

Coal as Raw Material for Carbon Production: Some New Aspects [and Discussion]

S. Jasienko, G. M. Kimber and J. W. Patrick

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Coal as raw material for carbon production: some new aspects

By S. Jasieńko

Institute of Chemistry and Technology of Petroleum and Coal, Technical University of Wrocław, Wrocław, Poland

[Plates 1 and 2]

Characteristic changes in the constitution of hard coals (such as the nature and abundance of functional groups, the degree of order and the development of capillary structure) that occur in coalification and carbonization processes have a profound bearing on the rational utilization of coal in the coke and carbon industries. For blastfurnace coke manufacture the selection of blend components and their influence on the development of coke structure are very important. Modification of coke structure by additions of bituminous substances of coal and petroleum origin to blends opens up the possibility of preparing synthetic coking coal. In formed coke production, the nature of coal and binder and their distribution within the mixture are significant. The use of hard coals and their extracts and pitch as raw materials for improved carbon manufacture are discussed in terms of graphitizability. The adsorptive and mechanical properties of active carbons are dependent upon coal constitution and rank. It is concluded that the rapidly developing new technologies of coal utilization require a more elaborate classification system that would include the more important structural parameters of coal constitution.

1. Properties and structure of coals

The changes appearing in the structure of hard coals in coalification from flame coal to anthracite have great importance for their rational utilization in all technological processes and particularly in the technologies of coke and of production of improved carbons.

In the coalification process from flame coal to anthracite, the content of ordered structure increases, the less ordered arrangements decrease. The dimensions of aromatic lamellae, the degree of their packing and number of aromatic lamellae in groups increases, following the increase in the ordering of lamellae groups to each other. The content of reactive oxygen groups methoxyl, carboxyl, carbonyl and hydroxyl – decreases and simultaneously the bridge connections disappear. The ratio of thioether sulphur groups with respect to those of thiophene decreases from 3:1 in flame coals to 1:6 in anthracites (Roga et al. 1955; van Krevelen 1961).

The cokes of low-rank coals are non-graphitizing. The inability to graphitize is the result of the small dimensions of the stacks of aromatic layers, their poor mutual orientation and the existence of cross links.

Cokes from coking coals are graphitizing. These cokes have suitable dimensions and orientation of the aromatic layers and high mobility of the structure.

The cokes obtained from anthracite heated below 2300 K behave as non-graphitizing substances, because of the cross links and high porosity, and at temperatures above 2300 K after thermal decomposition of the cross links the parallel orientation of stacks of layers leads to their fast growth, producing a graphitic structure (Franklin 1951).

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The internal surface and porosity of coals changes very characteristically in the coalification process, showing the lowest values for medium-rank coals and assuming high values for low-rank coals and anthracites.

Characteristic changes of properties and structure of petrographic components are also observed. The component with the lowest degree of coalification is exinite, which is distinguished by the largest content of volatile matter, hydrogen and non-aromatic hydrocarbon groups, and by the poorest ordered structure. Vitrinite is characterized by a large quantity of aromatic CH groups and oxygen compounds. The participation of non-aromatic arrangements in petrographic components of a given type of coal decreases from exinite \rightarrow vitrinite \rightarrow fusinite and also diminishes with the increase of their degree of coalification, particularly for vitrinite and exinite. Fusinite differs markedly in structure from the remaining petrographic components in showing an insignificant content of characteristic functional groups and being built of large aromatic lamellae. It does not change substantially in the process of coalification (Kröger 1957; Jasieńko 1965; Kidawa 1976).

In my paper I should like to present some new problems connected with the possibility of utilization of hard coals in the coking industry and improved carbons industry, mainly based on the results obtained by our research group in Wrocław.

2. COAL AS A RAW MATERIAL FOR THE COKE INDUSTRY

Coke making is still the basic industry in coal processing. In 1977 the world production of coke reached 373 Mt, using 540 Mt of coal and yielding as by-products 0.5 Mt of crude benzole, 1.9 Mt of tar and 190 km³ of gas. The source of raw materials and technologies of the coking process have changed but many problems remain.

Roga (1950) said: 'Two tendencies may be observed in the coking industry: a constant increase in the production of coke due to the vigorous development of the industry (especially in the metallurgical field), and a steady increase in the requirements of the quality of coke. The coke industry is thus consuming more and more coal, in spite of the fact that the percentage of output of coking coals is decreasing.'

