STUDY ON MESOPHASE PITCH FOR C/C COMPOSITE'S MATRIX

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1. Introduction

A C/C composite was one important member in the family of composites materials. It has many outstanding properties, such as low density, high specific (tensile) strength, high specific modules, high temperature resistance, excellent thermo shock behavior, high thermal and electric conductivity and good wear-resistant ability. C/C composites have been applicant in wide area[1-4]. Manufacture aircraft brakes is one of its important application. Although C/C composites for aircraft brakes have been industrialized, complicated equipment, long production cycle, and high production expense limit their extensive uses.

In this paper, the manufacture and properties of mesophase pitch was studied. The aim is to use mesophase pitch as matrix of C/C to increasing the C/C composite properties and lowering the cycle time and cut down the cost.

2. Experimental

2.1. Mesophase Pitch manufacture process.

2.1. Raw Material

Petroleum residue was chosen as raw material for manufacture of mesophase pitch.

2.2. Thermo-Condensation Process and Technological Parameter.

Two stage thermo-condensation method was used to manufacture mesophase pitch.

2.3 Properties test

The properties test of mesophase pitch include: Mesophase content, softening point, rheological properties, viscosity, DSC, TG, IR, element analysis, Ash content.

3. Results and discussion

3.1. The effecting of condensation temperature to mesophase pitch properties.

Condensation temperature has great effecting to the properties of mesophase pitch. Table 1 show the mesophase pitch which were made under different temperature.

Tab. 1 Effecting of condensation temperature to properties of mesophase pitch

Sample	Condensation Condition			S.P. (℃)	AC, %	Element Content		
	Temperature ℃	Vacuum Degree(Torr)	Time			C,%	H,%	H/C
DCC-MP -1	407	40	6	283	100	95.2	4.5	0.47
DCC-MP -2	405	40	6	280	100	94.8	4.5	0.57
DCC-MP -3	400	40	6	268	98	95.9	4.2	0.52
DCC-MP -4	395	40	6	260	60	95.2	4.7	0.59
DCC-MP -5	392	40		251	50	95.2	4.8	0.61

According to above date, following Figures were given:

