CARBON FIBER FROM SOLVENT REFINED COAL

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1. Introduction

Pitch based carbon fiber was important one of carbon fiber family. According to mechanical properties pitch-based carbon fiber can be divided into two type: high performance carbon fiber (HPCF) and general performance carbon fiber (GPCF). Due to high modulus, high tensile strength and other excellent performance, mesophase pitch-based carbon fibers become one important engineering materials[1-3], and wide used in aerospace and air space field。The advantage of GPCF is low price and had been wide used in the field of: 1) Absorb materials: used for water and air purified, environmental protect product, etc. 2）abrasion materials: brake for automobile, etc. 3）Reinforcement: be used as reinforcement of plastic, rubber and cerement, etc. 4）Seal materials: high temperature seal gasket and packing set. 5）heat insulation materials: high temperature furnace for semiconductor industry and graphite purify industry. 6）antistatic materials. The study of GPCF was started in 1960s，but the research of to low the price, improve the properties are still going on。In this paper, the manufacture of carbon fibers from Solvent Refine Coal (SRC）were discussed.

2、Experiment

2.1 Raw materials and equipment.

Raw materials：

1）Solvent Refine Coal (SRC）：Soft point: 175 C.

2）Modified agent；

Equipment：

1）2LAutoclave；

2）Spinning equpment；

3）Infused and carbonization equipment.

2.2 Test methods

1）Soft point and spin ability：Soft point test equipment.

2）FT-IR：NICOLET 60.

3）Element analysis：Flash EA 1112;

3）Thermal analysis：TG of GE Corporation.
4) Mechanical properties test：Instron 1211

3、Results and discussion

3.1 Study on the thermal modified of SRC

Fig.1 is FT－IR spectrum of SRC, It show that there lots of condensed aromatics structure, and also there many methyl and methane structure. The elemental analysis results of SRC and spinnable petroleum pitch show in table 1. From table 1, it could be seen that carbon content of SCR is lower than that of petroleum pitch, on the other hand, the hydrogen and oxygen content of SRC is higher. Those results due to two reason: one is effect of remainder solvent of SRC process, the other is that the lower molecular proportion of SCR is higher than that of petroleum pitch.

