Studies Concerning the Pressure Developed During the Carbonization of Coal

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I

Introduction

It has been known for some time that any coal capable of giving coke will exert a pressure against the walls of the oven upon carbonization. The development of pressures attendant upon the process of carbonization in by-product ovens has assumed increased importance not only because reserves of good coking coals with low-pressure characteristics are rapidly becoming depleted, but also because higher and narrower silica brick chambers have been introduced for increased production. In a number of instances, pressures were realized which damaged the brickwork of the oven. There are, however, oven failures which are not caused by these pressures. The oven walls may have been constructed from silica bricks of inferior quality, or from poorly cemented silica bricks; on the other hand the walls may have become eroded by certain mineral constituents of the coal. Poor operating practices also may be responsible for oven failures (45). Apparently, there exists considerable difference of opinion concerning the maximum pressure to which a coke oven wall may be subjected without damage. According to earlier statements of Koppers and Jenkner (31) pressures over 1.5 pounds per square inch are dangerous to the oven wall. More recently, Rueckel (45) has stated that “a unit resistance of 6 pounds per square inch has been found adequate to restrain expanding forces of most low volatile coals and the brick ‘per se’ possesses strength greatly in excess of this figure.” In our experience, blends coked according to United States practice show a maximum pressure not exceeding 1.5 pounds per square inch of oven wall when carbonized in our latest testing oven.

Investigators who studied the question of coking pressures found themselves confronted with three problems: the underlying reasons, the quantitative measurement, and the control of the phenomenon. Various workers in the field have been attracted by different aspects of the subject, and it is only by careful consideration of these investigations that a comprehensive knowledge of the problem can be obtained.

We have not considered the theory of coking pressures in the following pages for two reasons: In the first place, such treatment would merit much more space than we could give it in a bulletin of this type; second, it has only been very recently that the theory of coking pressures has been treated from a fundamental approach (26). This theory, however, has been of great value to us in obtaining a better understanding of the phenomena, and we feel that it should be equally important to other workers in the field.

We have restricted ourselves to a discussion of measurement
and control of pressure during carbonization because these can be accomplished without a full grasp of thermodynamic theory.

In the first part of this bulletin the essential results of the pertinent literature are presented and special equipment used in this phase of coal research is discussed. In the second part, the development and operation of a new laboratory oven designed to measure coking pressures as they occur in practice are described. The last part summarizes the results obtained in this laboratory with this testing oven.

II
Basic Facts, Terms, and Concepts

Fluidity and Plasticity: When a coal is heated at a constant rate of temperature rise, it will expand slowly until it reaches a temperature somewhere between 250°C and 300°C where, depending upon the nature of the coal, it will begin to contract. Upon continued heating the coal will again expand and become soft in the temperature range between 385°C and 450°C. At still higher temperatures the coal mass will harden, giving rise to solid porous coke and gaseous products (31). The temperature interval between softening and solidification is called the "plastic range" or "fusion zone." Because coal is a heterogeneous system, it is not surprising to note that neither the temperature of fusion nor the temperature of decomposition is a sharp physicochemical constant. However, by rigid adherence to a definite form of apparatus and procedure of testing, results can be duplicated with reasonable satisfaction.

Apparently, expressions such as plasticity, plastic state, and plastic range reflect the influence of common language rather than the results of a scientific analysis. According to Webster, the word "plastic" means "capable of being molded or modeled, as clay." In scientific language the concept should have a much more precise definition and should be clearly distinguished from related concepts, such as elasticity and viscosity. It seems pertinent to quote, in this connection, the discussion of elasticity and plastic, viscous, and turbulent flow by Bingham (10): "If a perfectly elastic solid be subjected to a shearing stress a certain strain is developed which entirely disappears when the stress is removed. The total work done is zero, the process is reversible, and viscosity can play no part in the movement. This is not a case of flow but of elastic deformation. If a body which is imperfectly elastic regarding its form be subjected to shearing stress, it will be found that a part, at least, of the deformation will remain long after the stress is removed. In this case work has been done in overcoming some kind of internal friction. We may distinguish the kinds of flow under three regimes. It is characteristic of viscous or linear flow that the amount of deformation is directly proportional to the deforming force, and the ratio of the latter to the former gives a measure of viscosity. It has been questioned at times whether this ratio is truly constant, but it appears that only one qualification is necessary. In very viscous substances time may be necessary for the flow to reach a steady state, aside from any period of acceleration, because with substances like pitch the viscous resistance develops slowly, so that the above ratio gradually increases when the load is first put on, but even in this case the ratio finally reaches a value which is independent of the amount of the load. As, however, the deforming force is steadily increased, a point may be reached where the above ratio suddenly decreases. At this point the regime of turbulent or hydraulic flow begins. There are substances, on the other hand, for which the value of the above ratio increases indefinitely as soon as the deforming force falls below a certain minimum. These substances are said to be plastic. In plastic flow it is generally understood that a definite shearing force is required before any deformation takes place. But whether this is strictly true or not has not been established."

Bingham (10) quotes an excellent example to the point from Clerk Maxwell's Theory of Heat:

"Thus a tallow candle is much softer than a stick of sealing wax; but if the candle and the stick of sealing wax are laid horizontally between two supports, the sealing wax will in a few weeks in summer bend under its own weight, while the candle remains straight. The candle is therefore a soft (or plastic) solid, and the sealing wax is a very viscous liquid."

The fused coals studied by Seyler (47) exhibited viscous flow within certain limitations. He states that "for an initial small deformation the rate of flow per unit length was directly as the load and inversely as the area, that is, directly proportional to the pressure, within certain limits. Under these conditions coal attains a definite fluidity (or viscosity) which is constant at a given temperature."

Swelling, Expansion, and Pressure: If a sample of coal is heated in such manner that it can expand freely, the coke button obtained is found to occupy more space than the original coal sample. The most striking demonstration of this phenomenon is probably the Lambris test (33): One gram of pulverized coal is placed upon a layer of sand in a platinum crucible. On heating the bottom of the crucible, the sample swells up, at times as high as several inches. The excess volume, or the difference, volume of coke minus volume of coal, may be termed expansion, or may be calculated as percentage expansion. On the other hand, if a charge of coal is car-
bonized in a container and free expansion is impeded, a pressure against the walls of the container is observed and in many cases even a shrinking of the charge results.

Originally it was believed that the expansion in the first case and the pressure in the second case are proportionate. This may explain the fact that expressions such as expansion, swelling, and swelling pressure have been used almost interchangeably by many authors. Such lack of precise language has occasioned a certain confusion which has been further aggravated by the fact that the assumed simple relationship between expansion and pressure does not exist.

Lambris (34) showed as early as 1928 that a coal which gives a considerable expansion need not necessarily give a significant pressure; hence, expansion and coking pressure ought to be sharply distinguished. However, it is only in recent years that a trend toward greater clarity of expression and greater precision of thought is noted in the literature on this subject. A few quotations may serve to illustrate this development.

In 1938 Brown (14) presented the following introduction in a paper entitled “Coal Expansion”: “During the past few years there has been a great deal of interest in plasticity, swelling, and expansion of coal. We became interested in expansion about three years ago and have run more than 900 tests on coals and coal mixtures from the Pocahontas, Sewell, Beckley, Pittsburgh, Upper and Lower Freeport, Upper and Lower Kittanning, and other coal seams.

“We will confine this paper to expansion of coal as applied to the by-product coke oven. The words ‘swelling’ and ‘expansion’ have the same meaning, namely, the act of expanding and the difference as referred to the testing of coal is hard to explain. Therefore, let us define the term ‘expansion’ to mean the characteristic of coal when carbonized in a restricted space, such as in a coke oven or expansion tester, at a definite rate of heating (1700°F - 1750°F coke temperature) and under a definite pressure (2.25 pounds per square inch) and definite bulk density (55 pounds per cubic foot).

“We would suggest the term ‘free swelling’ to mean the characteristic of coal when heated in an unrestricted space (such as fuel bed), without a definite rate of heating, pressure or uniform bulk density. There seems to be no relation between expansion and free swelling.”

In 1940 Russell (46) made the following remarks: “It is a curious fact that the term ‘coal expansion’ has been widely accepted in the United States as descriptive of the phenomenon in question. This phenomenon, however, concerns the development of pressures within the coal during carbonization that are exerted against the coke-oven walls. An ‘expanding coal’ is considered to be one that will develop sufficient pressure during coking to weaken, distort or otherwise seriously damage the walls. It should be noted that the terms ‘coal expansion’ and ‘expanding coal’ do not carry any implication of pressure development but rather suggest increase in dimensions.” Recently, Simomura (48) suggested the term “driving” instead of “swelling” to designate pressures in carbonization.

Since it has been our purpose to study the pressure developed during coal carbonization in by-product ovens, it would seem advisable to avoid completely such expressions as swelling or expansion and to use the term “coking pressure.” Inasmuch as coking coals differ from noncoking coals by the existence of a plastic state, attention is also given to work dealing with the plastic range of coking coals.

III

Experimental Methods for the Study of the “Plastic Range”

The determination of the limits of the plastic range of coal is difficult because the plastic range cannot be expected to lie between sharply defined limits. Several different principles have been used in developing methods for this determination. They include measurements of permeability, expansion, and viscosity.

