Preparation of Carbon Fiber from Coal Tar Reacted with Nitrogen Containing Compounds

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Introduction

General purpose carbon fiber(GPCF) is normally used a precursor for activated carbon fiber(ACF) through oxidative activation. It was reported that the ACF containing enough nitrogen(1-3%) gave an excellent adsorptivity to NOx and SOx.[1,2]

In the present study, THF insoluble coal tar was polymerized with nitrogen containing compounds such as nitrobenzene and p-nitroaniline. The precursor obtained through the reaction was used for fiber preparation.

Experimental

The coal tar(Jung-u Coal Chemical Co, Korea) was reacted with nitrobenzene(NB) or p-nitroaniline (NA) at various concentrations at 300℃ for 1 hour under nitrogen atmosphere. 5wt.% of NA(NA5) was reacted with coal tar and followed by heat treatment 350℃ to raise the softening point from 135℃ to 28 7℃ in the preparation of precursor for fiber spinning. The precursor pitch was extruded through one hole spinneret(0.2mm-D/0.4mm-L) at 10kgf/cm^2, 300℃, and followed by stabilization by air blowing at 325℃. The stabilized fiber was carbonized at 600, 1000℃ for one hour under nitrogen and argon atmosphere respectively. The precursors and fibers were characterized by using FT-IR, ^{1}H-NMR, elemental analysis, optical microscopy and X-ray diffraction. The aromaticity, f_a, was calculated on the basis of the Brown-Lander method.

Results and Discussion

The softening temperature of the modified pitch increased after reaction of 20wt.% NA from 85℃ to 350℃ (Fig. 1). It was investigated that NA polymerized coal tar molecules. The f_a value increase was observed with NA concentration increase indicating the condensation reaction accompanied by dealkylation. The benzene insoluble(BI) - pyridine soluble(PS) fraction, parameter of processibility, was most abundant in the modified pitch at 5 and 15 NA wt.%. The 5wt.% NA introduced 1.3% nitrogen through the reaction(Table 1) and remained 0.75% after 1000℃ heat treatment.

Carbonization yield was also increased with an increase of NA concentration. The increase of molecular size gave an effect on the optical texture of the carbonized material form large fluid texture through fine mosaic to totally isotropic with an increase in NA concentration (Fig. 2). This would be resulted from reduced mobility in the carbonization procedure because of enhanced molecular weight through the chemical modification.

The X-ray parameters in Table 2 represent that the less ordered texture was formed at the higher concentration of NA though the Lc(002) values for carbonized materials were relatively small and they were not much dependent on the NA concentration. The carbon fiber with isotropic cross section was prepared as shown in Fig. 3.

References

1) Mochida, I., An, K. H., Korai, I., Extended Abstracts Program., 1995, pp. 48-49
2) Jansen, R. J. and Bekkum, H. V., *Carbon*, 1995, **33**, 1021

Table 1. Elemental analysis of pitches reacted with nitroaniline.

Samples	Elemental Analysis (wt.%)				Atomic ratio		fa
	C	H	N	S	H/C	N/C	
NA5	92.38	4.24	1.27	0.49	0.551	0.012	0.940
NA10	91.19	4.11	2.16	0.47	0.541	0.020	0.958
NA12	91.78	4.12	2.24	0.44	0.539	0.021	0.965
NA15	90.97	4.08	2.95	0.42	0.538	0.028	0.984
NA17	90.73	3.92	3.36	0.43	0.519	0.032	0.987
NA20	90.09	3.80	4.58	0.41	0.506	0.044	0.990

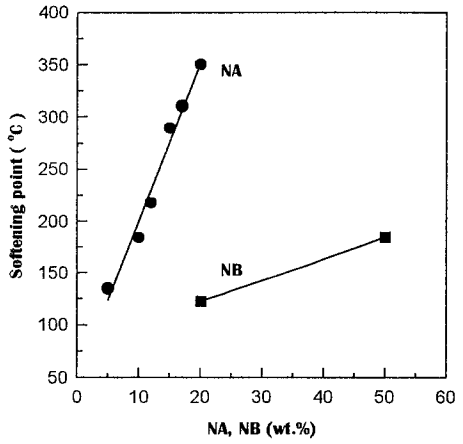

Fig. 1. Softening point dependence on the concentration of NA and NB.

