Rapid Mineral Identification by Electrographic Contact Printing

K. F. G. Hosking, M.Sc., Ph.D., A.M.I.M.M.

Procedures applicable in the laboratory, field, or mill are described

Abstract

The electrographic contact print method has been employed as an aid to the identification of conducting minerals in polished sections of ores and in samples of grains embedded in "conducting bakelite" and polished. Extensions of this method are described which facilitate the rapid identification of many economically important minerals in the laboratory, field, and mill by utilizing readily constructed pieces of apparatus.

Introduction

Hiller (1937), Gutzeit (1942), and Williams and Nakhla (1950–51)[1] have demonstrated that the electrographic contact print method can facilitate the identification of conducting minerals in polished sections of ores; the last-named have also shown that it could be used to investigate mill samples, provided the grains were first embedded in "conducting bakelite" and polished. The value of the method depends largely on the fact that many of the opaque minerals are by no means always readily identified in polished sections by the optical and ancillary methods of recognition usually employed.

Fundamentally the technique of taking contact prints of cations from polished sections is as follows:—

The polished specimen is partly embedded in an anode consisting of plasticine wrapped in aluminium foil. A copper plate, attached to the current source, is inserted beneath the aluminium-covered electrode and the specimen is levelled in a small press. A piece of gelatin-coated paper which has been just previously soaked in an appropriate attacking reagent is placed on the polished surface so that the gelatin is in contact with the minerals. The paper is covered with an aluminium foil/plasticine cathode and it in turn with a plate of glass or plastic. The whole assemblage is then subjected to a pressure of 10 lb. per sq. in. and a current of appropriate intensity is passed for a period which rarely exceeds 30 seconds. The applied voltage varies with the mineral under examination, but is usually between 4 V and 30 V, while the current intensity should be between 20 mA. and 40 mA. for the best results.

After the passage of the current for the requisite time the circuit is broken and the paper is removed and immersed in an appropriate developing reagent. Provided the choice of attacking and developing reagents is correct, this procedure results in a mirror image distribution "map" of one or more cations in the polished section. By repeating the process with other attacking and developing reagents it is often possible to obtain a series of distribution "maps" from which the nature and disposition characteristics of some or all of the major cations can be determined.

A similar procedure can be applied to grains of conducting minerals which have previously been embedded in "conducting bakelite" and polished. Reference should be made to the paper by Williams and Nakhla (1950–51) for a comprehensive account of both these techniques.

It seemed to the writer that the development of simple electrographic contact printing techniques which would enable *rapid* identifications of conducting minerals to be made in the laboratory, field, and mill, *without* polishing and embedding the specimen in conducting bakelite, constituted a logical extension of the work already noted. Consequently the methods described below were devised.

Identification of Conducting Minerals

The method described here is best suited to the examination of conducting fragments

[1] These references are appended at the end of the article.

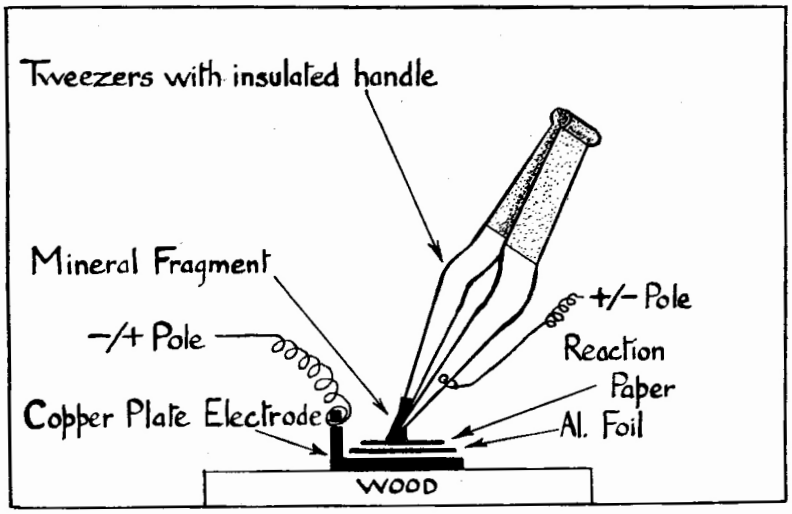

Fig. 1.

measuring between a quarter- and a half-an-inch long but a smaller fragment of an appropriate mineral can also be examined provided that it can be securely held by the acutely-tapered steel forceps which form an integral part of the apparatus.

Fig. 1 shows the apparatus used. It consists of steel forceps bound with insulating tape and connected to the source of current by an insulated wire attached to one leg of the forceps about 1 in. from the tip. An L-shaped piece of copper sheet, nailed to a wooden base, constitutes the other electrode and can conveniently be connected to a wire leading to the source of electricity by a small metal clip.