The progress in the last 20 years in research on the formation of coke structures obtained from different types of coal and of coals with addition of bituminous substances may have important significance when elaborating new production technology of the coking process.

From these achievements I should like to mention as the most important the recognition of types of anisotropic structure appearing in cokes obtained from coals, in the whole coalification scale, and determination of the relation of coke structures to the coking properties and structure of coals (Ramdohr 1928; Mackowsky 1955; Alpern 1956; Taylor 1961; Jasieńko 1963, 1965; Patrick et al. 1973).

Transition zones appear in the coke from blends of gas coking coals with orthocoking coal and metacoking coal. The observation that the cokes obtained from primary coking coals have the best degree of structure ordering is significant in determination of the nature of this group of coals (Jasieńko 1963; Kidawa 1976) (figure 1).

The formation of structural areas that exhibit optical anisotropy in the coking of coals with addition of bituminous substances seems to be very important for the development of new methods for production of metallurgical coke.

An important development in this field of research is the work of Sanada (1978), Mochida &

Marsh (1979) and others. An introduction of a bituminous substance (petroleum pitch, pitch, solvent refined coal) of adequate properties to hard coals during carbonization causes a modification of the optical texture of the resultant cokes (Grint et al. 1979). The mechanism of modification of the optical texture of resultant cokes is related to the formation of nematic liquid crystals, mesophase and semicoke. The choice of bituminous substances corresponding to each type of coal is very important. Both the compact and capillary structures have an influence on behaviour. A substance of petrochemical origin with a high content of hydrogen or the product of chemical disintegration of the coal exerts a more advantageous influence than typical substances of carbochemical origin (pitches, extracts-products of physical disintegration). Low molecular mass substances also have an important influence on the formation of anisotropic structure of the cokes (Jasieńko & Gerus-Piasecka 1978).

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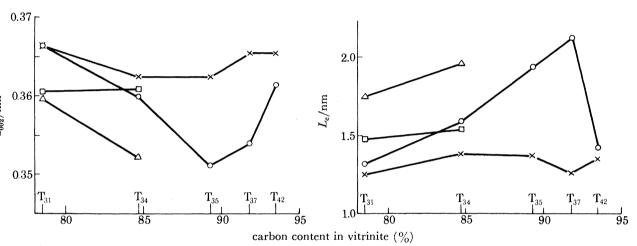


Figure 1. Structural parameters of cokes from hard coals of different rank (Jasieńko & Kidawa 1977). T₃₁, flame coal; T₃₄, gas-coking coal; T₃₅, orthocoking coal; T₃₇, semicoking coal; T₄₂, anthracite; O, coke of vitrinite; A, coke of exinite; D, coke of micrinite; X, coke of fusinite.

The full recognition of the beneficial effects of bituminous additives on the formation of the anisotropic structure of cokes may constitute a fundamental advance in the utilization of lower-rank coals as components of the blends.

In the investigations on the influence exerted by high and lower-rank additives, we found that there is a very advantageous influence of semicoking coals showing some plasticity and also of finely ground low-rank coal, when using orthocoking coals with an increased content of inertinites as enriching components.

In the process of coking, the components that exert the most important influence on the properties and structure of cokes are the vitrinites and exinites. The exinite, even in lower-rank coals, shows an agglutinating ability. Micrinites should be considered as poorly coking components, softening only at grain edges; fusinites are entirely inert components (Jasieńko 1965; Taylor et al. 1967).

Not only are the content and properties of petrographic components very important, but also their distribution in the coal sample. The components should be comminuted and uniformly distributed. A high concentration of inertinites in small areas has a disadvantageous influence on the coking properties.

3. METALLURGICAL FORMED FUELS

In the last 10 years, much research on the development of new technologies of metallurgical formed fuels has been carried out.

These investigations have been initiated for the following reasons:

- (i) necessity of extension of coal material resources for the production of blast-furnace coke, owing to dwindling reserves and unequal distribution of coking coals;
 - (ii) environment protection, difficult to put into practice in an intermittent coking process;
- (iii) social aspects: difficult working conditions during coke production by the conventional method.

The methods for production of formed cokes are based on the following principles.