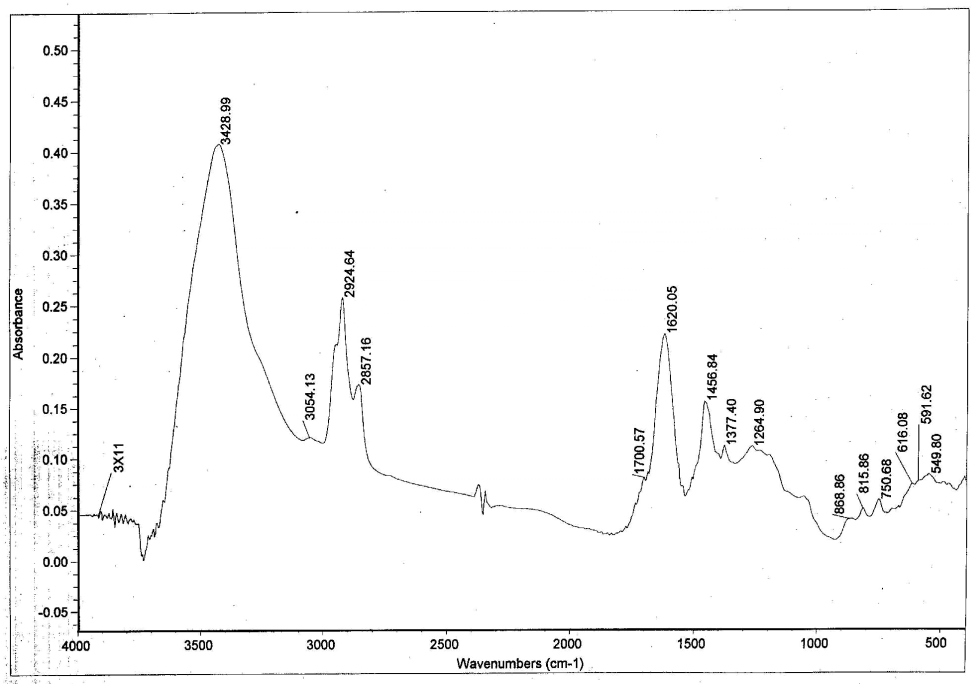


Fig. 1 FT-IR spectrum of SRC

Table 1　element content of SRC and Petroleumpitch

Samples	C,%	H,%	O,%
SRC	81.4	6.6	7.8
Petroleum isotropic pitch	94.3	5.2	0.5
Petroleum mesophase pitch	94.8	4.5	0.4

Test result show the soft point of SRC is 175 C and during test process, when temperature higher than 170 C, SCR give off volatile vapor, and the amount of vapor

increasing with the increasing of temperature, that due to the solvent in SRC, the evaporation and decompose of low molecular proportion. The instability molecular in SRC make it spinning unstable. In order to manufacture carbon fiber, modified treatment is necessary.

TG spectrum in Fig.2 show, the weight lose start around 80 C, that coursed by evaporation of solvent remain in SRC, when temperature reach 260 C, the weight lose about 10%, those part include some of low molecular weight portion and unstable methyl and methane structure decomposed. From 260 C to 310 C, SRC weight loses decreased, that due to the evaporation of solvent finished. Start from 310 C, the weight lose of SRC obviously with the increasing of temperature, that due to large amount of methyl and methane structure decomposed, and the molecular rearrangement to form condensed aromatics structure. The weight lose at 500 C is about 47%. The TG spectrum also give the conclusion of SRC could not spinning directly, and the modified treatment is needed.