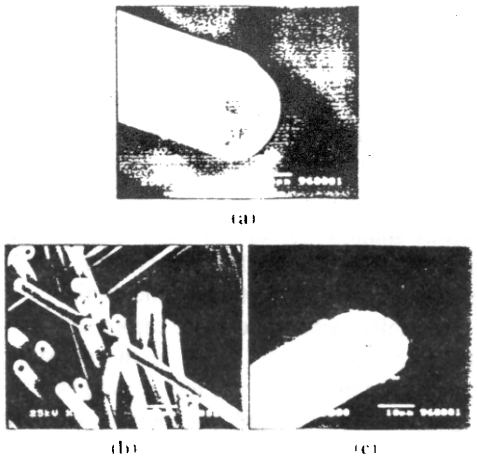

Fig. 3. SEM microphotographs of a stabilized fiber and a carbonized fiber.
(a) stabilized fiber, (b) & (c) carbonized fiber.

Table 2. X-ray parameters of carbonized NA-modified pitches heat-treated at 600℃ and 1000℃ for 1hr.

Samples	600℃ - HTT		1000℃ - HTT	
	*Lc(002) (Å)	d(002) (Å)	*Lc(002) (Å)	d(002) (Å)
NA5	20.4	3.46	21.0	3.51
NA10	20.4	3.45	21.0	3.53
NA12	18.7	3.45	21.1	3.48
NA15	18.7	3.45	21.2	3.48
NA17	18.8	3.45	20.4	3.53
NA20	17.6	3.49	17.3	3.50

*Lc(002) = $(K\lambda)/(\beta\cos\theta)$, λ=1.5405Å, θ=diffraction angle, β=full width of half maximum in radian, K=constant

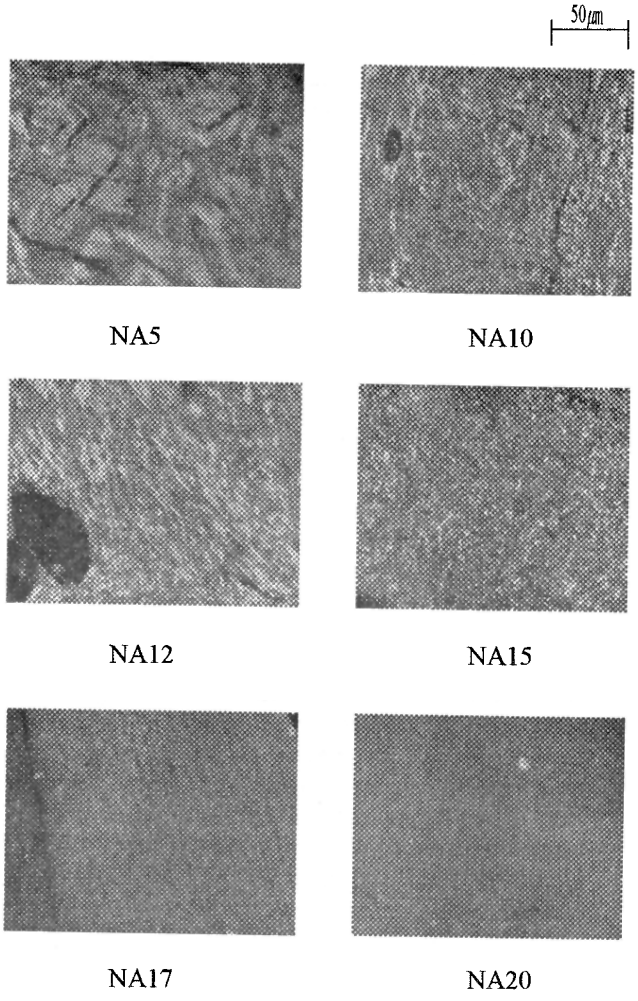

Fig. 2. Polarized light microphotographs of cokes heat-treated at 600℃ for 1hr.