Multi-Staged Carbonization of Aramid Fibers

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1. INTRODUCTION

Aramid, particularly, Kevlar fibers have attracted much attention of researchers as a precursor for high modulus carbon fibers[1, 2] and high efficiency active carbon fibers[3] because they are composed of a linear single aromatic rings and no stabilization reaction in the oxygen atmosphere is necessary. However, high price and low carbon yield are major obstacles for broad application of them as a precursor of carbon materials. The carbon yields of Kevlar fibers range from 25 to 40% after carbonization at 1000℃ under inert atmosphere[1,3].

In this study, the present authors examined multi-staged carbonization of the Kevlar and Nomex fibers to increase the carbon yields. Thermal analyses(TGA :thermogravimetry, DTA :differential thermal analysis) were used to track the degradation reaction of amide bonding and change of carbon yield for mechanistic study.

2. EXPERIMENTAL

Poly(p-phenyleneterephthalamide :PPTA) short fibers(KIST) and Nomex(Du Pont) were used as a precursors in this study. 4mg of PPTA and Nomex fibers were set in a platinum cell(5mm in depth), placed in thermal analysis system(SEIKO Instrument Co. SSC-5200) and heated to 1000 C under pure nitrogen atmosphere (flow rate:400ml/min) by various heating conditions.

Scanning electron microscope (SEM) was used for observing the carbonized PPTA short fibers.

3. RESULTS

Strong endothermic peaks ascribed to thermal decomposition and rearrangement of amide main chains were observed at around 530℃ and 440℃ in the PPTA and Nomex fibers, respectively. Other weak exothermic peaks related to the formation of aromatic planar molecules were also observed at around 700 C in both PPTA and Nomex fibers. The starting temperature for the decomposition of amide bondings in PPTA fiber was found higher by 90℃ than that of Nomex.

Table 1 and **Fig.1** show the change of weight in the carbonization of PPTA fibers in various heating conditions. Kevlar fiber showed 21.7wt% and 37.1wt% of carbon yields after heat treatment of 1000℃ in a one staged carbonization process with a heating rate of 1℃/min and 10℃/min, respectively. A three staged carbonization of PPTA fibers surprisingly increased carbon yield up to 60 wt%(T-500). Soaking temperature was most influential to carbon yield. Compared to the one stage carbonization of Nomex fiber, the carbon yield increased only 9% after using the three staged carbonization, a results indicating that the three stage step carbonization was more effective in PPTA fiber.

Table 2 shows .atomic compositions and ratios of carbonized PPTA and Nomex fibers. Carbonized PPTA fiber through three staged carbonization showed very similar atomic compositions and ratios except for a little more oxygen contents compared to that through one staged carbonization. Carbonized Nomex fiber showed very different atomic compositions and ratios, showing more hydrogen, nitrogen, and much more oxygen contents than carbonized PPTA.

4. CONCLUSION

Carbon yield after 1000℃ heat treatment of PPTA fibers surprisingly increased up to 60wt%, exhibiting more than 20wt% increase than conventional one step carbonization. The atomic composition of carbonized PPTA fibers appeared very similar regardless of carbonization method. Carbonized Nomex fiber contained very different atomic compositions and ratios than the carbonized PPTA fibers. The Nomex fiber contained more hydrogen, nitrogen, and much more oxygen, a result suggesting different types of carbonization chemistry involved in these aramids.