One of the earliest methods, that of Foxwell (23), was further developed by its originator (24), by Layng and Hathorne (35), and by Coffman and Layng (18). It is based upon the fact that coal in the plastic range possesses a very low permeability to gas. Therefore, if a stream of gas is passed through a porous bed of coal and the coal is heated, a sharp increase of gas pressure will be noted as soon as a coherent plastic layer is formed. If the heating is continued until the mass solidifies to porous coke, the gas pressure will again drop.

A suitable apparatus is shown in Fig. 1; the procedure is fully described in the “Methods of the Chemists of the United States Steel Corporation,” 3d ed., p. 40. The apparatus “consists of a 1-in. by 12-in. Pyrex combustion tube, A, fitted with a side neck 1½-in. from one end. This tube is closed at the ends with 1-hole rubber stoppers, through one of which is inserted a rare metal pyrometer couple encased in a ½-in. silica tube, B, while a glass tee is inserted through the other. One arm of this glass tee, C, is connected to a supply of purified and dehydrated nitrogen, and the other arm, D, to a manometer tube graduated to 48-in. water pressure. The side neck of the combustion tube extends into a 1½-in. water seal, E.
coals regarded as dangerous in coke manufacture, and exhibiting large coking pressures in pressure-measuring ovens. It seems reasonable to assume that coals of the former type give rise to a rather homogeneous mass which is impermeable to gas, while coals of the latter type do not easily flow together, consequently remaining pervious to gases.

Another method that has been applied to the study of the plastic range is the measurement of expansion during carbonization: the dilatometer principle has been used by numerous workers in the field, e.g., Lessing (37), Korten (32), Audibert (5); and Bunte, Brüeckner, and Ludewig (17).

The apparatus recommended by Mott, Wheeler (41), and their associates (the so-called Sheffield laboratory apparatus) is shown in Fig. 2. In order to carry out a test, 6 grams of coal, freshly ground to pass a 60-mesh screen, are introduced into a silica tube of 16 mm internal diameter so as to form a column 40 mm long. A plunger weighing 100 grams, on top of the coal charge, is lifted by the expanding coal against a counterpoise connected with the plunger. The tube is heated in the vertical furnace at a rate of 1°C per minute. After an initial contraction, the coal begins to expand; the maximum expansion is measured by means of a fixed scale and a pointer moving with the plunger.

Mott and Spooner (39) discuss certain mechanical disadvantages of the Sheffield laboratory apparatus, such as fragility, cost, and delays in obtaining spare parts. They describe a much more durable setup that was developed in the laboratories of the South Metropolitan Gas Company, London, England.

Mott and Wheeler state that the percentage expansions of the coals examined by the use of the Sheffield machine “conform fairly well with the quality of their cokes, a high value for the expansion denoting a high quality of their coke.” Exceptions to this rule have been noted by the authors themselves.

In our experience, coals which give a negligible back-pressure in the Layng-Hathorne test may show a large expansion in the Mott-Wheeler test and may or may not yield satisfactory coke.

In a number of cases, as the investigators were not satisfied solely with establishing limits of the plastic range, or amount of expansion, they attempted to develop a more detailed description. The experiments of Audibert, and more recently of Massinon, are offered as examples. Audibert (5) studied the fusion of coal in a dilatometer which “consisted essentially of a cylindrical copper or iron tube, in which was placed the sample of coal to be studied, previously made into a cylinder 60 mm long and 6 or 7 mm in diameter. This cylinder was obtained by compressing the powdered coal slightly moistened with water, in a steel mould, care being
taken in the process that the density of the material was the same in all sections of the pellet. The tube being supported vertically, a glass rod was placed on the coal. The rod was almost completely counterpoised, and carried a long pointer moving in front of a scale. The tube was plunged into a lead bath, or a mixture of melted salts, the temperature of which could be adjusted at will by an electric winding. Provided the internal diameter of the metal tube was not exactly equal to the diameter of the briquette it contained, the movements of the pointer, in these circumstances, gave evidence of a change in the form of the heated coal sample.

"The bath was first heated to the temperature T; this being reached, the heating current was adjusted so as to compensate exactly for losses of heat to the surrounding air, after which the dilatometer was immersed in the bath. The thermal capacity of the tube being very small compared with that of the bath, no appreciable variation in temperature was thereby caused, and it may be considered that the temperature remained constant throughout the experiment.

"The phenomena observed may be conveniently represented graphically, taking as abscissae the times from the instant when the dilatometer was placed in the bath, and as ordinates the displacements of the pointer, expressed as fractions or multiples of the initial length of the cylinder."

Thus, a curve corresponding to every value of the temperature T was established. Moreover, all the coals examined showed the same temperature characteristics in that they had:

1. A temperature of beginning melting.
2. A temperature of beginning expansion, swelling, or intumesence.
3. A temperature above which thermal decomposition becomes appreciable.
4. A temperature of solidification.

As a result of his investigations, Audibert distinguished two classes of coals: Class I, comprising the good coking coals, is characterized by a temperature of decomposition above the temperature of fusion, while Class II contains the coals for which the temperature of fusion is higher than the temperature of decomposition. Hence, the coals belonging to Class I melt before decomposing, while the coals of Class II decompose more or less before properly melting. Since by changing the rate of heating, coals of Class II may behave as if they were representatives of Class I, the borderline between the two classes cannot be regarded as fixed.

Massinon (38) studied a number of Belgian coals by measuring the amount of expansion, the gas yield, and the viscosity between
300° and 500°C. The curves obtained suggested a division of the effect of temperature into three zones:

1. **Softening.** Between 405 and 455°C the coals first slightly contracted, then rapidly expanded, these phenomena being accompanied by a rapidly increasing gas evolution and a slight decrease in viscosity.

2. **Fusion.** From 455 to 475°C. The coals contracted; the gas evolution became still more vigorous, and a large decrease in viscosity was observed.

3. **Solidification.** From 475 to 500°C. The volume of the coals remained constant, the gas evolution slowed down, the viscosity increased to infinity.

In order to determine characteristic temperatures of the plastic range, Berl and Schildwächter (9) and Gieseler (27) have utilized an “Erhitzungsmikroskop.” In view of the initial expense and the qualitative nature of the results the method has not been extensively used. As an example of the qualitative results, some of the observations of Gieseler are cited.

From finely powdered coals small bodies were compressed and fragments of these were observed with the microscope while being heated. Unfortunately, the planes of fracture did not permit good observations of the softening and other processes. A number of coal grains (from 0.25 to 0.40 mm) on a platinum foil were placed in the concavity of a suitable device. Ten to twenty grains could be seen in the field of the microscope, which permitted a good observation of the changes upon heating. In case of an Upper Silesian coal with 29.6 per cent V.M. the first changes of form were noted at 390°C when edges of the coal grains became rounded; above 400°C one grain after the other became soft. The coal remained plastic for a longer period of time and the gas developed blew up the mass into hollow spheres. With a second Upper Silesian coal with 38.3 per cent V.M. only portions of the grains softened at 405°C; the surface and shape of the grains remained practically unchanged. A lower Silesian coal with 24.7 per cent V.M. showed the first changes at 402°C; complete softening occurred between 430° and 450°C and extended up to 500°C. The formation of hollow spheres and tar bubbles did not occur to the extent noted with the Upper Silesian coal.

A Ruhr coal with 26.0 per cent V.M. softened between 375° and 410°C and remained plastic up to 500°C. Single tar bubbles were seen occasionally. Another Ruhr coal with 17.3 per cent V.M. remained unchanged up to 470°C; and between 495° and 500°C all grains softened at once, then immediately solidified.

A number of methods for the determination of characteristic temperatures of the plastic range may be classified as penetrometer and plastometer methods. The definition of a penetrometer method demands that the velocity or depth of penetration of a needle into a yielding substance should be measured. Specific methods have been proposed by Damm (19), Agde and v. Lyncker (1), Gieseler (28), and others (43, 44); the results have not always been evaluated in the manner as indicated. Rather, they have been used to measure volume changes and temperatures characterizing the coal in its plastic stage.

The Agde and v. Lyncker apparatus is described briefly as follows: An electrically heated porcelain beaker (85 mm diameter and 165 mm high) is filled with a suitable salt for a bath and covered with a brass lid into which two openings have been bored, one for a thermocouple or thermometer and the other for the actual measuring device, a brass cylinder of 20 mm diameter and 60 mm high. This brass cylinder serves to hold the so-called measuring tube and the measuring rod. A hole 4.2 mm in diameter is bored on top of the brass cylinder and a 11 mm-diameter hole bored on the bottom. On the side there is a gas outlet and a set screw to fix the measuring tube in place. The measuring tube, which contains the sample, is of thin blue steel 10 mm O.D. and is fixed in place at the bottom of the brass cylinder by means of the aforementioned set screw so that a gas outlet hole in the measuring tube coincides with the gas outlet in the cylinder; the bottom of the measuring tube is closed with a tightly fitting screw cap. A brass rod 4 mm diameter is fitted into the 4.2 mm top hole of the brass cylinder so that it slides smoothly. At the lower end of the rod a polished steel needle 1.2 mm diameter and 60 mm long is fixed in place. Over the upper part of the brass rod, various weights (50 g, 100 g, 50 g + 100 g) may be fastened; the upper end of the rod is attached to a flexible copper wire and connected to a counterpoise over the wheel of a circular dial, the counterpoise exactly balancing the weight of the unloaded rod. A modified form of this apparatus is shown in Fig. 3.

