British Geological Survey

Industrial Minerals Laboratory Manual

FLAKE GRAPHITE
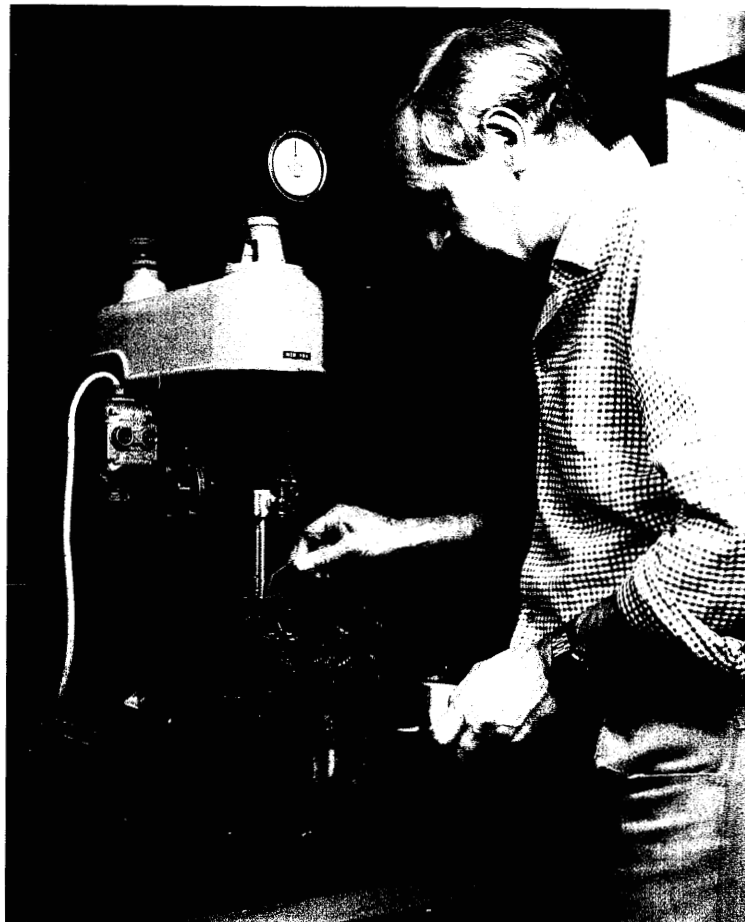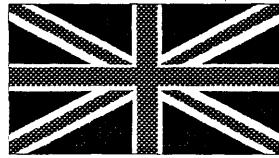
C J Mitchell

Natural
Environment
Research
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British Geological Survey

TECHNICAL REPORT WG/92/30
Mineralogy and Petrology Series

Industrial Minerals Laboratory Manual

FLAKE GRAPHITE

C J Mitchell

A Report prepared for the Overseas Development Administration under the
ODA/BGS Technology Development and Research Programme, Project 91/1

CONTENTS

Preface

Industrial mineral raw materials are essential for economic development. Infrastructure improvement and growth of the manufacturing sector requires a reliable supply of good quality construction minerals and a wide range of other industrial mineral raw materials.

Although many less developed countries have significant potential industrial mineral resources, some continue to import these materials to supply their industries. Indigenous resources may not be exploited (or are exploited ineffectively) because they do not meet industrial specifications, and facilities and expertise to carry out the necessary evaluation and testwork are unavailable. Unlike metallic and energy minerals, the suitability of industrial minerals generally depends on physical behaviour, as well as on chemical and mineralogical properties. Laboratory evaluation often involves determination of a wide range of inter-related properties and must be carried out with knowledge of the requirements of consuming industries. Evaluation may also include investigation of likely processing required to enable the commodity to meet industry specifications.

