FUEL RESEARCH INSTITUTE OF SOUTH AFRICA.

TECHNICAL MEMORANDUM NO. 35 OF 1967.

THE MICROSCOPICAL EXAMINATION OF CHAR PREPARED AT THE KLIPPOORTJE PLANT.

By:

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INTRODUCTION:

Samples consisting of various specimens of char prepared at the Klippoortje plant from No. 5 and No. 4 Seam coal were submitted with the request that the Institute should ascertain, if possible, whether the agglomeration of the particles was due to the "plasticity of the coal" or to the "carbonisation of the tar".

GENERAL CONSIDERATIONS:

The plastic properties of a coal depend on its petrographic constitution and the degree of coalification (rank).

The petrographic constituents can be divided into fusible (vitrinite and sporinite) and inert (semi-fusinite, fusinite, micrinite and sclerotinite) macerals. South African coals having a ratio of fusible to inert constituents of 1.5:1 or more and an ultimate analysis (dry ash-free basis) such that the carbon content lies between about 81.5 and 88.5 per cent and corresponding hydrogen contents between about 5.6 and 4.5 per cent, invariably reveal coking properties.

The amount of tar formed on heating a coal depends on its volatile matter content as well as on its rank. Coals having a relatively low volatile matter content (high rank coals) will not develop plasticity and therefore have no coking properties although some tar may still be liberated when the coal is heated. Coals having a relatively high volatile matter content will usually also have a high tar content since a large proportion of the tar is derived from the sporinite which is an important contributor to the volatile matter obtained during carbonisation. Very low rank coals have a high volatile matter content but yield relatively little or no tar.

The volatile matter content of Klippoortje No. 5 Seam coal is in the order of 33% and it has a ratio of

fusible ...../

fusible : inert constituents of well over 2.5:1. The rank of this coal, although still falling within the range of coals having coking properties, is somewhat on the low side and it has a swelling number (also an indication of the coal's coking propensities) of only 1-1½. It can thus be expected that the coal will have only weakly developed softening properties.

The tar yield of this coal is in the order of 10 to 12 per cent (low temperature assay).

In a coking process plastic softening and the formation of tar under conditions of temperature rise are, of course, integrated phenomena occurring simultaneously and thus not independently of each other. Mixing of the softened coal and tar phases will occur and this will be enhanced by the escaping gases and vapours. The cementing of the particles on heating can therefore be expected to be due to the combined effect of the plasticity derived from the fusible macerals present in the coal and the tar evolved.

The cell wall substance of coke originating from carbonised tar is optically isotropic and thus similar to the cell wall substance of coke manufactured from low rank coking coals. It is therefore virtually impossible to distinguish between the two types of cell wall substances by means of the optical properties.

PREPARATION OF THE SPECIMENS FOR MICROSCOPICAL EXAMINATION:

After immersing the specimens in Schneiderhöln wax at about 115°C and cooling, they were cut in such a way that cross sections of the particles could be observed. The specimens were then further prepared by smoothing the surfaces, drying and retreating with wax under vacuum and final polishing for microscopical examination.

DISCUSSION OF RESULTS:

Judging by the appearance of the specimens examined, which were approximately nut size, fusion could not have taken place as a result of the complete softening and subsequent deformation of the lumps of coal. The formation of the gas in the fusible macerals inside the particles secreted at least some of the fused substance which then flowed into the voids separating the particles; the particles themselves remained relatively firm as a result of the skeletal structure formed

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by the inert i.e. unfusible macerals. If the large particles had contained sufficient fusible macerals developing an adequate degree of plasticity to provide enough fluid substance to fill the voids completely the particles would have been cemented together firmly when resolidification took place.

The microscopical study of these cemented links between the particles yields information with regard to the types of material from which the links originated, since the nature of the cell walls resulting when tar or coking coals are carbonised are reasonably well known.

