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Technical report: Petrology of Korean Anthracites

Prepared for the Government of the Republic of Korea
by the United Nations Industrial Development Organization,
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Based on the work of Mr. M. Shibaoka

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Vienna

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SUMMARY

This report contains the author's findings and recommendations after spending a month at the Seoul Laboratories of the Korea Institute of Energy and Resources (KIER) in September 1986 as a United Nations Industrial Development Organization (UNIDO) expert in the field of anthracite petrology.

As a first step a tentative classification and nomenclature for macerals in Korean anthracites is proposed and new macerals - graphinite, impregnite and mottlite - are identified in photomicrographs. Detailed analyses, based on the proposed classification system, of four Korean anthracites are presented and less detailed information is reported for many other samples. Preliminary experiments in the application of microscopy to the investigation of fluidized-bed combustion of anthracite are briefly discussed. Finally, recommendations for the management of samples and data, preparation of microscopic specimens and microscope bench arrangement are given. Sample preparation instructions prepared by a CSIRO technical officer are attached as an appendix.

1. INTRODUCTION

During September 1986, I worked at the Seoul Laboratories of the Korea Institute of Energy and Resources (KIER) as a United Nations Industrial Development Organization (UNIDO) expert in the field of coal petrology (specializing in anthracite). My brief was to assist KIER staff working on fluidized-bed combustion of low-grade Korean anthracite. This report summarizes my findings and includes recommendations for procedures to be implemented at KIER in the future.

The establishment of a classification system for anthracite macerals is the first step in anthracite petrology. For this reason, I spent most of my time at KIER working on this problem, although reflectance measurements and other investigations were also carried out. I believe that I have been successful in this respect, and my results are briefly explained in this report.

Application of microscopy to the investigation of fluidized-bed combustion of anthracite is an interesting subject. I have collected several coal and ash samples from the KIER pilot plant at the Daejeon Laboratories. However, because these specimens need to be prepared for microscopy by CSIRO technicians, they will be examined in Australia, and the results will be reported to KIER later.

This report also contains my recommendations concerning the management of samples and data, preparation of microscopic specimens and microscope bench arrangement.

2. CLASSIFICATION OF MACERALS IN KOREAN ANTHRACITE

It is, no doubt, desirable that macerals in Korean anthracite are classified using a system based on the one recommended by the International Committee for Coal Petrology (I.C.C.P). However, the current I.C.C.P. system was originally established and based on observation of lower rank coals (from brown coal to bituminous coal). It is not always easy to judge to which categories of I.C.C.P. macerals the coaly materials in Korean anthracites belong. Furthermore, Korean anthracites contain some materials which are not defined in the I.C.C.P. system. For these reasons I propose a tentative classification system and

nomenclature for macerals in Korean anthracites. This proposal should be discussed with the I.C.C.P. or other appropriate organizations in future.

2.1 Graphinite

In highly metamorphosed coals some unique material occurs. This material shows a very high reflectance with a metallic yellow colour (Fig. 1), however, because of its granular texture its reflectance cannot be measured accurately. Its genesis is not known, but is probably related to graphitization of coaly material and formation of impregnite (see below). It is most likely that both the deposition and removal of carbonaceous material in and from the coal seam took place during the graphitization. This highly metamorphosed material is tentatively called graphinite.

2.2 Impregnite

The name impregnite has been chosen for one of the newly observed materials forming part of some Korean anthracites. Impregnite occurs as filling in various void spaces, such as cell cavities, fine gaps in and between other macerals and minerals, and also in brecciated coal (Figs. 2 and 3). The maximum thickness so far measured is about 2 μm . The maximum reflectance and bireflectance of this material are variable, but usually greater than those of vitrinite and inertinite, and it is usually yellowish in colour. In anthracite this material often occurs in close relationship with typical pyrolytic carbon, and, in some instances, the two materials are almost continuous. This fact suggests that some of the impregnite has the same genesis as pyrolytic carbon. However, some of the impregnite has a much lower reflectance than the pyrolytic carbon and may be a mesophase-type carbon material derived from tar. Both the pyrolytic and mesophase carbon-like materials are likely to have been derived from coal in the process of metamorphism.

With the exception of the above-mentioned graphinite and impregnite, all the other coaly materials are classified into three large groups: mottlite, vitrinite and inertinite.

2.3 Mottlite

As it is hard to judge whether this sort of material belongs to the vitrinite or inertinite group, it is tentatively called mottlite. If the size of an optical domain in a coaly material is smaller than 20 μm , this material is called mottlite (Fig. 4) because the particle has a mottled appearance. This is due to the presence of many different optical domains. The mottlite usually occurs as a layer, or layers, and, in some instances, shows a fusinite or a semifusinite-like morphology. The physical properties, e.g. elasticity, of coaly material in bituminous coal for example, may gradually vary within a single particle (i.e. without a sharp boundary). If such a particle suffers tectonic stress the resulting strain may be variable within the particle, and this may result in gradual variations of optical anisotropy. If a coal consists of many particles of this type, the coal, as a whole may show a mottled appearance. Such a situation is expected when vitrinite and inertinite-type materials alternate without sharp boundaries. The mottled appearance is likely to become more significant when the rank of the coal exceeds a certain level, such as medium volatile bituminous coal.

2.4 Vitrinite and Inertinite

If the size of the optical domain of a coaly material is greater than 20 μm , the material is regarded as either vitrinite or inertinite in this report. Differentiation between these two macerals is based mainly on their morphology. Some vitrinite occurs as a large homogeneous layer, others show striation or granular texture, while inertinite can take any shape, rounded, elongated or angular, and does not show clear striation.

The morphological features of vitrinite and inertinite in bituminous coals can give good guidance for the identification of these macerals in anthracite. Usually the bireflectance of anthracitic vitrinite is greater than that of inertinite. but this is not always the case. If the polished surface of coal is parallel to the bedding plane, or close to it, the bireflectance can be small.

Graphnite, vitrinite, mottlite and inertinite are the major maceral groups in Korean anthracites. Some or all of these four, as well as minerals, form the ground mass, and are embedded in each other.

Impregnite usually occurs as a minor component in vitrinite, mottlite and inertinite. Often high reflectance material with thread-like morphology occurs in the vitrinite. It may have derived from sporinite and other exinite macerals, but at this stage detailed discussion for its diagenesis is not attempted.

2.5 Subdivision of the Four Macerals

Finely brecciated graphinite (i.e. with an interval between cracks of $< 10 \mu\text{m}$), called graphinite-b, is counted separately from the massive graphinite.

The mottlite with fusinite and semifusinite morphology is called mottlite-1. Mottlite which shows any feature of botanical texture, even if no cell cavity is left, is included in mottlite-1. Mottlite without any clear botanical texture is called mottlite-2, and a fine mixture of mottlite and vitrinite is also included in this category. If the mottlite is finely brecciated, it is called mottlite-b.

Plain vitrinite without any internal texture is called vitrinite-1, while vitrinite with a granular texture (like degradinite or desmocollinite in bituminous coal) is called vitrinite-2. Finely brecciated vitrinite is called vitrinite-b.

2.6 Proposed Classification

The relative proportions of vitrinite, mottlite, inertinite and minerals in the coal samples appears to be a useful method for characterizing the primary constitution of the coal seams, and could be used to identify them. However the extent of lateral variations of petrographic composition in the coal seams should be carefully investigated, and in future many samples should be taken from the same seams, but in different locations. However it should be remembered that neighbouring seams tend to have similar petrographic compositions.

Microscopic examination of coal samples is also useful in determining the extent of metamorphism that the coal seams have suffered. The occurrence and genesis of graphinite and impregnite should be investigated, in the future, in relation to the transformation of minerals in the roof and floor rocks, and to igneous intrusion.

The large proportion of brecciated coal present is an indication of tectonic disturbance. In some samples, e.g. No. 10, many microfaults were found, and the effect of residual strain on the optical properties of the coal was observed. The brecciation of coal seams is particularly important in connection with coal quality, and the samples which are rich in brecciated coal tend to be rich in minerals. Samples Nos. 11 and 17 are good examples. This is probably due to filling of the cracks in brecciated coal by secondary minerals, such as clay and pyrite. Scanning electron microscopy (SEM) observation of the brecciated particles provides good evidence for this, however, some coal seams, e.g. No. 38, contain large amounts of the primary minerals which were deposited together with plant detritus.

In the case of inertinite, only brecciated inertinite (inertinite-b) forms a separate category from all other types of inertinite.

The proposed classification of anthracite macerals is tabulated below:

Graphinite
Graphinite-b

Impregnite

Vitrinite-1
Vitrinite-2
Vitrinite-b

Mottlite-1
Mottlite-2
Mottlite-b

Inertinite
Inertinite-b

Minerals

2.7 Examples of Analysis of Korean Anthracites

On the basis of the classification explained above, four of the KIER samples listed in Table 1 were analysed in detail. The results given in Table 2 and Figure 5 show that these four samples have significantly different petrographic compositions.

