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THERMAL ANALYSIS OF EXOTHERMIC PROCESSES IN COAL PYROLYSIS *

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ABSTRACT

Differential scanning calorimetery demonstrated the occurrence of exothermic processes associated with the production of volatile matter in or near the plastic region of the coal samples studied.

The temperature and magnitude of the exothermic peak was observed to be strongly affected by the heating rate, sample mass and, to a lesser extent, by sample particle size. Thermal properties also were found to be influenced by oxidation or weathering effects.

INTRODUCTION

Research is under way to develop screw extrusion as a means of continuous injection of coal into high-pressure systems [1—4]. Coal is extrudable only when it reaches a temperature which is associated with the onset of its plastic state in which it can be handled as a viscous fluid. This temperature varies from about 390 to 490°C for most coals [5]. Coal will not extrude below its plastic range and it forms hard coke, which is not extrudable above this temperature range.

The attainment of conditions required to extrude coal requires the development of extruder design criteria which match the thermal characteristics of the coal. The thermal properties of major interest are (1) the total enthalpy required to heat the coal to extrusion temperatures, including both specific heat effects and thermal transitions resulting from physical or chemical changes, and (2) the thermal conductivity of coal through the plastic range.

Coals, because of their mode of genesis would be expected to be resistant to thermal changes below temperatures at which pyrolitic breakdown occurs. There has been very little experimental work regarding the thermal properties of coal at temperatures below 500°C. The development of coal extrusion requires detailed information, especially in the plastic region, where small thermal transitions could affect the plastic characteristics of the coal.

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There have been few applications of differential scanning calorimetry (DSC) to the study of coal pyrolysis due to its relatively recent development. Recently, Mahajan et al. [6] reported DSC thermocurves for twelve U.S. coals of various ranks in a helium atmosphere at 5.6 MPa (gauge) and temperatures up to 580°C. Using about 20 mg of dried sample at a heating rate of 10°C min^{-1}, they concluded that the thermal effects during pyrolysis of coals ranging in rank from anthracite to HVC bituminous were endothermic. Exothermic heats were observed only in the case of sub-bituminous or lignite coals. The net thermal effect was found to be strongly rank-dependent. This work was unique in that an effort was made to correct the raw thermocurves for the effect of volatile weight loss using the results of concurrent thermogravimetric analysis (TG) of the samples tested. These corrections proved to cause a significant change in the shape of the thermocurves, in some cases leading to at least a partial reversal of the thermal effects.

Some recent DSC and TG scans were reported for samples of an HVB bituminous coal [4]. These data show low-temperature endothermic reactions at about 260 and 410°C and an exothermic reaction at 490°C. The low-temperature endotherm was interpreted as a drying curve. The exotherm was observed to be highly dependent on heating rate, being only one-sixth as large when the heating rate was lowered from 100 to 20°C min^{-1}.

Very recently, DSC and TG scans were reported for a variety of bituminous coals heated in nitrogen at various heating rates [7]. The decomposition was observed to be endothermic up to 500°C and exothermic thereafter. Quantitative repeatability of the energetic measurements was adversely affected by baseline shifts arising from condensation of decomposition products on the surfaces of the sample holder.

There is ample evidence of the existence of substantial exothermic processes above 400°C during coal pyrolysis. The temperature and magnitude of these processes, particularly in relation to any accompanying endothermic reactions are particularly dependent upon heating rate and whether the sample is heated in its own pyrolysis gases, an inert diluent, or a vacuum.

The mechanism by which the exothermic peak is produced remains unclear. If, as suggested by Van Krevelen [8], the exothermic peak results from resolidification of the plastic coal mass, the location of the peak should correlate with the resolidification temperature.

On the other hand, pyrolysis produces a substantial mass of volatile matter which is readily oxidized in the presence of very small quantities of oxygen [9]. Such reactions could well be responsible for the exothermic peak. Whatever the actual mechanism, it is clear that the exotherm occurs within, or very near, the plastic region and is associated with the production of volatile matter preceding and accompanying the softening process.

EXPERIMENTAL

A Perkin Elmer DSC-1B differential scanning calorimeter was used to heat various samples of coal in a nitrogen environment up to the maximum operating temperature of 500°C.

Coal samples were provided mainly by the Pennsylvania State University Coal Research Section. Samples were stored in an inert atmosphere until used, then sieved to provide the particle size distributions required.

The DSC experiments described here were not accompanied by a thorough thermogravimetric analysis. However, the production of volatile material was observed using the effluent analyzer of the DSC-1B. This thermal conductivity detector is particularly sensitive to contamination by condensable vapors in the effluent. The effect of contamination on detector performance is to substantially reduce its sensitivity resulting in an uncertainty regarding the temperatures at which volatiles appear.

RESULTS AND CONCLUSIONS

General characteristics of DSC thermograms of coal and coal chars

Figure 1 depicts a typical sequence of DSC scans which includes the instrument base-line and thermocurves of the fresh coal sample as well as the low-temperature char for a high volatile bituminous coal (Utah A, PSOC-238). Low-temperature char thermocurves were obtained by reheating the original sample after the initial scan to 500°C. The fresh sample and char thermocurves, normalized for base-line shift are shown in Fig. 2.

The general positive slope of the two normalized thermocurves in Fig. 2 results from a combination of sample weight loss and the temperature-dependent specific heat. Although the effects cannot be separated unless the thermocurves are corrected for weight loss, variation of the specific heat is