Over the last 10 years, funding from the Overseas Development Administration has enabled the British Geological Survey to provide assistance to less developed countries in the evaluation of their industrial mineral resources. This series of laboratory manuals sets out experience gained during this period. The manuals are intended to be practical bench-top guides for use by organisations such as Geological Surveys and Mines Departments and are not exhaustive in their coverage of every test and specification. The following manuals have been published to date:

> Limestone
> Flake Graphite
> Diatomite
> Kaolin
> Bentonite

A complementary series of Exploration Guides is also being produced. These are intended to provide ideas and advice for geoscientists involved in the identification and field evaluation of industrial minerals in the developing world. The following guide has been published to date:

> Biogenic Sedimentary Rocks

A J Bloodworth
Series Editor

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Project Manager

Industrial Minerals Laboratory Manual

Flake graphite

1. INTRODUCTION

This manual concentrates on the laboratory assessment of flake graphite, though other forms of graphite are considered briefly. Overviews of the geological occurrence and the uses and applications of graphite are given. These are supported by more detailed information in the Appendices. Procedures for mineralogical assessment and laboratory processing trials of graphite-bearing rocks are discussed, along with those for evaluation of graphite concentrates produced in the laboratory. Detailed step-by-step procedures for laboratory processing trials are given in the Appendices. Case histories detailing two typical laboratory investigations with 'worked examples' are also included. This manual is one of a series produced as part of the BGS/ODA R&D Project 'Minerals for Development'.

Graphite is one of two naturally occurring allotropes of crystalline carbon, the other being diamond. In contrast to diamond, graphite is soft, greasy to the touch, and soils fingers and paper. Table 1 summarizes the principal properties of graphite whilst Figure 1 illustrates the crystalline structure of graphite. Graphite generally occurs in one of three forms: microcrystalline or amorphous; crystalline lump or vein; and crystalline flake. The characteristics of the different forms of graphite are listed in Table 2. The major consumer of flake graphite is the refractories industry. It is also used in friction materials, lubricants, foundry core and mould washes, and may undergo property modification to form expanded graphite and graphite foil.
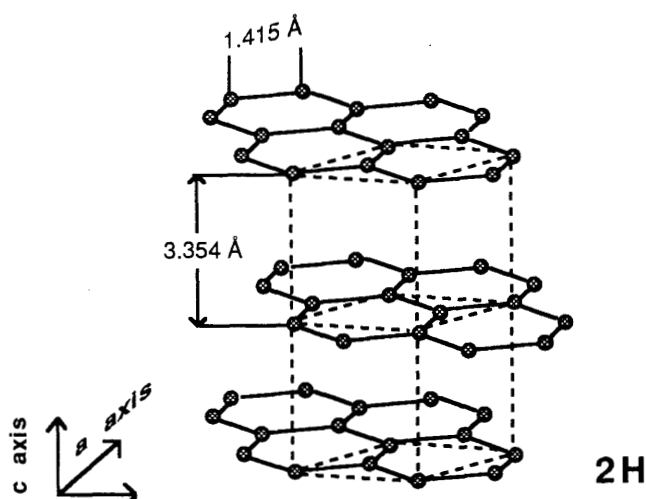
Figure 1. Arrangement of carbon atoms in the hexagonal (2H) unit cell of graphite. (After Krauss et al., 1987).

Flake graphite

Table 1. Properties of graphite

Composition	C **Hardness** 0.5 - 1 (Mohs scale). **Density** 2.09 - 2.26
Colour	Dark steel grey to iron black; metallic lustre; grey to black streak.
Morphology	Hexagonal crystal system (rhombohedral symmetry); usually platy, occasionally acicular; perfect basal cleavage, commonly striated, occasionally twinned.
Melting point	Decomposes at 600°C in oxidising conditions and melts at 3550°C in non-oxidising conditions; infusible in most common fluxes; vaporisation temperature 4500°C.
Miscellaneous	Good thermal and electrical conductivity; chemically inert; greasy texture (natural lubricant); low thermal expansion and shrinkage; compressible and malleable; low absorption coefficient for X-rays; nontoxic; generally not 'wetted' by metals.

Flake graphite

2. GEOLOGICAL OCCURRENCE AND BACKGROUND

Graphite generally occurs as a result of metamorphism (regional or contact) of organic matter in sediments. Flake graphite is assumed to be derived from fine-grained sediments rich in organic matter. As metamorphic grade increases, carbonaceous material converts to 'amorphous' graphite. Flake graphite forms from its amorphous precursor in rocks at or beyond amphibolite grade metamorphism (Landis, 1971). Vein graphite is assumed to form by partial volatilization of graphite and subsequent recrystallization during regional granulite and / or charnockite facies metamorphism. Amorphous graphite is generally considered to have originated by thermal or regional metamorphism of coal or carbonaceous sediments.

Positive vanadium and nickel anomalies and negative boron anomalies are possible signatures for graphite if geochemical survey data are available. The presence of sulphides and trace amounts of uranium may also be an indicator.