The plastometers of Davis (21) and of Gieseler (28) embody one of several basic principles for the measurement of viscous flow, namely, that of measuring the resistance offered to a movable body by a viscous or plastic substance, e.g., molten coal. The Davis plastometer shown in Fig. 4 consists of a cylindrical steel retort 5½ inches long and 1½ inches in diameter with 3-inch wall, arranged for rotation horizontally at constant low speed in the middle of a tube furnace 12 inches long. A tube is passed into the retort through one of two tubes in the cylinder, which serve as a shaft. Within the retort this tube carried five T-shaped rabble arms, dipping into the coal charged therein and clearing the retort wall by ⅛ inch. The arms are spaced at equal intervals around the
shaft, which is prevented from turning with the retort by a tension coil spring attached by a lever to the external end of the tube. As the coal under test becomes viscous the spring is stretched and the amount of tension is indicated in pound-inches on a circular scale.

"Eight grams of coal sized between 20 and 40 mesh are charged into the retort which is closed by screwing the head in place. The retort is placed in the furnace and is started rotating. The current is switched on and the heating rate begun at 7° per minute. Over the plastic range—380° to about 460°—the heating rate is maintained constant at 3.4° ± 0.2 per minute. Temperature and torque indications are read and recorded at 5-minute intervals from 380° until the solidification point is reached. Thus, for a Powellton coal, at 400° viscosity began to develop and at 410° reached the first maximum torque (19 pounds); at 412° the torque had fallen to 6 pounds. From this temperature to 440° the torque gradually decreased to a fraction of a pound. At 440° it began to increase rapidly and reached a maximum of 47 pounds at 460°. At this temperature a rapid drop in viscosity occurred (47 pounds to zero torque at 460° to 467°). Thus, 460° is the solidification temperature of the coke when the coal is heated, sized as specified, over the plastic range at 3.4° per minute." (52)

The Gieseler apparatus is similar to that of Davis in that it uses a torsional principle though applied in a different manner. The rate of stirring of coal charge is proportional to the fluidity of the coal mass, inasmuch as stirring begins only after the coal has become plastic by heating. Brewer and Triff (12) have described a modified Gieseler apparatus (Fig. 5) as follows: "The retort is 21 mm in inside diameter and 16 mm in depth. In testing strongly contracting coals a steel pin 1.6 mm in diameter is inserted through a hole in the retort at a point 5 mm from the top and to a distance of 5 mm radially toward the center from the inside wall. Without
This pin arrangement the stirring shaft carries the contracted coal charge around with it and thereby falsely indicates a highly fluid mass. At the same time, the pin arrangement constitutes an improvement over the sieve-plate cover first used at the top of the retort, since the binding between the stirrer shaft and the sieve-plate caused by the accumulation of partly carbonized heavy tars is entirely eliminated.

![Modified Gieseler Apparatus](image)

**Fig. 5.—Modified Gieseler Apparatus.**

“A radial type of ball spindle bearing assembly taking a horizontal thrust only was first used. Because of high friction in this bearing, a weight of 145 grams on the loading pan was required for satisfactory operation. Except for the fusion temperatures, which showed the same order of agreement, the data obtained with this bearing and 145 grams on the loading pan were less concordant than those obtained with the new ball-bearing assembly shown.

This latter bearing takes both a vertical and horizontal thrust and shows but little friction in its operation. A 20-gram weight on the 18.7-gram loading pan is sufficient to cause uniform rotation of the stirring shaft in the empty retort, no rotation in the coal charge until initial softening of the coal is reached, and a smooth movement during passage through the preplastic and plastic temperature ranges.”

Brewer and Triff have compared plasticity data obtained with their modified Gieseler plastometer with data found by the Agde-Damm dilatometer and the Davis plastometer methods. The following Table I taken from the original data illustrates the nature of the results derived from the comparison and points to “a good order of agreement.”

Summarizing, if phenomena such as expansion and viscosity of coal are studied in regard to dependence upon such variables as temperature or time, curves are obtained which may show characteristic breaks. These breaks in the curves have been interpreted or defined in various ways by a number of workers. Some of these observations are perhaps merely indicative of the fact that neither the lower nor the upper limit of the plastic range can be established by an unequivocal determination and, therefore, should be established by applying the theory of means to the points of inflection at the lower and upper ends of the curves. The plastic ranges thus obtained are of the order from 20 to 40°C. Similar values may be deduced from practical experience. Recently, two medium volatile coals were found to have plastic ranges of 106° and 100°C (22). However, it is stated that these “wide ranges of plasticity are not characteristic of medium volatile coals,” and it is reported that the plastic ranges of two representative medium volatile coals from the Sewell Bed are 50° and 61°C as determined in the Davis plastometer.

**IV**

**Development of Apparatus for the Determination of Expansion and Coking Pressure**

Although the investigations concerning the plastic range and behavior upon softening are of undoubted scientific value, they do not convey any quantitative information of the actual pressures developed during the carbonization of coal in modern by-product ovens.

Among the first to develop an apparatus to attack the problem was korten (32). Korten's method consisted of measuring expansion under a load by means of a piston and lever arm arrangement
connected to a recording revolving drum (Fig. 6). Other workers who utilized the essential features of the Korten apparatus but with added refinements were Koppers (30), Hofmeister (29), Altieri (2), and Brown (14).

Altieri has built several ovens (2, 3, 4). His latest oven, Model C-2 which differs from Model C-1 by a hot movable wall, has been designed to measure simultaneously the change in depth and the change in width of the coal charge during the period of carbonization. It is significant to note that Altieri has changed his ovens from a cylindrical shape to a rectangular shape, which is more in keeping with the condition in by-product coke manufacture. It should be realized, however, that this Model C-2 oven is the latest and most modern design of the principle advanced by Korten.

The "Bethlehem tester" developed by Brown (14) is a sole-flue oven with a capacity of 52 pounds of coal (Fig. 7). It is quite evidently an improvement upon the Altieri tester type "A" and "B." Brown has also built an electrically heated oven for laboratory use with a capacity of 9 to 10 pounds of coal. Although Brown's ovens do not measure pressure, they have some merit since they measure expansion or contraction of the coal during the last stages of carbonization.

It appears that Nedelman (42) was the first to modify the Korten-Koppers oven so that the actual pressures developed by the coking coal were measured (Fig. 8). This was accomplished by the direct transfer of pressure from the lever arm to a hydraulic piston, allowing the lever arm to move only a fraction of a millimeter. The hydraulic piston was connected to a continuous recording instrument.

J. Taylor (49) proposed one of the first methods to assay coals for pressure developing upon carbonization. His apparatus consisted essentially of a small retort in which 3 grams of coal were heated
between layers of asbestos paper. The coal, ground to pass a 90 I.M.M. sieve and dried for two hours at 105°C, was charged into the retort and compressed by placing a 10 kg weight on the piston for a few seconds. The temperature of the retort is then rapidly raised to 250°C, at which point it is regulated to a constant rate (10°C every 3 minutes). At 350°C the 10 kg weight is once more placed upon the piston for a few seconds, and the position of the piston is noted from graduations on its stem. When the coal begins to swell, weights are placed upon the piston to keep it in the position as recorded when the 10 kg weight was in place at 350°C. Taylor has stated that in this way experimental pressures up to 60 kg were measured. His results are not recorded as weight per square inch or square centimeter.

Koppers and Jenkner (31) describe a laboratory oven for the measurement of coking pressures which is somewhat similar to the oven used by Nedelman. There chief contribution, however, has been the introduction of an apparatus for the determination of coking pressure in a large-scale oven which approaches the conditions as they exist in practice. Davis and his associates of the United States Bureau of Mines (21) used two electrically heated testing ovens of different design to investigate the expanding properties of a number of coals. One, a slot-type oven with a movable wall, carbonizes about 200 pounds of coal, while the other, a sole-heated type similar to Brown's oven, has a capacity of about 40 pounds of coal. C. C. Russell (46) has designed a large-scale testing oven for the measurement of pressures which develop during carbonization. His oven is "12 inches wide, 42 inches high and 28 inches long inside the doors. Each wall of the oven has an area of about 1000 sq in. The heating flues and the face of the oven
are constructed of standard 9-inch Alcor brick, which has the mechanical properties of silica brick without having the crystallization changes that affect the expansion of silica brick. This brick was selected so that the oven could be shut down or started up in a relatively short time. The oven is completely insulated with one course of Sil-O-Cel brick and the outside cover is constructed of first quality firebrick. The roof of the oven is suspended from the top of the 1 beams of the strong back, so that the movable wall will move without contact with the top. The movable wall is constructed on a steel carriage, which is equipped with roller-bearing rollers that rest on 1 1/4-in. cold rolled round steel; the wheels grooved so that they have the smallest possible contact with the rails to reduce friction. The entire oven sits within a strong back constructed of 8-in. I beams and channels.

