## MICRO-CHEMICAL STUDY OF ORE MINERALS.

. .

Ъy

# J. ADAM WATSON, F.I.C., A.C.G.I.

## CONTENTS.

| I.   | INTRODUCTION.  | 1.  |
|------|--|-----|
| II.  | PREPARATION OF POLISHED SECTIONS OF ORES.                                | 4.  |
|      | (a) Characteristics of polished<br>surfaces - grinding - polishing.      | 4.  |
|      | (b) Abrasives and Polishes -<br>characteristics - grading.               | 7.  |
|      | (c) Preparation of sections - cutting - grinding - polishing - mounting. | 11. |
| III. | EXAMINATION OF POLISHED SECTIONS.  | 16. |
|      | (a) Microscopical examination -<br>etching.                              | 16. |
| +    | (b) Micro-chemical examination.  | 20. |
| IV.  | THE ORE DEPOSIT AT TALNOTRY, NEWTON-<br>STEWART.                         | 23. |
|      | (a) Geology of the area.   | 23. |
|      | (b) The Ore mass.  | 27. |
|      | (c) The Ore minerals.  | 28. |
| v.   | SUMMARY.   | 37. |
| VI.  | ILLUSTRATIONS.   | 40. |
| VII. | REFERENCES TO LITERATURE.  | 46. |

#### THE MICRO-CHEMICAL STUDY OF ORE MINERALS.

#### I. INTRODUCTION.

The work recorded in the following pages is a continuation of that previously carried out in the Geological Department and published in the <u>Mineralogical Magazine</u> (1) in 1935 under the title "Colour reactions in the micro-chemical determination of minerals". A copy of that paper accompanies this report as the methods elaborated in it were applied in the present study.

The decision to continue the above work into the region of Ore minerals was made in the full realisation of the fact that preliminary work would be required on the preparation of polished sections of the ores and their examination using reflected instead of the transmitted light ordinarily used in petrological work. As the polishing of minerals requires a special technique that is not used extensively in this country a considerable part of what follows is devoted to describing this preliminary work on abrasives, polishing agents and the preparation of polished sections.

The pioneer work on the subject was done by Murdoch (2) in 1916. Following this were important

publications by Davy and Farnham (3), Schneiderhohn (4), Van der Veen (5), and Short (6). These are either American or German workers and the processes have been developed and applied mainly in these countries. On that account many of the grinding materials recommended by them are unobtainable at home. Again these methods vary in many ways so that it was considered advisable to start at the beginning and examine the problem of polishing in the light of the principles laid down for metallographic work and use such abrasives as were commercially obtainable. These commercial articles not being sufficiently uniform in grain size for the finer grinding operations a method had to be elaborated for obtaining from them a series of uniform products. When this had been done it was possible to formulate a scheme of grinding and polishing. The scheme given here combines features of several already published methods and gives polished sections that are satisfactory for the study and identification of the constituent minerals.

The next step was to gain experience in identifying the common ore minerals. This was obtained by polishing and studying specimens of these minerals in as pure a state as possible.

Finally a small Ore deposit at Talnotry, near Newton Stewart, was studied, samples were collected and these, after polishing, were examined, the chemical examination being carried out according to the scheme given in the paper mentioned above. II. PREPARATION OF POLISHED SECTIONS OF ORES.

### (a) Characteristics of polished surfaces.

In order that the following procedure may be fully appreciated the general characteristics of polished surfaces must be kept in mind. Sir George Beilby (7) showed that the smoothness and lustre of polished surfaces is caused by the material liquefying under the stress of polishing agents and solidifying at once while still under the influence of surface tension to a surface characteristic of a liquid. Thus a certain thickness probably of molecular dimensions is either vitreous or micro-crystalline compared with the macro-crystalline nature of the original mineral. Comfirmation of this has been given by G.I. Finch (8) by X-ray investigations. The depth to which this alteration proceeds is a function of the polishing agent. In discussing the preparation of polished metallurgical specimens Professor Desch (9) states that rouge has an excessive dragging effect on a metallic surface so that while giving a high polish its powerful action produces flowing to a considerable depth. He therefore recommends the use of less powerful polishes. If the course of polishing be watched by examining a specimen frequently as the operation proceeds, the gradual rounding of the edges

of cracks will be noted and further it will be found that too prolonged or vigorous polishing will give a wavy surface. This fundamental work of Beilby's appears to have been overlooked or ignored by some writers on polishing methods (10).

There are two processes involved in the production of a satisfactory ore section. First the Grinding of the specimen to a fine matte surface. This is attained by using successively finer abrasives. An abrasive appears to act by splintering the surface, and material, commensurate in size with the abrasive grains, is only removed at the intersection of two or more lines of splintering. This splintering action affects the material below the bottom of the cavity formed and as the extension downwards is determined by the size of the splintering grain it is unadvisable to use coarse abrasives since they may give rise to strain phenomena which are foreign to the mineral affected. It is better, therefore, to cut the section by means of a rotating disc than to chip it and subsequently flatten the surface with coarse ( carborundum.

In grinding the form of the splinter depends on whether the abrasive is free to move or fixed. Free abrasive grains give splinters which are convex to the direction of grinding, while with fixed grains the splinters are concave to this direction. When the grain is fixed the action is restricted to that portion of it that comes into contact with the surface so the width of the splinter produced is reduced. At the same time when using a very fine abrasive cohesion may take place between it and the surface and the complex so formed gets removed bodily, and a small consequent molecular rearrangement results in a slight flowing of the surface and polishing (11). The condition of fixed grain grinding is attained by spreading the abrasive on wet linen or flannel. This way of using an abrasive is employed in the last stage before passing to the second operation, that of Polishing.