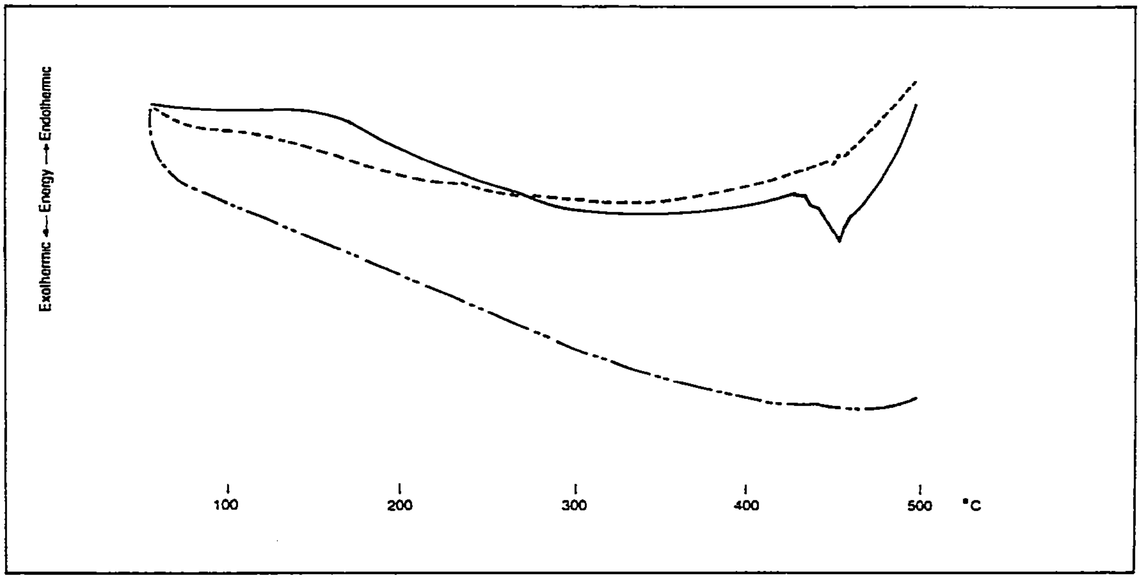

Fig. 1. Typical thermocurve for a high-volatile bituminous coal (Utah A, PSOC-238). ———, Fresh sample; ------, reheated char; —·—·—, base line. m_0 = 25.6 mg; particle size = 40—20 mesh; heating rate = 40°C min^{-1}.
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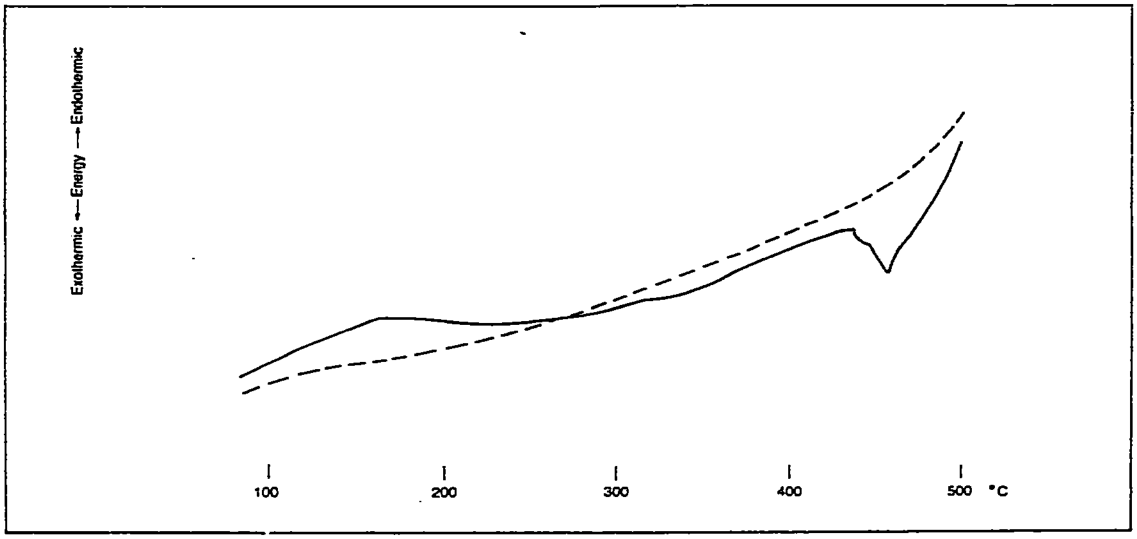

Fig. 2. Normalized thermocurve for a high-volatile bituminous coal (Utah A, PSOC-238). ———, Fresh sample; - - - - - -, reheated char. m_0 = 25.6 mg; particle size = 40—20 mesh; heating rate = 40°C min^{-1}.

the more important of the two. This conclusion follows the observation that the two thermocurves have roughly the same overall slope, while the weight loss incurred by the reheated char was less than 10% of that observed for the fresh coal sample.

Thermal effects of chemical or physical transitions are characterized by positive (endothermic) or negative (exothermic) peaks. The broad endotherm observed peaking at about 150°C can be attributed to the release of water since its amplitude correlates with the moisture content of the sample tested. The sharp exotherm commencing at about 430°C was preceded by the release of volatile organic matter observed at about 360°C and above.

Comparison of coal samples

A number of coal samples were selected for analysis. The selection was intended to be a representative one designed to highlight behavior in the plastic region. The coals chosen are listed in Table 1. The detailed characteristics of the thermocurves of the coals listed do not differ in any of the essential elements from those shown in Fig. 1.

Figure 3 depicts thermocurves of various coal samples obtained under the conditions shown. These thermocurves were obtained for purposes of comparing the behavior of the different samples, and no effort to adjust the instrument base-line was made. In the case of PSOC-238 and KY No. 9, the appearance of an endotherm following the exotherm just below 500°C is suggested. This observation is confirmed later.

A comparison of coal samples under instrumental conditions producing a relatively horizontal base-line in the temperature range of the expected exothermic peak (360—500°C) is shown in Figs. 4 and 5. The highest accessible

TABLE 1

Coal samples investigated *

No.	Sample identidication		Mine location	Rank	% Fixed C (DMMF)	% Volatiles (DMMF)	Calorific value (MMMF) (Btu lb.$^{-1}$)	% Moisture **	Softening temperature (°C)	Max. fluidity temperature (°C)	Resolidification temperature (°C)	Plastic range (°C)
	PSU No.	Seam or mine										
1	PSOC-233	Wadge	Colorado	HVC	58.20	41.80	12 591	7.54				
2	PSOC-238	Utah A	Utah	HVA	54.66	45.34	14 018	3.60				
3	PSOC-300	Pee Wee	Tennessee	HVA	61.12	38.88	14 562	2.77	416	444	465	49
4	PSOC-310	New Mexico No. 7	New Mexico	HVC	56.61	43.39	12 208	11.07				
5	PSOC-314	Blind Canyon	Utah	HVA	52.68	47.32	14 004	4.08	408	432	456	48
6	PSOC-316	Fish Creek	Colorado	HVC	59.38	40.62	12 059	12.12				
7	PSOC-318	Upper Freeport	Pennsylvania	Low. Vol.	80.70	19.30	15 278	2.93	471	498	508	37
8	PSOC-336	Upper Freeport	Pennsylvania	Med. Vol.	74.12	25.88	14 847	6.13	415	462	493	78
9	PSOC-399	Fort Scott	Oklahoma	HVA	53.05	46.95	14 492	3.99	378	450	479	101
10	PSOC-414	Darco Lignite	Texas	Subbit C	50.53	49.47	8446	31.05				
11		Orient No. 3 Ill. No. 6	Illinois	HVC	55.9 *	36.8 *	10 500–11 500	1.4				
12		Kentucky No. 9	Kentucky	HVB								

* All data (except for samples 11 and 12) were provided by the Pennsylvania State University Coal Research Section. Data for samples 11 and 12 were provided by Dr. C. England, Jet Propulsion Laboratory, Pasadena, CA.