Sample No. 20 contains only the vitrinite and mottlite groups and small amounts of coaly shale and minerals. The most significant features of this sample are the very high proportion of brecciated vitrinite and mottlite. Although the proportions of coaly shale and isolated minerals are small, the ash yield of this sample is relatively high (36.1%). This is due to filling of the cracks in brecciated particles by minerals. The proportion of graphinite in sample No. 21 is very high (36%), and many of the graphinite particles are brecciated. About 6% of graphinite/mottlite intermediates are included in mottlite-2. Sample No. 25 contains impregnite, and although the total percentage of the impregnite is only 2%, its occurrence is fairly extensive. About 2% of the vitrinite particles contain networks of impregnite and are included in vitrinite-b. The last sample, No. 26, is extremely rich in vitrinite, and contains essentially no mottlite or inertinite.

Quick microscopic examination of other samples has revealed the following facts:

Nos. 22 and 24 are rich in graphinite

Nos. 7, 9, 24, 29 and 30 contain impregnite

Nos. 26, 37, 38, 41, 43, 49 and 51 are rich in vitrinite

Nos. 19 and 34 are rich in semifusinite and fusinite

Nos. 46 and 47 are rich in mottlite and inertinite

Nos. 11, 17, 31, 32, 41, 42, 44, 45 and 50 are brecciated, and

Nos. 7, 8, 18, 19, 41, 43 and 51 are rich in pyrite

Reflectance of vitrinite has been used as the most reliable parameter for coal rank. Only vitrinite-1 reflectance should be used for this purpose. However, as the vitrinite reflectance of anthracite is likely to be affected by many factors (see page 48, "Stach's Textbook of Coal Petrology"), results of the measurement should be carefully interpreted.

3. APPLICATION OF ANTHRACITE MICROSCOPY TO COMBUSTION RESEARCH

In the Seoul Laboratories of KIER chemical analyses and ash fusibility tests are carried out on Korean anthracites. These are very important for providing the basic data for combustion research. However, it is most desirable that microscopy is applied to the investigation of anthracite combustion processes in the future. The author has carried out some preliminary observations with Korean anthracite samples provided by Mr. K. Jung, CSIRO Division of Mineral Engineering. The anthracite (original size 1/4 in to 3/6 in) was burnt in a fluidized-bed combustion furnace. After burning 5, 10 and 15 minutes, half burnt lumps of coal were extracted from the furnace. They were embedded in plastic and polished for microscopic observation (see Appendix for sample preparation instructions).

Under the microscope no preferential combustion of any particular maceral was observed, behaviour which is quite different from that of lower rank coals. At this stage of anthracite metamorphism, even vitrinite does not swell (or vesiculate), and the pores in fusinite are almost completely filled by minerals. Thus the combustion behaviour of the macerals was very similar. In this sense the microscopic study is not very useful.

When cracks in brecciated coal were not filled by minerals, combustion took place on the surface of each brecciated grain (Fig. 6). However, in most cases the cracks were tightly filled by minerals (Figs. 7 and 8), and in such cases combustion did not proceed along the cracks, in fact the crack filling mineral veins appear to have become a kind of combustion barrier. Obvious changes in some minerals were also noticed during microscopic observation of half burnt samples. A more detailed investigation would be most desirable sometime in the future to obtain a complete understanding of the mode of combustion under the conditions experienced in a fluidized bed.

4. RECOMMENDATIONS

4.1 Management of Samples and Data

1. All samples to be examined should be registered in a SAMPLE BOOK, given serial numbers, for example Lab. (laboratory) numbers and Pet. (petrographic) numbers. In the sample book the origin (e.g. coal seam, coal mine, coal bearing formation, geological age), nature of sample (e.g. particle size and specific gravity), name of collector, title of project etc. may be shown.
2. All samples, in general, should be kept in suitable (air tight) containers (e.g. plastic or glass bottles) and stored in an appropriate place as permanent reference samples.
3. All analytical results should be assembled in a systematic way in record books/files or cards or computer discs.

Establishment of this system is most urgently needed. All records of samples and analyses should belong to KIER and be centrally organized. They should not be kept personally.

4.2 Preparation of Microscopic Specimens

1. At present, cleaning of grain mount moulds and charging of powdered coals into the moulds is done on the laboratory floor. This sort of work should be done on a proper laboratory bench. Plastic resins, for example araldite, should be used in a fume cupboard in order to protect workers from the toxic gases.
2. Most Korean anthracites are rich in minerals. The mineral particles tend to be easily pulled out of the coal and then cause scratching of the polished surfaces during the grinding and polishing of microscopic specimens. For this reason it is highly recommended to vacuum impregnate the microscopic specimen with plastic at a late stage of grinding. After the impregnated plastic becomes hard, the final grinding and polishing should be carried out.

3. After each stage of grinding and polishing, the specimen and hands should be washed with clean water.
4. Upgrading of grinding and polishing machine is recommended. If, for example, three grades of sand papers are to be used, three grinding machines should be used in order to avoid scratching due to contamination by coarser sands.
5. Sample preparation instructions prepared by a CSIRO technical officer are attached as an appendix.

4.3 Microscope Bench Arrangements

1. The stool which is currently used for microscopic observation should be lowered by approximately 15 cm. As microscopic work is usually continued for many hours, a comfortable chair or stool should be used (Fig. 9a).
2. The microscope bench should also be about 15 cm lower, and it is desirable not to have any drawer under the bench top. Arm rests (about 5 cm higher than the bottom of the microscope) on both right and left sides (Fig. 9b) are needed.
3. For reflectance measurement and fluorescence work a semi-dark room is needed. However, as a temporary measure, a black curtain may be sufficient.
4. It is desirable that the light microscope is used in a separate room from the heating microscope, and that smoking in the light microscope room be discouraged.
5. It is recommended that water not be used in the microscope room, and also that objective lenses and other optical parts are kept in desiccators during the rainy season.

5. ACKNOWLEDGEMENTS

I am most grateful to Dr. Park, Keung-Shik, President of KIER, Dr. Park, Sukwhan, Dr. Cheong, Taejin and Dr. Lim, Deoksoon for offering all kinds of help to me during my stay at KIER.

APPENDIX: INSTRUCTIONS FOR SETTING, GRINDING AND
POLISHING GRAIN MOUNTS OF COAL

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Dear Ms. Garcia,

I am making a preliminary reply to your letter to Dr. M. Shibaoka of May 26th, 1983, your reference Ref/n/Nota:46:83. I apologise for the long delay in replying to you. In order to get some information to you now I will outline briefly the steps to take in setting, grinding and polishing grain mounts or block mounts of coal, with brief reference to the company names of the products we use etc. At a later date I can supply much more detailed information if you request it, with samples of materials used, and also of coal samples at various stages of preparation so you can form your own criteria for each stage of the work, and as well, the details when you request them of preparation of other types of material than coal.

Setting (mounting) I will divide into two portions, one to do with grain mounts, the other with block mounts.

Until recently the standard for grain mounts which we used stated that everything be used which was retained on a Number 10 sieve, approximately 1.7 mm, having passed through the next size larger and excluding the smaller fractions which pass through the No. 10, i.e. as few "fines" as possible. The preparative method now in use requires the use of grains retained on a No. 16 sieve, approximately 1 mm, having passed through the next sieve up in size, and excluding smaller material which drops through the No. 16. This retained fraction is air-dried before we get it, on long trays. I am not altogether familiar with the details of sampling procedure necessary to get a representative portion of the material being examined, as this is done by a specialist department, but I can obtain all such information at your request and pass it on. *

To make up the grain mounts we use a cold-setting araldite and usually something over 1 g of coal stirred well in a teflon cup which holds about 5 g of the already mixed araldite, and tip this mixture immediately into a silicone-rubber mould with openings in it 25 mm square and about 13 mm deep (the teflon cup should be cleaned afterward with acetone and tissue, and all this setting work should be done in a very good fume cupboard and only whilst wearing butyl rubber gloves, as these materials until polymerised are very dangerous carcinogens). Let me add as a note that when you have become familiar with the general amount to be placed into the cup, that the cup stage can actually be left out and material can be scooped directly into the rubber mould space, or can be transferred into a metal balance scoop of the old type, easily fashioned out of some bent aluminium foil or the like, and thence to the rubber mould. At the bottom of each mould opening I usually fix an aluminium plate 25 mm square, the plate thickness being about 1.4 mm, though this is hardly critical. Before mould and aluminium squares are used it is best to have painted them with a waxy mould release compound: they should be dried separately overnight, or for 30 min. in say a 55 degree C oven before insertion of the aluminium square so that no mould release solvent will remain and later interfere with the polymerisation process of the araldite resin. This interference is to be avoided at all cost especially as a sample of limited quantity may be lost due to difficulties introduced by faulty resin. Poorly polymerised resin will cause poor grinding, smearing of material,