"A lever system with a 7:1 ratio mounted on this strong back as indicated in Fig. 1 (Fig. 9 this paper) is used for the transmission of the pressure or movement to the mechanism for measurement. Weights attached to the movable wall through cables as shown counterbalance the weight of these levers. Instead of the hydraulic cylinder used by Koppers and Jenkner for measuring the pressure developed, a simple mechanical principle has been applied, which is far less expensive and without doubt as sensitive as the hydraulic cylinder. It has the additional advantage that the equipment required is available in practically all laboratories. The principle involves the placing of a heavy weight (1200 lbs) on a rigid carriage on a fairly sensitive platform scale. The levers as shown are rigidly connected to this weight through two turnbuckles. Before the test is begun the weight of the load is accurately determined, and then carefully connected to the levers, so that the pull is not more than 20-lb. After the coal is charged into the ovens, any pressure developed is transmitted to the wall, thence to the levers and to the load. As the pressure increases, the load on the platform scale is reduced proportionately, and this is determined by weighing the load at frequent intervals. In this way a continuous record of the course of the pressure developed during carbonization of a test charge can be obtained. Substantially no movement of the movable wall takes place as long as the pressure in the oven is less than the effort required to lift the weight. What movement occurs is determined by a gauge accurate to 0.001 inch.

"The oven has only one door which is lined with 9 in. of brick for insulation. The back of the oven is an integral part of oven structure and contains a carbon block 5 in. high having 13 holes spaced 1 in. apart, center to center, that lead into the oven chamber. These are used for the introduction of thermocouples to measure the temperature progression at 1-in. intervals throughout the width of the oven. In order to prevent erroneous temperature readings due to transmission of heat through steel protecting tubes, the thermocouple wells are staggered in length. The center couple is longest and each couple proceeding from the center toward each wall is 1 1/4 in. shorter than its adjacent couple.

"The oven is heated with coke oven gas conducted to the flues through open pipe burners. The gas main to the movable wall is of flexible steel tubing, to avoid strain. This also allows the movable wall to be moved out for cleaning. Temperatures of heating flues are obtained by means of an optical pyrometer at three locations in each wall.

"Coal is charged into the oven from a hopper 6 ft above the top of the oven, through a pipe, into a hole in the center of the suspended top. After charging and leveling, the pipe is removed and a gas vent installed in its place. The charge is coked until the center thermocouple indicates a temperature of about 600°C. At that temperature the coal has passed through its plastic stage and has become solidified coke. It has been found that above that temperature no further increase in pressure is obtained. With low volatile coals that produce high pressures, the maximum pressure has been reached below that temperature and the rapid pressure decrease is well in progress."

Test devices to be introduced into actual industrial ovens during operation have also been proposed. F. Ulrich (50) has used in large-scale operation an apparatus patterned after one of Koppers and Jenkner (31) which measures coking pressures. Essentially, this

Fig. 8.—Russell’s Oven for Measuring Coking Pressure.
device, which has the shape of an artillery shell and is made of heat-resistant material, measures pressure directly by means of a manometer. A metal diaphragm located at the pointed lower end is depressed against a lever which transfers the pressures to a diaphragm valve connected to the manometer. The apparatus is suitably and efficiently water-cooled and equipped with a thermocouple. Special precautions are taken to prevent flow of the plastic coal into the cylinder past the diaphragm. In this connection it is interesting to note the critical remarks of Foxwell (24). "The method used by Ulrich is a valuable research method, but it cannot be used to forecast the behavior of coals before use. I am, moreover, just a little uncertain whether the insertion of a water-cooled tube into the oven is permissible, as it slows up the flow of heat at the point of measurement just as the plastic layers are coalescing."

"In my own view, and apart from any results that Ulrich has obtained, the importance of his technique lies in the fact that if a reliable method can be found of measuring the lateral expansion pressure within the coke-oven continuously up to the maximum value, it is no longer necessary to wait until coals have destroyed ovens in order to devise a suitable method of testing. The pressure thrown by coals in the oven can be compared with smaller scale results, and thus we can know what results should be given by our laboratory or small-scale testing apparatus."

It should be noted that some workers have doubted the significance of pressure measurements in evaluating coals for coke production. Mott and Spooner (40) in emphasizing the shrinkage of coal state: "The primary cause of damage to oven walls is not swelling power per se but swelling power accompanied by low shrinkage, and low shrinkage may be regarded as being important, indeed more important than swelling power." Foxwell (24) has disputed the importance of this statement and the majority of workers are not likely to subscribe to the viewpoint of Mott.

The importance of shrinking as a factor in coke production need not be neglected. However, the effect of excessive pressures developed during carbonization is a well-established fact which emphasizes the desirability of pressure measurements in advance of operation.

V

The New Laboratory Testing Oven

Development: A consideration of the testing equipment which has been described in the technical literature and presented in the preceding section indicated the desirability of developing a new laboratory apparatus which would meet the following requirements:

1. The shape, dimensions, materials of construction, and method of operation should resemble a large-scale by-product coke oven chamber.
2. Coking should take place with little change in volume of the charge and with free escape of volatile matter.
3. Bulk density, petrographic composition, and size distribution of the samples should be comparable to those used in actual practice.

However, even though our oven has been built to meet the above specifications, it may seem to have certain disadvantages usually attributed to small-scale testing apparatus: the oven chamber is not built in the same proportions as those in practice, the rate of heating is usually faster than in commercial ovens, small samples are difficult to duplicate, and the pressures are higher than those usually found in practice. Nevertheless, it is felt that the advantages of this small-scale tester by far outweigh its disadvantages.
tion, while the other is mounted on four ball-bearing wheels which run on angle iron tracks perpendicular to the oven walls. The width of the coking chamber may be varied either by original design or by appropriate inserts. The floor of the chamber is lined with one-quarter inch silica brick to prevent excessive wear and all other refractory surfaces that come in contact with the coal charge are either coated with silica cement or lined with silica brick.

The development of satisfactory heating for this small-scale tester involved a considerable amount of thought and experimentation. It was necessary to achieve a uniform temperature over the whole face of the chamber which could be quickly and accurately controlled within rather narrow limits. This condition was accomplished by the construction of the special, yet simple burners, proper combustion chambers, and dependable control equipment.

Two burners, each of which consists of two concentric pipes, are assembled into a unit by screwing the ends of the gas inlets into a tee. The air inlets are arranged in a similar manner. For details of construction see Fig. 11. The advantage of this burner assembly is that there is only one gas and one air line leading to each unit. The service lines from each unit are connected to form a single gas and a single air line to the main valves. The units are fastened in place with four small pieces of strap iron which are bolted to the frame. The ends of the burners project about one-half inch into the burner ports and the open space around the burners is closed with packing of wet asbestos paper or cement.

The two identical surface combustion units are narrow chambers 8 x 8 x 1 inch filled with ¼ x ½-inch silicon carbide crystals. The silicon carbide, being a good conductor with a large heat capacity,

tends to even out the temperature of the hot walls; besides, its mechanical strength reinforces these walls. The units which may be held at any temperature between 400°C and 1300°C ± 10° by means of a heat control determine the operating temperature of the oven chamber. The temperature is regulated by means of a Pt-Pt-Rh thermocouple balanced in a potentiometer circuit. A galvanometer in this circuit operates a photoelectric relay which controls solenoid valves in the gas and air lines.

Coal samples are prepared for the test by grinding to pass through a quarter-inch sieve. The main sample is either cut down with a riffle or by means of some other approved sampling method to the approximate amount that is needed in the test and the individual samples are adjusted to exact weight just before use. When a blend is made, the weighed portions are thoroughly mixed by rolling on an oilcloth or canvas. The bulk density is determined by carefully pouring the prepared sample into a sheet iron box that has the approximate dimensions of the furnace chamber, and weighing. The apparent density of the charge is then calculated from the net weight of the coal and the known volume of the box. After the bulk density of the sample has been determined, the charge is placed in the hopper and dropped according to a prearranged time schedule.

The oven is charged so that one inch of hot silica brick will be exposed above the coal, as this procedure tends to keep the volume constant.

A headpiece of two whole bricks which cover the top of the chamber is placed on top during the run. These two bricks are grooved so that when they are placed together a small rectangular slot ½ x 1½ inches is formed for the escape of volatile matter. The purpose of the headpiece is to make conditions similar to those in actual practice and to permit combustion of volatile matter without a direct flame coming in contact with the charge.

The manner in which the pressure is determined is of considerable interest. In early types of apparatus the pressure was transmitted through a piston pushing against a rubber diaphragm connected to a Bourdon type gauge. The pressure reading with this type depends upon the diameter of the diaphragm and the range of the gauge. The disadvantage was that the rubber aged, had to be kept cool, and was occasionally punctured by pressures which we now measure without difficulty.

In our present design a triple-layer stainless steel bellows or sylphon 1½ inches in diameter is used (obtainable from Chicago Metal Hose Corp., Maywood, Illinois), which is capable of withstanding a maximum pressure of 600 pounds per square inch. (Fig. 12). The pressure is transmitted to the bellows from the furnace
3. The pressure connecting rod is put in place and the gauge set to read 15 pounds per square inch. This setting is made up by hand rotation of the rod.