<u>Polishing</u>: For efficient polishing the polishing agent must be in a fine enough condition to permit of cohesion taking place between it and the surface. Since the thickness of the flowed skin increases with the time and pressure of polishing the operation must be carried out as lightly and quickly as possible. A further point is that the polishing agent must be chosen with reference to the mineral being polished. For most minerals magnesium oxide has been found to be the most satisfactory but some require chromic oxide while with pyrite, rouge must be used.

#### (b) Abrasives and Polishes.

There is a definite difference between Abrasives and Polishes and this difference is essentially one of grain size. Beilby (12) has shown that the finest abrasive, as exemplified by emery, consists of individual crystalline grains observable under the mocroscope. A polish, such as rouge, is made up of particles showing Brownian movement, which particles are themselves aggregates of still finer particles. The efficiency of a polishing agent depends on the size and hardness of these ultimate particles.

With one exception the abrasives and polishes used in this work are oxides. The exception is Carborundum - a silicon carbide - which is used for the early stages of grinding. Since even the finest carborundum will not give a surface smooth enough for polishing, a grinding operation using flour emery or preferably calcined alumina must follow the use of carborundum. The final polish is obtained with magnesium oxide, chromic oxide or rouge (ferric oxide), and of these the most generally useful is magnesia. These three substances have specific uses; thus the majority of ore minerals will take on a good polish with magnesia, a few require chromic oxide, while pyrite requires the continued action of rouge. This difference in behaviour is not entirely dependant on

- 7 -

the hardness and it would be misleading to lay down limits in terms of the usual hardness scale.

Grading: The majority of workers on the subject agree in advocating the use of alumina for the last stages in grinding. Unfortunately the grades and brands recommended are not easily obtainable in this country so a start was made using Sira for the purpose, this being a commercial product. As it did not prove satisfactory a number of samples of calcined alumina of varying grades of fineness were obtained through a friend connected with that industry. Experiments with these showed the fineness necessary for the work in hand. After this a supply of the finest calcined alumina was obtained from the British Aluminium Company. As obtained this was neither fine nor uniform enough for use and it was subjected to elutriation in order to obtain a series of products of increasing fineness and each one of uniform grain size. The simple method of shaking up with water and allowing it to settle for a definite time was tried. As, however, the great bulk of the material had settled in a few minutes leaving only a small quantity in suspension, this method was abandoned as being impracticable for obtaining any quantity of fine material without great loss of time. A Crooks Elutriator was then fitted up and used. By this

- 8 -

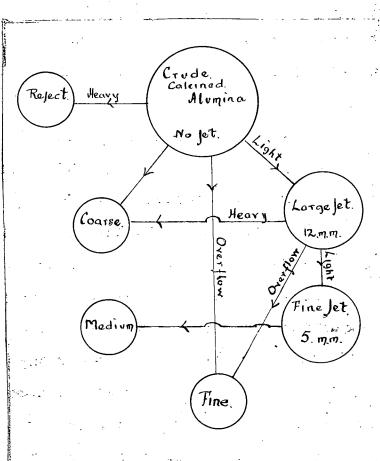
means a satisfactory product of great fineness was obtained. But the apparatus does not permit of the grading of the heavier part, and at the same time. if any quantity of alumina be treated at once the efficiency of the washing falls off owing to the formation of channels in the solid up which the water passes carrying little or no solid with it. Later on an Andrews Elutriator became available which enabled the alumina to be separated into three grades. The design of the apparatus with its three vessels connected through stop-cocks permits of the separation of any material into three portions together with the finest portion that is carried over with the overflow. As the rate of flow of water can be altered it is obvious that a very efficient separation can be made. The rate of flow of water is governed by the size of the outlet and the pressure of the water supply. Two jets are supplied with the apparatus which is fitted with a manometer whereby an arbitrary measurement can be made of the pressure.

The apparatus was standardised by running it with water alone in order to connect the rate of flow through the jet (c.c.s per sec.) with the manometer reading (in mms.). These figures serve as a basis on which to start the apparatus. No actual measurements of grain size were made of the grades of alumina used in grinding but a microscopical examination showed that there were no outsize grains present. Having determined the conditions of rate of flow which gave the required grading of the crude alumina it was possible to repeat this at any time and be certain of obtaining an identical product.

Three grades - coarse, medium and fine - were prepared from the commercial alumina by the following procedure.

The water supply was turned on to the apparatus, no solid being present. The constant level tank was so adjusted that using the large jet a flow of 5 c.c. per sec. was maintained under a manometer reading of A quantity of alumina was introduced, the 12 mm. jet was removed and the operation continued with this flow of water as long as the overflow contained any This overflow was collected and allowed to solid. settle, the solid separation forming the Fine grade. The heavy portion which had collected in the bottom vessel was withdrawn and rejected. The apparatus was started again with the large jet inserted and the manometer maintained at 12 mm. Any overflow was collected, allowed to settle and added to the Fine The heavy portion was removed from the fraction. apparatus at the bottom and formed the Coarse grade. The apparatus was started again with the fine jet and

- 10 -



•• ...

a pressure of 5 mm. Nothing passes over in this operation but the material separates into two fractions in the two upper vessels of the apparatus. That in the lower vessel forms the <u>Medium</u> grade, that in the upper vessel is bulked with the <u>Fine</u>.

The diagram opposite is a flow sheet of the operation.

The three grades of alumina so obtained were found to be uniform products and behaved satisfactorily in the grinding scheme which follows.

It was not found necessary to grade any of the polishing materials used. Magnesium oxide is in the form of the pharmaceutical preparation. Magnesia Ponderosa is a uniform powder that requires no treatment. It should be noted that the Magnesia Levis is quite unsuited for polishing and care must be taken always to obtain the heavy material. Experiments on the elutriation of fine jewellers' rouge and commercial green chromium oxide showed that they both contained so small an amount of coarse material that it could be neglected.

(c) Preparation of polished Sections.

<u>Cutting</u>: The advantages of cutting instead of chipping sections have already been mentioned and explained. Fortunately the cutting can be done expeditiously and economically by using a revolving disc of soft iron running in a trough containing a thin paste of medium carborundum and rockflour in water. This method is much cheaper than using diamond dust to edge the cutting disc as is commonly done.