* Since that date we
have once again included
the "fines"

etc., and not only make it impossible to polish the sample, but tear the expensive cloths in the process. As well as this, lesser degrees of fault will cause contamination or other damage to other samples being done by hand, or in machine operation where a number of samples are being handled at once. I should mention here that water will also interfere seriously with polymerisation. To continue, after pouring the well mixed sample in its araldite into the mould space, or after mixing it in the mould space itself, the material will be seen to have filled only about half of the mould space. Where the sample has been mixed in the mould space itself, it is important to wipe the upper walls, above the level of the setting resin, clean of any resin plus sample which may have adhered to these areas, cutting down on belt grinding of the sides at a later stage. As the material in the bottom part of the mould space hardens over the next few hours, bubbles should be poked out with a steel probe, such as a needle set in a glass or wooden handle or the like. Poking out the bubbles once after 15 minutes and again after another 20 to 25 minutes is usually all that is required. It is important to get rid of bubbles at both this and the later stages of setting as the bubbles when hardened and opened on grinding, present cavities which carry grinding and polishing media from one stage to another, spoiling the sample itself, other samples, and in fact the polishing cloths themselves. When the bottom layer of the mount has reached a thick consistency a freshly mixed araldite resin can be poured over to top it up, slightly overfilling; after about 10 minutes a spatula is used to wipe away excess bubbles. After wiping clear the top bubbles, a small square of aluminium foil with the number stamped on it and bent (see drawing at end of letter) can be hung from the top of the mould over the edge into the araldite, number facing out and upside-down. Thus when finished the number will be readable when the polished surface is under the microscope. The best polymerisation is obtained over 24 hours. The reason for the aluminium square at the bottom of the mould is this: that without this, the resin tends to pull away from the bottom of the mould and leaves pulled-back corners and other artifacts which may be undesirable. The moulds as well tend to degrade over time and this is undesirable along the bottom in particular. As the aluminium squares can be reground to flatness as necessary another problem is avoided.

We make the silicone rubber moulds from a commercial rubber which we mix ourselves and pour into a form made in our workshops. These moulds have six spaces each, last well, and many settings can be done at one time, i.e. in a number of moulds.

Now some petrologists like to have side-mounts (side-sets) and for them I have silicone rubber moulds made up so that the 25 mm is the depth of the mould, and seen from the top the opening is 25 x 13 mm. In this case small measuring cylinders are filled to the 8 ml mark with the coal from the tin and poured into a small beaker containing about 5 ml of already mixed araldite; the coal and the araldite are vigorously mixed and poured into the mould and let to harden, poking out the bubbles, leaving a small space at the top to add onto later with the numbered aluminium strip, again placed so that it will be right side up when the polished surface is facing up in the microscope. The coal obviously fills the whole mount except for the narrow number strip. The aluminium foil used for these numbers and also for the boxes described below is about 125 micrometres thick, though of course anything which serves the purpose may be used.

Now to the mounting (setting) of blocks. The block wanted is usually cut off with a hacksaw or diamond saw and shaped on a grinding belt. In general the polished face should not exceed 30 x 50 mm, or at most 50 x 50 mm unless there is a very good reason for it; it is better to divide larger samples into several smaller blocks. The reason for this is that the difficulty of preparation goes up something like the square of the surface area prepared (my own subjective judgement) and larger samples are rarely worth the extra effort which is necessary. Very large samples can of course be made with special machinery, but here I speak only of hand work.

The faces of blocks for mounting are always cut perpendicular to the bedding plane of the coal. From face to back of sample can be 1 to 2 cm or slightly larger if necessary. The face to be worked is flattened on a grinding wheel with P320 or P400 silicon carbide paper and running water, for which see details below, and the block should be well dried in an air oven at 55 degrees C for several hours or even better overnight before setting in araldite. The boxes for mounting blocks are usually made from heavy aluminium foil, in our case 125 micrometres thick, formed into a box around a wooden block of the appropriate size, and sticky tape is put around the box, wooden block in place, once fully around the sides, then again overlapping the bottom and folded onto the bottom in case of later leaks in the foil box. The number is stamped with dies onto the foil on the side of the box, wooden box still in place, so that the number on the plastic at the end of the process will be right side up when the final polished face is facing up on the microscope. Take the wooden box out. When the coal block is dry, mix up cold setting araldite and pour some onto the bottom of the box, put a bit on the face of coal block to go down, set the coal block in the box and add the rest of the araldite. This should cover the coal block or nearly so, yet not reach to the top of the foil box: if it does you may experience difficulty in getting the foil off afterwards. Poke out the bubbles at intervals during setting and let set hard over 24 hours. After setting hard, the aluminium foil can be cut with a scissors to start it and usually stripped off by hand without too much difficulty. The making of these foil boxes in numbers is so time-consuming that the commoner sizes above the normal size mount made may be produced in silicone rubber by pouring into a mould former made up in your own workshops. I can send you the plans for some of these sizes if you wish. Let me here note that any box or other mount which requires over about 35 or 40 grams of araldite should be handled with the exothermic nature of the reaction in mind: above this size the heat cannot escape efficiently and drives the reactions faster and faster, in effect causing a flash polymerisation or a boilover plus polymerisation and ruining your sample. This problem may be dealt with by layer-setting, using say 20 to 25 grams per layer, waiting until fairly well set and cooled, and then adding in the next layer. There is more to say on this topic, but for now it is enough to be aware of the problem.

I do not here use hot press or fast setting araldite for grain or block mounting as advocated by some laboratories and even in some standard specifications, as the heat and/or pressure involved may alter the coal or coal products or introduce other artifacts in our opinion.

A note on removing the aluminium squares which usually adhere to the grain mounts after removal from the mould: bunch up a towel or other cloth to act as a shock absorber, rest the sample on it and tap the aluminium square off with a small hammer: sometimes several directions of tapping are necessary.

Now as to grinding and polishing either grain mounts or block mounts: the backs - but not the faces - of the mounts can be trimmed very usefully and quickly on a water lubricated continuous silicon carbide grinding belt with roughness from P80 to P240; within limits the belt roughness for this doesn't matter. Flatten the back of the mount to get rid of the sharp meniscus, bevel each back edge well and evenly, then grind off the back corners as well. The back trimming is done to protect the hands during the many further manipulations. Whilst the faces are seldom opened on the belt, it is advisable to bevel the edges of the face as well, and to corner them. This has to do with problems which may arise later in the polishing stages, whether by hand or machine. Rinse the samples well. It should be noted that in the absence of a continuous grinding belt, this trimming and bevelling may be carried out on a wheel as described below, fitted with a rougher grade paper than is normally used on the wheel, e.g. the grades P80 to P240 mentioned above.

To open the faces of the samples I use a 300 mm wheel on which is placed P400 silicon carbide paper. I use non-adhesive paper (Naxos, Germany) which I cut myself to the size wanted from the roll supplied by the manufacturer. The paper is wetted on the back and placed on another wet paper with its abrasive side down, then loosely clamped. Exactly how you do this will depend on the apparatus you have available, but the point is to keep the paper from wrinkling and/or breaking during the course of your operations. It is not necessary to have such big wheels, many people have 200 mm diameter or smaller. The machines I use are the Struers Planopol and Planopol-2 (Denmark). However the following description of the operation may be adapted to just about any type of equipment.

The initial grinding of any coal sample by hand takes place as the wheel with P400 paper affixed turns anti-clockwise with a good flow of water onto it at 150 rpm. The sample is held with both hands and drawn through an arc from left to right from the centre of the wheel, also turning the sample with the wrists as the sample passes through the arc. After the first draw through the arc, the orientation of the sample is changed in the hands by 90 degrees and again drawn through a second arc in the same way, and again and again, each time changing orientation, until the face of the sample whether grain mount or block is open and flat, as judged by wiping the face of the sample dry with the palm of the hand and holding it up to a well placed light and occulting it to see if any facets are to be seen, and whether plastic still covers any of the embedded sample on the face you're working. Now the reason for the arcing, turning during the arc, and the continual 90 degree changes of orientation is this (and please pay special attention to this as those who practice this never seem to articulate it): each sample preparer has a bias in his stroke, and as well some bias may be introduced by irregularities in the sample, and this continual turning distributes the bias evenly. If you do not follow this practice you may easily end with a wedge or even worse a many-faceted sample which will prove impossible to polish or use. Although this may seem a simple-minded explanation it is made for the benefit of those who are beginning the work rather than practiced experts, and it may explain and help you through many otherwise inexplicable occurrences in the early stages of your work. It may also be useful to note here that the streak which appears behind the sample on the wheel is a good indicator of sample opening and the headway made: also watch that you don't grind right through your sample!