4. The sample is weighed and mixed, its bulk density determined and then it is transferred to the charging hopper (2300 grams in the 3-inch oven and 3200 grams in the 4½-inch oven).

5. Charging is made according to a time schedule; the top brick is removed 10 seconds before the minute, the hopper is placed on top of the oven directly over the chamber, and the charge is dropped.

6. The headpiece is placed over the oven, the fume hood is closed, and any insulation or protection for the hood is put in place.

7. If there is no provision for automatic recording, pressures are noted at regular intervals.

8. Any pressure peaks and minima that do not occur at the normal time for a reading should be recorded. The data may be plotted as they are taken.

9. After the final peak has been recorded and the pressure has reached a steady value, the headpiece is removed and the volume of the coke is determined. A reliable indication that coking is completed is that the burning volatile matter changes from a smoky, yellow color to almost colorless.

10. If the coke is to be studied further, the charge should remain in the oven for five minutes after the smoky flame has disappeared. If the coke is to be discarded, it may be discharged as soon as the top volume has been determined.

11. The oven is discharged as follows: the air and gas are turned off, the service lines between the two sections are disconnected, a suitable pan containing about ⅛ of an inch of water is placed under the oven, the pressure rod is removed, the oven is opened, and the coke is raked out.

12. The oven is prepared for another experiment by pushing the oven sections in place, connecting and adjusting the gas and air lines, placing the insulating brick over the oven chamber, and returning the pressure rod to its proper position.

One hundred to two hundred tests may be performed without any major repairs being necessary with the present oven, if proper care is taken in bringing the oven to temperature and discharging the coke. At first, the oven is heated with a very small flame, which is carefully and gradually increased until the desired temperature is reached. Several adjustments are necessary before the temperature of 900-1000°C is attained; this usually requires approximately 5 hours. By merely increasing the air to gas ratio, the oven can be
adjusted to higher temperatures, if so desired. During the first few hours excess gas is used in the burners, the resulting incomplete combustion insuring a gradual increase in temperature. An excessive rate of heating will cause extensive cracking of the silica bricks and should be avoided.

If a series of tests extending over several successive days is contemplated, it is advisable to keep the oven at the temperature of operation for the entire period. This will lengthen the life of the oven by minimizing the danger of cracking of the silica bricks, and will also save time in operation.

The coke obtained in the oven appears to be regular high-temperature coke (only of smaller top size), and may be used to gather advance information concerning the quality of coke to be expected in large-scale operation. Specifically, determinations of apparent density, true density, porosity, and microstrength have been used for this purpose in this laboratory. Adaptations of the shatter and tumbler tests to the smaller samples are contemplated.

VI
Pressure Measurements

Accuracy and Precision: Pressure measurements, just as any other type of quantitative measurements, should meet certain standards of accuracy and precision. In order to obtain a critical estimate of the accuracy of pressure data obtained with one definite sample in a particular setup with fixed conditions, it would be necessary to know the theoretical maximum pressure to be expected with the sample under consideration, or at least the actual maximum pressure under the circumstances. At present, theoretical maximum pressures cannot be computed, and actual maximum pressures can only be found by a statistical analysis of a large number of data, e.g., 100 measurements with the same coal. Because of these conditions, the only criteria now available for judging the merits of a given method of measurement are the comparison with empirical standards and the duplicability of numerical values. In this connection, attention is called to the large number of variables which enter into coking pressure measurements. A discussion of these variables should show that rigid duplicability can hardly be expected.

Variables of Carbonization Experiments: These may be classified under three main groups:
  1. Nature of coal or blend.
  2. Preparation of sample.
  3. Construction and operation of ovens.

1. Nature of Coal or Blend—It has been established in industrial practice that for the purpose of carbonization in by-product ovens, coals may be classified as safe, borderline, or dangerous, insofar as possible damage to the oven walls is concerned. Various investigators have confirmed the fact that the inherent nature of a coal is of prime importance in regard to maximum pressure to be expected upon carbonization. Several workers have focused their attention upon individual characteristics which are descriptive of the nature of coal. Brown (14), for example, has carried out a large number of expansion tests in his Bethlehem tester, (described on page 17) relating his results to various characteristics such as volatile matter, moisture, ash, fusain, and other variables. It should be noted that the time necessary to reach maximum expansion (or contraction) varied greatly in these experiments. Fig. 13, taken from Brown’s work, illustrates a study relating the maximum expansion (or contraction) values to the volatile matter content of coals taken from various seams. While the curves representing the Upper Kittanning and Pocahontas Seams show not only a steady increase of expansion with decrease of volatile matter but also different slopes characteristic of each seam, the curve given for the Lower Kittanning Seam shows that such a simple relation is at least not general. This is all the more striking if the data are inspected more closely: the points shown in Fig. 23 of the original paper of Brown show a wide scattering and obviously defy pre-

Fig. 13—Comparison of Pocahontas, Upper and Lower Freeport Coals, Expansion versus Volatile Matter, according to Brown.
sentation of a curve in a two-dimensional system. Bauer (8) commenting on Brown's results with Lower Kittanning coals states that "a continually decreasing curve could be drawn having no greater average deviation than Brown's multiple-valued function; this curve would depart from linearity in the interval 20 to 30 percent."

It is our opinion that expansions, contractions, or pressures observed in coking experiments depend upon more than one variable. Brown states that, "there is considerable variation in expansion or contraction of coals from the same or different seams, therefore, all coals should be tested." This statement also holds for pressure measurements; e.g., in a study of coals from the Lower Kittanning Seam we found an increase of maximum pressure with a decrease of volatile matter, although without exceptions.

Inherent (or added) mineral matter, fusain, and moisture are factors that decrease the swelling pressure according to their amount.

2. Preparation of Sample—Prior to carbonization, coking coals are often subjected to preparatory treatment such as cleaning, crushing, sizing, mixing, and drying. As a result of these operations such variables as bulk density, size distribution, moisture content, ash content, and petrographic composition are subject to changes which may influence the pressures developed upon subsequent carbonization.

The bulk density of the charge is an important variable in determining the pressure developed during carbonization of a given coal or blend. An increase in bulk density causes a rise in pressure. This point has been emphasized by Koppers and Jenkner (31), Fig. 14, and it has been the experience of all investigators regardless of the methods used for the study of the phenomena. The work of Brown (14) shows that the same relationship exists between bulk density and expansion under a load. Altieri (3), in a study of a coal blend with 30 per cent volatile matter, and an expanding low volatile coal found that the lateral expansions increased about 1 per cent for each pound increase of bulk density between 54 and 62 pounds per cubic foot. In discussing some practical aspects of this relationship, Russell (46) suggested that the tests be performed at the highest bulk density that may be presumed to exist in the oven. He believes that the bulk density of about 54 pounds per cubic foot should be the minimum value for use in test procedures, because this seems to be a fairly uniform bulk density characteristic of bone-dry coals.

Bauer (8) pointed out that it is necessary to distinguish between swelling pressure, which is a force per unit area, and the extent of swelling, which is a displacement, or change in volume. On the basis of the assumption that the total increase in volume is directly proportional to the initial true volume occupied by the coal particles, he derived the equation:

\[ \text{Per cent expansion} = -a + b (\text{Bulk Density in the oven}) \]

In this equation the signs of the constants a and b are predicted but their magnitudes depend on conditions.

Auivil and Davis (6) assumed that for purposes of calculating the effect of bulk density on expansion, a cubic foot of actual solid coal under any given set of conditions would expand a fixed amount regardless of the density of the charge. Later, Auivil, Davis, and McCartney (7) gave another form of this assumption: namely, that the expansion obtained in the oven is the algebraic sum of the voids in the charge and the external expansion. They present an equation:

\[ E_s = V_s (1 + E_r) - 1 \]

where \( E_s \) = actual solid coal expansion,
\( V_s \) = total volume of charge in cubic feet per cubic foot of solid coal at test conditions,
\( E_r \) = fractional expansion observed in the test.

When the assumption holds exactly, that is for an ideal coal, \( E_s \) will be a constant where the bulk density is the only variable, and the values would fall on a horizontal straight line upon plotting solid coal expansion \( E_s \) against bulk density. "It will be seen that
all coals depart somewhat from ideal behavior and that the direction and magnitude of the deviation varies with the coal. These deviations are to be expected because the thermal conductivity of the charge varies with the bulk density and the change in conductivity of the uncoke and charge adjacent to the plastic layer will influence the thickness and rate of heating in the layer and consequently affect the expansion of the coal.

The authors present evidence to show that solid coal expansion varies with bulk density only through variations caused by induced changes in the rate of heating and conclude that the extent of solid coal expansion $E$, is essentially the same whether it takes the voids in the charge or expands externally. On the basis of experimental evidence, it is said that “probably all coking coals are expanding and any statement as to the shrinkage of a coal is meaningless unless conditions of carbonization are carefully specified.”

It is further shown that by the use of the concept of solid coal expansion the effects of inerts such as fusain and coke breeze may be calculated; the effects of mineral matter and moisture cannot be computed in the same manner because these “would affect the heating in the charge and thus create secondary effects.”