Grinding: The cut section is smooth enough to commence grinding on a glass plate using fine carborundum (220 grade) and plenty of water. When the surface is flat and uniformly ground it is washed and transferred to another glass plate and grinding continued with a finer carborundum (600 grade). After a short grind with this abrasive the surface will again be uniformly ground but of finer texture. This is followed by a third grinding still on glass using the medium grade of Calcined Alumina described When examination under the microscope shows above. the surface to be uniform but still finer in grain the last grinding is proceeded with using the fine calcined alumina still on glass. When all large cracks and pits have been removed by this grinding the section is ready for polishing. All the grinding has been done by hand but rotating hard steel laps may be used if so desired. The speed of these should not be higher than 500 - 750 R.P.M. The author does not consider that the time saved by

using mechanical means compensates for the loss of control that is inherent in hand working.

The first stage in polishing is Polishing: carried through using fine calcined alumina spread on wet flannel laid on a glass plate (cf. page 4 above). As this process will at most give only a slight polish it is only continued for a very short time (not more than one minute). The final polishing that follows it immediately is started using magnesia spread thickly on a piece of broadcloth, or two thicknesses of good flannel well wetted with water. During this operation the section must be examined frequently. Provided all the minerals present are only of moderate hardness (H =  $4 \cdot 5$ ) a good mirror-like surface will be obtained in about 4 minutes. The section will then be ready for examination.

If, however, after this time a good polish has not been obtained it may be concluded that the minerals present are not such as will polish satisfactorily with magnesia and a change must be made to chromic oxide or in extreme cases to rouge. Needless to say this must be done on a separate pad which pad should have an additional layer of flannel.

When there are two minerals present that differ greatly in hardness, e.g. arsenopyrite and galena, it will not be possible to obtain a uniformly polished surface without producing smearing of the soft mineral over the junctions and considerable relief between the two minerals. In such a case it is advisable to polish the soft one first and examine the section before proceeding to polish the harder one.

The following facts regarding polishing will be found scattered through the literature and from the writer's experience they are of great importance in obtaining a satisfactory section:-

- 1. A hard mineral requires to be polished on a <u>soft</u> pad using a powerful polish, e.g. chromic oxide or rouge.
- 2. A soft mineral requires a hard pad and a polish such as magnesia.
- 3. The use of a soft pad tends to increase the relief between minerals.
- 4. Prolonged polishing tends to smear the softer mineral over the junctions, to produce a wavy surface and increase the thickness of the "flowed" skin.

The surface that will yield the most information under microscopical examination will be produced by a procedure that balances these conflicting influences and this can only be achieved by experience.

Mounting: Unless the specimen is a large one it is advisable to mount it on a microscope slide. The advantage of mounting is that it can be so done that the surface under examination is parallel to the slide although the two faces of the section may not be strictly parallel.

It was found that Canada balsam was not a satisfactory mountant as, being very fluid when molten, it was not easy to support a section of which the faces were not parallel. This difficulty was got over by mixing the balsam with rouge. This gave a material which did not flow when melted yet was sufficiently soft to allow the section to be pressed into it. By using a small screw press having a large face parallel to the base, the section may be bedded into the softened mountant in a position such that the face of the section is parallel to the slide.

This mountant when cold is rigid enough to withstand the necessary handling for examination purposes but it will not allow of any considerable polishing. Hence the section should be finished before it is mounted. III. THE EXAMINATION OF POLISHED SECTIONS.

#### (a) Microscopical Examination.

The order in which the various minerals begin to show up should be noted during the polishing process as this order is a good indication of the relative Amongst the first to take on a polish is hardness. the common mineral galena (H - 1.5). Again as polishing proceeds various characteristic markings appear which are removed as the final stage is approached and these are associated with the crystalline properties, being related either to the form or habit of the crystals. Outstanding examples of these are the triangular cracks on the surface of galena which are the traces of the corners of the cube faces and the flaky surface of pyrrhotite due to the tabular habit of the crystals. In the early stages of polishing the form of the constituent grains of certain minerals often show clearly, but they disappear as polishing proceeds and are only made evident by subsequent etching.

While many minerals have a characteristic colour in hand specimens this property is less definite in a polished section owing to the fact that a large amount of the light is reflected unchanged from a well polished surface. There are, however,

differences in shade even in the silvery or greyish minerals that can be distinguished with practice. As is well known the effect of a colour depends on its surroundings so that a mineral will appear to be a different tint against differently coloured Like all visual identifications of associates. minerals this colour effect is a question of experience and this experience was gained by studying polished sections of known mineral associations. To avoid unnecessary complications artificial light was used throughout. This light was obtained from a 12 volt lamp attached to the microscope and fitted with a blue screen of sufficient density to counteract the yellow colour of the light. The bulb of the lamp was ground in order to diffuse the light and avoid an image of the filament appearing in the field.

Etching: The etching effects produced by various solutions have been studied by all investigators in this work and a review of the methods suggested is given by Short (13). The results may be classified roughly under three headings:-

- 1. Colour changes due to tarnishing.
- 2. Pitting of the surface due to solution of the mineral.
- 3. Development of markings due to boundaries of mineral grains or crystalline structure such as twinning and zoning.

It would be thought that a method giving results of such a varied character should be of great diagnostic value. Unfortunately, in practice, the reactions are somewhat uncertain so that this value is materially reduced. Thus a given mineral does not always react to the same extent with a reagent. Sometimes no result at all is obtained and at other times the effect is so feeble as to be of little use. This may be due to the different orientation of the crystals or it may result from electrolytic effects between adjacent minerals.