The first group of methods is based on low-temperature carbonization of low-rank coals, carried out to obtain semicoke and a bituminous substance (low-temperature tar), briquetting of the semicoke with the bituminous substance (fraction of own low-temperature tar) as a binder and further oxythermal or thermal treatment of the briquettes.

The second group of methods involves carrying out the process in conditions that prevent thermal decomposition of the bituminous substance until transition into the plastic stage, followed by hot briquetting of the formed plastic mass and a further thermal treatment of the briquettes.

The third group of methods is based on the introduction of a foreign bituminous substance as a binder for the formation of formed fuels from non-coking coals. As foreign bituminous substances, coking coals in the hot briquetting process, bituminous substances of petrochemical (petroleum residue) or carbochemical origin (pitch, solvent refined coal) can be used.

Analytical and structural examinations (microscopical and X-ray) show that, in comparison with conventional coke, formed cokes always have a higher content of volatile matter, moisture and hydrogen, lower carbon content, lower development and inhomogeneous distribution of pore systems and a considerably lower degree of structural arrangement (Jasieńko & Bujnowska 1979).

Considerable structural heterogeneity is one of the main characteristics of formed cokes. As opposed to conventional cokes, formed cokes contain numerous different types of structure depending on the kind of raw materials used and the technological process (Bujnowska & Jasieńko 1979).

Petrographic composition of input coals has an essential influence on the quality of formed cokes. High contents of vitrinite and exinite and their uniform distribution in coal create the possibility of formation of a formed coke with a homogeneous structure. Formed cokes obtained from low-rank coals with high exinite content have a relatively homogeneous structure, their pore system is better developed and uniformly distributed owing to better plasticizing of the coal substance during heat treatment, and their compact substance shows optical anisotropy. High exinite content in low-rank coals makes it possible to obtain formed cokes with properties and structures similar to conventional cokes. Conversely a high content of inertinite needs a higher quantity of binder.

By using a suitable base coal and binder and by controlling the technological process, it is possible to obtain formed cokes of a high homogeneity showing optical anisotropy on large areas of the structure, approaching in their structure and properties good metallurgical cokes. In our research we stated that the formed cokes of a high homogeneity are obtained by the F.M.C.

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method and a structure approaching that of classical coke by the Lurgi-Bergbau Forschung method. Use of the coke produced by these methods in a blast furnace gave satisfactory results.

4. COAL AS A RAW MATERIAL FOR PRODUCTION OF CARBON AND GRAPHITE

The pioneer works of Blayden et al. (1944) on the carbonization process of solid fuels and their group components, and the fundamental works of Franklin (1951, 1953, 1956) about the susceptibility of organic substances to graphitization, opened a new way for utilization of hard coals in the carbon and graphite industry. Some important considerations on the subject 'carbon from coal' were given by Walker (1972) and Owen (1975, 1979).

I believe that our work on the graphitization of petrographic components also introduced some new elements in the development of carbon science.

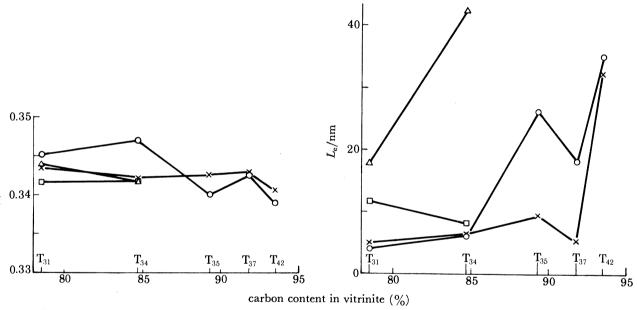


Figure 2. Structural parameters of graphitization products (2773 K) of petrographic components of hard coals (Jasieńko & Kidawa 1977), symbols as figure 1.

4.1 The graphitizability of petrographic components of coals

The cokes obtained from the main petrographic components, vitrinites, exinites, micrinites and fusinites, show different graphitizability (Jasieńko & Kidawa 1977).

The graphitizability of coke from vitrinites increases with the rank of coal from flame coal to primary coking coals. Vitrinite cokes of low-rank coals are non-graphitizing, whereas those orthocoking coals show very high susceptibility to graphitization. The graphitizability of cokes from vitrinites of anthracites is also high within the temperature range 2300-2800 K. A very high graphitizability is shown by cokes from exinites, even those from gas-coking coal. The cokes obtained from micrinites and fusinites of coals of different rank are non-graphitizing (figure 2).