It appears that more than one equation may be designed to fit the data; e.g., Brown has published several curves showing the relationship between bulk density and expansion, and between expansion and applied pressure. We found that the relationship between expansion and applied pressure may be represented by an equation of the type:

$$\log \text{Load} = a \log \text{expansion} + \log \text{constant}$$

Apparently, most of the workers in the field seem to believe that the bulk density in the oven, though perhaps not uniform throughout, remains unaffected in the process of carbonization. This does not imply that the bulk density of the coal charge is uniform in the coke oven after charging. It has been shown by several workers that there is an increase of bulk density under the charging holes and an increase down from the top of the coal in the oven due to packing by the leveling bar. However, Altieri (4) has clearly recognized that during “carbonization in coke ovens there is a tendency toward consolidation and compression increasing the bulk density of the charge, thus tending to increase side thrusts.” In small-scale experiments this statement should be modified by a consideration of the possible effects of changes in volume; otherwise, we are in substantial agreement with Altieri.

The size distribution of a charge seems to have a bearing on the coking pressure only insofar as it affects the bulk density.

Before carbonization, many coking coals are subjected to a cleaning operation. If that operation would result only in a decrease of mineral matter, and fines which contain fusain, then the main effect would be an increase in coking pressure proportionate to the amount of inert material removed. However, there are other counteracting factors, such as decrease of bulk density, or slight oxidation, which would tend to decrease the coking pressure. Therefore, the net overall effect of cleaning cannot be predicted, unless a definite pronounced change in petrographic composition is accomplished. A brief discussion of this topic follows:

Lehmann and Hoffman (36) have designed a whirling mill for the technical dressing of coal which accomplishes a rough petrographic separation. They have shown that after treatment in their whirling mill a Ruhr gas coal had such an enrichment of the “bright” coal constituent that the resulting coke gave an increase from 60 per cent to 80 per cent (4 mm screen) in drum resistance as specified by the German Syndicate.

It is recalled that, generally speaking, “bright” coal is the best coking coal, “dull” coal is a rather poor coal for carbonization, and fusain will not coke at all. Most coals, used in the production of by-product coke are subjected to some sort of cleaning which usually results in an enrichment of the “bright” coal. Such a cleaning procedure is illustrated in the flow sheet in Fig. 15.

These enriched coals, as well as hand-picked isolated petrographic constituents, have been the object of extensive scientific study.

According to Seyler (47) most vitrains become fluid at relatively low temperatures while most durains do not. Durains are composed of fine granular masses of opaque materials which approximate fusain in composition and may act as inerts. As such, they allow free escape of gases and do not swell.

Van Ahlen (53) has shown that “bright” coal has a lower softening point than “dull” coal and that fusain will not soften at all if these components are computed on the basis of the same degree of coalification. This is in line with the work of Seyler. Van Ahlen further states that the decomposition temperature of the bitumina are different as they increase from “bright” coal to “dull” coal and fusain. However, in Seyler’s view it is a “fundamentally erroneous conception that coke is a briquet formed by a cementation of an infusible part of the coal by a fusible component extruded from the particles and acting as a ‘binder.’”

Brückner and Greve (15) and Brückner and Ludewig (16) have stated that vitrains show a much greater expansion or degree of free swelling than durains. In mixtures, however, experimental results do not correspond to the rule of mixtures, but show con-
siderable deviation therefrom because of mutual physical and chemical reactions. It seems from their data that the "bright" coals studied seemed to flow together resulting in a coherent plastic layer while the "dull" coals did not. It would also seem from their results, on the basis of the Foxwell test for resistance to passage of gases, that the "bright" coals offered much more resistance than the "dull" coals.

Brewer, Holmes, and Davis (13) studied the influence of petrographic composition on the plastic properties of American coking coals. Six representative bituminous coking coals and thirteen characteristic layer samples of these coals were investigated. The fusion temperatures increased with the rank of the coal, but the relative order within a given coal series varied with the predominating type of coal in the given sample. The relative order of decrease in "expansion" by the Aggle-Damm dilatometer test for a typical low-volatile coal series was from bright coal layer to the mixed sample of the entire column to splint coal layer.

There is an objection to this type of work. Methods for the quantitative determination of fusain (25) have been developed; satisfactory quantitative methods for the determination of other petrographic constituents are not available; therefore, a quantitative correlation of the changes of coking pressure concomitant with changes of petrographic composition cannot be accomplished as yet. It should be clear that the effect of coal preparation on coking pressure will only be fully understood when research on the constituents of coal has progressed from the qualitative to the quantitative stage.

3. Construction and Operation of Ovens—It is a common belief that no pressure is exerted against the walls in beehive coke ovens, and it has been repeatedly emphasized that it was only with the advent of the modern by-product oven that pressures developed which might be dangerous to the ovens. These pressures may be influenced either by the construction or by the operation of the oven.

The rate of heating which is an important factor in the development of pressure against the walls of the coke oven may be influenced in several ways; (1) by a change in the design of the oven such as, (a) increasing or decreasing the width, (b) changing the thickness of the wall, or (c) changing the heat conductivity of the walls; (2) by a change in the operation of the oven such as, (a) an increase or a decrease of the flue temperature, (b) a change in the coal or type of blend being coked, or (c) by a change in the method of charging the oven. A decrease in the rate of heating which may be effected as above indicated will result in a lowering of the pressure against the walls. Brown (14) has recognized that "a coal carbonized at low temperature and long coking time may be dangerous when coked at higher temperatures." Even more elucidating are the following remarks of Mordecai (24)—"My experience over many years in the manufacture of high-grade foundry coke indicates that such coke must not only be strong as
indicated by mechanical tests, but also must be large in size, and must have small pores. Such a coke is made from Durham coals containing 24 to 26 per cent volatile matter, usually termed 'strongly coking,' and reputed by some laboratory tests to be in the class 'dangerous swelling.' The conditions for manufacture have proved best at (1) high moisture in coal, say 13 per cent, and (2) slow rates of heating, say carbonizing a 21-inch oven in 40 hours. Under these conditions I have never encountered dangerous swelling pressures in practice."

In the light of all this, it is to be expected that even with a laboratory oven the change of a detail of construction will result in a different numerical value for the pressure developed. This conclusion is fully borne out by experimental evidence.

Furthermore, a close inspection of the Fuchs' equation (26),

\[ P = \frac{\alpha}{\beta} K (T_0 - T_r) - \frac{1}{\beta} (V_{\text{max}} - V_{\text{min}}) \]

indicates that all values between zero and \( \frac{\alpha}{\beta} (T_0 - T_r) \) may be expected, since \( k \) may vary between zero and 1 depending upon the temperature drop in the plastic layer, and since no experimental arrangement exhibiting the essential features of a commercial coke oven can possibly permit the volume of the plastic layer to be kept constant.

**VII Experimental Results**

**Coal Samples Investigated:** A description of the source, special method of selection, and analysis of the samples used in this investigation is presented in Table II. The samples were prepared for use in the pressure-measurement-ovens by crushing to pass a \( \frac{1}{4} \)-inch mesh. A.S.T.M. standard procedure was followed to obtain samples for analytical determinations.

**Typical Pressure Curves:** Fig. 16 shows three typical pressure curves obtained with a Pittsburgh-Lower Kittanning coal blend 80:20 (by weight) upon carbonization at 1100°C in the 2-inch, 3-inch, and \( \frac{1}{4} \)-inch ovens. Similar curves were obtained with other blends and coals. It is readily seen that an increase of oven width causes a large decrease of the pressure developed during carbonization. It is obvious that an operator, judging on the basis of the shape of the time-pressure curve, might classify the blend as very dangerous from its performance in the 2-inch oven, as quite dan-
Fig. 16.—Pressure Measurements on a Blend of 80 per cent Pittsburgh and 20 per cent Lower Kittanning Seam Coals in the 2-, 3- and 4½-inch Ovens. 

...from the curve obtained in the 3-inch oven, and as safe, from the results in the 4½-inch oven. It is evident, therefore, that in order to assay an unknown coal for possible oven damage due to coking pressure the result obtained in any given testing device can only be evaluated by reference to the results obtained under identical conditions with a standard coal or blend of known behavior in large-scale production.

Fig. 17 shows pressure curves that have been registered on a continuous recording instrument upon carbonization in the 4½-inch oven of three different binary blends (composed of the above-mentioned Pittsburgh and Lower Kittanning coals). The fluctuations of the top curves due to alternate drop and rise of pressure are noteworthy. This lends additional support to the idea that coking pressure is a dynamic phenomenon, and not a static one.

In Figs. 16 and 17, the top curves show certain features which are easily recognized with most experimental curves. The pressure rises quickly to a value from 20 to 50 per cent of the observed maximum, remains fairly constant for the greater part of the run, rises to a quickly transient maximum, and then drops sharply. These curves may be explained on the basis of phenomena in the plastic layer.

(a) The pressure rises from zero to a certain value: the plastic layers are being formed.

(b) The pressure remains fairly constant at the value reached sub (a): the plastic layers travel through the charge with a constant temperature gradient.

(c) The pressure rises to a maximum: the plastic layers meet in the middle of the oven, their average temperature rises sharply to a maximum.

(d) The pressure drops: conversion of the plastic layer into coke is completed.