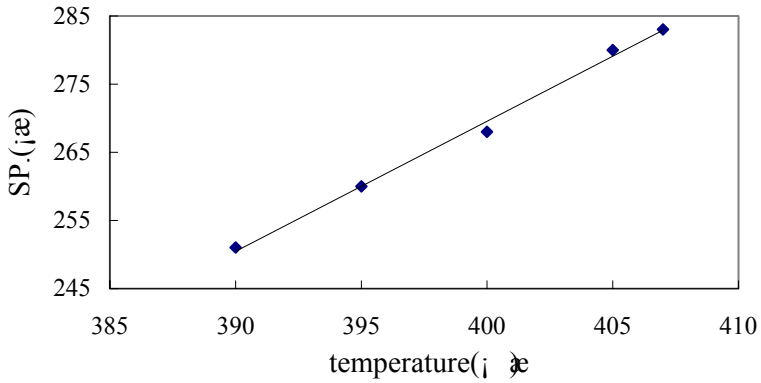

Fig.1 Effecting of Condensation Temperature to Softening Point

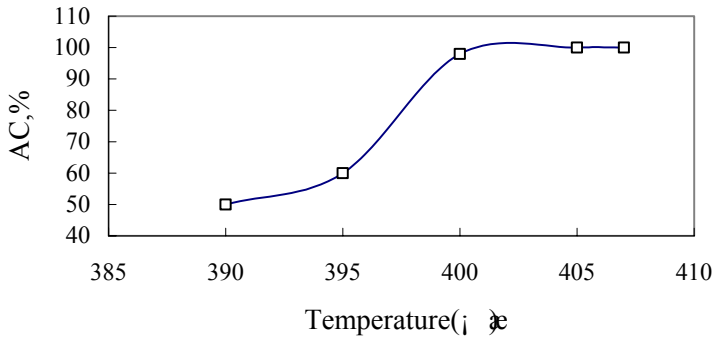

Fig. 2 Effecting of condensation Temperature to AC

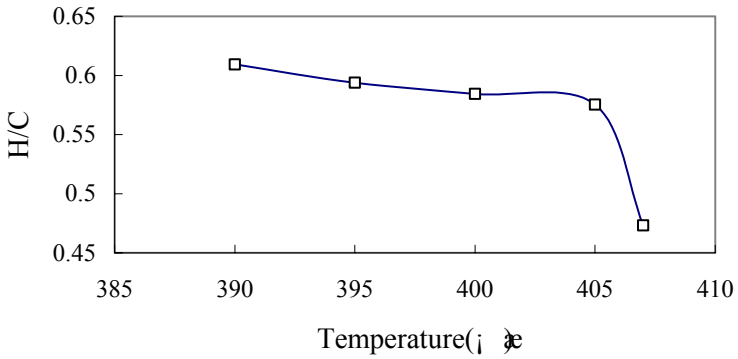

Fig. 3 Effecting of Condensation Temperature to H/C of mesophase Pitch

Tab.1 and Fig 1 to 3 show that with the increasing of condensation temperature the softening point and AC increased, while H/C decreased. The results due to dehydregen and condensation reaction more completely, the molecule weight and aromacity are higher at higher condensation temperature. During condensation reaction, mesophase formed. It was single-sphere at beginning, with the increasing of temperature, the single-sphere collide and coalesce into complex-sphere, at the end, complex-sphere disintegrate into coarse fibrous structure continuos phase.(see Fig.4)

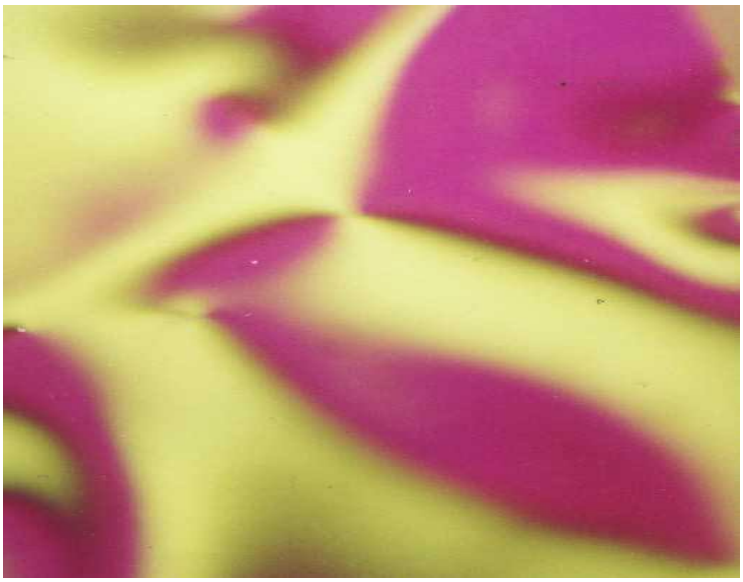

Fig. 4 Mesophase pitch polarize coarse fibrous structure

3.2. The effecting of vacuum degree to the properties of mesophase pitch.

Vacuum degree is one of the important technological parameter of thermo-condensation. Table 2 show the results of the effecting of vacuum degree to the properties of mesophase pitch.

Tab. 2 The effecting of vacuum degree to the properties of mesophase pitch

Sample	Condensation Condition			S.P. (℃)	AC, %	Element Content		
	Temperature ℃	Vacuum Degree(Torr)	Time			C,%	H,%	H/C
DCC-MP -3	400	40	6	268	98	95.9	4.2	0.52
DCC-MP -6	400	300	6	262	90	95.3	4.4	0.56
DCC-MP -7	400	500	6	260	85	95.2	4.6	0.58
DCC-MP -8	400	760	6	162	5	93.9	6.1	0.64

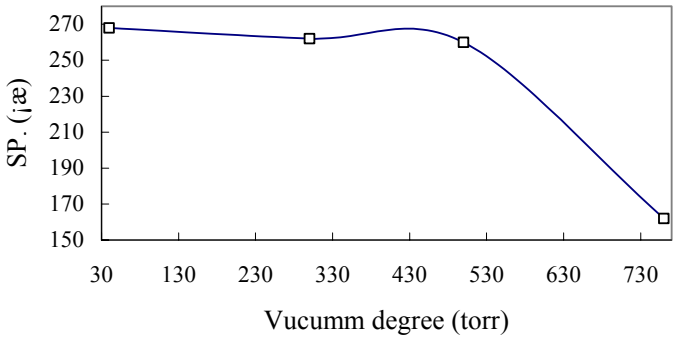

Fig. 5 Vacuum degree to softening point

Table 2 and Fig. 5 show that with the increasing of vacuum degree the softening point and AC increased, while H/C decreased. Under higher vacuum degree, the small molecule which had not been condensed during reaction process was excluded more completely and condensation degree of mesophase pitch was higher, so the molecule weight and aromacity were higher, the softening point and AC were higher, while the H/C was lower.