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These results are a logical consequence of the structure of coals and petrographic components and they also give information about the possibility and method of utilizing hard coals in industry for improved carbons.

Primary coking coals having a low content of mineral substances and inertinite, with added reactive bituminous substances – post-distillation residues of crude oil, coal extracts or pitch – may constitute raw material for production of pitch-type cokes.

The quality of anthracites used in the electrode industry may be considerably improved by removing not only the mineral substances but also the inert petrographic components.

Vitrinites of low-rank coals and the carbonization products obtained from them may be considered as raw materials for glassy carbon production.

4.2. Properties and structure of coal extracts as raw materials for the electrode industry

The depolymerization of the organic matter of hard coals induced by hydrogen-donor solvents under pressure at temperatures close to thermal decomposition of the coals, studied by Pott-Broche several years ago, recently became the subject of intensive studies in numerous countries including our own. The basic aim of these investigations is the preparation of coal extracts useful for hydrogenation into liquid fuels and as raw materials for the carbon industry.

The research performed in the Institute of Chemistry and Technology of Petroleum and Coal, Technical University of Wrocław, in cooperation with the Institute of Carbochemistry of the Main Mining Institute, contained recognition of the carbonization and graphitization processes of extracts from coals of different ranks, their group components, mesophase formation during carbonization of the extracts, and determination of properties and structure of solid carbonization and graphitization products.

Briefly, extracts obtained from hard coals by using degrading and reactive solvents can be considered as raw materials for the carbon and graphite industry. The carbonization products of extracts have a low content of ash and sulphur, low content of heavy metals and α₂ component, low oxidation susceptibility, high mechanical properties and low puffing. Negative features of the coal extracts are low content of fibrous phase in the cokes, high thermal expansion and lower graphitizability (Jasieńko & Biegańska 1979 b, Rusin & Potyka 1978; Hermanowska & Fica 1978).

The possibility of obtaining extracts from different ranks of coal showing different graphitizabilities is certainly advantageous, and these extracts cannot be overlooked as a raw material for the carbon and graphite industry.

Extracts from low-rank coals can be used for impregnation, extracts from gas coal as binders and for coke production, extracts from gas-coking and orthocoking coals as binders and for production of coke of fibrous and needle structures.

The results of investigations up to now show that extracts from coking coals have the best properties as a raw material for the graphite industry (Jasieńko et al. 1975).

The extracts from petrographic components differ in group composition; it therefore seems that extracts obtained from coals enriched in vitrinite content will show better and more homogeneous properties, which may be important in using coal extracts for fibre production.

4.3. The graphitizability of pitch group components

Pitch is a very important raw material in the carbon and graphite industry, where it is used as a binder for pitch coke production and for impregnation. The structure of cokes formed from

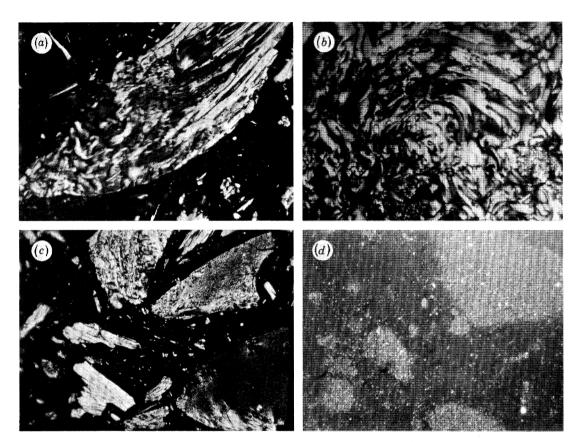


FIGURE 3. Optical micrographs of cokes obtained from pitch group components (Jasieńko et al. 1977 b). Polarized light, magn. ×250, crossed nicols. (a) Coke from γ component; (b) coke from β component; (c) coke from α₁ component; (d) coke from α_2 component.