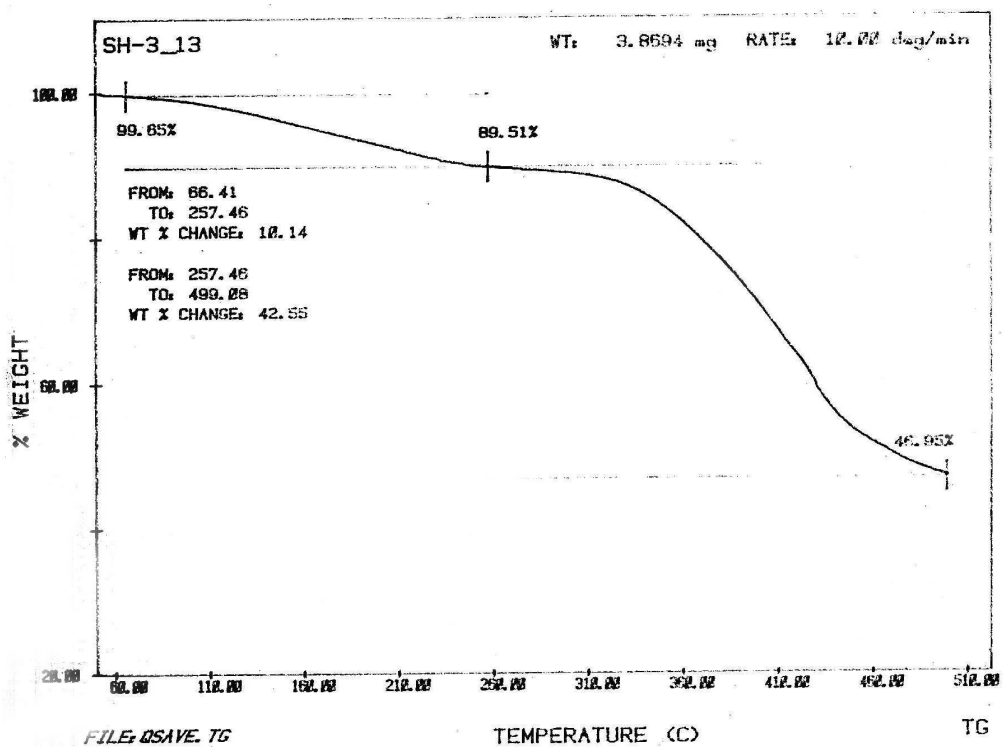


Fig. 2 TG spectrum of SRC

3.1.1 The effect of thermal modified condition

After thermal modification of SRC, isotropic pitches were obtained and list in table 2. With the increasing of treatment temperature, the soft point of modified pitch increased, and in the same time, the yield decreased, and the spinnability of higher modified temperature decreased. Thermal treatment of SRC is a process of radical reaction, and

with the increasing of treatment, the amount and activity of radical increased and condensation degree increased, so the molecular weight of resulted product become higher, and soft point increased, viscosity increased, spinniability decreased.

Table 2 the thermal treatment result of SRC

sample	Treatment condition		Soft point (C)	Yield (%)	Spinnability
	Temperature (C)	Time (hr)			
SRC-G1	320	3	234	70.0	Good
SRC-G2	330	3	239	68.0	Good
SRC-G3	360	3	267	64.4	Bad

3.1.2 Spinning of pitch

The spinnability of three isotropic pitches in above table were studied. The results show that the spinnability of SRC-G3 was poor and could not obtain fibers, SRC-G2 could spin into fiber at high temperature, but the continuous time is short, SRC-G1 with lowest SP. Could spin at lower temperature (290~310 C) and higher pressure (0.5-1MPa), the fiber could continues 1000 meters.

3.1.3 Oxidization and carbonization treatment

Pitch fiber from SRC － G1 were oxidization at different temperature and carbonization at 1000□, carbon fibers were obtained. The carbon fibers properties of different oxidization temperature show in table 3. The oxidization temperature effect carbon fibers properties obviously, and 280□ was optimum temperature, and highest tensile strength of carbon fiber were obtained at this oxidization temperature. The results were different with petroleum pitch, in general, the optimum oxidization temperature of petroleum isotropic pitch was higher than 300□, generally was 340□, and the optimum oxidization temperature of petroleum mesophase pitch was around 280□ [4]. Those results illuminate that SRC pitch has higher oxidization activity than petroleum isotropic pitch and can finished oxidization at lower temperature, that will be propitious to industrialization.

Table 3 Properties of carbon fibers from SRC pitch

Samples	Oxidization temperature □	Diameter μ	Tensile strength (MPa)	Tensile Modulus (GPa)	Elongation %
SRC-CF1	350	25	312	32.4	0.95
SRC-CF3	330	20	300	29.2	1.04
SRC-CF3	280	25	346	28.2	1.24
SRC-CF4	260	31	186	21.1	1.06

The carbon fibers' properties in table 2 were very low, and diameter were large, could not fulfill the demand of practicality, so it need improvement.