When quite satisfied that the sample is open and flat, it consolidates the position to move the sample anticlockwise, the same direction as the wheel and on its right hand side. Move rapidly a few times anticlockwise, change the orientation of the sample in your hands by 90 degrees, repeat, and so through the four orientations, just a few seconds in each. This randomises the scratches and gives an added guarantee of flatness. Examine well in your x5 hand lense after drying.

Rinse the sample well in a strong spray of water and change the paper to the grade P800. Go through exactly the same procedure, using 12 or 14 arcs, and check the surface with your x5 hand lense again. Make a habit of examining your surfaces with the same hand lense and in the same light so that you build up the criteria by which you may make judgements at each stage of the various kinds of samples you handle. The P800 should have cut out most of the P400 scratches, and some of the coal particles, depending on the coal, may be assuming a dull lustre by this stage. If you can, pick out and remember some distinctively shaped grains to see how their surfaces change from stage to stage of your work, using now your x5 hand lense, and later your low-power microscope. These suggestions in detail are for your learning, and of course they become almost automatic and very quickly done in later stages of your work, when you will be doing larger numbers of samples at one time.

Following a strong spray rinse of the sample, change the paper to P1200 and go through the same procedure, 12 to 14 arcs, followed by the anticlockwise motion on the

right hand side of the also anticlockwise wheel, then check the surface with your glass. Some of the grains may be shiny by this stage. Then on the stationary wheel with water still running over it, carry out an anticlockwise motion on the paper for five or six seconds in each orientation. It is then often a help to have a very worn P1200 paper sitting aside on a glass plate, with running water available, and repeat these last mentioned motions on the very worn paper but for 15 or 20 seconds in each of the orientations - in this lab I continue to use the same carefully chosen worn paper piece for up to a year. Note the difference in the surface appearance before and after using the worn P1200 paper: this is also very striking in the microscope.

At this point examine also the state of the face bevelling previously put into the sample to make sure that some bevel is left, and if none is left, reintroduce it either on belt or fast wheel and re-corner as necessary. This bevelling is especially important during the polishing stages to come so that the sample will be less likely to catch in the cloths and tear them, or catch and be thrown about the lab at great speed. Not only personal injury but damage to and even loss of the sample may be avoided by making this quick bevelling and cornering of the face and making sure it's still there before you go to polish.

Spray rinse, clean in an ultrasonic bath if available, spray rinse again, and you are ready to go on to polishing. If an ultrasonic bath is available, let me note here that these seem to work better with a few mls of ordinary kitchen detergent in the bath, or with something like the Kodak product called Photoflow.

There are three stages to hand polishing as I will describe it and as I usually carry it out, two of them on wheels as described below, and one on a stationary hand lap. I will call the steps by the names of the polishing agents associated with them:

1. Linde C, which is 1.0 micrometre Al_2O_3 (or alpha alumina),
2. Linde B, which is 0.05 micrometre Al_2O_3 (or gamma alumina), and
3. magnesium oxide, the product used here being Buehler Magomet. All of these are supplied by the Buehler Corporation of Evanston, Illinois, United States.

For hand polishing I use 8 inch wheels supplied with the Buehler Polimet which is sold as a continuously variable speed machine running from 0-1200 rpm. I favour the aluminium rotors because the load on the motor at starting time is so much less and the motors therefore last longer; while we do have bronze wheels also, it must be remembered that it is necessary to start them off more slowly and you may wish to circumvent this problem altogether if you are buying in new equipment. If you are establishing a new lab with limited funds, a good general machine which is capable of expansion to machine operation and very adaptable to grinding and polishing of a great variety of materials is the Struers DP-U4. This is what I would buy to get the greatest adaptability and expandability to a great variety and load of work, though of course, two are always better than one, and lots of spare parts no matter what you buy. This can be treated elsewhere at more length.

The Linde C wheel's metal surface is prepared by holding a straight iron bar wrapped in P1200 paper over the metal surface and spun wet, then wiped dry. In order the following are placed over the wheel: first a sheet of the thin plastic food wrap known here and in North America and Britain as Gladwrap or Saran Wrap, or whatever equivalent is available; then a piece of fresh canvas cloth (small sample enclosed) being very careful to see that no loose threads are hanging underneath from the edge onto the wheel, as they will soon cause breaks in use; and then the Buehler Metcloth. Force the rim evenly over the assembled materials. It can be helpful to pick the wheel up in one hand and hit the rim gently with a rubber-headed hammer after the rim is on to be sure it's all the way down. Then trim off the excess materials hanging below the rim with sharp scissors of the dressmaking variety, and tap once again with the rubber-headed

hammer. The Buehler Metcloth is not available with adhesive back, but it is well worth using.

The Linde B wheel's metal surface is prepared in the same way, the plastic wrap is set over, but no canvas is used: just set the Buehler Microcloth directly over the plastic wrap. Set the rim over in the same way and tap into place with the rubber hammer and trim the excess with scissors as with the previous wheel. A sample of the Microcloth is enclosed. Although we use the non-adhesive variety of the Microcloth here, it is available from the company in an adhesive-backed version. Other cloths are available which produce more or less the same result as Microcloth, e.g. Metron from Metaserv Company of the United Kingdom, but I find this cloth very grabby in hand use and have ceased to use it in favour of the other.

The cloth used with magnesium oxide is called Selvyt, sample enclosed, made in England, but unfortunately I don't have the English source. I will enclose the address of our source here in Melbourne for what it's worth. This cloth is usually set over glass which in turn is clamped onto a wooden block, and unlike the others, this cloth must never be allowed to go dry; it is stretched out only when in use and when not in use is kept in a beaker of water nearby. If it should dry out and any trace of MgO is left it will form into magnesium carbonate, which is hard and will ruin your polished surfaces. This carbonate can be got out by soaking the cloth in about 0.24 N HCl for a long time, but in practice I never reuse such suspect cloths but after soaking in HCl and rinsing just save them to give away to people who call in to see us about odd polishing projects of their own.

I use squeeze bottles of distilled water for both Linde C and Linde B, transferring the material in with spatula to almost empty bottles, then almost filling them with distilled water. The most convenient squeeze bottle dispenser to use is a 500 ml with a very wide top so as to avoid the necessity for a funnel for loading; and the best type of bottle is that which forces the material up from the bottom so you can use it right down to emptiness. The Linde C works out to about 8g/500 ml distilled water, and Linde B to about 4g/500 ml of distilled water. The utmost care must be taken to get the right materials into the right bottles and to avoid cross-contamination!

Now we are prepared to polish: but before doing so, look at your sample not only with your hand-lense but in the microscope you must have in your preparation area. Look at some standard low magnification, whether x20 or x40, so long as it is always the same and will build up a fund of knowledge in you as to about what your surface should look like at this stage. Naturally, it looks just terrible at this stage, but you will find that there is a certain way it should look, and come to recognise this as it appears in various materials and conditions, and also any deviations from it, and come to use these deviations as diagnostic signs for clearing up problems in future. I don't know how I can stress the last few lines enough- the details are important for starting off, but the most important thing I can tell you is the value of observation, and the forming of criteria which you carry around with you for the different stages in your work, and the feel of how those criteria will vary in appearance from material to material. This is the start of becoming completely independent in this work.

Turn on your Linde C wheel supplied with Metcloth and bring it up to the 7 setting, or about 700 rpm. Shake the Linde C bottle well and then wet the cloth, then apply the sample with a scrubbing motion clockwise, opposite to the wheel direction which is customarily anticlockwise (note that I here speak of polishing a 25 mm square sample, and that larger blocks will require a different treatment). A clock standing by with a big sweep second hand, as used in the older darkrooms, is very handy. Scrub evenly and firmly opposite to the direction of the turning wheel for about 15 seconds, lift and turn the sample through 90 degrees, shaking the bottle and applying more Linde C,

and scrub another 15 seconds or so in the new orientation. I generally carry out this stage for a total of 75 seconds in all, in other words about 15 seconds in each orientation and then repeat the first orientation once. In between each stage of orientation more Linde C in water is usually applied. At the finish, spray clean in water from the tap, clean in ultrasonic bath, spray clean again, dry back and sides with a towel and blot the face dry on fresh filter paper. Mount and observe in your microscope. Note the changed appearance: most of the P1200 and larger lines are now gone, replaced by Linde C lines, and the surface seen is much brighter, reflecting much more light.

Go to the Linde B wheel supplied with Microcloth, turn on to the same speed and repeat the same procedure as with the previous stage, using however the Linde B squeeze bottle with frequent shaking. Speed about 700 rpm, time 75 seconds, 5 orientations, etc. Then spray clean, clean in ultrasonic bath, spray clean, dry and blot on filter paper and observe in your microscope. The change between Linde C and Linde B is very dramatic, indeed almost unbelievable. Almost all lines are out although it never happens that all lines are gone.