In the publication quoted above, Short advocates placing a drop of the etching liquid on the mineral under examination and viewing the surface microscopically through the liquid for the whole duration of the reaction. He recognises the possibility of electrolytic interference by bordering minerals and tries to avoid it by working on a mineral of such size that the drop will not spread beyond it. This obviously cannot be done with small grains. But the main objection is the inadvisability of bringing the microscope objective within the influence of acid He also makes use of the tarnished aureole fumes. surrounding the mineral where the fumes have attacked the surface. While these aureoles might be definite with more or less pure minerals, the presence of other minerals than that being tested makes the stains so difficult to interpret that, so far as the experience of the present writer goes, the method is too uncertain to be really useful.

The most useful etch reactions are those which show up the grain boundaries and internal crystalline structure and on the whole the most satisfactory way of obtaining these is to adopt the method of Schneiderhohn (14) and immerse the whole surface in the etching solution. After leaving it for a standard time it is removed, washed and polished very lightly to remove any staining. Quite good results are obtained in this way, but even so there are often patches of a mineral in a section which show no After the examination is completed the etching. section may be repolished and tested with another reagent.

Many solutions have been suggested for etching but in the writer's experience the most generally useful are potassium permanganate with either acid or alkali, potassium bichromate, ferric chloride and alkaline hydrogen peroxide.

It is possible to etch certain minerals electrolytically using a weak current such as may be obtained from a 4 volt dry cell, the section being immersed in a weak electrolyte and connected to one pole of the battery. This method is described by Schneiderhohn (**DS**), who connects the section to the positive pole of the battery. Some unexpected results were obtained with this method, and as a preliminary investigation gave very interesting results it is proposed to reserve a description of them for a later paper.

#### (b) Micro-chemical Examination.

While the etch reactions already described should strictly be included under this heading, it is more convenient to consider them separately and restrict to this section the qualitative tests for individual elements.

All the writers on polished ore work devote considerable space to microchemical tests carried out according to the method formulated by Behrens (15) and elaborated by Behrens and Kley (16) and Chamot (17). The tests here depend on recognising the crystalline or coloured precipitates given by the elements with specific reagents, the operations being carried out on glass slides on the microscope stage. As, however, the appearance of the crystals tends to vary with the conditions of formation and the presence of other elements, considerable experience is required before the method can be used with certainty, while the necessary precautions are so elaborate that one valuable attribute of microchemical methods, that of rapidity, is largely lost.

It was to overcome this defect that the present writer carried out the work on "Spot Reactions" embodied in the paper mentioned in the introduction (1). The history of that research is briefly outlined in the paper which accompanies this report, so it need not be repeated here.

While this method was elaborated for use in ordinary mineralogy, it is admirably adapted for the work presently in hand. Working under a 1 inch objective on the microscope small quantities of a mineral can be removed with a dissecting needle and after solution in an appropriate acid can be analysed by the scheme of "Spot Reactions" described in the paper. By this means it is possible to analyse qualitatively minerals occurring as small grains or inclusions within the ore mass. These colour reactions give the best results in comparatively weak solutions so that only very small quantities of the mineral are required for the analysis. Thus it is comparatively easy to examine small grains of any mineral and to be sure at the same time that there is no admixture of adjacent minerals in the sample taken. The advantages of micro-chemical analysis, by

- 21 -

whatever method it be carried out, are obvious, since to make an ordinary qualitative chemical analysis would require a much larger amount of material which in the case of small grains would be difficult to obtain in an uncontaminated state. An examination on the lines above indicated will give the same information as a bulk qualitative analysis of the ore and in addition it will indicate how the various elements occur in the mass. This information is indispensable to a complete mineralogical study and at the same time is of considerable economic value when dealing with mixed ores having a fine grained structure.

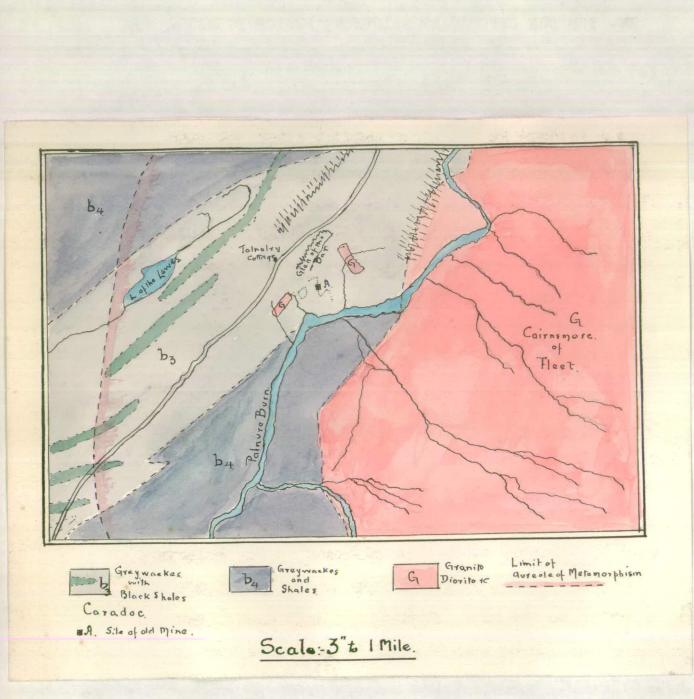
22

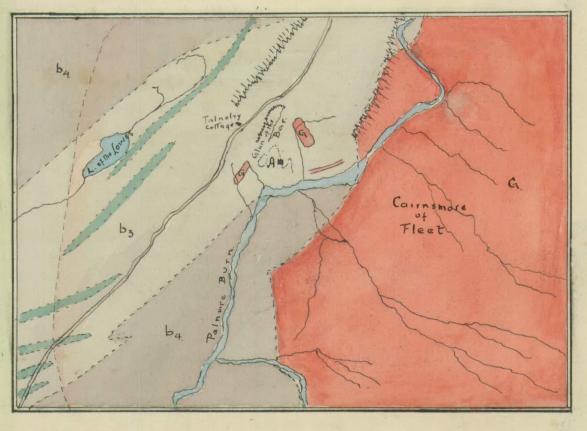
## GEOLOGICAL SKETCH MAP.