3.2 Study on SRC hydrogenization

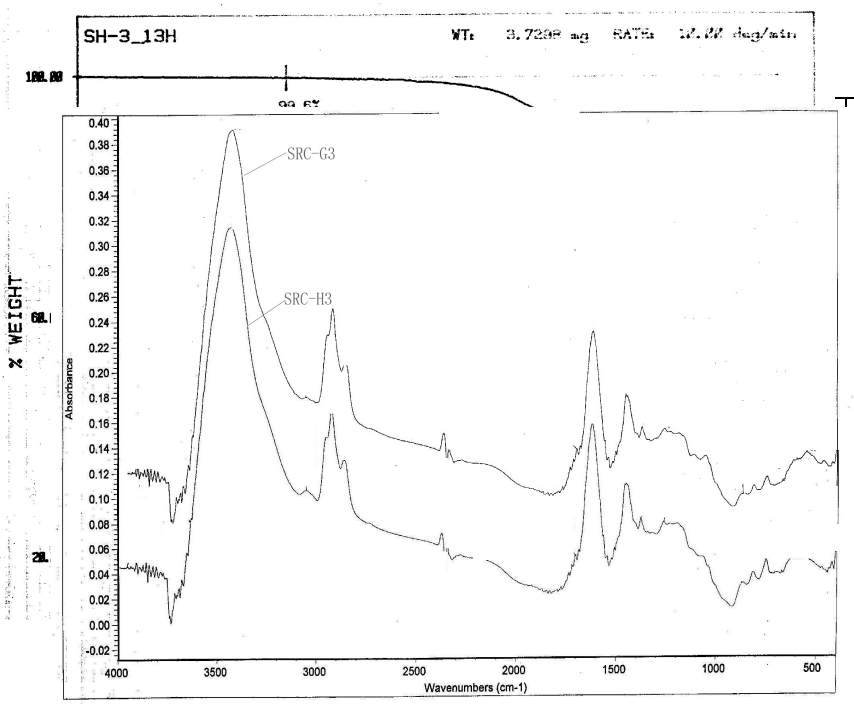
3.2.1 Study on the SRC hydrogenization condition

In order to improve the spinnability of SRC pitch and improve the mechanical properties of SRC pitch based carbon fibers, hydrogenization treatment was applied. Hydrogenization agent was used to co-carbonization with SRC, and improved spinnability of SRC pitch. The hydrogenization results list in table 4. After co-carbonization reaction, the hydrogen of hydrogenization agent transfer to SRC and changed part of aromatic structure into naphthene structure, so spinnability of SRC pitch improved.

Table 4 SRC hydrogenization results

Samples	Reaction condition		Soft point, □	Spinnabiltiy
	Temperature, C	Time ,hr		
SRC-H1	400	3	267	Bad
SRC-H2	360	3	259	Medium
SRC-H3	330	3	230	Good
SRC-H4	300	3 220 Good □Table 4 show, that with the increasing of hydrogenization temperature, the soft piontofSRC pitch increased. Compare with thermal treatment, at same reaction temperature such as 330	T. M. (GPa)	elongation（%）

	SRC-H4	300	3 220 Good □Table 4 show, that with the increasing of hydrogenization temperature, the soft piontofSRC pitch increased. Compare with thermal treatment, at same reaction temperature such as 330 C, reaction 3 hours, the soft point of SRC-H3 is 230 C, lower than SRC-G29□. And same results also show at 360□, reaction 3 hours, SRC-H2	T. M. (GPa)	elongation（%）	



		RC pitch increased. Compare with thermal treatment, at same reaction temperature such as 330 C, reaction 3 hours, the soft point of SRC-H3 is 230 C, lower than SRC-G29□. And same results also show at 360□, reaction 3 hours,		

		nd carboniz ation□□ □□table 5 Propertie s of carbon fibers from SRC-H3 □Sample s Oxidizati on temperat ure （□） diameter （um) T. S.□(MPa) T. M. (GPa) elongatio n（%） SRC-CF 1 260 8.3 740 49 1.5 SRC-CF 3 280 7.8 780 47 1.6 SRC-CF 3		

SRC-CF1	260	8.3	740	49	1.5
SRC-CF2	280	8.8	688	47	1.6
SRC-CF3	330	8.8	683	44	1.6

Results in table 5 show that the optimum oxidization temperature of SRC-H3 is 280 C, that same with petroleum mesophase pitch, that indicate SRC has high react activity. Compare with the results in table 3, the diameter of fiber from SRC-H3 much less than that of SRC-G2, and mechanical properties are much higher. Those results indicate that the effect of hydrogenization were obviously.

4. Conclusion

In order to manufacture carbon fibers from SRC, hydrogenization is necessary. SRC react with co-carbonization agent, isotropic pith with good spinnability were obtained. Diameter of pitch fibers of hydrogenization SRC were less than that of thermal modified SRC, and mechanical properties of carbon fibers from hydrogenization SRC were two times than that of thermal modified SRC.

Reference:

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