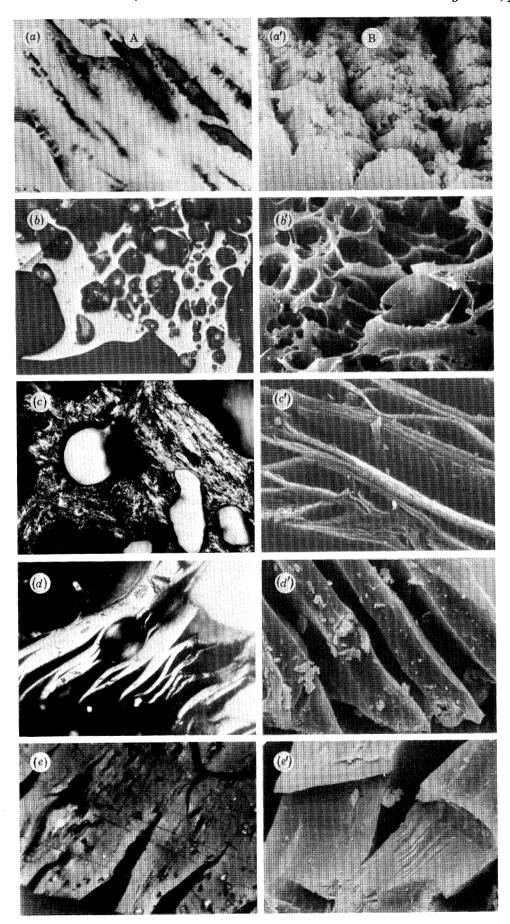


FIGURE 6. For description see opposite.

pitches is very important, particularly in connection with their susceptibility to graphitization and their mechanical properties. In an appraisal of the pitches as raw materials for the electrode industry, studies of the properties and structure of the group components are very useful.

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The group components of pitches show large differences in properties and structure. The γ component has a hydroaromatic structure and the lowest degree of structure ordering. The β component is characterized by a more aromatic structure, a higher degree of structural ordering and good caking properties. The component α_1 is distinguished by a slightly higher degree of aromatization and of structure ordering, but it has a lower caking power. The α_2 component shows the most aromatic and condensed feature of the structure but it does not have caking power (Karabon & Więcek 1975; Jasieńko *et al.* 1977*a*).

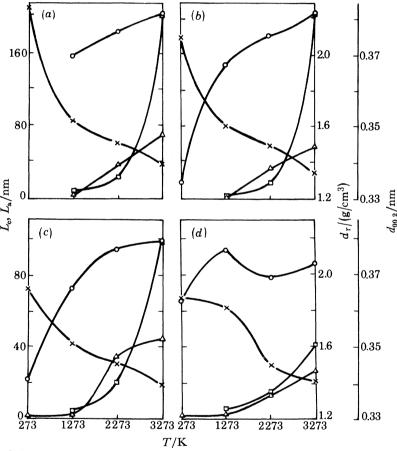
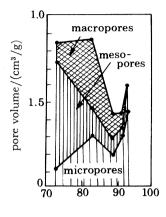


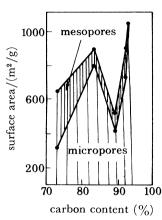
FIGURE 4. Changes of the structural parameters and density of coke from pitch group components in the graphitization process: (a) coke from γ component; (b) coke from β component; (c) coke from α_1 component; (d) coke from α_2 component. \Box , L_a ; \triangle , L_c ; \bigcirc , d_r ; \triangle , d_{002} (Jasieńko et al. 1977 b).

DESCRIPTION OF PLATE 2

Figure 6. Micrographs of activation products of vitrites from hard coal: 75% burn-off (Biegańska & Jasieńko 1979). A, Optical microscopy (magn. ×250); B, s.e.m. (magn. ×1040). (a, a') Vitrite from flame coal (73.3% C d.a.f.); (b, b') vitrite from gas coal (83.1% C d.a.f.); (c, c') vitrite from orthocoking coal (89.2% C d.a.f.); (d, d') vitrite from semicoking coal (91.7% C d.a.f.); (e, e') vitrite from anthracite (93.1% C d.a.f.).

The relative amount and the properties and structure of group components have an important influence on the properties of the resulting pitch. Pitches are, however, complex colloidal systems and their properties depend also on the spacing and association of the group components.