The pressure curves presented in Fig. 18 refer to measurements with coal No. 7 in the 3- and the 4½-inch ovens. These curves deviate from the more frequently encountered curves as discussed above in that they show a continuous rise of pressure with time. This type is often met with in low-volatile, high-pressure coals. An explanation may be based on the assumption that in these cases, the advancing plastic layers are meeting with steadily increasing resistance, which may be ascribed to an increase of charge bulk density with time.

**Duplicability of Maximum Pressures:** Besides keeping constant those factors which relate to the nature of coal or blend, the sampling, and the oven, the following conditions were found to require attention:

(a) The coking time. It should be the same in duplicate runs.

(b) The ratio of coal charge volume to coke volume. This factor was found to be important in our earlier work. A method was devised for estimating the volume charge taking place upon car-
bonization. Experimentally, this method consists in establishing the differences between the original volume of the coal charge and the final volume of the coke. The original volume is determined from weight and bulk density of the coal charge, and the final volume by filling the coking chamber after completion of the carbonization with coke breeze sized between 3 and 6 mesh. The final volume was usually larger than the initial volume. In this method, the possible contribution of cracks to the final volume is disregarded; it should be noted, however, that coke of the size selected for the test will not fall into areas opened by cracks. The slight movement of the oven wall, which may amount to as much as 1/16 of an inch, is also neglected.

An example of the calculation is presented:

1. Bulk density of coal, g per cc = 0.750
2. Coal charge, g = 1580
3. Coal charge, cc = 2110
4. Bulk density of 3-6 mesh coke, g per cc = 0.373
5. Coke needed to fill the oven, g = 1280
6. Oven volume, cc = 3430
7. Volume of space above coal charge, cc = 1320
8. Coke needed to fill empty space after run, g = 395
9. Volume of space above coke charge, cc = 1060
10. Volume change, (7)-(9), cc = 260

A volume change of 260 cc is considerable and clearly visible upon inspection.

These data obtained in the 2-inch oven are easily evaluated if the volume change is either negligible or constant, and if no plastic mass flows out of the oven during the run. Under these circumstances closely checking results may be obtained. As an example, data are presented descriptive of three determinations carried out with a blend composed of 80 per cent Pittsburgh coal and 20 per cent Lower Kittanning coal in the 2-inch oven.

### TABLE III

<table>
<thead>
<tr>
<th>Change of Pressure with Time in the 2-inch Oven</th>
</tr>
</thead>
<tbody>
<tr>
<td>(Pittsburgh-Lower Kittanning Blend 80:20)</td>
</tr>
<tr>
<td>Time, Minutes</td>
</tr>
<tr>
<td>0 10 20 29 30 31 32 35 36 147 139</td>
</tr>
<tr>
<td>Gauge Reading I</td>
</tr>
<tr>
<td>15 77 120 160 200 260 250 147 139</td>
</tr>
<tr>
<td>Gauge Reading II</td>
</tr>
<tr>
<td>15 90 110 235 245 220 185 120 110</td>
</tr>
<tr>
<td>Gauge Reading III</td>
</tr>
<tr>
<td>15 58 85 145 260 245 220 140 130</td>
</tr>
</tbody>
</table>

Data necessary for estimating the volume changes that have taken place in the experiments recorded in Table III are compiled in Table IV.

### TABLE IV

<p>| Data Descriptive of Volume Changes             |
| (Experiments of Table III)                    |
|                                               |</p>
<table>
<thead>
<tr>
<th>I</th>
<th>II</th>
<th>III</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bulk density of coal, g per cc</td>
<td>0.763</td>
<td>0.765</td>
</tr>
<tr>
<td>Coal charge, g</td>
<td>1580</td>
<td>1580</td>
</tr>
<tr>
<td>Coal charge, cc</td>
<td>2070</td>
<td>2065</td>
</tr>
<tr>
<td>Oven volume, cc</td>
<td>3430</td>
<td>3430</td>
</tr>
<tr>
<td>Bulk density of 3-6 mesh coke, g per cc</td>
<td>0.445</td>
<td>0.448</td>
</tr>
<tr>
<td>3-6 Mesh coke charged after run, g</td>
<td>530</td>
<td>420</td>
</tr>
<tr>
<td>Space above coal charge, cc</td>
<td>1360</td>
<td>1365</td>
</tr>
<tr>
<td>Space above charge after run, cc</td>
<td>1191</td>
<td>1160</td>
</tr>
<tr>
<td>Volume change, cc</td>
<td>169</td>
<td>205</td>
</tr>
<tr>
<td>Maximum gauge reading</td>
<td>260</td>
<td>245</td>
</tr>
<tr>
<td>Lbs per sq in. of oven wall</td>
<td>8.1</td>
<td>7.7</td>
</tr>
</tbody>
</table>

For the 2-inch oven the conclusion may be drawn that in case of identical change in volume, identical pressure readings may be expected for a certain coal or blend; and furthermore, that increased volume will cause a decrease of coking pressure, and
vice versa. As the oven width was increased, this relationship became less pronounced; and in the 4½-inch oven differences in volume change seemed to be without influence upon the duplicability of results.

**Maximum Pressures:** In Tables V, VI and VII, maximum pressures (in lbs/in² of oven wall) as obtained in the 2-inch, 3-inch, and 4½-inch ovens are tabulated. Tests in the 2-inch oven required the shortest time; in this case, coking was completed in 35 to 40 minutes as compared to 60 minutes in the 3-inch oven and 90 minutes in the 4½-inch oven. The pressure values recorded in the 4½-inch oven seem to represent close approximations to values encountered in practice. The maximum pressure of blends is not generally predictable as the arithmetic means of the constituent coals.

**TABLE V**

**Measurements in the 2-inch Oven**

(Blends of Pittsburgh and Lower Kittanning Coal,
Table No. II, Samples No. 3 and 1)

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Pittsburgh Coal - %</th>
<th>Bulk Density g/cc</th>
<th>Max Pressure Lbs/in² of oven</th>
<th>Volume Change cc</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>100</td>
<td>0.775</td>
<td>48.3</td>
<td>3.4</td>
</tr>
<tr>
<td>2</td>
<td>100</td>
<td>0.775</td>
<td>48.3</td>
<td>3.4</td>
</tr>
<tr>
<td>3</td>
<td>100</td>
<td>0.779</td>
<td>48.5</td>
<td>3.1</td>
</tr>
<tr>
<td>4</td>
<td>100</td>
<td>0.778</td>
<td>48.4</td>
<td>5.3</td>
</tr>
<tr>
<td>5</td>
<td>82.5</td>
<td>0.777</td>
<td>48.3</td>
<td>6.6</td>
</tr>
<tr>
<td>6</td>
<td>82.5</td>
<td>0.780</td>
<td>48.6</td>
<td>8.1</td>
</tr>
<tr>
<td>7</td>
<td>82.5</td>
<td>0.759</td>
<td>47.3</td>
<td>6.6</td>
</tr>
<tr>
<td>8</td>
<td>82.5</td>
<td>0.769</td>
<td>48.0</td>
<td>7.0</td>
</tr>
<tr>
<td>9</td>
<td>82.5</td>
<td>0.778</td>
<td>48.4</td>
<td>9.7</td>
</tr>
<tr>
<td>10</td>
<td>80</td>
<td>0.763</td>
<td>47.5</td>
<td>8.1</td>
</tr>
<tr>
<td>11</td>
<td>80</td>
<td>0.767</td>
<td>47.9</td>
<td>8.1</td>
</tr>
<tr>
<td>12</td>
<td>80</td>
<td>0.765</td>
<td>47.8</td>
<td>7.7</td>
</tr>
<tr>
<td>13</td>
<td>80</td>
<td>0.765</td>
<td>47.8</td>
<td>9.0</td>
</tr>
<tr>
<td>14</td>
<td>75</td>
<td>0.769</td>
<td>48.0</td>
<td>12.0</td>
</tr>
<tr>
<td>15</td>
<td>75</td>
<td>0.769</td>
<td>48.5</td>
<td>13.8</td>
</tr>
<tr>
<td>16</td>
<td>75</td>
<td>0.755</td>
<td>47.2</td>
<td>11.6</td>
</tr>
<tr>
<td>17</td>
<td>75</td>
<td>0.769</td>
<td>48.0</td>
<td>11.4</td>
</tr>
<tr>
<td>18</td>
<td>75</td>
<td>0.770</td>
<td>48.1</td>
<td>13.8</td>
</tr>
<tr>
<td>19</td>
<td>65</td>
<td>0.762</td>
<td>47.5</td>
<td>9.8</td>
</tr>
<tr>
<td>20</td>
<td>65</td>
<td>0.776</td>
<td>48.3</td>
<td>12.9</td>
</tr>
<tr>
<td>21</td>
<td>65</td>
<td>0.759</td>
<td>47.3</td>
<td>13.4</td>
</tr>
<tr>
<td>22</td>
<td>65</td>
<td>0.762</td>
<td>47.6</td>
<td>15.4</td>
</tr>
<tr>
<td>23</td>
<td>60</td>
<td>0.772</td>
<td>48.2</td>
<td>15.3</td>
</tr>
<tr>
<td>24</td>
<td>60</td>
<td>0.768</td>
<td>47.9</td>
<td>15.2</td>
</tr>
<tr>
<td>25</td>
<td>60</td>
<td>0.768</td>
<td>47.9</td>
<td>12.9</td>
</tr>
<tr>
<td>26</td>
<td>60</td>
<td>0.779</td>
<td>48.5</td>
<td>11.3</td>
</tr>
<tr>
<td>27</td>
<td>60</td>
<td>0.781</td>
<td>48.6</td>
<td>14.0</td>
</tr>
<tr>
<td>28</td>
<td>50</td>
<td>0.754</td>
<td>47.2</td>
<td>13.9</td>
</tr>
<tr>
<td>29</td>
<td>50</td>
<td>0.750</td>
<td>46.8</td>
<td>12.2</td>
</tr>
<tr>
<td>30</td>
<td>50</td>
<td>0.743</td>
<td>46.5</td>
<td>13.3</td>
</tr>
<tr>
<td>31</td>
<td>50</td>
<td>0.743</td>
<td>46.5</td>
<td>8.5</td>
</tr>
</tbody>
</table>