With the char manufactured from the No. 4 Seam coal agglomeration took place to a much lesser degree than with the char made from No. 5 Seam coal. This is due to the fact that the No. 4 Seam contains less bright coal bands (vitrinite). The agglomerations that were visually and microscopically inspected indicated that in every case agglomeration took place where bright coal bands were present, which could supply cementing material during the plastic stage. In all samples inspected the cementing material that held the particles together could however not be clearly observed under the microscope because the cementing that took place can be compared to a process of "spot" welding where the surfaces joined lie very close together.

Inspection of photomicrographs I to VI provides evidence of how the particles are cemented together.

<u>Photomicrograph I</u> (No. 5 Seam char) shows three particles of coal A, B and C. Particles A and B are cemented to each other by means of a zone marked in broken lines. Point X is a reference point which can be observed in photomicrographs I, II and III at magnifications of 5X, 10X and 25X, respectively. Particle C is not cemented to B and the black dividing line can be clearly seen.

<u>Photomicrograph II</u> shows a portion of the same field but at a magnification of 10X. The zone cementing the particles is again marked in broken lines. It can also be noted that a piece of inertinite (marked In) consisting of carbonaceous shale nearly touches the cemented zone but is in fact separated from it by a thin elongated void. It appears as if there had not been sufficient fluid substance to fill the void and to provide cementing material.

Photomicrograph ...../

Photomicrograph III (Magnification 25X) shows more detail of the cemented zone. Its nature and structure resemble the typical structures found in cokes manufactured from relatively low rank coking coals. The cells are irregular in size and the cell walls are intermittent. Coke structures resulting from the carbonisation of tar can normally be distinguished by regular cells and continuous cell walls. In this particular case, the coke substance responsible for the cementing of the particles must therefore have been supplied by particle B which must have contained a large amount of vitrinite judged by the voids present in the particle.

Photomicrograph IV (Magnification 25X) shows a very weak zone between 2 particles marked A and B. Coke substance cementing the particles can be clearly observed.

Photomicrograph V (Magnification 25X) shows portions of two coal particles where no cementation has taken place. The space between the particles has been filled with wax.

Photomicrograph VI (No. 4 Seam char, magnification 5X) Particle A (consisting of coal and carbonaceous shale) is very poorly cemented to particle B and practically no cementing material can be observed. Where agglomeration was noted, the particles were invariably cemented to each other by means of "spots" of cementing material supplied by bright coal bands in the particles.

CONCLUSION:

In conclusion it can be stated that from the observations carried out on the specimens, it appears as if the coal tar did not play a very significant part in the cementation of the particles in question. In general, however, the influence of coal tar cannot altogether be disregarded. As pointed out earlier the cementing of particles is probably due to the combined role played by softened coal and tar.