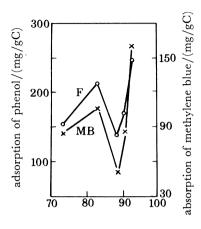


FIGURE 5. The parameters of capillary structure of activation product of different rank vitrites (Jasieńko & Biegańska 1979a).

In the carbonization and graphitization of pitch, the group components display a marked difference in the level of ordering in the structure (Jasieńko et al. 1977 b). The susceptibility to structural ordering and the degree of graphitization of products obtained at 3273 K is, in increasing order: component α_2 , component α_1 , pitch, component γ , component β . The real density increases in a similar way. For the α_2 component the curve of the relation of the real density to temperature shows the appearance of a minimum at 2273 K. Taking the susceptibility to graphitization as the basis, the group components of pitches may be divided into two classes of a high and low susceptibility to graphitization. The cokes of pitch, γ , β and α_1 components belong to the class of well graphitizing substances, while the coke of α_2 component shows a much lower susceptibility to graphitization.

It seems that the optimum conditions for obtaining a highly graphitizable coke are a high content of β and γ components and of the α_1 component, which acts as a filling material, and a sufficiently low content of α_2 component.

The composition of the blends used in the coking process, technology of coking and pitch preparation, seem to be very important for properties of the resulting pitch. Blends with a high content of coking coals (rich blends) may be the source of tar for production of pitches with good properties for the graphite industry.

5. The properties and structure of activation products obtained from vitrites of coals of different rank

The characteristic changes of capillary structural parameters (density, porosity, pore volumes and surface areas) in coalification show that hard coals should be considered as a raw material for the production of active carbons. Investigations on the activation process of different rank vitrites by steam have confirmed this suggestion (Jasieńko & Biegańska 1975, 1979a). The changes occurring in the porous structure of vitrites and carbonization products of vitrites from hard coals of different rank activated up to 75% burn-off by steam are presented in figure 5.

The results of the investigations confirmed that the properties and structure of the activation products from vitrites of different rank coals depend mainly on properties and structure of original coals, as does the mechanism of porous structure formation in the process of activation (Biegańska & Jasieńko 1979) (figure 6, plate 2). The mechanism of formation of the porous structure may have an important influence on the mechanical strength of activation products.

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The fact that the compact structure of activation products obtained from coking coals shows an optical anisotropy is also very interesting. This makes it possible to obtain from products of coking coals activation by additive bituminous substances and a repeated process of carbonization of blend cokes of high graphitizability.

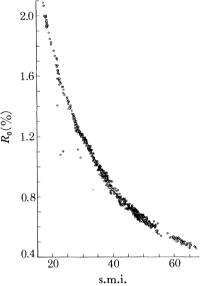


FIGURE 7. Correlation between reflectance of hard coals, R_0 , and structural index of metamorphism (s.i.m.) (Chruściel 1980).

6. New parameters in the classification of hard coals by rank

The question of the classification of coal by rank has been one of the fundamental problems in coal industry both from the coal science point of view and from the economic, especially now at a time of rapid development of new technology of coal processing.

The new tendency of coal processing – liquefaction, rapid pyrolysis and hydropyrolysis, gasification, production of metallurgical formed fuels and utilization of coals as raw materials in the carbon and graphite industry – requires an intensive study on the classification of coals. Introduction of new parameters in coal classification should be considered.

Determination of reflectance of coal as a parameter in classification is widely taken into consideration. A very important parameter is the heat of combustion. Attention should be paid to the new parameter proposed by Chruściel (1980), the structural index of metamorphism (s.i.m.):

s.i.m. = Q_s^{daf}/R_0 ,

where Q_s^{daf} is the heat of combustion in kilojoules, and R_0 is the reflectance of coal. This is based on parameters that can be determined with high accuracy and changes very characteristically in coalification. It may therefore be considered as a parameter for class determination (figure 7).

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It is also interesting to consider the parameters that can be determined by X-ray structural analysis: the interplanar spacing, d_{002} , and crystallite size, $L_{\rm c}$.

Coal properties depend highly on petrographic composition, and attention is called in many propositions to the introduction in classification of a parameter based on petrographic analysis.

When one is appraising the coal as a raw material in the coking industry and also in the carbon and graphite industry, it would be worth while to consider the application to the assessment of coking properties of the structural examination of the cokes, and determination of the degree of ordering of the compact substance expressed by optical anisotropy and development of porous systems.