References

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2. E. Fitzer, D. Kompalik, and M. Kunz, Proc.Int. Sym. Carb., (Baden-Baden, 1984) p. 847.
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Table 1 Heat treatment conditions and carbon yield of PPTA and Nomex fibers

Code	1st stage HTT S.Temp.*1 (°C)	1st stage HTT S. Time*2 (min)	2nd stage HTT S.Temp.*1 (°C)	2nd stage HTT S. Time*2 (min)	Final stage HTT S.Temp.*1 (°C)	Final stage HTT S. Time*2 (min)	Carbon yield(wt%)
A-1.0	One step carbonization with a heating rate of 1.0 °C/min						21.7
A-10	One step carbonization with a heating rate of 10 °C/min						37.1
T-480	480	100	700	60	1000	10	38.1
T-490	490	100	700	60	1000	10	44.2
T-500	500	100	700	60	1000	10	60.0
T-510	510	100	700	60	1000	10	41.3
T-60	500	80	700	60	1000	10	51.6
T-150	500	120	700	60	1000	10	58.3
T-200	500	200	700	60	1000	10	54.2
T-700	500	100	700	100	1000	10	58.8
T-520	520	100	700	60	1000	10	27.2
N-10	One step carbonization with a heating rate of 10 °C/min						39.1
NT-380	380	100	700	60	1000	10	48.2

*1: Soaking temperature, *2: Soaking time
*A series: One step carbonization of PPTA, T series: Three step carbonization of PPTA, N series: Carbonization of Nomex.

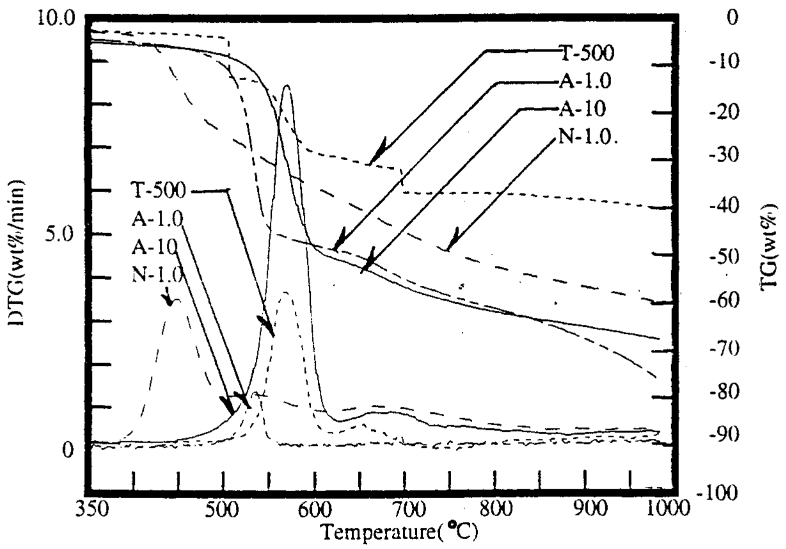

Fig.1 TG/DTG thermo-profile of PPTA and Nomex fibers

Table 2 Results of elemental analysis and carbon yield of heat treated fibers

	Carbonization conditions	C	H	N	Odiff	H/C	H/N	H/O	N/C	O/N	Carbon yield(wt%)
		Atomic contents(wt%)				Atomic ratios					
PPTA	No Heating(Dried)	70.60	4.10	11.80	13.10	0.71	4.48	5.13	0.16	0.88	-
	500℃, No soaking	70.20	3.82	11.40	14.58	0.65	4.69	4.19	0.14	1.12	96.5
	500℃, Soaking(100min)	70.34	3.55	11.20	14.91	0.61	4.44	3.81	0.14	1.17	88.4
	700℃, No soaking	78.23	2.12	8.78	10.87	0.33	3.38	3.12	0.098	1.08	51.9
	700℃, Soaking(70min)	78.28	1.92	8.45	11.35	0.29	3.18	2.71	0.091	1.17	63.6
	1000℃, No soaking	84.55	1.12	4.42	9.91	0.16	3.55	1.81	0.045	1.96	37.0
	1000℃, Soaking(10min)	84.21	1.14	4.21	10.44	0.16	3.79	1.75	0.042	1.96	60.0
Nomex	1000℃, No soaking	75.64	1.75	5.13	17.48	0.28	4.78	1.61	0.059	2.97	39.1