The difference in strategy suggested for larger blocks at these first two polishing stages is this: the wheels may be considerably slower, say 500 rpm or less, both hands are used to grasp the sample, and the scrubbing motion described above, while it may be possible, may be replaced by another which is safer and quite effective. With both hands gripping the sample it is applied to the right hand side of the wheel and moved slowly back toward the operator, continuously turning in the hands. After riding down toward the front of the wheel, the sample may drift back up again and is arced to the right and toward the operator again. It must be turned slowly through the full 360 degrees of its possible orientations related to the polishing plane, and then turned again. I find it most convenient to turn the blocks slowly clockwise in my hands, regrasping it slowly and hanging on well as it passes each 90 or 100 degree change. It is convenient to apply more polishing medium after each 20 seconds or so, allowing about 4 or 5 seconds to do this as the sample is either lifted altogether from the cloth, or held very lightly with one hand so it rides high as the medium is applied, then regrasped and more pressure put on. With very large or unusually grabby samples the speed may be considerably lower and still achieve quite a good result. One way of improving this in these cases is to flush the lap with distilled water between applications to get rid of broken polishing medium which is normally thrown off at the higher speeds, and which when left on the cloth can produce various artifacts such as higher relief than normal, so-called comets tails or streaking superimposed on the viewing surface, etc.

Now to continue: at this point change the objective on the microscope to a x40 air objective or somewhere in that neighbourhood to get a final magnification of about x400 - no doubt anything from x250 up would do, depending on availability of equipment. Scan around the sample examining the microscratch systems, and then dim the light using the iris or diaphragm: in the dimmed light the microscratch systems show up well and give at least some idea of what will be seen with an oil immersion objective. This is of special importance when reflectance measurements are to be made, especially in samples where only tiny fragments of coal are available (not as here) as the reflectivity on vitrinite is the key to the rank of the coal and an index of its possible thermal output, economic viability and its general geology. If microscratches are so thick in the field that clear spaces cannot be found for reflectivity measurements, they will reduce the reflectance and cause incorrect assessments to be made. These measurements are characterised by a rather wobbly histogram structure, which gives some guidance at least that repolishing is indicated. This problem is dealt with in the MgO step which follows.

The MgO powder should be kept tightly stored and dry. It is a help to this to have an intermediate bottle to be used for daily access, while the main storage vessel is

opened only occasionally to recharge the intermediate bottle. From the intermediate bottle take several dry spatula loads of MgO powder and transfer to the wetted Selvyt cloth stretched over glass, nap side up; or the material may be shaken directly from the intermediate jar. Work the MgO powder a bit with your clean fingers until water rises out of the cloth and a medium to easily manipulable slurry is available - it should not however be too fluid. Each sample should be worked by hand for about 30 or 40 seconds in each of the orientations, in general a small anticlockwise movement. If a drop or two of water is needed it can be added, and note how little is needed to change the nature of the slurry; or a little extra powder if the slurry is too watery. If something pulls out of the sample and grit is felt between sample and cloth, shift to another area on the cloth. If either pullouts from the sample or suspected magnesium carbonate are felt or the results are seen afterward in the microscope, either carefully clean the cloth in the case of pullouts, or put on a fresh one if carbonate is suspected: it is not worth chancing any further samples on such a cloth as so much labour is already tied up in each sample.

After the MgO treatment, spray clean especially well, treat in the ultrasonic bath, spray clean again, and then pull across the so-called wet cloth - another Selvyt cloth stretched nap up across a glass plate and with water running onto it. (I wish to note here that the entire water supply to the lab should be filtered through a replaceable cartridge filter with 1 micrometre or indeed even smaller openings. This is easily and cheaply placed in the lines and well worthwhile.) To go on, the cloth should be prepared by scraping under running water with a plastic or other scraper without any cutting edges to get any dirt off, then the sample should be pulled hard across the cloth 8 or 9 times, changing orientation 90 degrees each, the length of the draw 25 or 30 cm. Immediately spray clean with vigour and then rinse the sample with distilled water from a squeeze bottle, dry the back and sides with a towel and blot the face on a fresh filter paper. Mount for the microscope (another fresh filter paper for the sample leveller is held between sample and the anvil of the leveller) and examine your sample. The most important thing to note at this stage is the cleared up microscratch systems at x400 (or whatever you have) in dimmed light. In no case will all the microscratches be gone but a substantial improvement should be noticeable, so much so that a beam can be placed in a scratch free area without too much of a search to get a correct reflectance value, or group of values. Although the nominal size of MgO is 1 micron I find that in slurry it crushes to smaller sizes and cuts out lines which have been left in previous polish stages. Although not every sample is wanted for reflectance, in general it is impossible to predict which ones will be and I put all samples through this stage as a matter of course. In addition to making a very good polish, this stage loosens and takes up much of the random dirt which will have accumulated on the sample face, so that together with the ultrasonic and the wet cloth stages which follow, it will be seen that there is very little dirt left to be taken up in the oil used in the microscopic observation of coal and related materials. The final check of samples always includes a low magnification scan around the edges and surface to be sure that all such dirt is taken up, and the MgO stage is seen to be very efficient at cleaning if an experimental comparison is made with samples on which this stage has not been used.

In routine work the edges of the whole sample should now be scanned at low magnification in air to look for any dirt on the edges, and being dirt free on the edges the body of the sample should be quickly scanned at low magnification to be sure no dirt is elsewhere and to look for any other artifacts or signs, e.g. water stains, mostly cured by the distilled water rinse, or traces of oil which may be in the sample. If dirt is seen, repeat the spray clean, wet cloth, spray clean and distilled water sequence and blot dry and reexamine. The reason for this check is that these samples must necessarily be viewed under oil with an oil objective on the microscope; if dirt is present it will mix with the oil and cloud the field, and not only that, it will also invalidate any reflectances which may be measured. As at this stage to clean the dirt out would involve deoiling and

repolishing to some extent and another MgO treatment, a complex treatment which may be taken up another time, it is best to have the specimen clean to start with.

So much for polishing ordinary coal grain mounts and small coal blocks. Topics which I can take up later at your request would be trouble-shooting on these surfaces, i.e. how to find out what's going wrong at any given stage and get around the problems, a subject only touched on here so far; deoiling; and the many other types of samples you will undoubtedly be involved with in any coal petrology preparation lab, e.g.

cokes
"ditch cuttings" (samples of exploratory bore cores)
hydrogenation residues
oil shales in block and grain mounts
in situ preparations of cross-sectioned reactors with
their residues
special problems with mounting and preparing very small
amounts of specimen

and in addition to the above, which can be described in some detail, the use of machines to help in grinding and polishing almost all types of specimens in well-matched groups. As I am mentioning these machines anyway, I should point out that there is no "technological fix" in this field which can get around the necessity for having a skilled and interested operator, or training yourself to these skills: some manufacturing companies seem to claim otherwise.

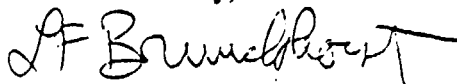
I can also give you some guidance on developing your own approaches to solving problems on materials which you may never have seen, or which no one may ever have seen before, if you request it. The approach to problem-solving is everything.

In closing let me say also that the grinding stages described above can if necessary be carried out on stationary papers with water flowing, i.e. by muscle power, though polishing really must be done with a moving lap to reach a good standard. Moreover instead of paper, the grinding can be carried out with various sizes of free silicon carbide grit on either stationary or moving cast iron laps, with water, finishing on the finest grades with glass plates and hand grinding. These materials are still used here, but nowadays only for special purposes. The surface appearance of the specimen is quite different to that seen with the use of silicon carbide papers at the various grinding stages: rather than scratched it could be said to be stippled. The advantage obtained by using the papers, based on my own scanning electron microscope studies, is to be found in the narrower range of cutting faces presented to the surface to be ground by the silicon carbide when held in the resin base over the paper, compared to the sizes and shapes of cutting faces presented by free grit; as well, there is a substantial advantage from the higher cutting speeds attainable with papers, the grains on which are fixed and not thrown off the wheel at high speeds, as free grit would be.

Attached please find a brief bibliography of materials dealing with coal petrology. Much of the best material, e.g. Stach-Teichmueller, is written out of lifetimes of experience and is best appreciated after rather than before such experience. As far as I know there is not much around for beginners; if you know of anything yourself in this or in the specimen preparation area I would be glad to hear of it.

I do hope that this will have been of some help to you.