** As received.

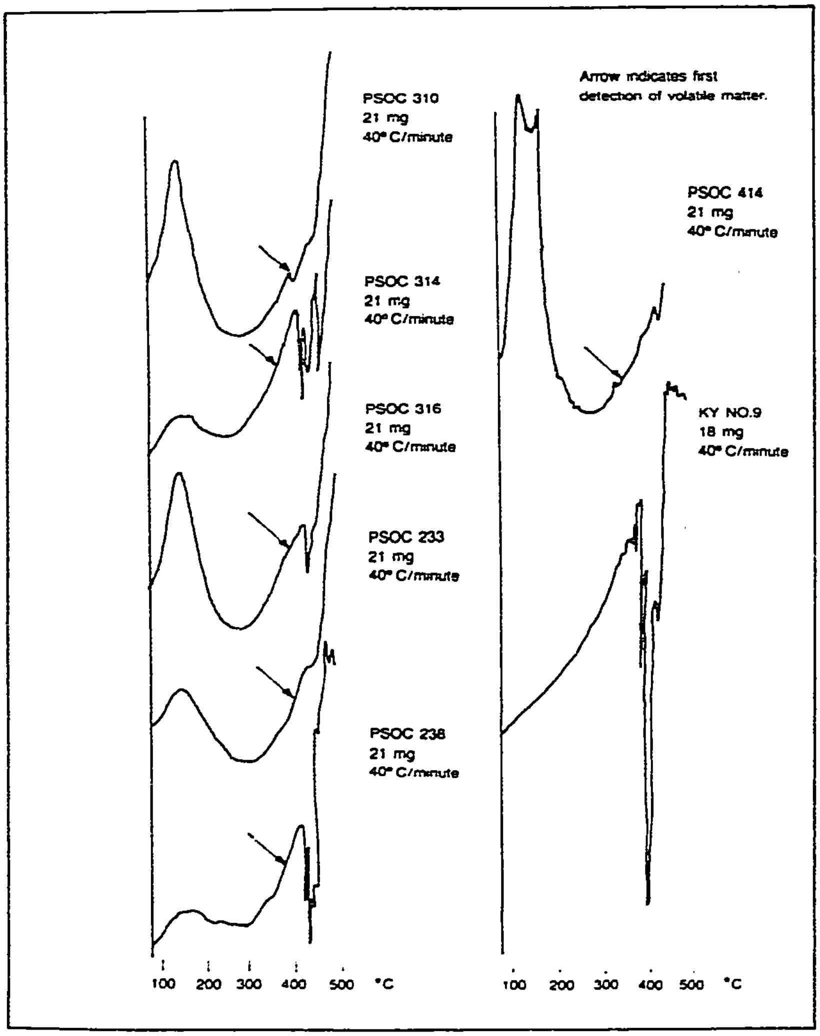

Fig. 3. Comparison of coal samples.

heating rate (80° C min^{-1}) was used in the cases depicted in Fig. 4. The resulting exotherms are strikingly apparent. The onset of the exothermic process is characterized in these thermocurves by a (relatively) sharp change of slope which occurred in the temperature range 415—475°C.

There appears to be no correlation between rank and the magnitude or location of the exotherm. However, when the available data are superimposed on the thermocurve, the plastic range is seen to very nearly coincide with the exothermic peak. Although this observation is certainly suggestive, the effect of heating rate on both the plastic range and the location of the exotherm is not accounted for. Plastic range data were obtained from the PSU Coal Data Base which does not specify the heating rate used. The ASTM Standard Method of Test D1812-69 specifies a heating rate of 3° C min^{-1} which is the probable condition for the reported data. In any event, it is unlikely that the heating rate exceeded 10° C min^{-1} for the reported data, and

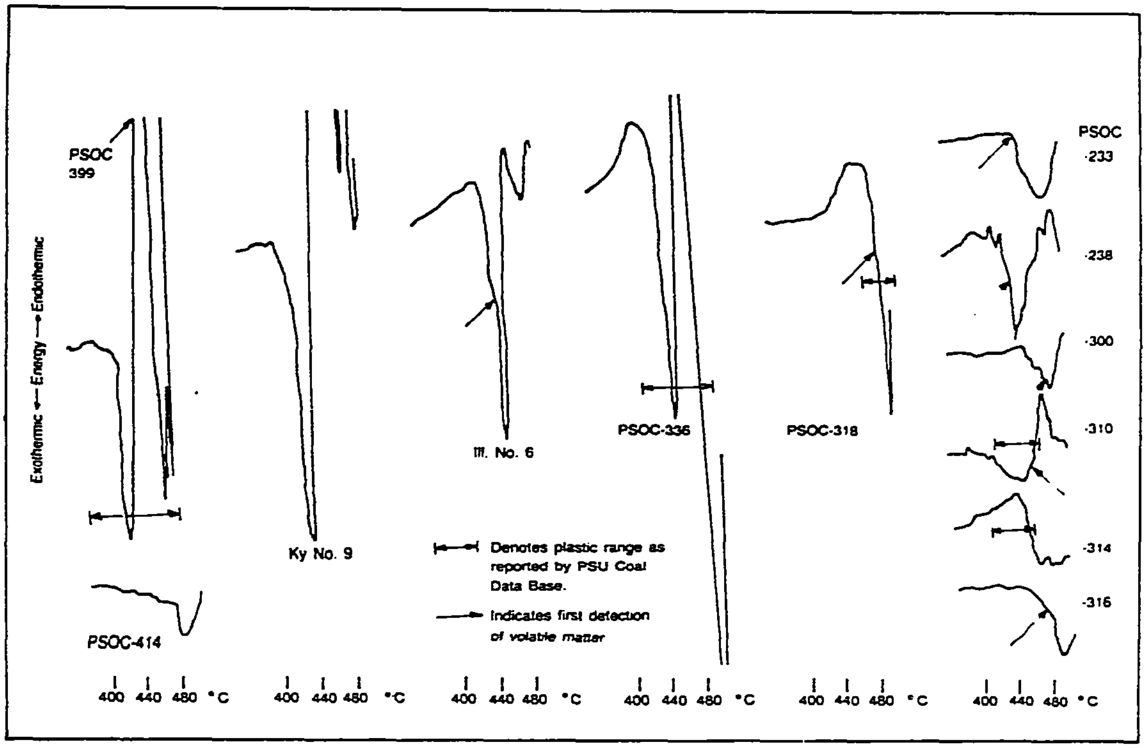

Fig. 4. Comparison of coal samples. $m_0 = 20$ mg; heating rate = 80°C min^{-1}; particle size = 50—40 mesh.