3.3. The relationship between softening point and carbon yield

Carbon yield of mesophase pitch was tested by tube furnace under protect of high pure nitrogen with temperature increasing rate 10□/min. The tested results show in table 3

Tab. 3 Carbon Yield of Different Mesophase Pitch

Sample	SP.(□)	AC, %	Carbon Yield, %
DCC-MP-1	283	100	89.61
DCC-MP-2	280	100	88.55
DCC-MP-3	268	98	87.47
DCC-MP-4	260	60	86.63
DCC-MP-5	251	50	86.30
DCC-MP-8	162	<5	62.28

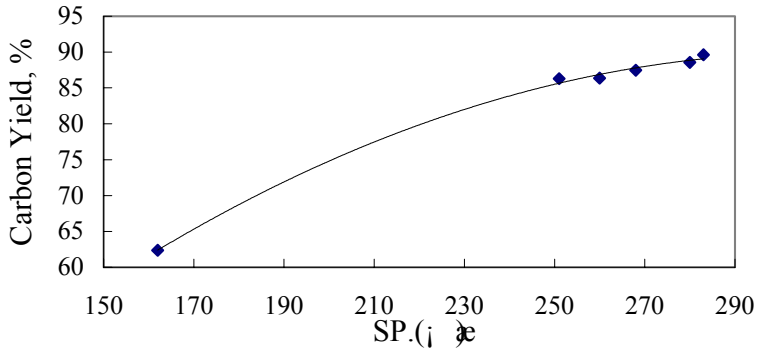

Fig. 6 Softening point of mesophase pitch to carbon yield

Table 3 and Fig. 6 show that with the increasing of softening point the carbon yield increased. The higher of softening point, the higher of molecule weight and aromaticity, so the carbon yield was higher.

3.4. The properties test of mesophase pitch

As matrix of C/C composites, it must has following properties: good rheology, high thermo-stability, high AC, high carbon yield and low ash content. Rheology is the most important character, it will effect the molding process and the C/C composites' properties. High carbon yield, high AC content and low ash content will give the C/C composites high mechanical properties. High carbon yield also can reduce the cycle times in the dentification process. According to above principle, DCC-MP-3 which with AC 98%, carbon yield 87.47%, and good rehological property was selected to do deeply research.

3.4.1. The consist and structure of mesophase pitch

The properties of DCC-MP-3 was show in Tab. 4

Tab. 4 properties of DCC-MP-3

Sample	SP □	AC %	C %	H %	H/C	QI %	carbon yield %	Ash %
DCC-MP-3	268	98	94.965	4.625	0.5844	22.96	87.47	0.0198

Table 4 shows that mesphase pitch (DCC-MP-3) consist mainly by carbon and hydrogen, carbon content about 95%, the ash content only 0.0198%, the QI content is about 23%.

Fig.7 was the FT-IR spectrum of DCC-MP-3, it shows that mesophase pitch DCC-MP-3 consist mainly by dense circle aromatic hydrocarbon and small part of linear hydrocarbon.