7. Conclusions

Hard coals can be considered as a raw material for production of different type carbons: graphitizable and non-graphitizable, glassy carbon, carbon fibres and active carbon.

In the future a more intense utilization of coal solvent extracts for graphite electrodes and other forms of carbon production, as well as an increased use of hard coals in the production of active carbons, is to be expected.

Further development of new forms of carbon from coal will greatly depend on progress in coal science, particularly in the recognition of coal structure and determination of the correlation between coal structure and its properties.

The nature of original coals – their properties and structure, i.e. type of coal and petrographic composition - is very important for the estimation of their use in all technologies and also in the carbon and graphite industry.

The rapidly developing new technologies of coal utilization require a more elaborate classification system that would include the more important structural parameters of coal constitution such as reflectance, petrographic composition, interplanar spacing and crystallite dimensions, and the structure of coke.

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Discussion

G. M. Kimber (Coal Research Establishment, Stoke Orchard, Near Cheltenham, Gloucestershire, U.K.). With regard to the production of high-purity carbons from coal extracts, we have gained considerable experience on this aspect on plants of up to 500 kg of coal per day. This has enabled graphite electrodes of 300 mm diameter to be fabricated; these electrodes have performed well in a furnace of the British Steel Corporation operating at 25 kA in spite of predictions based on the empirical specifications used for assessing petroleum cokes.

However, not all solvent extracts of coal are the same in this respect, and the specific processing conditions as well as the starting coal, mentioned by Professor Jasieńko, have an effect; for instance, to make needle coke it is essential to start with a coking coal if no hydrogen is used, whereas if some hydrofining or hydrocracking is involved lower-rank coals can also be used.

S. Jasieńko. The results of our investigations (Jasieńko et al. 1975) showed that the graphitizability of coke from coal solvent extract increases with rank of coal used from flame coal to orthocoking coal (hydrogenated anthracene oil was used as a solvent). But there were no great differences in graphitizability between coke from gas coal and orthocoking coal extracts. I believe that with hydrofining or hydrocracking processes, mentioned by Dr Kimber, in the

preparation of coal extracts, even extract from gas coal could be used for making coke of fibrous needle structure. Considering coal structure, I doubt if coke of needle structure can be obtained from low-rank coal solvent extract.