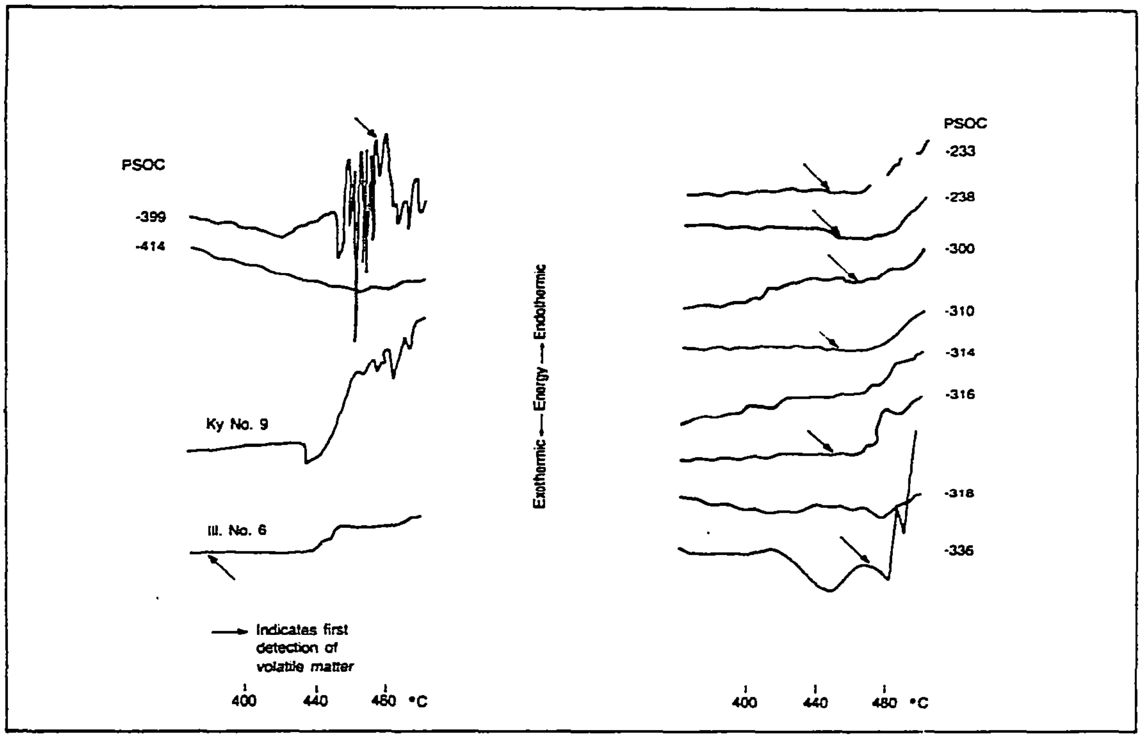

Fig. 5. Comparison of coal samples. $m_0 = 10$ mg; heating rate = 40°C min^{-1}; particle size = 50—40 mesh.

the plastic properties would be expected to differ markedly at 80° C min^{-1}. The very substantial influence of heating rate on the thermal transitions of coal will be described shortly.

In several of the cases depicted in Fig. 4 (Ky No. 9, PSOC-238, and PSOC-310), an endotherm is observed immediately following the exotherm. It is apparent that this endotherm would have appeared in the remaining thermocurves had the heating proceeded beyond the instrument limit of 500° C.

Figure 4 depicts two cases (PSOC-336 and PSOC-399) in which the exotherm appears to be interrupted by a sharp endotherm which gives way in turn to another exotherm. This type of behavior was observed periodically in the thermocurves of samples exhibiting strong exotherms from a combination of the effect of temperature gradients within the sample, inhomogeneity of the sample, and splattering of material out of the sample holder. (See also the PSOC-399 thermocurve in Fig. 5.)

Finally, several of the thermocurves (PSOC-336, PSOC-318, and others) indicate that the exotherm is preceded by the development of a relatively broad endotherm.

These observations are in agreement with those of Glass [10] that the exotherm itself is superimposed on a volatile matter endotherm, which commences at a somewhat lower temperature. As pointed out earlier, the actual temperature at which volatile matter was first observed in these experiments is subject to some uncertainty because of contamination of the thermal conductivity detector.

Effect of system operating parameters

Coal samples were placed in sample pans without compaction and covered with a loosely fitting sample pan holder. This arrangement was chosen to prevent splattering of the sample during heating while permitting the free escape of volatile matter into the blanketing inert gas stream.

Nitrogen gas was fed into the sample pan holder at the highest rate possible without disturbing the sample.

The remaining operational parameters investigated were heating rate, particle size distribution, and sample size. Table 2 outlines the experimental matrix performed to illustrate the effects of these parameters. Illinois No. 6 (HVC) was selected for this experiment.

The effect of heating rate on the magnitude and location of the exothermic peak is shown in Figs. 6 and 7 for 30—20 mesh and 100—80 mesh samples, respectively. These thermocurves clearly show the increased magnitude of the exothermic peak with increasing heating rate. The effect of particle size is also demonstrated by a comparison of the two figures. This effect will be discussed shortly; however, it is noteworthy to observe in Fig. 7 that the exothermic reaction which occurred when the sample was heated at 80° C min^{-1} is not present in the thermocurve of the sample heated at 10° C min^{-1}.

The thermocurves obtained at 20 and 10° C min^{-1} for 30—20 mesh coal, depicted in Fig. 6, exhibit erratic behavior caused by the apparent masking of competing exothermic and endothermic peaks. In view of the earlier explanation of this behavior observed in Figs. 4 and 5, it seems surprising to

TABLE 2

Experimental matrix

Particle size (mesh)	20 mg sample				10 mg sample			
	80 °C min^{-1}	40 °C min^{-1}	20 °C min^{-1}	10 °C min^{-1}	80 °C min^{-1}	40 °C min^{-1}	20 °C min^{-1}	10 °C min^{-1}
20— 10	X	X	X	X	X	X	X	⊗
30— 20	X	X	X	X	X	X	⊗	⊗
40— 30	X	X	X	X	X	X	X	⊗
50— 40	X	X	X	X	X	⊗	⊗	
60— 50	X	X	X	⊗	X	X	X	
80— 60	X	X	X	⊗	X	⊗	⊗	
100— 80	X	X	X		X	⊗	⊗	
—100	X	X	X		X	⊗		

Note: where succeeding runs show no thermal reaction, as indicated by the symbol ⊗, subsequent runs in the sequence were omitted.