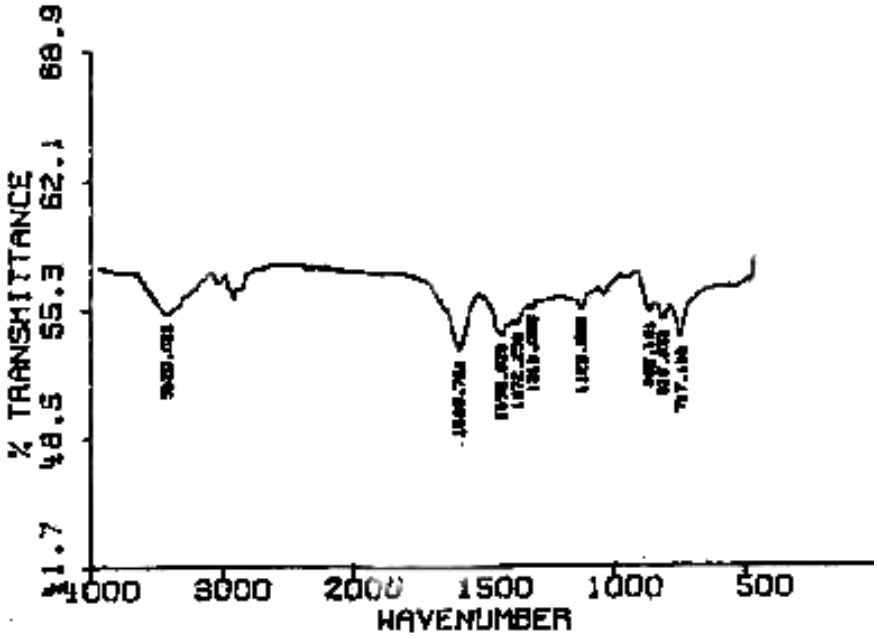

Fig. 7 FT-IR spectrum of DCC-MP-3

3.4.2 Rheological property and viscosity of mesophase pitch

Rheological property and viscosity are very important character of mesophase pitch. They effect the molding process and C/C composite properties. Fig. 8 shows the rheological property of mesophase pitch. With the increasing of temperature the viscosity of mesophase pitch decreased, and increasing shear rate has the same result as increasing temperature. At lower shear ratio, temperature effect viscosity obviously, but at higher shear rate, the temperature does not effect the viscosity obviously.

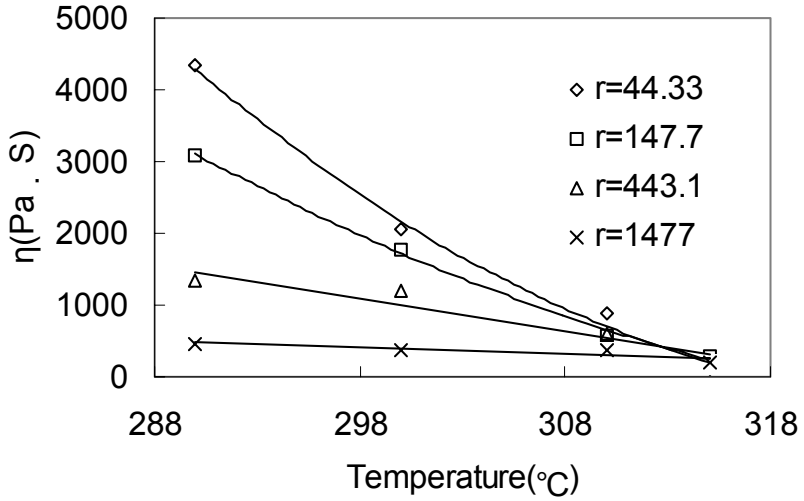

Fig.8 Rheological property curve of DCC-MP-3

The relationship of viscosity to temperature of mesophase determine the C/C composite molding process. Fig. 9 shows that mesophase pitch DCC-MP-3 was a kind of non-thixotropic fluid. With the increasing of the temperature the viscosity of mesophase pitch decreased. When temperature is higher than 450□ the increasing of

viscosity is due to condensation reaction occurred, the structure of mesophase was changed.

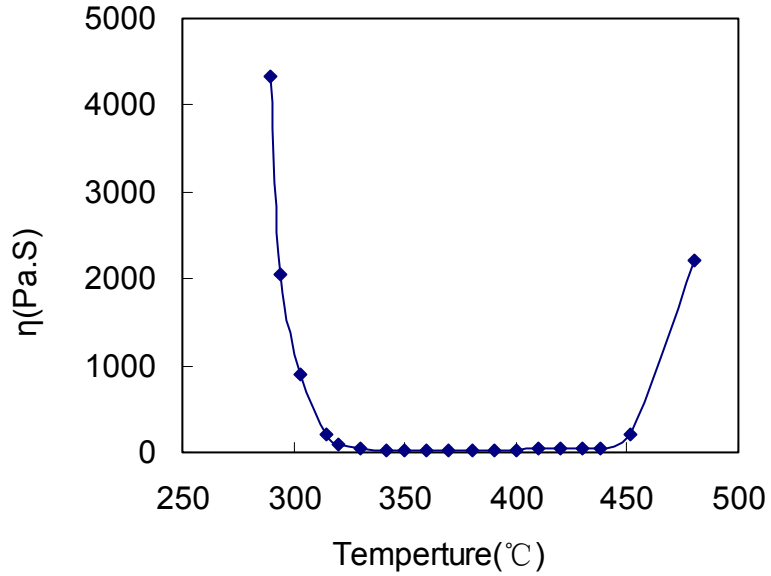

Fig. 8 Viscosity-Temperature curve of Mesophase Pitch DCC-MP-3

4. CONCLUSIONS

Use petroleum residual as raw material, by two stage-thermo-condensation method, mesophase pitch with SP. 268 C and AC 98% was obtained. This pitch has good rehological property, high carbon yield (87.47%) and high thermal stability. The mesophase pitch is a good matrix raw material for C/C composites.

Reference:

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[3] Savage G. Carbon-Carbon Composites, Chapman and Hall, NY (1993).

[4].Fitzer E, LM. Manocha LM, Carbon Reinforcements and Carbon/ Carbon Composites, Springer, Berlin, 1998.