0.05
700	114	1.42	85.69	0.21	12.51	0.17
800	139	0.91	84.59	0.03	11.57	2.9
900	100	0.63	82.26	0.93	15.81	0.37

K_2CO_3/CF=4, Holding time 1h. , Flow rate 0.3l/min

Table 2 Elemental analysis of KOH treated fibers

HTT(℃)	Surface area[m^2/g]	H	C	N	O(diff.)	Ash
As-received	-	2.57	94.45	0	2.98	-
600	620	2.33	88.63	0.13	7.86	1.05
700	1586	74.47	0.07	21.43	2.38	0.17
800	2255	0.78	83.48	0.43	8.21	7.10
900	1674	1.16	83.30	0.26	14.44	0.84

KOH/CF=4, Holding time 1h. , Flow rate 0.3l/min

Table 3 Change of surface area (m^2/g)

KOH/CF(Ratio)	1	3	4
Holding time 1h	76	1350	2255
Holding time 5h	80	-	1230

Activation Temp. :800℃ 0.3l/min holding times

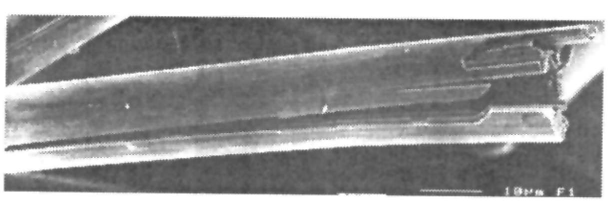

Fig. 1 HR-SEM photograph of ACF fiber type

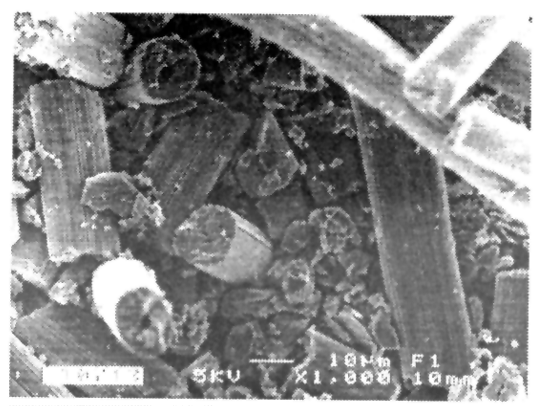

Fig. 2 HR-SEM photograph of ACF powder type