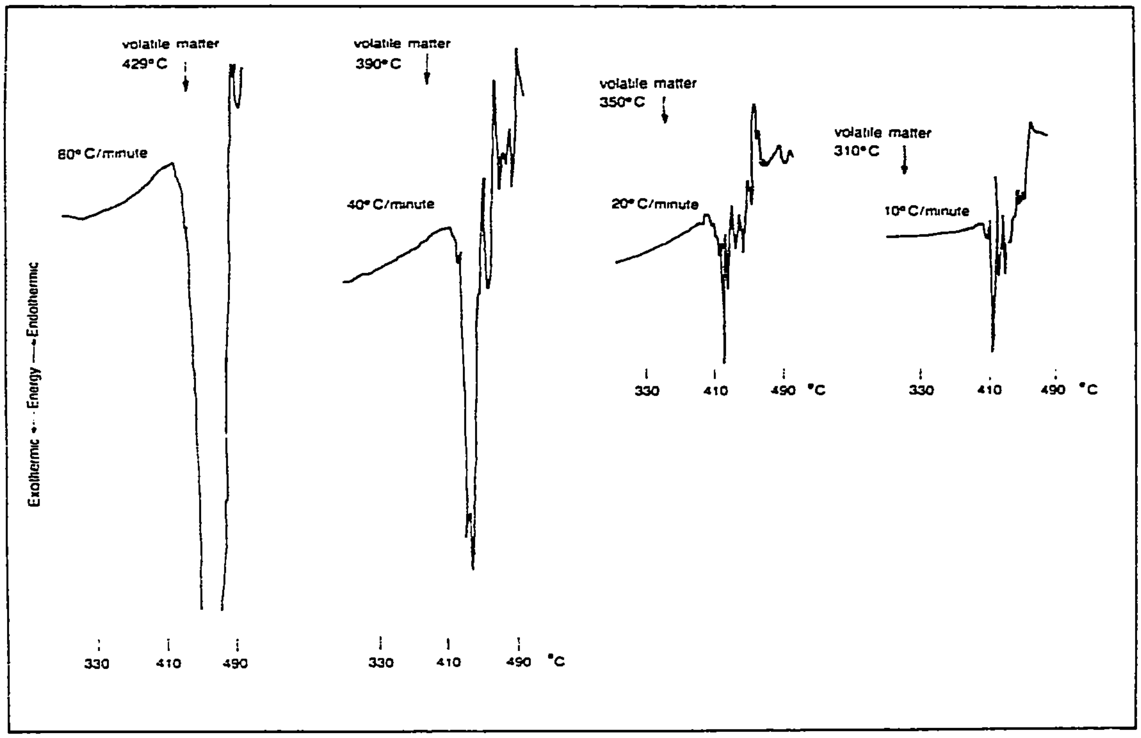

Fig. 6. Effect of heating rate on the DSC thermocurve of Ill. No. 6 (HVC). Particle size = 30—20 mesh; $m_0 = 20$ mg.

note its absence at the higher heating rates. It is probably, however, that the effect was simply overwhelmed by the exothermic peak which was very substantial at the higher heating rates.

Figures 6 and 7 also reveal the effect of heating rate on the temperature at which the exothermic peak began and the temperature at which volatiles were first detected. The volatilization temperatures and the peak temperatures depicted both shift to higher values at higher heating rates. This observation is consistent with the reported behavior of the maximum devolatilization rate, temperature and magnitude of maximum plasticity, dilatation, all of which experience a shift of the respective characteristic temperatures to higher values at higher heating rates [8].

Figures 6 and 7 indicate a greater shift of the detection temperature of volatiles than the peak temperatures. In Fig. 6, for example, volatiles are shown at 310°C at a heating rate of 10°C min^{-1} and 429°C at 80°C min^{-1}. That is, volatiles were detected fully 100°C before the exotherm at 10°C min^{-1} while they were approximately coincident with the start of the exotherm at 80°C min^{-1}. The limitations of the volatile matter detector described earlier cast some doubt on the results obtained at higher heating rates. It remains for a more thorough thermogravimetric analysis to settle this question. This investigation is currently under way.

A comparison of the thermocurves depicted in Figs. 6 and 7 shows the effect of heating rate on samples of 30—20 mesh and 100—80 mesh coal. As

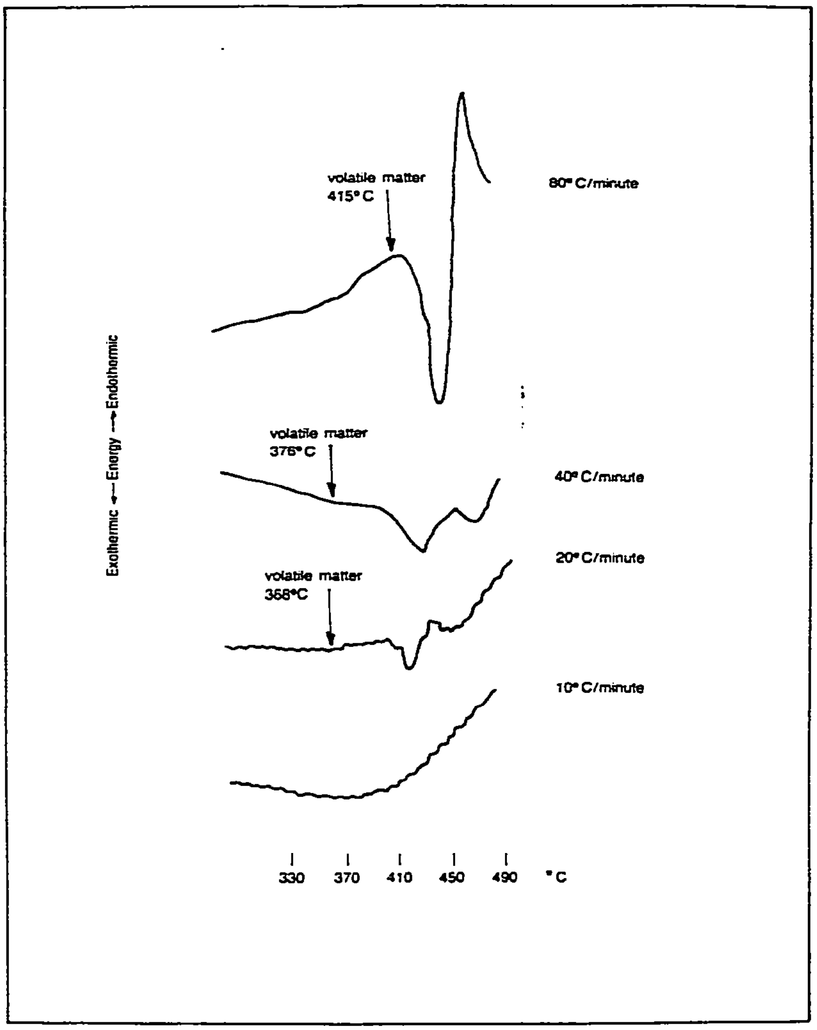

Fig. 7. Effect of heating rate on the DSC thermocurve of Ill. No. 6 (HVC). Particle size = 100—80 mesh; m_0 = 20 mg.

illustrated, the peaks were substantially reduced for the smaller particle size sample at all heating rates. Figure 8 shows this effect directly for samples over a range of particle sizes at a heating rate of 80°C min^{-1}. Here, the effect on peak size is seen to be concentrated at the higher end of the particle size spectrum.

Partial oxidation or weathering of the coal samples is the likely cause of this decrease in peak size. Coal is readily oxidized in air [8,9], and the extent of oxidation would be greater for the smaller particles due to the increased surface area. As seen in Fig. 8, samples passing through a 100 mesh screen exhibited no exothermic peak.