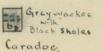
TALNOTRY, KIRKCUDBRIGHT.

Scale -  $3^n = 1$  mile.

included in 1" Geological Survey Map Sheet 4.











Limit of ---- Qureate of Metamorphism.

Am Site of Old mine .

IV. THE ORE DEPOSIT AT TALNOTRY, NEUTON STEWART.

(a) Geology of the area.

In order to try out the methods of examining ores described in the above pages a study was made of the ore deposit at Talnotry, Newton Stewart. This occurrence is particularly suitable for the purpose being of limited extent and fairly accessible, while the mining that has been carried out allows of the easy examination of a deposit which is of interest as being one of three known occurrences of nickel ore in the country.

The area is included in Sheet 4 of the 1 inch geological map of the Geological Survey. Reference to this map will show that the ore mass, which is of small dimensions, is situated about six miles northeast of Newton Stewart and some two hundred yards east of Talnotry Cottage on the road to New Galloway.

A sketch map is given here, on the scale of 3 inches to the mile, showing the main geological features. From this it will be seen that the country rocks consist of greywackes and black shales of lower Silurian age having the N.E. - S.W. strike normal to the region. The ore deposit lies at the base of a mass of diorite within the aureole of metamorphism that extends for about a mile from the

- 23 -

edge of the large Cairnsmore of Fleet granite mass.

A series of specimens was collected across the strike of the rocks from beyond this aureole into the granite mass. A short account of these will serve to illustrate the conditions obtaining.

The slides are described in order from S.W. to N.E.

 $\underline{T4}$  - collected at the 40th milestone on the Newton Stewart to New Galloway road (the mileage is reckoned from Dumfries), is a normal greywacke showing no sign of thermal metamorphism.

<u>T3</u> - collected from the roadside  $2\frac{1}{2}$  miles N.E. of the locality of T4 is a hard sandy shale of normal type without evidence of thermal metamorphism.

<u>T2</u> - collected on the roadside at the 37th milestone, that is well within the metamorphic aureole, is a rather fine but uneven grained greywacke showing clearly the development of secondary biotite.

 $\underline{T5}$  - collected from a tunnel driven into the country rock some 15 ft. below the hanging wall of the ore body and within 400 yards of the granite margin is an uneven grained greywacke showing very clearly hornfelsing and formation of secondary biotite.

 $\underline{T1}$  - collected from the Palnure Burn below Talnotry cottage is from the Cairnsmore of Fleet mass and shows it to be a muscovite, biotite granite. It has been described as an adamellite by Tyrrell (18).

The ore mass is described by the Geological Survey (19) as occurring as "a so-called magmatic segregation occurring at the base of an irregular sill-like intrusion of diorite". The following specimens were collected across the strike and through the ore to the underlying sediments.

<u>T 9</u> - collected from the N.W. summit of the sill-like mass, appears to have been a normal diorite in which the main ferromagnesian mineral was hornblende in large crystals. The rock, however, has been much altered with the saussuritization of the felspar, the development of sphene and particularly the development of secondary biotite. This is apparently due to the thermal action of the Fleet granite and therefore the diorite is somewhat earlier in age.

The mine and the mineralisation to be described appears to lie on a local line of crushing traversing the diorite, that is easily traceable in the field. It appears to be the foot wall of the ore mass but may not be the actual base of the diorite. Two small adits have been driven into the mass, one at ground level, the other some 15 feet higher up. The specimens were collected from the upper adit. T 10 to T 17 were taken in descending order from the diorite to the sediments.

<u>T 10</u> - is a diorite somewhat richer in hornblende than, and showing the same alteration as, T 9.

<u>T 11 and 12</u> - are similar to T 10 but show more markedly the formation of biotite.

<u>T 13</u> - is similar but shows both in hand specimen and slide signs of shearing and bending of the hornblende crystals.

<u>T 14</u> - collected where the mineralisation begins, shows a rock richer than the normal in hornblende. Shearing is more evident, being seen best in the hornblende where the crystals are bent and small fractures have developed. The ore minerals appear to be uncrushed and have often invaded the rock-forming minerals along cleavages and fine cracks.

 $T_{15}$  - is similar to T 14 but richer in ore. <u>T 16 and 17</u> - represent the sediments some five feet below the ore mass. They are a greywacke and a shale showing a marked hornfelsed structure.

Study of these slides leads to the following conclusions. The diorite is earlier than the granite and has been metamorphosed by it. The ore minerals are localised in the diorite while pyrite is generally met with inside the metamorphic aureole. This mineralisation seems to have occurred along a narrow

- 26 -

belt of crushing and the emplacement of the minerals appears to be later than that crushing but probably not by a great time.

#### (b) The Ore Mass.

The ore occurs in a narrow band along the base of a mass of igneous rock. The junction between the igneous and sedimentary rocks outcrops on the top of a knoll on the slopes of which the mine workings are A little way down the slope a small cavesituated. like excavation has been dug cutting both the igneous and sedimentary rocks so that the junction forms part of the roof. Ore occurs in both rocks with the main mineralisation in the ignoous, but as the ore thins out to the outcrop very little ore is seen in the excavation and the junction is taken provisionally along a line where the rocks show marked slickensiding. At the bottom of the slope and on the line of the junction an adit has been driven into the hill. This was filled with water so it was impracticable to examine the ore face. According to the information given in the Survey Memoir (19) a shaft was sunk for ten feet from this adit along a string of ore. None of the ore raised was removed and at present it is lying in dumps which interfere with an examination of the ground. A second small adit has been driven in

- 27 -

on the other side of the knoll. The debris from this consists of greywacke and shale but shows no sign of ore. No estimate could be formed of the extent of the mineral mass but from the evidence of the workings it must be small.