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PRETORIA.
23rd August, 1967.
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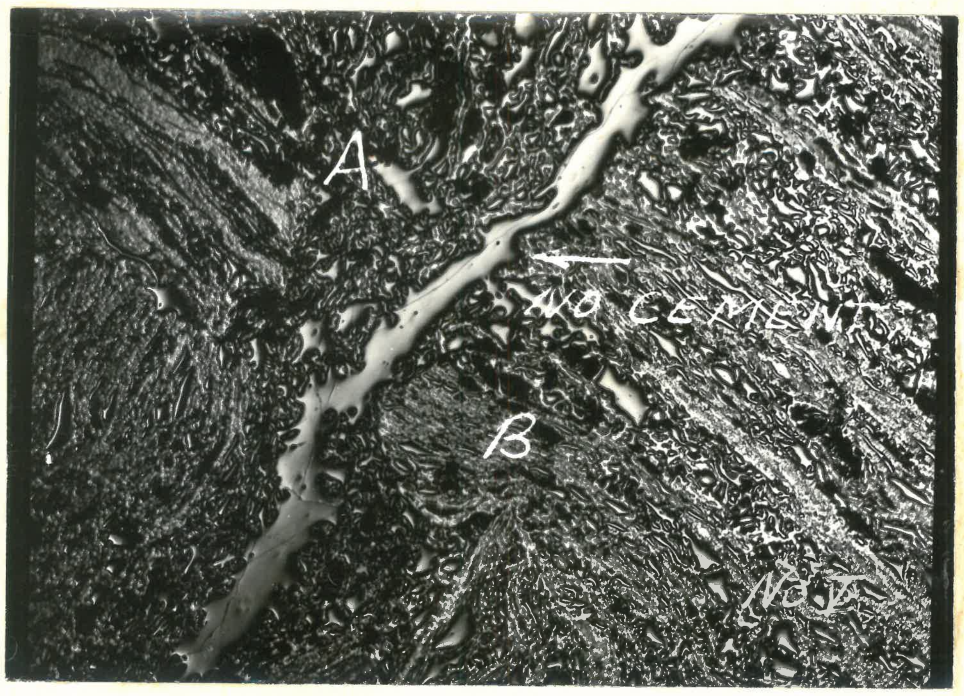
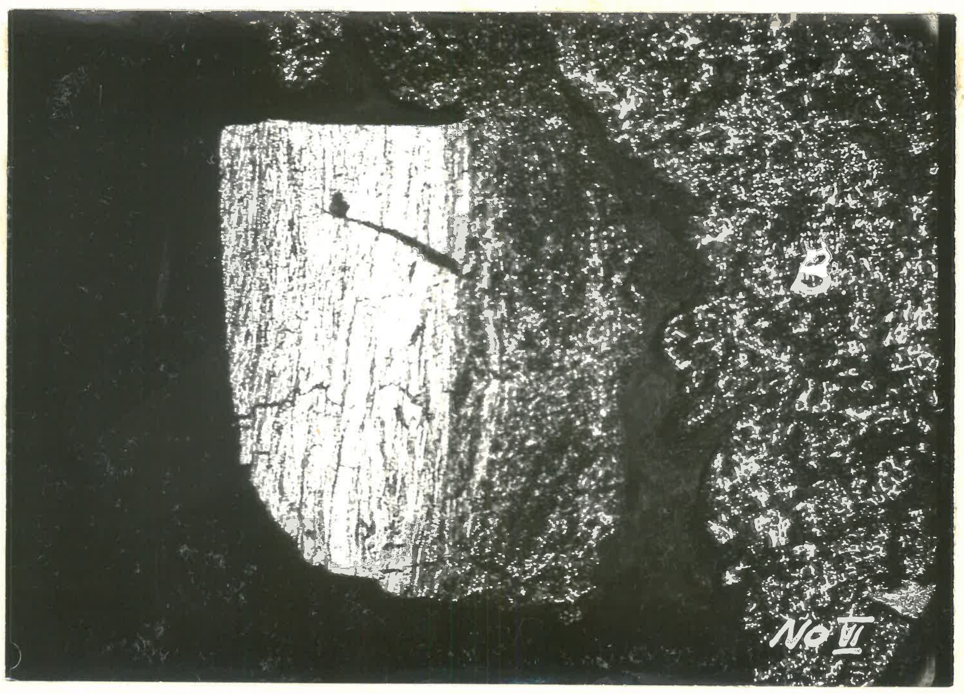
Photomicrograph No. III. (No. 5 Seam char) (Mag. 25X)
Showing the nature of the cells and cell wall substance of the zone cementing pieces A and B together.

Photomicrograph No. IV. (No. 5 Seam char) (Mag. 25X)
Showing a weak zone (indicated by arrows) between two particles marked A and B.



<u>Photomicrograph No. V</u>. (No. 5 Seam char) (Mag. 25X)
Showing portions of two particles A and B where no cementation has taken place.



<u>Photomicrograph No. VI</u>. (No. 4 Seam char) (Mag. 5X)
Showing the very poor cementing between particles A and B.