Sample size is also an important parameter in thermal analysis. Several factors influencing the choice of sample size are (a) the possibility of temperature gradients existing within larger samples especially at higher heating rates; (b) the inhomogeneity of coal; and (c) splattering of material out of

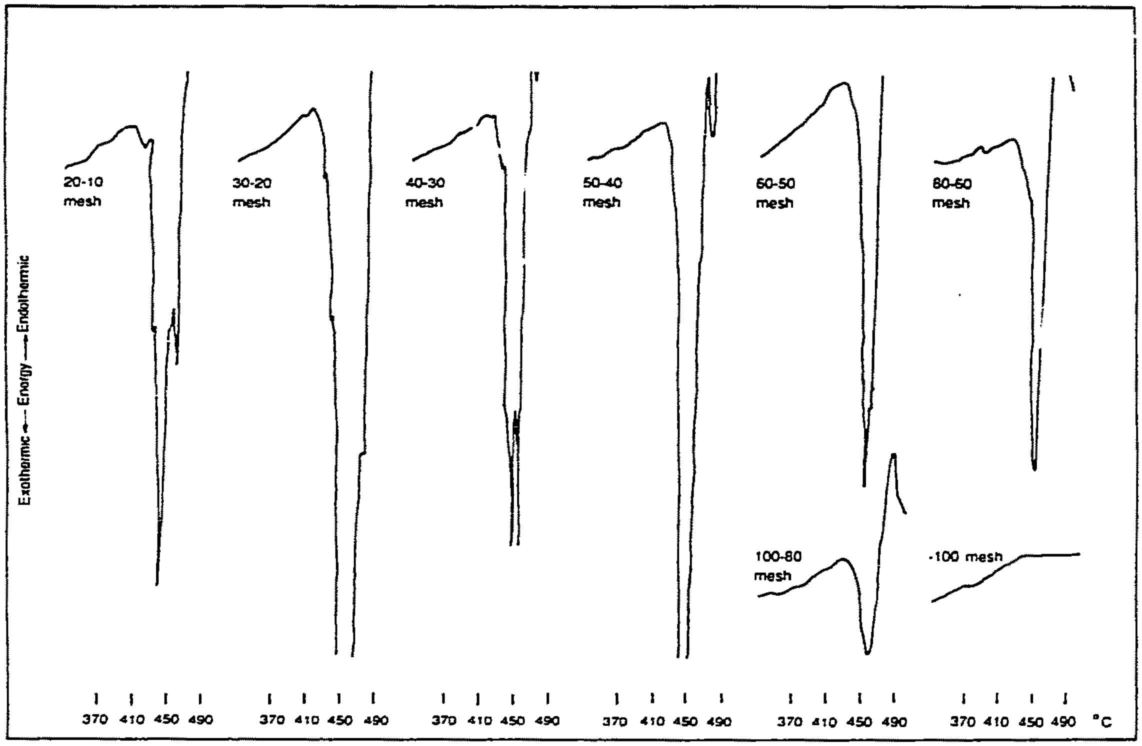

Fig. 8. Effect of particle size on the DSC thermocurve of Ill. No. 6 (HVC). Heating rate = 80°C min^{-1}; m_0 = 20 mg.

the sample pan as volatile matter is released. Thus, a sample size must be chosen so as to avoid spurious results due to such factors.

Experience with the DSC analyses of coal samples during this research indicated a more fundamental relationship of sample size to the appearance of exothermic peaks. This relationship is illustrated in Fig. 9, wherein the magnitude of the exothermic peak is a strong function of sample size. This is to be expected; however, data not shown in the figure indicate that the continuous decrease in magnitude with decreasing sample mass leads eventually to an abrupt disappearance of the exotherm. That is, the exotherm which was observed to decrease in magnitude with decreasing sample size, eventually disappeared at some small sample size at all heating rates. The discontinuous nature of this process could be verified by repeating the experiment using higher instrument sensitivities. In no case was an exotherm observed once the sample mass fell below this threshhold.

It is difficult to interpret this observation, especially in the absence of a concurrent thermogravimetric analysis. In general, however, it seems probable that this behavior is another manifestation of the relationship of the exothermic peak to the production of volatile matter. It seems probable that the existence of an exothermic peak is associated with the manner in which volatile matter is evolved and allowed to accumulate within or is flushed out of the sample pan.

The effect of heating rate on the magnitude of the exothermic peak described above can best be rationalized on the basis of an analysis of the

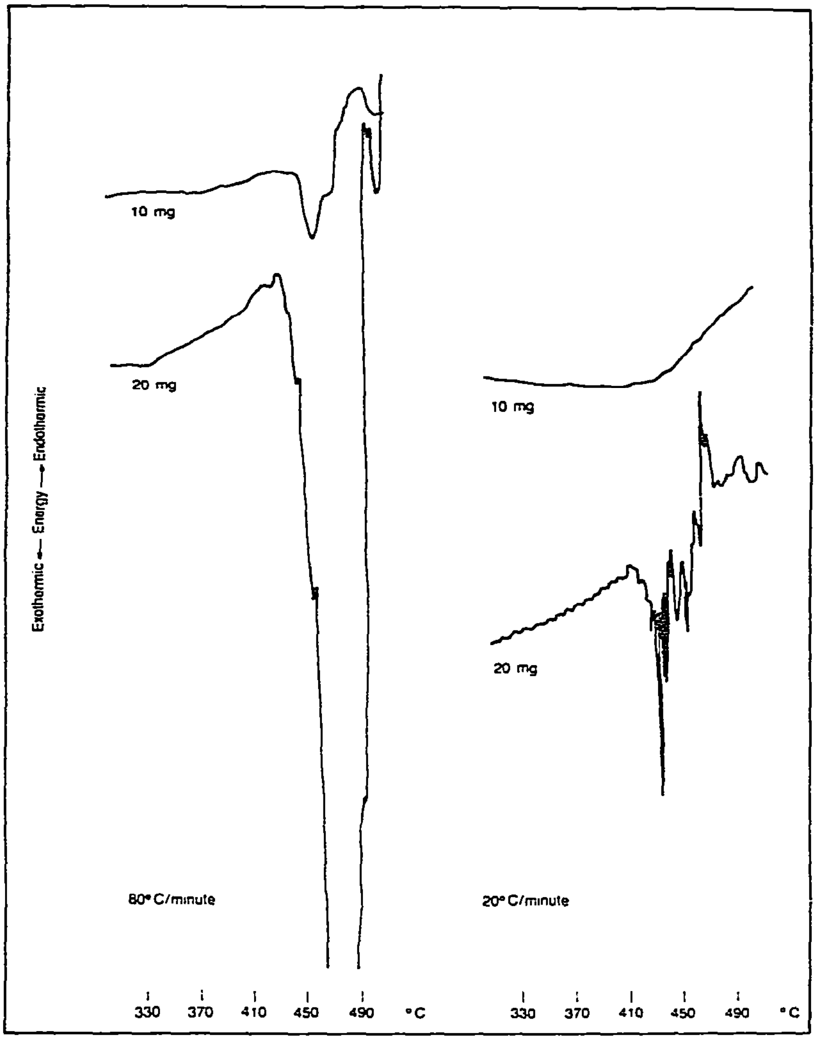

Fig. 9. Effect of sample size on the DSC thermocurve of Ill. No. 6 (HVC). Particle size = 30—20 mesh.