In order to make a print of a given cation in a fragment of mineral the forceps are connected to the anode and the copper sheet to the cathode. A piece of aluminium foil (that used for wrapping cigarettes is perfectly satisfactory) is laid on the copper plate and a small piece of spot-reaction (or filter) paper is placed on the foil and damped either with a few drops of attacking reagent or, when practicable, with a mixture of attacking and developing reagents. The fragment is held in the forceps and pressed on to the reagent-impregnated paper. A direct current is passed for about 30 seconds. The circuit is then broken, the paper removed, and, if necessary, treated with a few drops of

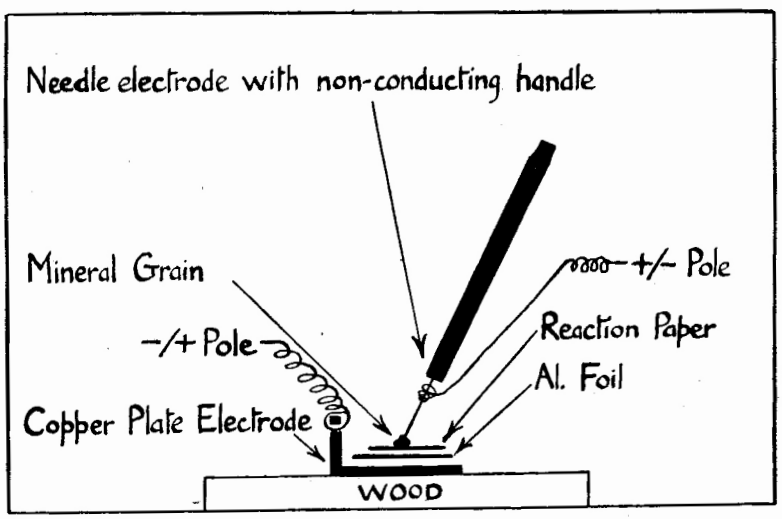

Fig. 2.

developing reagent. The print so obtained is somewhat diffuse but quite adequate for diagnostic work.

The following points concerning the method must also be noted :—

(i) In order to print anions the electrodes must be reversed—that is, the copper plate must then be the anode.

(ii) The aluminium foil should either be discarded after each test or well washed and dried in order to prevent any possibility of contamination.

(iii) Improved prints are often made if a small face is first ground on the fragment by holding it in the forceps and rubbing it on a block of polished cast-iron on which has been placed a little medium-grade carborundum powder and some water. Having developed a small face the fragment, still held in the forceps, should be washed repeatedly in water. Finally it should be immersed in alcohol and dried quickly over a small flame or by waving it in the air. This procedure ensures that there will be a reasonable area of the fragment in contact with the paper, that oxide and other films have been removed, and that the print will be taken from a surface of enhanced reactivity.

(iv) Much valuable diagnostic work can be carried out simply by using a small dry-battery as the source of electricity ; thus the method can be easily used in the field. It is, of course, more satisfactory to employ direct or rectified alternating-current from the mains, together with the necessary apparatus for measuring and controlling the current and voltage applied during printing.

(v) The attacking and developing reagents recommended by Williams and Nakhla (1950–1, pp. 270–7) for use in the preparation of electrographic contact prints from polished sections of minerals apply equally well when examining fragments.

Advantages of the Method.—The great advantage of the method described is that by its use, in conjunction with a few of the simpler physical tests, a large number of the opaque metallic minerals which are of major economic importance can be readily identified. Furthermore, the technique and apparatus are simple and a source of heat is not necessary. Finally, from the point of view of time and reagents the process is very economical.

Identification of Conducting Grains

The procedure and apparatus (Fig. 2) employed in the examination of the conducting components of mill-products, etc., only differ from those already described in that the grain to be identified is placed on the paper (preferably fine-grained filter-paper) impregnated with an appropriate attacking reagent and is touched for about 30 seconds with a needle electrode (which, if cations are to be printed, is the anode). The needle electrode consists of a fine sewing needle bound in contact with a wire leading to the source of electricity to a glass or wooden handle by means of insulating tape. After passage of the current a drop of an appropriate developing reagent is placed close to the grain and this causes the latter to become surrounded by a characteristically-coloured halo.

The entire test is best carried out under a binocular microscope and with a little practice grains considerably below 100-mesh can readily be identified. In the field *plus* 100-mesh grains can be identified by conducting the tests under a $\times$ 10 magnifying glass.

Other Notes.

(i) For practically all purposes a 4·5 V dry battery is an adequate source of electricity, although, as in all electrographic methods, a d.c. mains supply (with a variable resistance and milli-ammeter included in the circuit) is superior.

(ii) A current intensity which is considerably higher than that employed when taking prints from polished sections is advantageous, as it tends to increase lateral diffusion of the metal ions in the paper and assists the development of a halo which is not in any way obscured by the parent grain.