Samples of the ore vere collected from the dumps care being taken to make the collection as representative as possible. It was unfortunately not possible to obtain any quantity of the ore in position. A small quantity was obtained from the upper excavation this being the only place where it appeared. At the same time specimens were taken of the rocks on both sides of what appears to be the junction of the igneous and sedimentary rocks. These samples when examined proved to be highly interesting and worth a more complete study than is possible from the specimens collected. It is proposed to continue this investigation later so the conclusions put forward here may require modification in the light of a fuller study.

(c) The Ore Minerals.

The following minerals were found to be present in the ore mass on examination of the samples collected as above described:- Pyrrhotite, pentlandite, niccolite, cloanthite, chalcopyrite and pyrite. There is apparently an association of these minerals into two groups as they do not all occur in any one section. A number of sections show the presence of the pyrrhotite, pentlandite and chalcopyrite with small quantities of niccolite while other sections show niccolite, cloanthite and chalcopyrite. Pyrite is only present in very subordinate amount. The pentlandite always occurs as inclusions in pyrrhotite and niccolite is nearly always associated with cloanthite. Chalcopyrite occurs in practically all the sections although in varying proportions.

No large mass consisting entirely of ore was found, all the specimens showing an intermixture of country rock.

The following statement shows the mineral content of the specimens collected. In all cases several sections were cut from each specimen and the abundance of the minerals was judged from the average. The minerals are shown in order of decreasing quantity.

| T | 20. | Pyrrhotite with pentlandite, chalcopyrite.                              |
|---|-----|---|
| T | 21. | Pyrrhotite with pentlandite, chalcopyrite, pyrite (scarce).             |
| T | 22. | Niccolite, cloanthite, chalcopyrite.                                    |
| T | 23. | Niccolite, cloanthite, chalcopyrite,<br>pyrrhotite and pyrite (scarce). |
| T | 24. | Niccolite, cloanthite, chalcopyrite.                                    |
| T | 25. | Pyrrhotite with pentlandite, chalcopyrite.                              |

- T 26. Cloanthite, niccolite, chalcopyrite, pyrrhotite and pyrite scarce.
- T 27. Pyrrhotite with pentlandite, chalcopyrite.
- T 29. Cloanthite, niccolite, chalcopyrite, pyrrhotite in small quantities.

The prevailing minoral in the assemblage is pyrrhotite which occurs in the majority of the sections. This mineral polishes comparatively easily (H, 4.6) showing characteristic markings in the earlier stages due to the tabular habit of the crystals. The surface is smooth with a few cracks and has a purplish colour. It follows the outline of the bounding country rock without showing crystal It may be etched electrolytically by a weak edges. current using 5% HCl as electrolyte and attaching the section to the kathode. Under these conditions the surface breaks up into a mosaic of grains without definite crystalline shape (Plate la ).

A chemical examination along the lines described above reveals the presence of iron, sulphur and a trace of arsenic.

The most interesting feature of this mineral is the presence of inclusions (Plate 2a). These are aggregates with straight crystal edges against both the pyrrhotite and each other. They are usually segregated round fragments of rock or in fissures passing into that rock. In colour they are lighter

than the pyrrhotite and they only show up well when the pyrrhotite is well polished. Suggestion of alteration is seen along cracks in the crystals which are surrounded by a bluish zone. In entreme cases the crystals are reduced to a series of disconnected bright cores separated by the replacing minoral which appears to be continuous with the pyrrhotite. These inclusions etch electrolytically but only after long continued action. Chemical examination shows them to contain nickel, iron and sulphur. In all probability they are crystals of pentlandite occurring in the same manner as at Sudbury, Ontario.

It is interesting to note that the pyrrhotite shows no trace of nickel so that the nickel content of the pyrrhotite recorded in the analysis quoted in the Geological Survey Memoir must be referred to these inclusions of pentlandite.

The next most important mineral is niccolite. This polishes well (H, 5) to a brilliant surface although some time must be spent in polishing to remove the large cracks and cavities that occur. It appears as oval or rounded masses (Plate 3a). In the majority of cases it is embedded in a white mineral to be described later.

The colour is a brilliant ambor. Etching with an acid solution of potassium permanganate shows the masses of the mineral to be made up of small grains with irregular boundaries (Plate 4a). Twinning of the crystals is sometimes seen but this is usually obscure and owing to the lack of crystal edges cannot be given definite crystallographic orientation.

Chemical examination shows the presence of nickel, arsenic and a trace of cobalt.

Surrounding the niccolite and evidently closely connected with it is a silvery white mineral that takes on a polish later than the niccolite. In most cases this completely surrounds the niccolite and penetrates into fissures in the country rock. Crystal edges are often seen.

Etching of this mineral can be accomplished either with 50% Nitric acid or electrolytically using the section as the anode and an alkaline permanganate solution as electrolyte. Treatment by either of these methods shows up the internal structure as a number of zoned crystals (Plate 5a).

This mineral appears to have solidified later than the niccolite. The evidence for this is twofold. First, in most cases the niccolite is either completely surrounded by it or very nearly so while it is seen to invade the fissures of the country rock to a much greater extent than does the niccolite. Secondly, the outline of the niccolite is always smooth and rounded suggesting that the solidified niccolite had been subjected to the solvent action of a liquid. In one instance a zoned crystal was seen to be including a small mass of niccolite and the zone lines were parallel to the outline of the included niccolite. Chemically it is found to contain nickel, arsenic, sulphur, iron and a trace of cobalt.

In appearance the mineral suggests arsenopyrite but the comparatively small proportion of iron compared with the nickel is against this. The arsenic again is far in excess of the sulphur. Bearing in mind the close association with niccolite and the indications that this mineral has corroded the niccolite it may be concluded that it is one of the smaltite-cloanthite series, and as the proportion of nickel is much in excess of the cobalt it may be considered as an impure cloanthite. The hardness of cloanthite (H, 5.5) is in keeping with its polishing properties.