production of volatile matter as a function of temperature. At lower heating rates, volatiles are released at lower temperatures. The total amount of volatile matter produced is relatively independent of heating rate, although the maximum rate of loss occurs at lower temperatures for the lower heating rates. Furthermore, since the flow rate of inert diluent was held constant throughout, the rate of flushing of volatile matter out of the sample pan was relatively independent of heating rate. These circumstances would lead to a greater concentration of volatiles in the sample pan (at any temperature) at the higher heating rates. If this build-up of volatile material is essential to the occurrence of the exotherm, the absence of the peak at lower heating rates can be ascribed to the failure to achieve a sufficient concentration at higher temperatures due to flushing by the diluent gas. Moreover, the shift of the peak temperature upwards at higher heating rates can similarly be related to the shift of the maximum volatilization rate in the same direction.

Returning to the absence of an exothermic peak for smaller sample sizes, this too can be ascribed to the production of a concentration of volatile matter insufficient to sustain the exothermic reaction.

The only evidence which seems to contradict this interpretation is the failure to detect volatiles until after the inception of the exothermic peak at the higher heating rates. As explained earlier, this was probably due to deficiencies in the instrumental methods employed. A more thorough thermogravimetric analysis is currently under way to confirm this interpretation.

Weathering effects

Attention was focused on the influence of oxidation or weathering of coal samples on the exothermic peak observed in the plastic region. To this end, a number of specific tests were performed.

In an earlier paragraph, it was observed that coal samples containing mostly fines exhibited much diminished exothermic peaks, which disappeared entirely for samples passing through 100 mesh. This was attributed to the increased effects of weathering on small particle size samples. Many of the coal samples tested had been stored for as long as three years after having been ground to —10 mesh subsequent to sampling. A sample lump coal (Illinois No. 6, HVC) was ground to pass —10 mesh, and a series of thermocurves

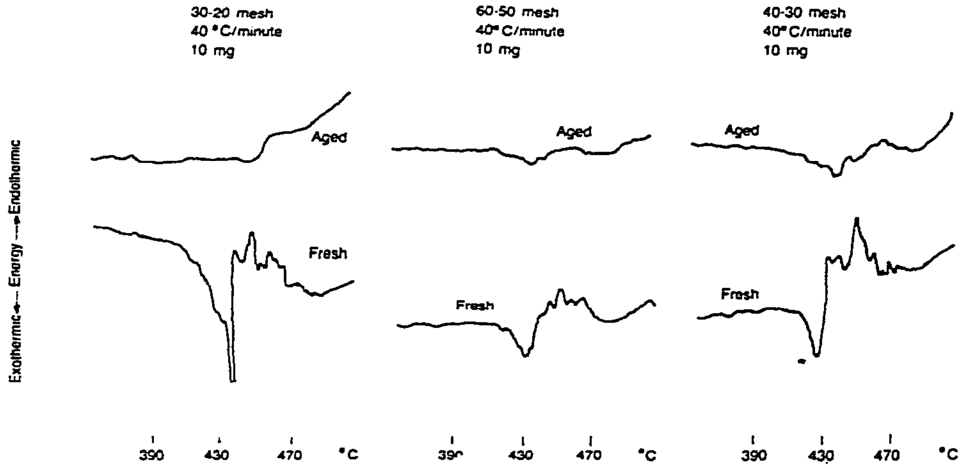

Fig. 10. Aging effects.

were obtained for comparison with thermocurves of the same coal ground and stored some months previously.

The results of this test are depicted in Fig. 10 wherein thermocurves of the fresh and aged coals are compared. The exothermic peaks of the freshly ground coal are clearly evident for all of the particle size distributions shown. The aged samples, on the other hand, display substantially diminished exothermic peaks. A similar diminution of the endothermic devolatization peaks is observed. It is clear that any quantitative interpretation of the energy absorption or evolution accompanying these processes must account for weathering effects.

Many of the coal samples tested had a substantial moisture content. Since the removal of this moisture was responsible for a substantial endothermic peak during the DSC scan, it was deemed advisable to remove the water by preheating at low temperatures prior to the start of a scan. The results of this procedure are depicted in Fig. 11. Figure 11(A) represents a typical scan including a very substantial endothermic water removal peak and Fig. 11(B) the resultant thermocurve when a similar coal sample was preheated at 100°C for 15 min prior to the start of the scan. The almost complete absence of the water removal peak is evident.

The possibility that such preheating could affect the exothermic peak was minimized by selecting a relatively low temperature for the water removal process. Preheating at higher temperatures could substantially reduce the amplitude of the exotherm as demonstrated in Fig. 12. Coal samples were

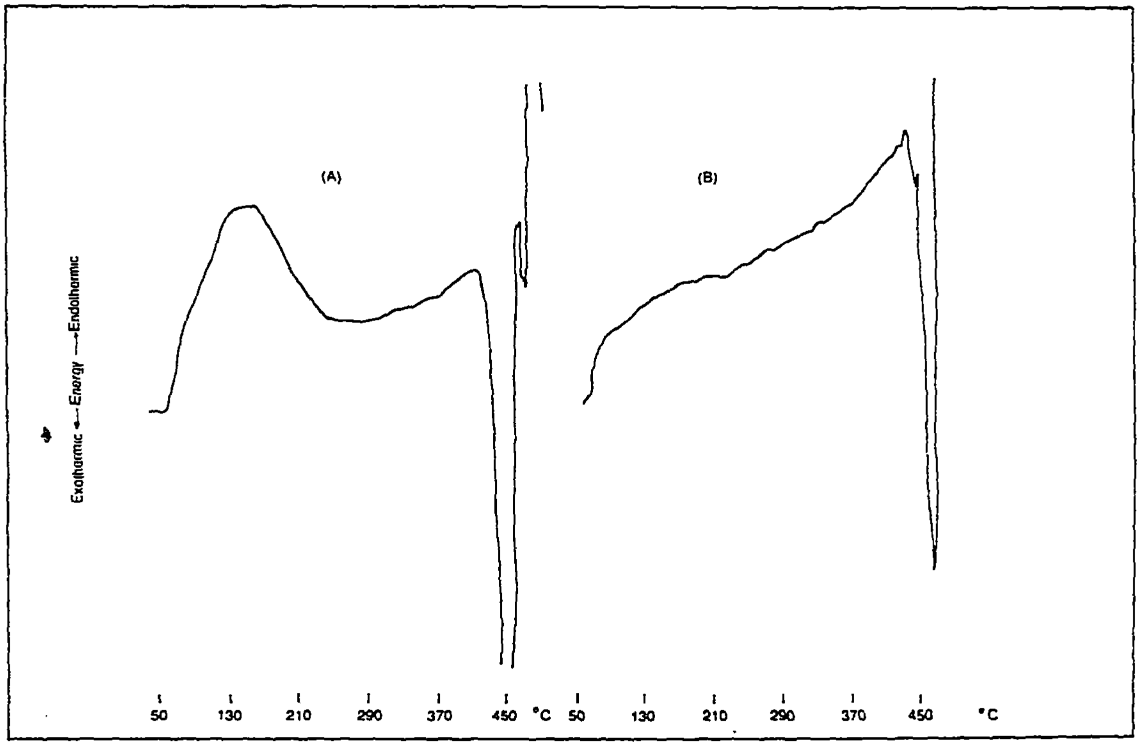

Fig.11. Water removal.

preheated for 30 min at various temperatures prior to commencing the DSC scan. Preheating at temperatures at which devolatilization occurred lead to a decrease in peak amplitude resulting in the eventual disappearance of the peak for the low-temperature char which had been heated to 450° C.

