Preparation of Metal Contaning Carbon Fiber from Coal Tar Pitch

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Introduction

The metal containing carbon materials expected to be applied in various application such as pore control because of its unique property. In some case metals is used to improve crystallization at low relatively temperature[1]. In this study we have attempted to prepare Ag-, Al- and Fe-filled carbon fiber by remaining the metal catalyst used in the process of precursor preparation. And also, the structures of carbon fibers were investigated.

Experimental

Coal tar pitch(softening point 110℃) was dissolved in THF(TSP-Table 1.) for removing primary QI in coal tar pitch. TSP was condensated in the presence of Br_2 and catalyst of 3 wt% each AgBr, $AlCl_3$, $FeCl_3$ at 180℃ and followed by N_2 blowing at 330℃.

Table 1. Elemental Analysis of TSP

	SP[1] (℃)	Elemental Analysis (wt.%)					C/H ratio
		C	H	N	S	O(diff.)	
TSP	85	91.97	4.31	0.94	0.53	2.25	1.79

1) Softening point

The precursor pitches containing the metal were spun into fiber through round type spinneret(L/D=0.6/0.3mm) and followed by stabilization at 300℃ and carbonization at 1000℃. The carbon fibers were activated at 900℃ with H_2O/N_2=0.4 atmosphere.

The softening point was measured by Mettler FP83 apparatus. The surface fibers were investigated by scanning electron microscopy(SEM)

Result and discussion

Softening points and yields of the reaction products were summarized in Table 2. The yield of samples which synthesized with catalysts was in the increasing order of Br_2 only, Br_2+$AlCl_3$, Br_2+AgBr, Br_2+$FeCl_3$. It imply that catalysts have contributed to increase in reactivity. Accordingly, the highest softening point of TSP/Br_2-$FeCl_3$ 3% is correlated to the highest of reaction yield.

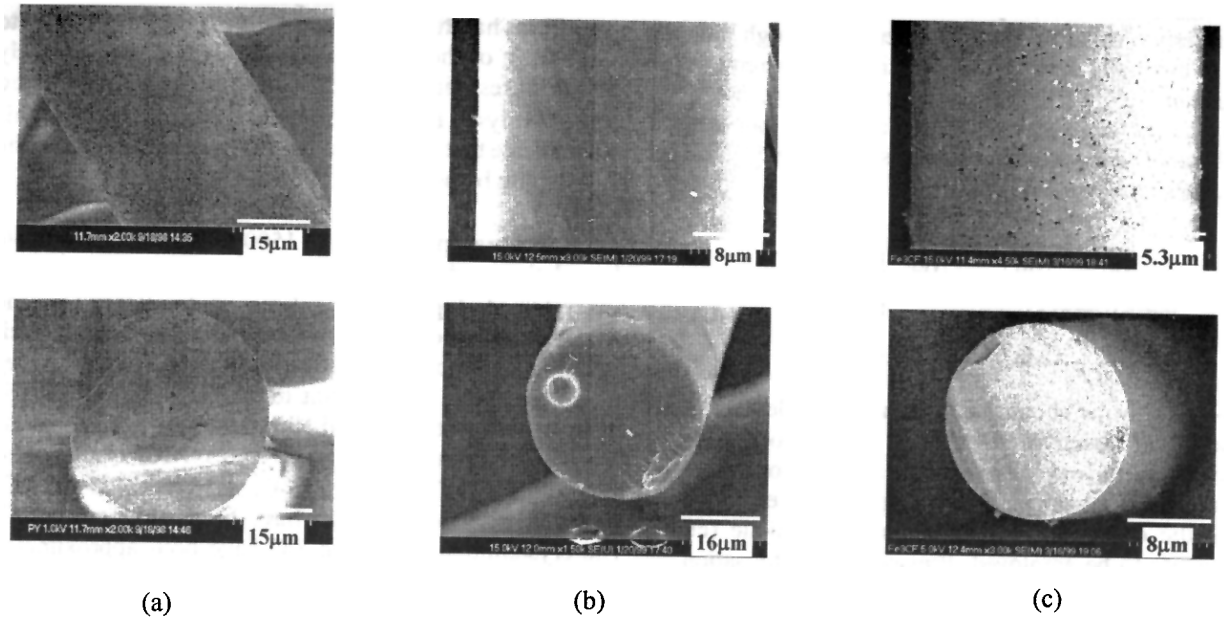
Fig 1. shows the SEM of samples that carbonized at 1000℃. The fibers applied metal catalyst exhibit pores on the surface of fiber after carbonization at 1000℃. This pores would be created in the process of diffuse out of the metal through carbonization. Only with carbnoization in Ar atmosphere, the pores were already formed and there surface area for FeCF was 530m^2/g. The burn off was relatively small at relatively severe conditions. The burn off ratio was in the order of AgCF, AlCF, FeCF, which was unexpected.

Reference

Antoine L, Gachon J, Sklovaky D, Filatov A, Nalimova V, International Symposium on Carbon, TOKYO, TANSO, 1998:130-131

Table 2. Softening point and yield of samples

		TSP/Br_2	TSP/Br_2 -AgBr 3%	TSP/Br_2 -$AlCl_3$ 3%	TSP/Br_2 -$FeCl_3$ 3%
Softening Point(℃)		253.8	260.2	263	302.5
Yield(%)	Modified Pitch	62.0	74.0	63.3	85.2
	Carbonized fiber	85.5	75.4	65.4	66.1



(a) (b) (c)

Fig 1. SEM microphotographs of carbonized fibers at 1000℃
(a) AgCF (b) AlCF (c) FeCF

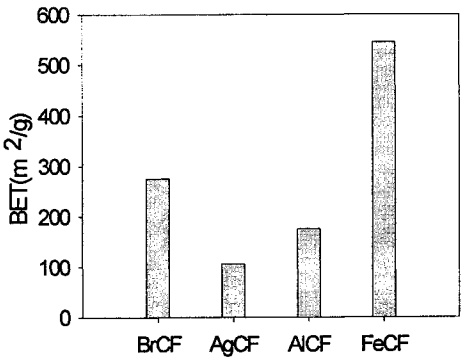


Fig 2. BET surface area of carbonized fibers.

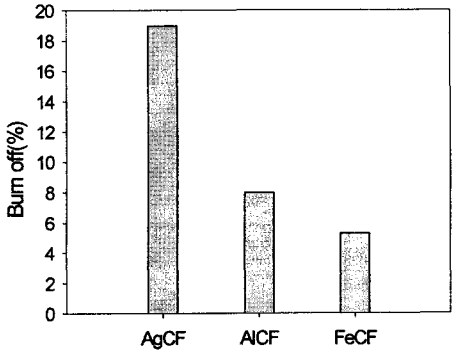


Fig 3. Burn off(%) of samples from activation under H_2O/N_2 atmosphere.