Table 2. Summary of characteristics of the different forms of graphite

	Flake	Vein	Amorphous
Description	Crystalline flakes; coarse >150 μm; fine <150 μm	Coarse crystals Mostly >4 cm	Microcrystalline <70 μm
Origin	Syngenetic; regional metamorphism	Epigenetic; regional metamorphism	Syngenetic; contact and / or regional metamorphism
Ore	5 - 30% graphite; strata-bound, tabular or lenses	98%+ graphite; veins and fractures	Seams, often folded and faulted
Product grade	75 - 97% graphite	98 - 99.9% graphite	60 - 90% graphite
Main uses	Refractories, brake linings, lubricants and batteries	Carbon brushes, brake linings and lubricants	Refractories, steel industry, paint and batteries
Major producers	China, Brazil, India, Madagascar, Germany, Norway, Canada and Zimbabwe	Sri Lanka.	China, S Korea, Czechoslovakia, Austria and N Korea

Adapted from Fogg and Boyle (1987)

Flake graphite

Flake graphite occurs as flat, plate-like crystals, with angular, rounded or irregular edges, disseminated throughout originally carbonaceous metasediments. The host rocks include quartz-mica schist, feldspathic or micaceous quartzite and gneiss. Flake graphite may also occur in metamorphosed carbonate rocks, though these occurrences are currently of little economic significance. Flake graphite deposits are usually strata bound, with individual beds or lenses from 30 cm to more than 30 m thick and extend for lengths of 2 kilometres or more. Ore bodies are normally tabular, occasionally lenticular, and occur locally as irregular bodies in the hinge zones of folds. Most economic deposits of flake graphite are of Archean to late Proterozoic age. These rocks may contain up to 90% graphite, although 10 - 15% graphite is a more typical ore body grade.

Graphite flakes range in size from 1 to 25 mm, with an average size of 2.5 mm. Commercially flake graphite is divided into coarse (150 - 850 μm in diameter) and fine (45 - 150 μm in diameter) flake. Fine flake may be further subdivided into medium flake (100 - 150 μm), fine flake (75 - 100 μm) and powder (-75 μm). Impurities include minerals commonly found in metasediments, usually quartz, feldspar, mica, amphibole, garnet and calcite, with occasional amphiboles, pyrrhotite, pyrite and magnetite

Vein-type ('high-crystalline' or 'lump') graphite is found as interlocking aggregates of coarse and/or microcrystalline platy or (less commonly) acicular graphite. The veins are hosted by igneous and metamorphic rocks (gneiss, schist, quartzite and marble).

'Amorphous' (microcrystalline) graphite is formed of aggregates of fine graphite crystals which give the material a soft, black, earthy appearance. This material is usually hosted by quartzites, phyllites, metagreywackes and conglomerates. 'Amorphous' graphite is defined as being finer than 40 μm in diameter, but some trade statistics define the upper limit at 70 μm (40 - 70 μm is the limit of resolution of the human eye). Deposits with grades over 80% carbon are considered to be economically viable.

3. USES AND SPECIFICATIONS

Specifications for graphite used in a wide variety of applications are given in Appendix 1. Particle-size data and carbon contents of a large number of commercial graphite products from major graphite producing countries are given in Appendix 2. The requirements of industries which use graphite as a raw material are discussed below. A brief review of current economic trends within the graphite market is also given.

3.1 Refractories

The major consumer of flake graphite is the refractories industry. Flake graphite improves the thermal shock and erosion resistance of refractory products. Flake orientation enhances the strength of the refractory. Graphite must be employed in reducing conditions because of its low oxidation temperature. Accordingly, graphite-bearing refractories such as magnesia-carbon bricks are extensively employed as linings in basic oxygen converters, electric arc furnaces and steel ladles. Magnesia graphite bricks used in electric arc steel making in the UK contain 15 - 20% graphite. Japanese magnesia graphite bricks may contain 20 -25% graphite which doubles the lifespan of the refractory. Alumina graphite bricks are generally used in continuous casting processes and torpedo ladles. Crucibles, for handling molten metal in foundries, are made from either clay graphite (large flake/85% carbon) or SiC graphite (fine flake/80% carbon) mixtures. Flake and amorphous graphite are used in washes on foundry moulds and cores. Sulphides are undesirable in graphite used as a foundry sand additive.