Yours sincerely,



L.F. Brunckhorst

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MANUFACTURERS AND AGENTS

Buehler Ltd.
2120 Greenwood Street
Evanston Illinois 60204
United States

Metcloth
Microcloth
Polimet polishing laps
including motors, inset
wells for laps, etc.
Linde C 1.0 micron alumina
Linde B 0.05 micron alumina
Magomet MgO

Struers A/S
Valhojs Alle 176
DK-2610 Rodovre/Kobenhavn
Danmark

Planopol-2 (I use for
grinding with papers).
Pedemax-2: used with
Planopol-2 for grinding
and polishing by machine

Ciba-Geigy

Araldite: contact your
local representative

Dow-Corning Corporation
Midland, Michigan 48640
United States

Silastic J for silicone-
rubber moulds

P. Blashki and Sons
I.M. Sage
109-113 Swanston Street
Melbourne Vic. 3000
Australia

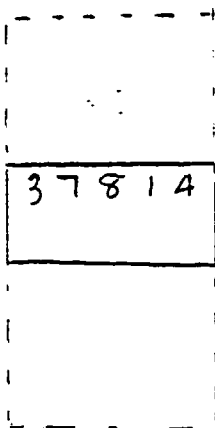
Selvyt Cloth "B" (made in
England but no address
available here)

G.M.B.H. and Co.
Post Fach 1260-4520
Melle I
near Hamburg, W. Germany

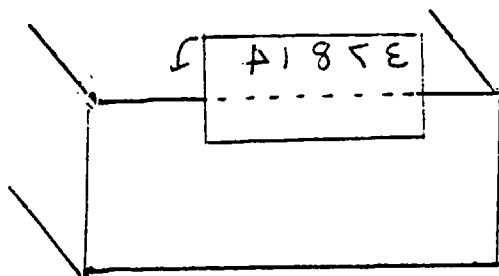
the silicon carbide papers
we use: we order in rolls
50 m x 300 mm and cut our
own circles.

Setting the Numbers

1. Stamp the numbers on aluminium foil

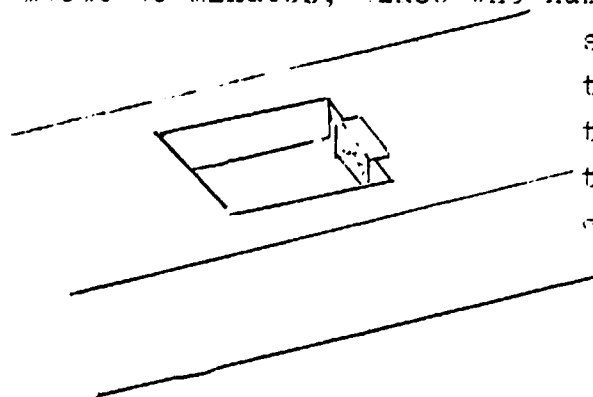


and then cut out the piece indicated.



2. Turn the piece over and smooth with fingers on a flat surface, then place over a squared-off piece of marble or the like and fold as indicated.

3. After the top layer of araldite has been added on and bubbles wiped off after about 10 minutes, place the number as seen here and be



sure the number is out into the elastic 0.5 mm or so, so that it will not be lost at trim stage by pulling out or grinding through.

Advantages: it is not necessary to wait for the bottom layer to set hard so that the number can rest on it - only hard enough so that adding the top layer will not disturb distribution at the bottom; and as well, the problem of the number strip falling over is avoided as it is supported from the top. At the finish just cut off the extending extra bit.

TABLE 1. LIST OF KIER SAMPLES

Sample No.	Coal seam	Age	Area	Group
7	Old ↓ Young	Upper Carboniferous	Samcheog coalfield	PYEONG AN GROUP
8		to	Sabug area	
9		Lower Permian	(western part of	
10		field)		
11				
13				
17	Old	Upper Carboniferous	Samcheog coalfield	"
18	Young*	to	Dogae area	
19	Young		(northeastern part	
20	Young)	Lower Permian	of field)	
21	Old	Upper Carboniferous	Jeong Seon coalfield	
22	Young	to Lower Permian	Gujeol area	
			(northeast part of field)	
24	Old ↓ Young		Chung Nam coalfield	DAE DONG
25			Seong Ju area	SUPER
26		Upper Triassic	(middle part of field)	GROUP
27		to		
28		Lower Jurassic		
29				
30				
31				
32				
34				
35				
36				
37				
38				
39				
40				

* The same seam, repeated by faults.

Sample No.	Coal seam	Age	Area	Group
41	Young	Upper Carboniferous	Samcheog coalfield	PYEONG AN
42	↑	to	Hambaeg area	GROUP
43		Lower Permian	(most western	
44			part of field)	
45		Old		
46	Young	Upper Carboniferous	Samcheog coalfield	"
47	↑	to	Jangseong area	
48		Lower Permian		
49			(middle part of	
50			field)	
51	Old			

TABLE 2. PETROGRAPHIC ANALYSES (vol %) OF FOUR KIER SAMPLES

Sample No.	20	21	25	26
Graphinite	-	24	-	-
Graphinite-b	-	12 36	-	-
Impregnite	-	-	2	-
Vitrinite-1	12 } 37	9 } 18	33 } 50	76 } 87
Vitrinite-2	4 }	2 }	5 }	10 }
Vitrinite-b	21 }	7 }	12 ^Δ }	1 }
Mottlite-1	- } 59	- } 29	2 } 39	-
Mottlite-2	14 }	19* }	36 }	tr
Mottlite-b	45 }	10 }	1 }	-
Inertinite	-	-	5 }	-
Inertinite-b	tr	-	1 } 6	-
Coaly shale	3	8	tr	7
Minerals	1	7	3	6

*Including 6% graphinite/mottlite intermediates

Δ Including 2% vitrinite particles which contain impregnite networks.

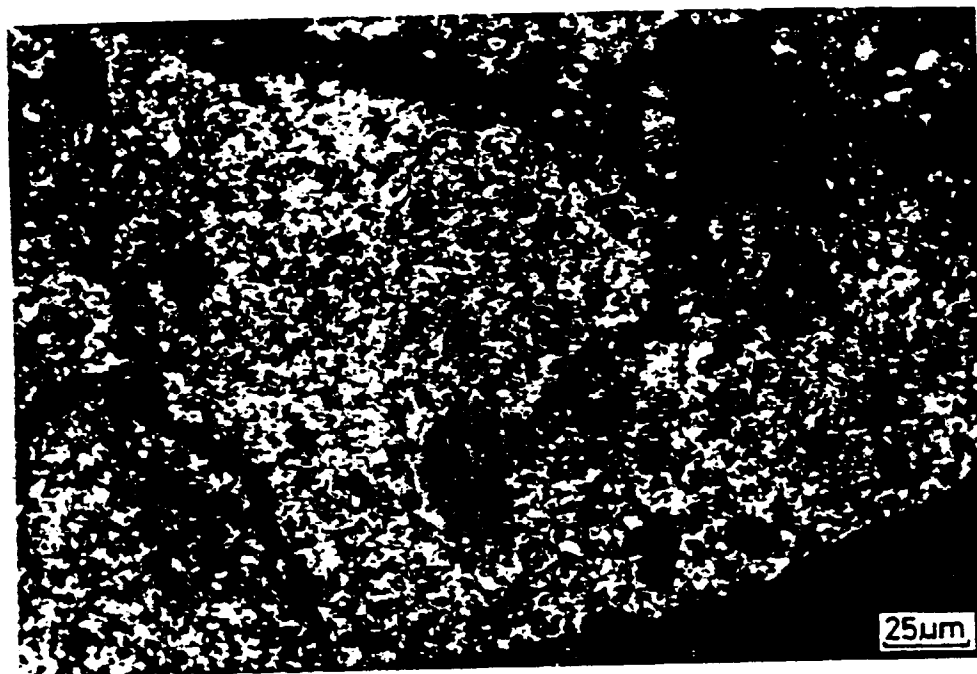


FIG. 1 GRAPHINITE IN SAMPLE 22 (JEONG SEON COALFIELD, GUJEOL AREA)
(Incident light, oil immersion)

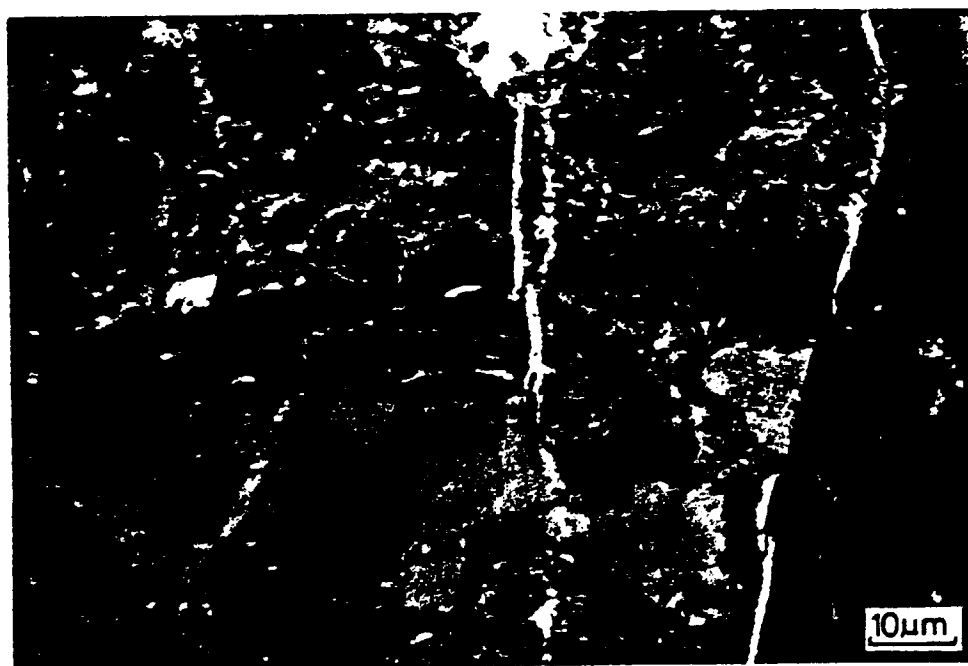


FIG. 2 CRACK FILLING IMPREGNITE (VERTICAL YELLOW VEINS) IN SAMPLE 22 (JEONG SEON COALFIELD, GUJEOL AREA)
(Incident light, oil immersion, partially crossed polarizers)