**TABLE VI**

**Measurements in the 3-inch Oven**

(Blends of Pittsburgh No. 3 and Lower Kittanning Coal, No. 1)

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Pittsburgh Coal - %</th>
<th>Bulk Density g/cc</th>
<th>Max Pressure Lbs/in² of oven</th>
<th>Volume Change cc</th>
</tr>
</thead>
<tbody>
<tr>
<td>32</td>
<td>100</td>
<td>0.769</td>
<td>48.0</td>
<td>2.4</td>
</tr>
<tr>
<td>33</td>
<td>80</td>
<td>0.777</td>
<td>48.4</td>
<td>3.7</td>
</tr>
<tr>
<td>34</td>
<td>80</td>
<td>0.780</td>
<td>48.6</td>
<td>3.7</td>
</tr>
<tr>
<td>35</td>
<td>80</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>36</td>
<td>80</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>37</td>
<td>80</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>38</td>
<td>50</td>
<td>0.767</td>
<td>47.9</td>
<td>13.5</td>
</tr>
<tr>
<td>39</td>
<td>50</td>
<td>0.756</td>
<td>47.2</td>
<td>13.5</td>
</tr>
</tbody>
</table>

**TABLE VII**

**Measurements in the 4½-inch Oven**

(Blends of Pittsburgh and low volatile coals)

<table>
<thead>
<tr>
<th>Test Number</th>
<th>Low Volatile Coal Per Cent</th>
<th>Sample No.</th>
<th>Bulk Density g/cc</th>
<th>Max Pressure Lbs/in² of oven</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td>0</td>
<td>4</td>
<td>0.661</td>
<td>41.3</td>
</tr>
<tr>
<td>41</td>
<td>0</td>
<td>4</td>
<td>0.779</td>
<td>48.5</td>
</tr>
<tr>
<td>42</td>
<td>20</td>
<td>2</td>
<td>0.754</td>
<td>47.2</td>
</tr>
<tr>
<td>43</td>
<td>20</td>
<td>2</td>
<td>0.783</td>
<td>48.9</td>
</tr>
<tr>
<td>44</td>
<td>20</td>
<td>2</td>
<td>0.778</td>
<td>48.5</td>
</tr>
<tr>
<td>45</td>
<td>30</td>
<td>2</td>
<td>0.775</td>
<td>48.3</td>
</tr>
<tr>
<td>46</td>
<td>30</td>
<td>2</td>
<td>0.762</td>
<td>47.8</td>
</tr>
<tr>
<td>47</td>
<td>40</td>
<td>2</td>
<td>0.736</td>
<td>46.0</td>
</tr>
<tr>
<td>48</td>
<td>40</td>
<td>2</td>
<td>0.754</td>
<td>47.2</td>
</tr>
<tr>
<td>49</td>
<td>50</td>
<td>2</td>
<td>0.761</td>
<td>47.5</td>
</tr>
<tr>
<td>50</td>
<td>50</td>
<td>2</td>
<td>0.757</td>
<td>47.2</td>
</tr>
<tr>
<td>51</td>
<td>70</td>
<td>2</td>
<td>0.770</td>
<td>48.0</td>
</tr>
<tr>
<td>52</td>
<td>70</td>
<td>2</td>
<td>0.771</td>
<td>48.1</td>
</tr>
<tr>
<td>53</td>
<td>100</td>
<td>2</td>
<td>0.749</td>
<td>46.5</td>
</tr>
<tr>
<td>54</td>
<td>100</td>
<td>2</td>
<td>0.750</td>
<td>46.6</td>
</tr>
<tr>
<td>55</td>
<td>20</td>
<td>7</td>
<td>0.742</td>
<td>46.4</td>
</tr>
<tr>
<td>56</td>
<td>100</td>
<td>7</td>
<td>0.765</td>
<td>49.1</td>
</tr>
<tr>
<td>57</td>
<td>100</td>
<td>7</td>
<td>0.779</td>
<td>48.5</td>
</tr>
<tr>
<td>58</td>
<td>100</td>
<td>6</td>
<td>0.789</td>
<td>49.2</td>
</tr>
<tr>
<td>59</td>
<td>100</td>
<td>6</td>
<td>0.797</td>
<td>49.6</td>
</tr>
<tr>
<td>60</td>
<td>100</td>
<td>6</td>
<td>0.790</td>
<td>49.2</td>
</tr>
<tr>
<td>61</td>
<td>20</td>
<td>6</td>
<td>0.781</td>
<td>48.8</td>
</tr>
<tr>
<td>62</td>
<td>20</td>
<td>6</td>
<td>0.784</td>
<td>46.9</td>
</tr>
</tbody>
</table>
Relation of Per Cent Volatile Matter to Maximum Coking Pressure: Table VIII indicates a simple proportionality between the volatile matter of seven coal samples taken at random from the Lower Kittanning seam and the maximum coking pressure observed in the 3-inch oven with blends composed of 80 per cent Pittsburgh coal and 20 per cent Lower Kittanning coal. Each figure represents an average of at least four determinations.

### TABLE VIII

Coking Pressures of 80-20 Pittsburgh-Lower Kittanning Coals in the 3-inch Oven

<table>
<thead>
<tr>
<th>Laboratory Number</th>
<th>Volatile Matter Dry-ash-free basis - %</th>
<th>Mean coking pressure of blend lbs/in.(^2) of oven wall</th>
</tr>
</thead>
<tbody>
<tr>
<td>52</td>
<td>18.6</td>
<td>9.5</td>
</tr>
<tr>
<td>51</td>
<td>18.4</td>
<td>8.4</td>
</tr>
<tr>
<td>55</td>
<td>18.5</td>
<td>7.5</td>
</tr>
<tr>
<td>53</td>
<td>24.3</td>
<td>6.6</td>
</tr>
<tr>
<td>50</td>
<td>27.3</td>
<td>5.3</td>
</tr>
<tr>
<td>56</td>
<td>27.4</td>
<td>4.7</td>
</tr>
<tr>
<td>54</td>
<td>35.6</td>
<td>2.5</td>
</tr>
</tbody>
</table>

A least square treatment of these results gave the following equation:

\[ y = 41.62 - 2.77x \]

where \( x \) = the maximum pressure observed with an 80:20 blend in the 3-inch oven,

and \( y \) = the percent volatile matter of the Lower Kittanning component on a dry-ash-free basis.

Such an equation is attractive by its simplicity but in all probability it does not describe any general relationship. E.g., the Lower Kittanning Seam Coal No. 2, Table II, when carbonized under the conditions of Table VIII gave a maximum pressure averaging only 3.8 lbs per in.\(^2\), while the equation would demand a value of at least 9.0 lbs per in.\(^2\).

Relation of Bulk Density to Maximum Coking Pressure: Our experiences confirm the results of other investigators that the maximum pressure rises with increasing bulk density though the relationship is not a simple one. We have evidence to show that the high volatile component of a blend (usually regarded as harmless) will cause an unexpected increase of the coking pressure if its bulk density is increased.

Summary and Conclusions

1. A survey of the literature concerning pressures developed during the carbonization of coal, and related phenomena, has been presented.

2. Several designs of pressure-measuring devices were studied, and the results indicated that seemingly small changes in construction or operation may occasion large differences in recorded maximum pressures. This conforms with the concept that coking pressure is a dynamic, not a static phenomenon.

3. A laboratory oven has been developed which embodies several novel features. It consists essentially of a rectangular silica brick chamber which will conveniently hold several pounds of coal of a size distribution comparable to that of commercial blends. Heating is effected perpendicular to the side walls, one wall being ostensibly movable so as to transfer the pressure created during coking to the pressure measuring device, the main part of which is a syphon-type bellows connected either to a Bourdon gauge or recorder. With a chamber 4½ inches wide and with a flue temperature of 1100°C, results are obtained of the same order of magnitude as in practice.

4. Duplicate experiments in the 4½-inch oven may be expected to check better than ± 10 per cent.

5. It is believed that laboratories engaged in assaying coals will find this new oven valuable for predicting coking pressures. The coke obtained in these tests may be used for further determinations related to the quality of the coke.
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