A Tube Bomb as a Model for a Delayed Coker

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The carbonization of a petroleum residue using a tube bomb in a molten tin bath under pressurized and rapid heating rate conditions was examined as a model for a delayed coker. Such a carbonization provided a lump coke of which macro and micro appearance, CTE and bulk density were comparable to those of a needle coke produced in a delayed coker for the same feedstock. The influences of carbonization conditions on the coke properties were studied.

INTRODUCTION

Needle coke which is an essential precursor of the graphite electrode for electric arc smelting has been produced by a delayed coker of large scale. Qualifying characteristics of needle coke have been recognized to be strongly influenced by the nature of feedstocks and operational carbonization conditions. However, the scientific relation between them was far from the establishment. The laboratory preparation of the real needle coke similar to the commercial one is most desired for such a study, although the optical anisotropy of the coke and its development have been extensively studied to elucidate the detail mechanism.

In the present study, we report pressurized carbonization of a petroleum residue in a tube bomb heated in a molten tin bath at the rapid heating rate. The lump coke, thus prepared, allowed the measurement of characteristic properties as a needle coke.

EXPERIMENTAL

Some analytical data of a petroleum residue are summarized in Table 1. Sample of about 10 g was weighted in a glass tube (deameter: 15 mm, height: 100 mm), which was put in the tube bomb (diameter: 20 mm, height: 100 mm). The bomb equipped with two locks was flushed with nitrogen flow several times before sealing. The pressure of nitrogen in the bomb was adjusted at room temperature before the carbonization.

The sealed tube was immersed into the molten tin bath which was kept at a prescribed temperature. The sample in tube was heated to a prescribed temperature within 2 min. Along with the carbonization, the gas evolved was purged through the control value to adjust the pressure to the prescribed one until the end of the carbonization, which took ca. 3hr. The procedure was repeated by charging the feed to obtain a lump coke (higher than 10 mm).

Table 1 Some Analytical Data of Sample

Sample	SPGR	H	C	H/C	CCR	TS	Vis(cst) at 100°C
LS_1 VR	0.931	12.45	86.41	1.73	9.98	0.19	70
Maltene	0.891	13.20	86.34	1.83	2.57	0.13	25
Asphaltene	1.034	10.36	87.32	1.42	17.60	0.35	

RESULTS AND DISCUSSION

Photographs of lump cokes produced in a tube bomb at 500 °C under 15 kg/cm^2G are shown in Figure 1. The coke exhibited needle-gathering appearance, carrying pores oriented in a direction parallel to the bomb axis similar to the commercial coke.

Figure 2 shows the microphotographs of several parts in the lump coke produced under 15 kg/cm^2: their locations are

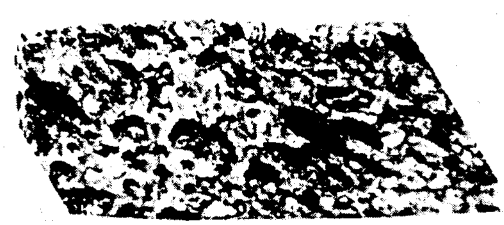

Fig.1 Coke Appearance from Tube-Bomb under pressurized condition

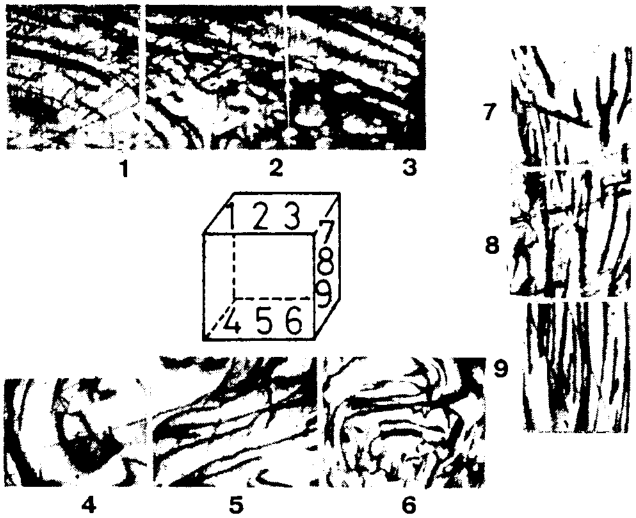

Fig.2 Optical Texture of Coke
(vertical & parallel Section)

indicated in the same figure. Horizontal sections of the coke exihibited winded flow texture: whole regions of the coke were of well developed anisotropy, although their orientation was random, varying at each location to form many discrinations.