The last mineral of any importance is chalcopyrite. This is about the first mineral in any of the sections to take a polish (H,  $3 \cdot 5 - 4$ ). The colour is the normal brass yellow and the surface obtained is perfectly smooth.

It appears in practically all the sections where

it is often found as a thin lining to fissures in the rock that are otherwise filled with pyrrhotite or cloanthite. It also frequently veins these minerals, filling fine cracks (Plate 6a).

Etching in dilute hydrochloric acid using the section as cathodo and a weak current brings up the crystal structure very well.

Chemically 1t contains iron, copper and sulphur.

A very small quantity of pyrite is to be found enclosed in the pyrrhotite but it is a very subordinate mineral. Chemical examination shows the presence of a trace of nickel along with the normal iron and sulphur.

Careful study of the sections and a consideration of the observations recorded in the previous pages has led the writer to believe that the ore minerals were introduced separately from and later than the igneous rock.

In support of this theory there is the appearance of the ore masses which, from the way in which they have invaded fine cracks in the country rock, suggest that the mass containing the elements of the ore minerals was very liquid. There has been a definite order in the solidification of the ores as shown by the presence of inclusions. Thus niccolite preceded

- 34 -

cloanthite and pentlandite preceded pyrrhotite, while chalcopyrite was the last to solidify, filling the interstices and veining the other ore minerals. The time sequence between cloanthite and pyrrhotite is difficult to determine but it would appear that pyrrhotite was slightly the later.

The picture that best seems to fit the observations is that of a liquid mass separating and forming globules much as does an oil and water emulsion. In one case the niccolite separated as globules which solidified into crystalline aggregates, but without the formation of large crystals with definite edges, and these were corroded by the liquid that later and more slowly solidified to cloanthite, this time with the formation of some definite crystals. In the same way pentlandite separated from and solidified before the pyrrhotite. The last and probably most fluid portion solidified as chalcopyrite which, prior to solidification, was able to make its vay into many fine fissures both in the country rock and the already solidified ore minerals.

None of the slides or sections show any evidence that the ore mass separated from the igneous rock became segregated at the bottom and cooled as part of the intrusion.

Further work is projected on this ore occurrence.

Field work is required to determine the extent of the ore mass and the condition of the surrounding rocks while the problem of the order of formation of the ore minerals must be considered on theoretical and chemical grounds.

## V. SUPPIARY.

The work recorded in the preceding pages is a continuation of that done on the micro-chemical analysis of minerals and deals with the study of ore minerals by microscopical and micro-chemical methods.

As such ores have to be examined in the condition of polished sections using reflected light, the literature on the characteristics of polished surfaces was consulted and the general theory on this subject is discussed briefly.

The technique of grinding and polishing such sections was also studied and it was found to be necessary to prepare the requisite abrasives in a uniformly fine condition as the commercial products were unsuitable. The manner in which this was done is described under the heading of Abrasives and Polishes.

Various published methods for preparing the actual sections were tried and eventually a scheme was worked out that gave the desired results.

Using this method, a series of polished sections of the common ore minerals in as pure a condition as obtainable, was prepared and these were examined and subjected to various tests in order to become familiar with their appearance and properties.

- 37 -

A system of micro-chemical analysis had already been worked out so that it was only necessary to follow the procedure laid down in the published paper which is attached to this report.

In order to test these methods in practice, a small ore deposit occurring at Talnotry, near Newton Stewart, was examined and studied. Specimens illustrative of the general geology and also of the ore were collected. These were sectioned and examined.

A short description of the general geology of the area is given.

Specimens of the ore were polished and examined. The following minerals were found to be present :-Pyrrhotite, pentlandite, niccolite, cloanthite, chalcopyrite and pyrite. These minerals apparently occur in definite associations. Thus pyrrhotite and pentlandite occur together and niccolite with cloanthite. Within the two groups, pentlandite has solidified before pyrrhotite and niccolite before cloanthite. The evidence as to the time relations of the groups is not conclusive but the niccolitecloanthite series appears to be slightly earlier. Chalcopyrite has definitely solidified after the other minerals.

A chemical analysis of the minerals gave the

following results:-

Pyrrhotite contains iron, sulphur and a trace of arsenic. 11 Pentlandite nickel, iron, arsenic. ŧ Niccolite nickel, arsenic and a trace of cobalt. 11 nickel, arsenic, sulphur, iron and a trace of cobalt. **Cloanthite** 12 Chalcopyrite 1ron, copper, sulphur. 11 Pyrite iron, sulphur, and a trace of nickel.

Thus in the pyrrhotite-pentlandite series nickel is confined to the pentlandite.

Lastly the identification of cloanthite rests on the relative proportions of its constituents.

It is intended to continue this work on the Talnotry ore deposit as there are several interesting points that require elucidation.