Flake graphite is also used to make expanded graphite for insulation in steel production and is added to polyurethane foam as a flame retardant. Expanded graphite is produced by using acid (chromic and sulphuric) to weaken the bonds between graphite plates and this allows expansion on heating to about 700°C. 'Amorphous' graphite is used as a foundry additive, in refractory bricks and in plastic and castable refractories. Graphite powder can be used as a thermal blanket for the continuous casting of steel.

3.2 Non-refractory uses

Flake graphite is used in friction materials (brake / clutch linings), lubricants and in the manufacture of graphite foil and long-life alkaline batteries. Brake linings use mixtures of natural (flake and vein) and synthetic graphite (60:40 blend) in order to lower the wear rate, and form up to 15% of the lining. A wide range of lubricants employ graphite (flake and vein), including high-temperature bearings and oils

Flake graphite

(which leave a thin film of graphite once the carrier has evaporated), die lubricants, and anti-seize agents in steel mills and railways. Abrasive components are undesirable for use in lubricants and carbon brushes. Flake and amorphous graphite is used in alkaline/manganese long-life batteries. Amorphous graphite is used in dry cell zinc/carbon batteries. Pencil manufacturers use a mixture of graphite (vein or amorphous), clay and organic binders to produce pencil leads. Amorphous graphite is also used to recarburize steel, in electric and generator motor brushes, in conductive coatings, anti-static floor paint, paint used to protect metal surfaces, in rubber, in explosives, to make electrodes and in metal bearings.

3.3 Market trends

The strong market for graphite throughout the late 1980s, mainly due to the demand for refractories from the steel industry, has tailed off. There is now a slight oversupply which is compounded by new flake graphite production capacity. However, the expected growth areas for natural graphite are in the use of flake graphite.

Growing markets for flake graphite include exfoliated graphite (for the manufacture of gaskets and seals used in cars), high-alumina and magnesia-graphite bricks, zirconia-graphite, alumina-graphite and friction materials. Table 3 gives the current (July 1992) prices of graphite.

Table 3: Graphite prices (July 1992)

Crystalline lump	92/95%C	$750 - 1,500
Crystalline large flake	85/90% C	$650 - 1,200
Crystalline medium flake	85/90% C	$450 - 1,000
Crystalline small flake	80/95% C	$400 - 600
Powder (75 μm)	80/85% C	$325 - 360
	90/92% C	$520 - 600
	95/97% C	$770 - 1,000
	97/99% C	$1,000 - 1,300
Amorphous powder	80/85% C	$220 - 440

Price quoted per metric tonne
Source: 'Industrial Minerals' magazine, July 1992.

Flake graphite

4. MINERALOGICAL CHARACTERISATION

Mineralogical characterisation of graphite-bearing rocks with economic potential should aim principally to determine the graphitic carbon content and graphite flake size. These two properties will determine the economic value of the graphite. Mineralogical characterisation also provides the basis for the planning of laboratory beneficiation trials.

An initial thin-section examination of graphite-bearing rocks is useful for determining the size and shape of the graphite flakes, their relationship to other minerals present, and likely liberation size. A full petrographic description of a typical graphite schist is given as part of the graphite 'case history' in Section 6 of this manual. A similar examination of poorly-indurated weathered graphitic rock and graphite rich soil can be carried out by binocular microscope. Identification of non-graphite minerals may be confirmed by X-ray diffraction (XRD). Landis (1971) proposed a graphitisation series between flake graphite and amorphous graphite based on XRD peak width and shift. Comparison by BGS laboratories of XRD data from selected commercial 'crystalline' and 'amorphous' graphite samples (Figure 2) shows a limited amount of peak broadening in the latter material, but no evidence for peak shift. It is likely that particle size differences between the two materials will account for this contrast in peak width. Care should therefore be taken not to confuse trade descriptions of graphite such as 'amorphous' and 'crystalline' with strict mineralogical terms.

Determination of the carbon content of graphite-bearing rock (graphite 'assay') is generally carried out by a simple loss-on-ignition (LOI) test. The laboratory procedure is given in Appendix 3. Graphite content is determined as the percentage weight loss of a sample heated in air to 1000°C. LOI should be performed on duplicate samples to monitor the precision of the analysis.