(iii) The grain to be examined may conveniently be transferred to the paper (and from one piece of paper to another if it is to be subjected to more than one electrographic test) either by means of a sharpened and damped match or a damped glass capillary with a sealed end.

(iv) Having conducted a test and allowed the paper to dry, it, with the grain *in situ*, may be preserved for reference purposes between two pieces of cellulose tape. Whilst most test-papers thus treated show little or no deterioration after considerable periods of time, a few are of little further use after a day or so.

Advantages of the Method.

(i) The method constitutes a valuable aid to the simple and rapid identification of the conducting components in a mill sample and

is especially useful when embedding and polishing equipment is not available.

(ii) In the field and laboratory it facilitates the recognition of small grains of conducting minerals which have been removed from a non-conducting matrix.

(iii) Either this method or the one described earlier may be conveniently used to investigate quickly the suitability of given attacking and developing reagents for use when taking electrographic contact prints from specific minerals in *polished* sections of ores and *embedded* grain samples.

Specific Mineral Tests

While the papers by the workers already noted should be consulted for a comprehensive account of the reagents, etc., to be used when taking electrographic contact prints from specific minerals, it is appropriate to include here directions for taking prints from a limited number of species in order that those not acquainted with the earlier work can evaluate the methods described without further information.

The following instructions have been extracted from the paper by Williams and Nakhla (1950–51) and refer to the taking of prints from polished sections; they, therefore, indicate the ideal procedure. As the conditions need not be so rigorously controlled when identifying minerals by the techniques described here and as it is inadvisable to mix the developing and attacking reagents when identifying grains the directions below should be modified according to the problem in hand.

Arsenopyrite, FeAsS.

(i) *Element Tested.*—Iron.
Attacking and Developing Reagents.—1 : 15 nitric acid *plus* a little 5% potassium ferrocyanide.
Volts.—8.
Time of Attack.—25 seconds.
Result.—A direct blue print.

(ii) *Element Tested.*—Arsenic.
Attacking Reagents.—A 1 : 1 mixture of 0·880 ammonia and hydrogen peroxide (6 vols.).
Volts.—10.
Time of Attack.—30 seconds.
Developing Reagent.—1% silver nitrate.
Result.—A brown print.

Chalcocite, Cu_2S.

(i) *Element Tested.*—Copper.
Attacking and Developing Reagents.—0·880 ammonia *plus* a little 5·0% alcoholic solution of alphabenzoinoxime.
Volts.—6.
Time of Attack.—20 seconds.
Result.—A direct green print.

(ii) *Element Tested.*—Sulphur.
Attacking Reagent.—2% sodium hydroxide; electrodes reversed.
Volts.—5
Time of Attack.—25 seconds.
Developing Reagents.—A solution of antimony trichloride in dilute hydrochloric acid.
Result.—An orange print.

Chloanthite—Smaltite, (Ni, Co) As_2.

(i) *Element Tested.*—Nickel.
Attacking and Developing Reagents.—0·880 ammonia *plus* a little 1% alcoholic solution of dimethylglyoxime.
Volts.—10.
Time of Attack.—20 seconds.
Result.—A direct red print.

(ii) *Element Tested.*—Cobalt.
Attacking Reagent.—0·880 ammonia.
Volts.—8.
Time of Attack.—30 seconds.
Developing Reagent.—0·1% acetic solution of α-nitroso-β-naphthol.
Result.—A brown print.

(iii) *Element Tested.*—Arsenic.
Attacking Reagents.—0·880 ammonia *plus* hydrogen peroxide (6 vols.).
Volts.—5.
Time of Attack.—30 seconds.
Developing Reagent.—1·0% silver nitrate.
Result.—A brown print.

Galena, PbS.

(i) *Element Tested.*—Lead.
Attacking Reagent.—1 : 10 nitric acid.
Volts.—12.
Time of Attack.—20 seconds.
Developing Reagent.—Freshly prepared 0·2% aqueous sodium rhodizonate.
Result.—A red-violet print.

(ii) *Element Tested.*—Sulphur.
As for the sulphur in chalcocite, but 6 V employed instead of 5.

Acknowledgments.—The first method noted in this paper was originally described by the writer in his thesis which was approved by the University of London for the award of a Ph.D. degree.

References

GUTZEIT, G. "Determination and localization of metallic minerals by the contact print method." *Amer. Inst. Min. Engrs.*, Tech. Pub. No. 1457, 1942.

HILLER, TH. "Sur l'application de la méthode des empreintes à la détermination des minéraux opaques en section polie." *Schweiz. Min. und Petrog. Mitt.*, **17**, 1937, 88–145.

WILLIAMS, D., and NAKHLA, F. M. "Chromographic contact print method of examining metallic minerals and its applications." *Trans. Instn. Min. Metall.*, Lond., 1950–1, **60**, 257–295.