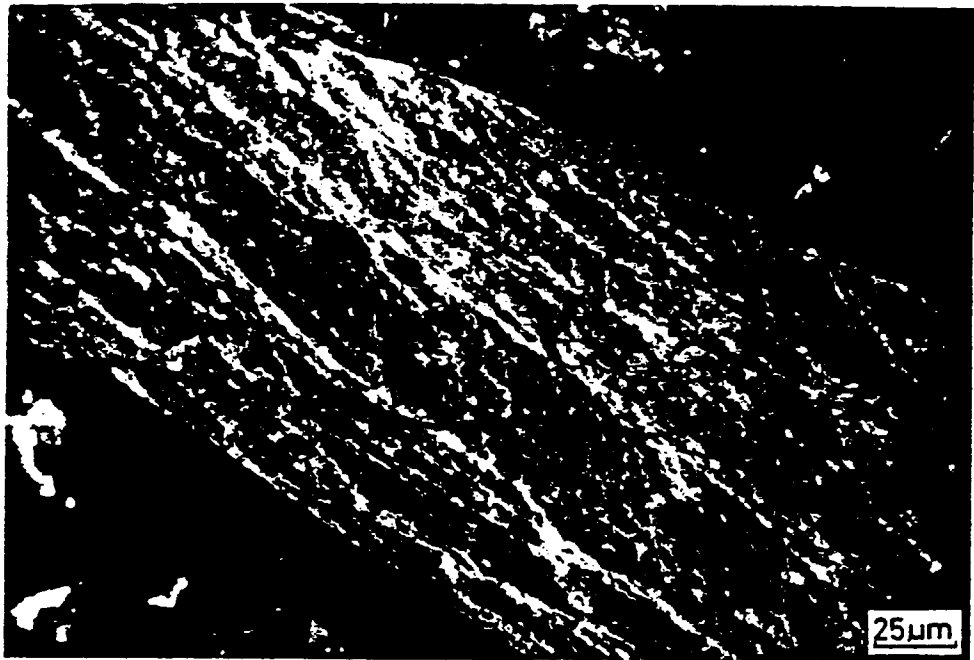


FIG. 3 NETWORK OF IMPREGNITE (PALE YELLOW) IN SAMPLE 25 (CHUNG NAM COALFIELD, SEONG JU AREA)
(Incident light, oil immersion, partially crossed polarizers)

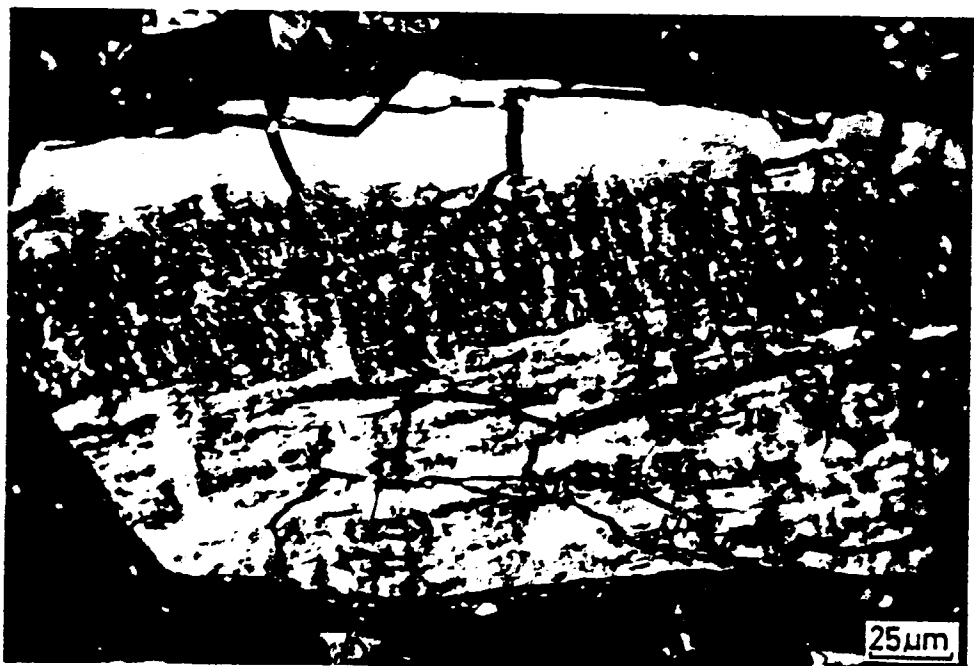


FIG. 4 MOTTLITE IN SAMPLE 25 (CHUNG NAM COALFIELD, SEONG JU AREA)
(Incident light, oil immersion, partially crossed polarizers)

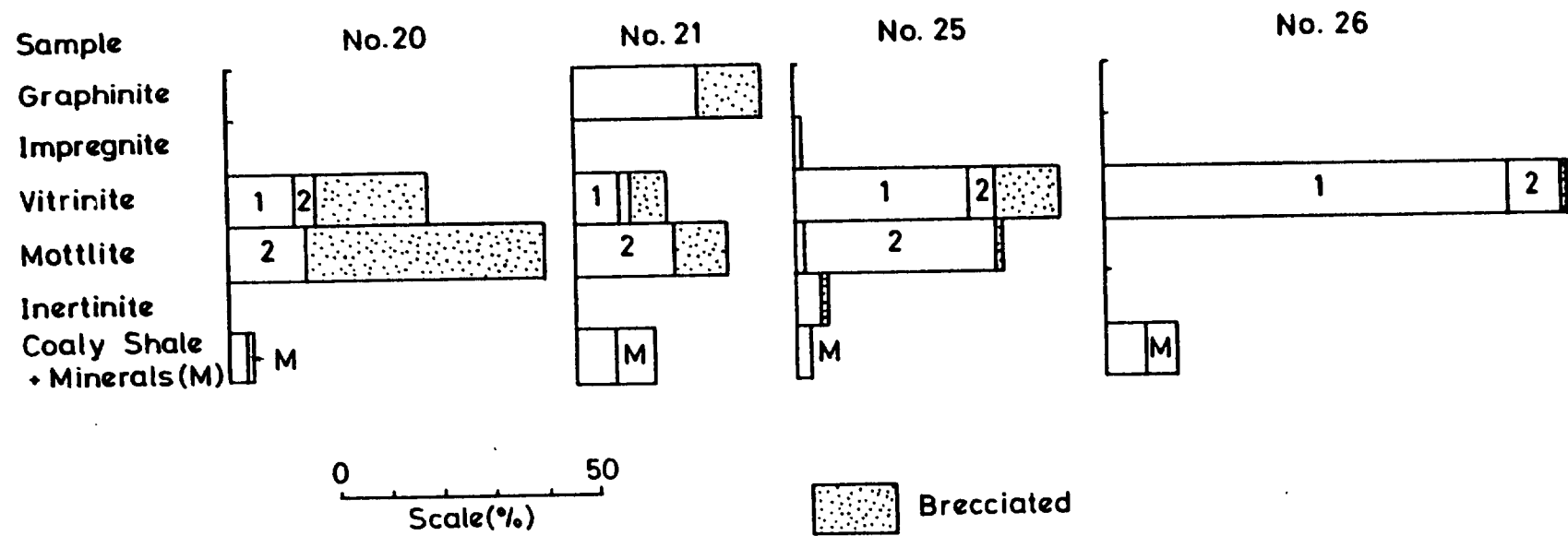


FIG. 5 PETROGRAPHIC ANALYSES OF FOUR KOREAN ANTHRACITE SAMPLES
 1, 2 denote vitrinite-1, vitrinite-2 etc.