Graphite-bearing soils contain clays and iron oxyhydroxides which will also lose weight on heating. Other minerals present in graphite-bearing rocks may also show this behaviour and must be accounted for. Microscope and/or XRD examination will indicate the presence of any minerals which might 'interfere' with the determination of graphitic carbon by LOI. A modified LOI test has been devised to take this into consideration. A list of some possible minerals which may cause problems is given in Appendix 3.

Thermogravimetry (TG) affords an alternative method for determination of graphitic carbon content. The technique is of particular value if other minerals which evolve volatiles on heating are present. The sample is heated at a controlled rate to 900°C under nitrogen, by which temperature any clays or carbonates present will have decomposed. The instrument records weight losses in the sample resulting from these reactions. The temperature is maintained at 900°C and oxygen is

Flake graphite

introduced to the sample. The graphitic carbon is oxidised leaving a residue of ash. Graphitic carbon content can be calculated from the resulting weight loss curve. Provided the sample is well-characterised optically or by XRD, it may be possible to quantify other volatile-evolving minerals from the weight loss curve. Figure 3 illustrates a typical weight loss curve.

LOI presents a very simple and rapid technique for assay of graphite. It is a technique which is useful in the preliminary assessment of large number of raw samples from the field, as well as in the monitoring of graphite products from any subsequent beneficiation trials. Relatively large sample weights used in this test ensure that sampling errors are kept to a minimum. Though it potentially provides more detailed temperature-dependent data, TG requires much more sophisticated equipment and tends to be more prone to sampling error because of the small sample weight required by the thermobalance. It is very difficult to obtain a small sub-sample of flake graphite-bearing material which is truly representative.