The vertical sections (Figure 2) regardless of the location in the coke exhibited flow textures running in a direction parallel to the bomb axis.

Microphotographs of the cokes produced in the tube bomb under variable pressures are shown in Figure 3, where the cross-sections parallel to the axis except for the coke produced under atmospheric pressure were photographed. The coke produced under atmospheric pressure was flaky to be difficult to find the cross-section as often experienced. All cokes exhibited well-developed anisotropy,

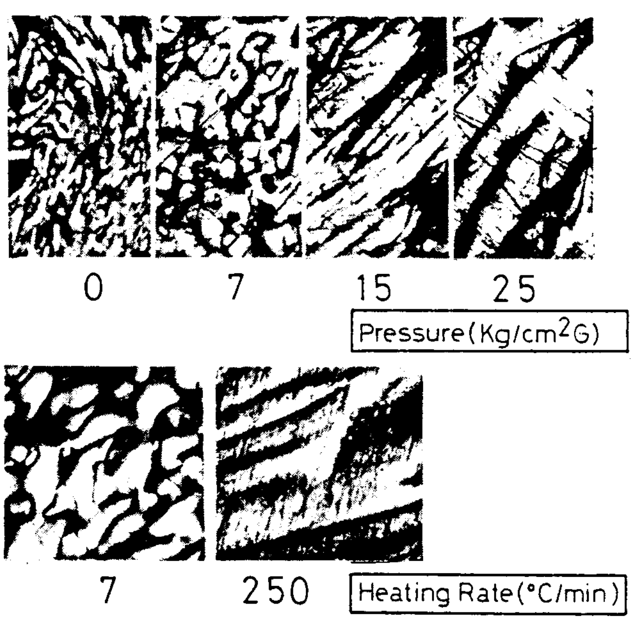

Fig.3 Optical Texture of Coke
(Several Pressure, Heating Rate

however, the orientation of flow texture was clearly improved to be of better uni-axial arrangement under higher pressure. Under atmospheric pressure the textures were winded, no uniform orientation being observable in the photograph. The pressure of 7 kg/cm^2G improved the degree of orientation a little, the flow textures being arranged roughly in one direction. Higher pressures of 15 or 25 kg/cm^2G allowed better development of flow texture in an almost perfect uni-axial orientation.

Figure 3 shows the microphotograph of the coke produced under 15 kg/cm^2G at 500 °C, where the heating rate was 7 °C/min. Being contrast to the coke produced under similar conditions except for the much higher heating rate of 250 °C/min it shows mosaic texture although the bulk density and coke yield were much the same.

Bulk density, CTE and volatile matter content of the cokes produced in the tube bomb before and after the calcination at 1000 °C are summarized in Table 2. Those

Table 2 Properties(BD,CTE,VM) of Cokes

	BD	CTE($\times 10^{-6}$)	VM(wt%)
Cokes from Delayed Coker (LS_1VR)	0.80 *1 (0.84)	10.21 (n.d.) *2	7.4
Cokes from Tube - Bomb (LS_1VR-7K)	0.87 (0.95)	17.71 (1.47)	5.5
Cokes from Tube - Bomb (LS_1VR-15K)	0.82 (1.02)	20.9 (1.51)	5.9

*1 values in parenthesis are those for the coke after calcination

*2 no expansion detected

properties are comparable to those of a commercial coke produced in a large scale coker from the same feedstock. Thus, the carbonization pressure and heating rate are very critical on the preparation of the needle coke.

DISCUSSION

The development of flow texture of high orientation has been recognized to follow the steps, appearance of anisotropic sphere, their growth and coalescence. Pressurized carbonization and the rapid heating are both favorable for the smooth progress of such steps into needle-like structure since the more volatile matters which can stay in the system until the final stage may moderate the viscosity increase of the system to allow the smooth movement of mesophase molecules into the better uni-axial-stacking.