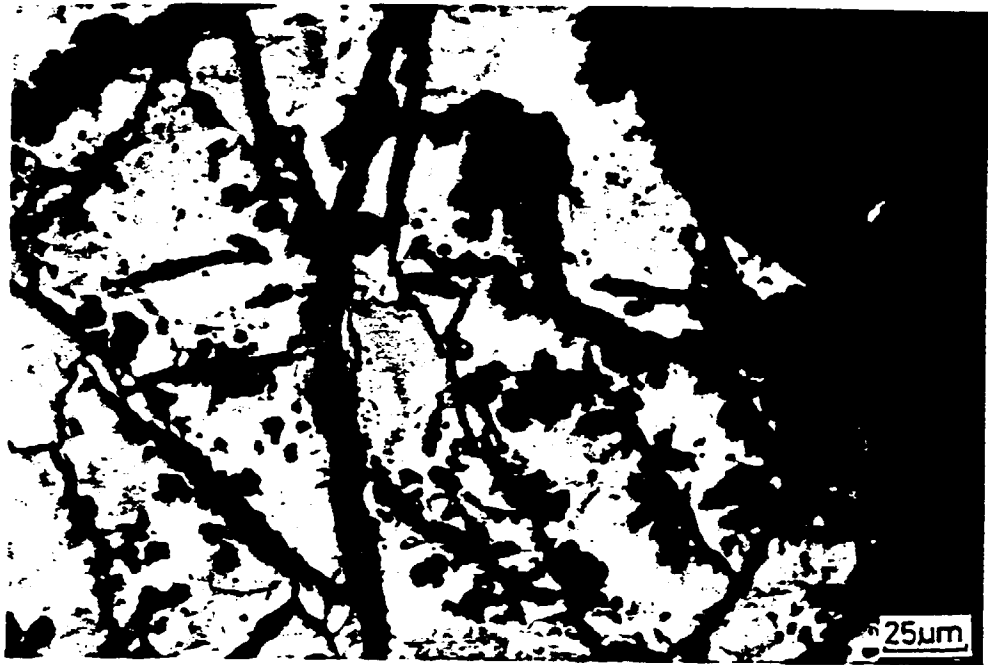


FIG. 6 COMBUSTION ON THE SURFACE OF EACH BRECCIATED GRAIN. PARTIALLY BURNED KOREAN ANTHRACITE IN CSIRO FLUIDIZED-BED COMBUSTOR (Incident, light, oil immersion)

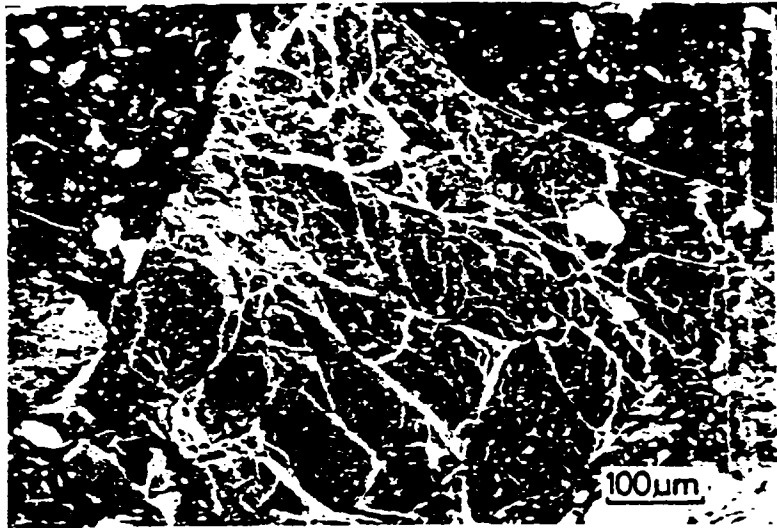


FIG. 7 CRACKS IN ANTHRACITE FILLED BY MINERALS (WHITE NETWORK) IN SAMPLE 42
(SAMCHEOG COALFIELD, HAMBAG AREA)
(Scanning electron microscopy)

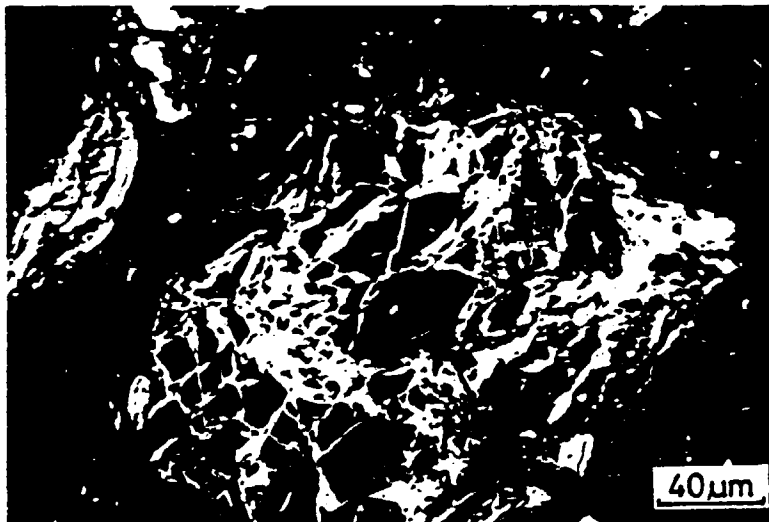
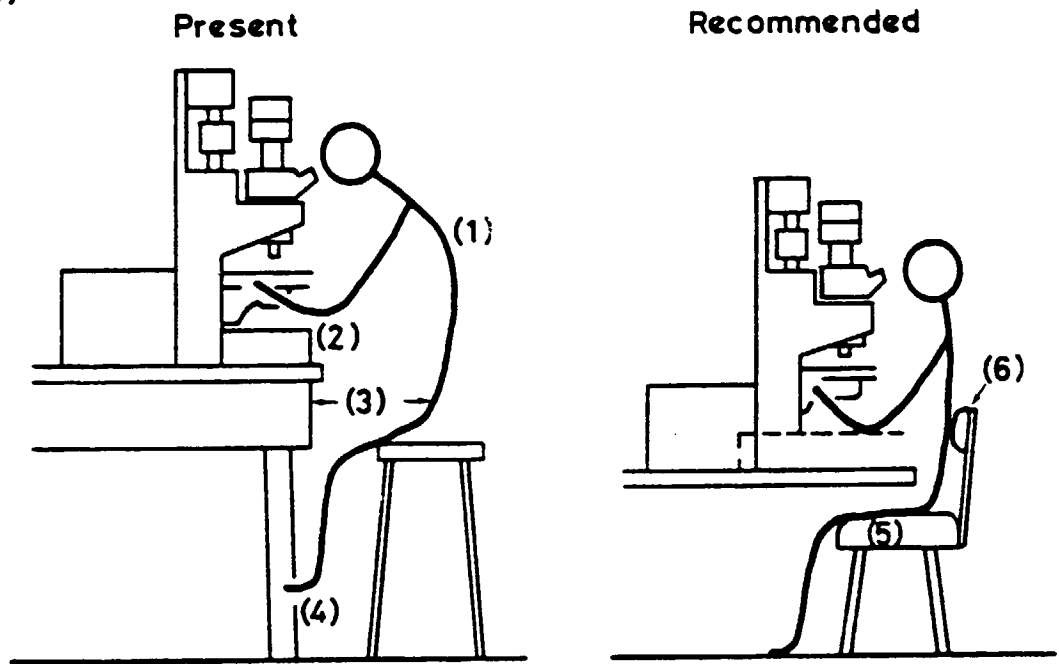


FIG. 8 CRACKS IN ANTHRACITE FILLED BY MINERALS (WHITE NETWORK) IN SAMPLE 42
(SAMCHEOG COALFIELD, HAMBAG AREA)
(Scanning electron microscopy)

(a)



- 1 Spine is bent
- 2 No arm rest
- 3 Too large gap
- 4 Feet are not touching the floor
- 5 Good cushion
- 6 Proper back support

(b)

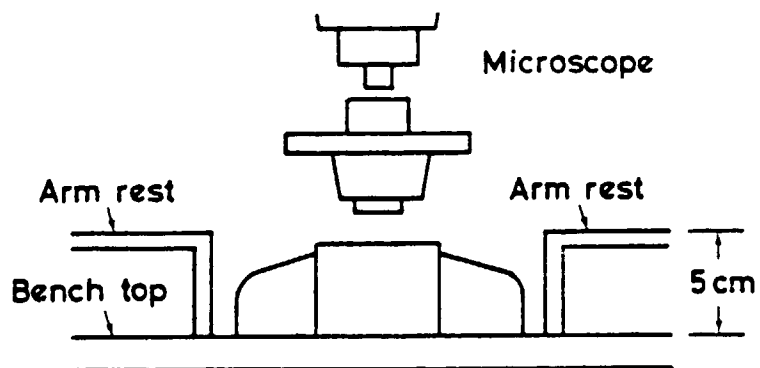


FIG. 9 RECOMMENDED MICROSCOPE BENCH ARRANGEMENT