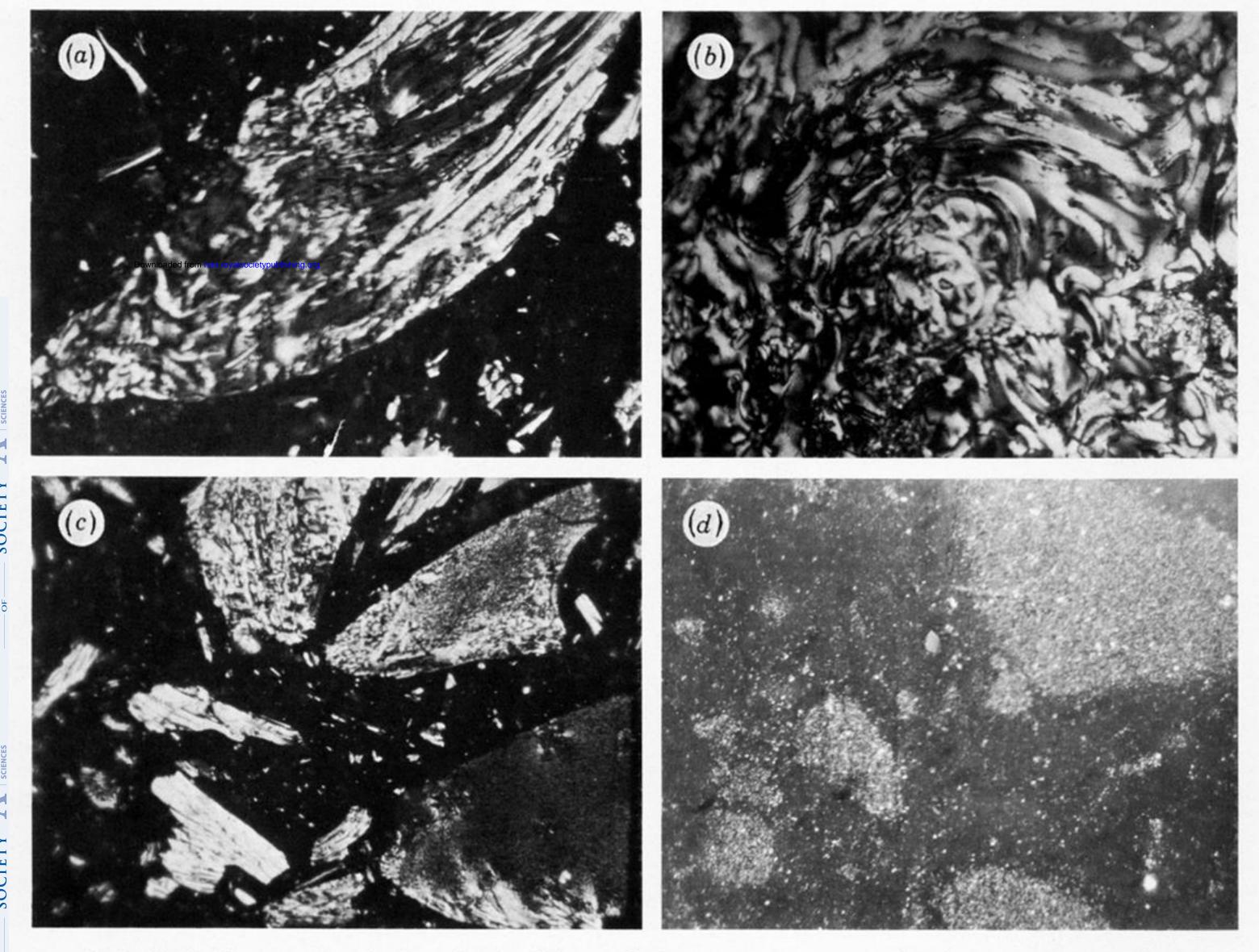
From our other investigations (Jasieńko $et\ al.\ 1979\ b$), it was shown that the cokes from the extract of gas-coking coal extract in hydrogenated anthracene oil have a high content of fibrous and needle-type structure, and high graphitizability. That can be explained by the arrangement and presence of sufficient nuclei of basic structural units in the extract and its lower viscosity compared with extract of primary coking coal.

- J. W. Patrick (British Carbonization Research Association, Chesterfield, Derbyshire, U.K.). Professor Jasieńko appears to place considerable emphasis on the use of optical anisotropy as a means of characterizing coke; I should like to ask if Professor Jasieńko believes that all the coke properties of industrial significance can be accounted for in terms of the optical anisotropy.
- S. Jasieńko. In the paper presented I proposed the use of microscopic examination, especially to determine the optical anisotropy to estimate the coking properties of hard coals as a supplementary examination, for example to the Gray-King method. I believe that using this examination we can obtain important information about the behaviour of coal in the coking process and also about the graphitizability of the resulting coke. This examination, after normalization of the method, may be considered as a parameter in the classification of hard coal by rank.

Microscopic examination also gives very useful and interesting information on industrial coke: porous structure, fissures and crack formation, and structure of compact coke substance such as the kind and quantity of isotropic, anisotropic and transitional phases. On the basis of this examination some properties of coke, like mechanical properties, reactivity, electrical conductivity can be deduced, but only qualitatively.

The structure of metallurgical coke, usually obtained from blends with four or more components, is inhomogeneous and is a very complex solid system. It is very difficult and often impossible to determine 'all the coke properties of industrial significance' using one physical or chemical method.

To summarize, my answer to the question given by Dr Patrick is: no.



IGURE 3. Optical micrographs of cokes obtained from pitch group components (Jasieńko et al. 1977 b). Polarized light, magn. ×250, crossed nicols. (a) Coke from γ component; (b) coke from β component; (c) coke from α₁ component; (d) coke from α₂ component.

1979). A, Optical microscopy (magn. \times 250); B, s.e.m. (magn. \times 1040). (a, a') Vitrite from flame coal (73.3% C d.a.f.); (b, b') vitrite from gas coal (83.1% C d.a.f.); (c, c') vitrite from orthocoking coal (89.2% C d.a.f.); (d, d') vitrite from semicoking coal (91.7% C d.a.f.); (e, e') vitrite from anthracite (93.1% C d.a.f.).