## VI. ILLUSTRATIONS.

| Plate I.         | Slide T 1b. Granite, Cairnsmore of Fleet      |  |  |  |  |
|------------------|---|--|--|--|--|
|                  | mass. Palnure burn 400 yds. east of           |  |  |  |  |
| 1                | Talnotry ore mass.                            |  |  |  |  |
| 1                | Slide shows quartz orthoclase and microcline, |  |  |  |  |
|                  | biotite (black) and muscovite showing         |  |  |  |  |
| 1<br>1<br>1<br>1 | cleavage. Crossed nicols. x 30 diams.         |  |  |  |  |
| Plate 2.         | Slide T 3. Fine-grained greywacke. Road-      |  |  |  |  |
|                  | side on edge of metamorphic aureole, about    |  |  |  |  |
|                  | 2 miles S.W. of Talnotry Cottage.             |  |  |  |  |
|                  | Slide shows no evidence of thermal meta-      |  |  |  |  |
|                  | morphism. Crossed nicols. x 30 diams.         |  |  |  |  |
| Plate 3.         | Slide T 4. Greyvacke, roadside quarry by      |  |  |  |  |
|                  | 40th milestone S.W. of Talnotry Cottage.      |  |  |  |  |
| •                | Coarse and uneven grained.                    |  |  |  |  |
|                  | Crossed nicols x 30 diams.                    |  |  |  |  |
| Plate 4.         | Slide T 9. "Diorite", hill-top above          |  |  |  |  |
| ÷                | Talnotry mine. More acid part of "diorite"    |  |  |  |  |
|                  | that carries the ore. Slide shows quartz,     |  |  |  |  |
|                  | some secondary; saussuritised plagioclase,    |  |  |  |  |
|                  | pale hornblende, and abundant secondary       |  |  |  |  |
|                  | biotite. Crossed nicols. x 30 diams.          |  |  |  |  |
| Plate 5.         | Slide T 10. "Diorite" from upper adit of      |  |  |  |  |
| ·<br>·<br>·      | Talnotry mine. More basic than T 9.           |  |  |  |  |
|                  | Slide shows abundant hornblende altered with  |  |  |  |  |
|                  | separation of sphene, highly altered          |  |  |  |  |
|                  |   |  |  |  |  |

- Plate 6. Slide 16a. Greywacke immediately underlying ore mass. Slide shows a normal fine grained rock that has been profoundly altered. The ground mass almost completely recrystallised with formation of abundant new biotite. New crystals of brown tourmaline are also present. Ordinary light. x 30 diams.
- Plate 7. Slide T 22. Ore from Talnotry mine dump. Slide shows a basic phase of "diorite" containing pyrrhotite. Minerals are altered as in Plate 5 with evidence of crushing. The ore (black) occurs veining the crystals and fine cracks. Crossed nicols. x 30 diams.

Plate la. Pyrrhotite etched x 45 diams.

Plate 2a. Pyrrhotite with inclusions, etched x 25. P = inclusion of pentlandite.

Plate 3a. Niccolite and chalcopyrite. x 40.

- Plate 4a. Niccolite showing grain structure, etched x 45.
- Plate 5a. Niccolite and cloanthite, etched x 80. Showing zoning of cloanthite crystal.

Plate 6a.

Chalcopyrite and cloanthite x 30. Showing veining of cloanthite by chalco-



Plate 1.



Plate 3.

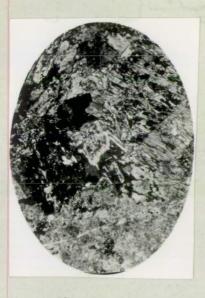






Plate 2.



Plate 4.

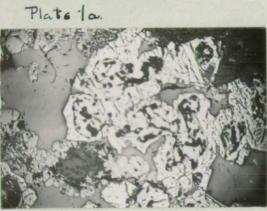


Plate 6.





Plate 2a.





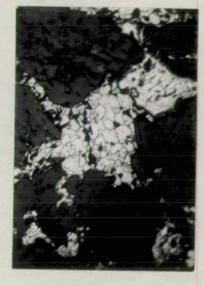


Plate 4a.



Platesa.



Plate 6a.

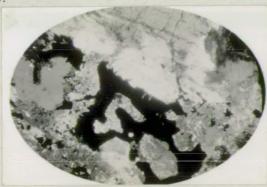


Plate 7

## VII. REFERENCES TO LITERATURE.

- J. Adam Watson. Colour-reactions in the microchemical determination of minerals. Min. Mag. XXIV, 148, p.21. 1935.
- 2. J. Murdoch. Microscopical determination of the opaque minerals. New York. 1916.
- 3. W. M. Davy and C. M. Farnham. Microscopic examination of the ore minerals. New York. 1920.
- 4. H. Schneiderhohn. Anleitung zur Mikroskopische Bestimmung und untersuchungen von Erzen. Berlin. 1922.
- 5. R. W. Van der Veen. Minerography and ore deposition. The Hague. 1925.
- 6. M. M. Short. Microscopic determination of the ore minerals. U.S. Geol. Survey Bull. 825. 1931.
- 7. G. T. Beilby. Aggregation and Flow of Solids. London. 1921.
- 8. G. I. Finch. The Nature of polish. Trans. Farad. Soc. 33, 425. 1937.
- 9. C. H. Desch. Metallography. p.139. 1922.
- 10. M. M. Short. op. cit. p.16.
- 11. F. W. Preston. The Structure of Abraded Glass Surfaces. Trans. Optic Soc. 23, 141. 1922.
- 12. G. T. Beilby. Aggregation and Flow of Solids. p.111. 1921.
- 13. M. M. Short. op. cit. p.50.
- 14. H. Schneiderhohn. op. cit. p.116.
- 15. H. Behrens. A Manual of microchemical analysis. London. 1895.
- 16. H. Behrens and P. D. C. Kley. Mikrochemische Analyse. Leipzig. 1921.

| 17. E. N | I. Chamot.<br>New York.   | Elementary<br>1921.      | chemical mi                  | croscopy.                |
|----------|---------------------------|--------------------------|------------------------------|--------------------------|
| 18. J. W | I. Gregory.<br>Inst. Min. | The Nicke<br>Met. Trans  | l-Cobalt Ore<br>• 37, 178-18 | of Talnotry.<br>2. 1928. |
| 19. The  | Lead, Zinc,<br>Memoir Geo | Copper and<br>1. Survey. | Nickel Ores<br>p. 128.       | of Scotland.<br>1921.    |
|          |                           |                          |                              |                          |
| •        |                           |                          |                              |                          |
|          |                           | -<br>-                   |                              |                          |
|          |                           |                          |                              |                          |
|          | . <i>"</i>                | · · · ·                  | •                            |                          |
|          |                           |                          |                              |                          |
|          | •                         |                          |                              |                          |
|          |                           |                          | · ·                          |                          |
|          | •                         |                          | · .                          |                          |
|          |                           |                          | · ·                          |                          |

•

• •

.

, . •