Thermogravimetric analysis

During the pyrolysis of coal, there is a gradual weight loss over the entire temperature range. The displacement of the DSC output curve relative to the instrument reference base-line is proportional to sample mass. As the mass of coal decreases with increasing temperature, the displacement of the output curve relative to the base-line will vary continuously. Thus, the output curves cannot be integrated to yield reaction enthalpies per unit sample weight using the above equation unless the output curve can be corrected for weight changes during pyrolysis [6].

During the experiments described above, no continuous thermogravimetric analysis was performed. An estimate of these effects was made by measuring the total weight loss of a number of samples over the entire pyrolysis run from 50 to 500° C. Table 3 shows the results of a total of 132 such tests. The percent weight loss shown correlates roughly with sample volatile matter content. Because of the large average weight loss (as much as

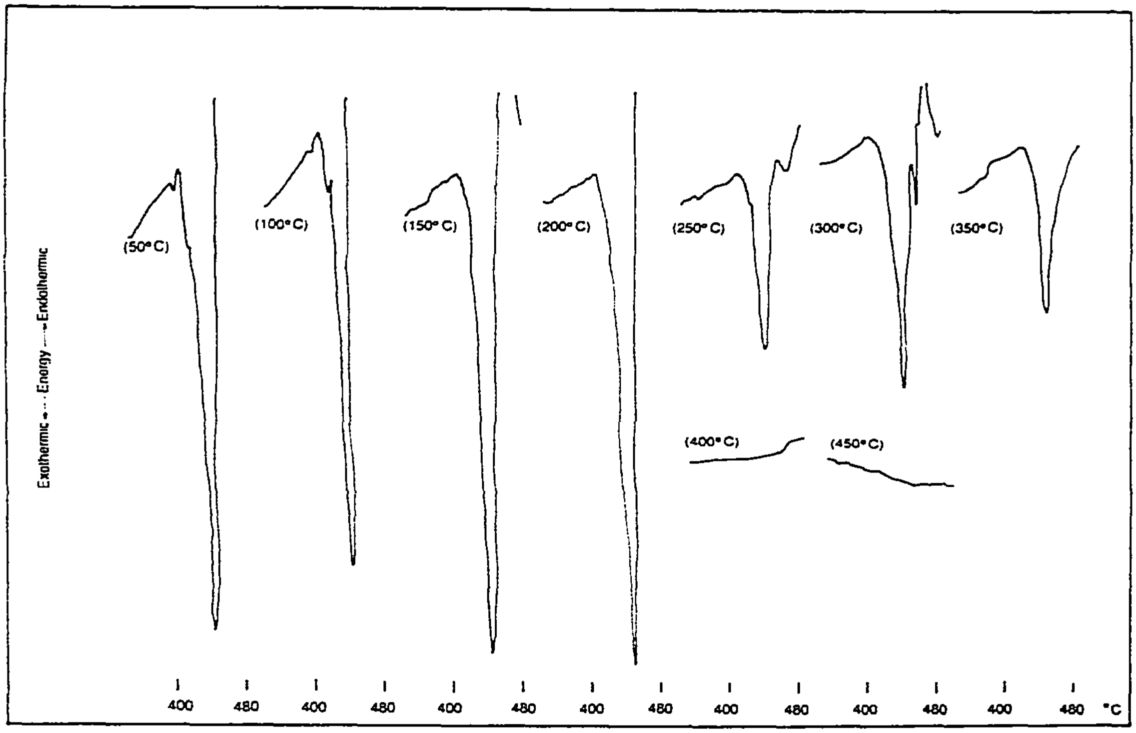

Fig.12. Isothermal tempering. Heating rate = 80°C min^{-1}; particle size = 50—40 mesh; m_0 = 20 mg. Each sample was preheated for 30 min at the temperature indicated in parentheses.

TABLE 3

Thermogravimetric analysis

Coal sample	Initial pyrolysis		Reheated char	
	No. in test	Ave. wt. loss (%)	No. in test	Ave. wt. loss (%)
233	3	26.88	1	0.96
238	13	28.87	6	1.02
300	2	22.51		
310	5	29.84	1	1.3
314	5	26.52	5	0.94
316	5	26.15	1	0.03
318	2	8.75		
414	10	40.40	6	1.70
Ky. No. 9	15	26.11	5	1.37
Ill. No. 6	72	27.58	5	0.93

40% for PSOC-414) a quantitative analysis of these results was not attempted.

Also shown in Table 3 are the results of 30 tests in which the weight loss of the reheated low-temperature char was determined. In no case did the char weight loss exceed 2%.

Summary of results

A number of coal samples ranging in rank from low-volatile bituminous to sub-bituminous were tested. Thermocurves for these samples displayed the existence of a sometimes very substantial exothermic transition within the temperature range 400—500° C. Although the appearance and magnitude of this exotherm did not appear to correlate with rank, available data were suggestive of a relationship between the reported plastic range and temperature of the peak.

Various experimental parameters were found to strongly influence the temperature and magnitude of the exothermic peak. Peak amplitude was increased at higher heating rates, which also caused a shift of the exotherm to higher temperatures. Volatilization rates and plastic characteristics of coal exhibit a similar behavior.

Sample particle size was also found to affect the exotherm with reduced peak sizes observed for smaller particle size samples at all heating rates. This effect was attributed to partial oxidation of the smaller particles.

Sample mass was also found to influence the appearance of exothermic peaks. The exotherm was observed to diminish proportionally with decreasing sample mass, only to disappear sharply at some small sample size, at all heating rates. Increase of instrument sensitivity confirmed the absence of any peak.

The combination of these factors seems to point to a relationship between the occurrence of the exothermic transition and the production of volatile matter. Although some apparent exceptions to this conclusion were observed, thermogravimetric analysis and volatile matter detection were not sufficiently precise to settle the question.

Partial oxidation or weathering was found to materially affect the observed thermal transitions as did preheating of samples at temperatures at which devolatilization was substantial.

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