Flake graphite
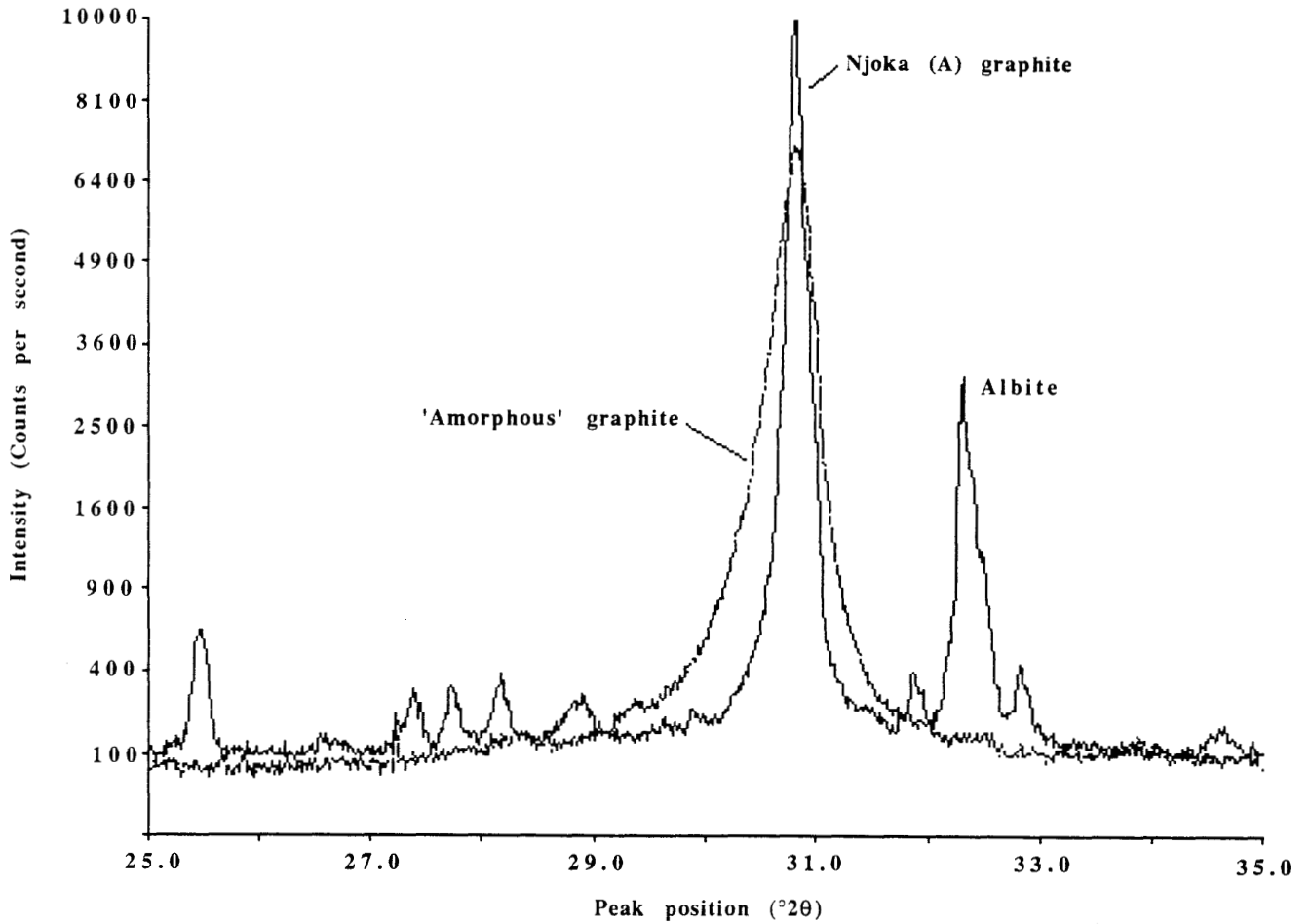

Figure 2. XRD traces of flake graphite from Zambia and 'amorphous' graphite from China showing contrasting peak width:height ratio.

Flake graphite
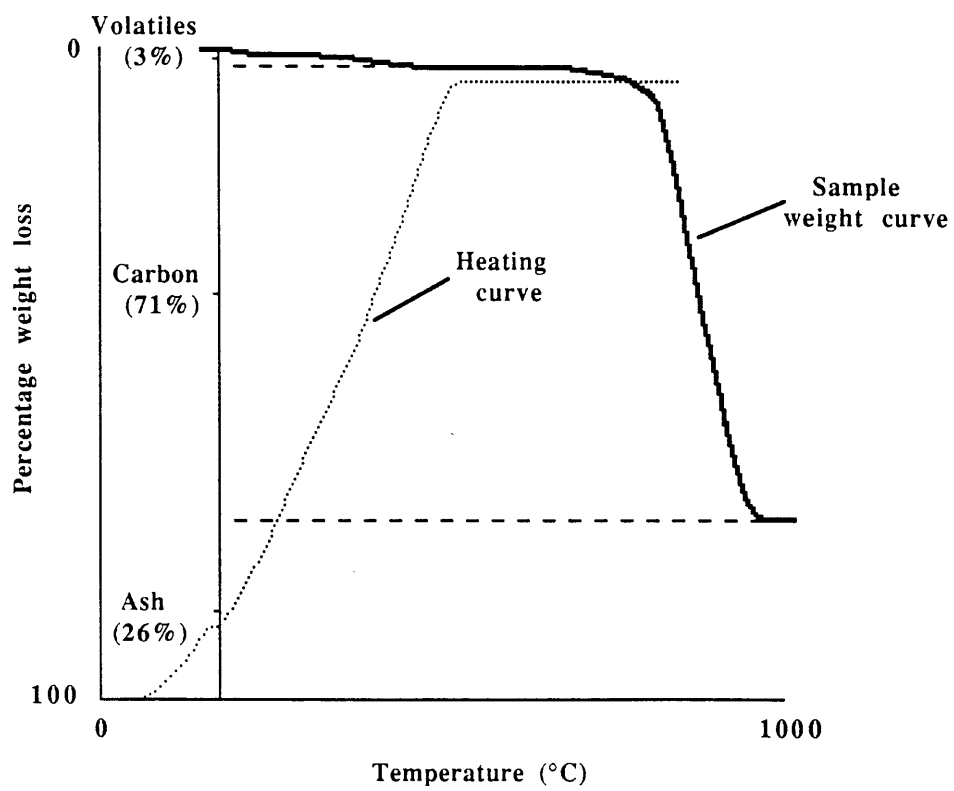

Figure 3. TG weight loss curve of a Zambian flake graphite.

Flake graphite

5. LABORATORY PROCESSING TRIALS

Laboratory processing trials of flake graphite-bearing samples should aim to establish the optimum method by which flake of a particular size range might be separated with the maximum possible grade and recovery. *Recovery* is the proportion of the total graphite present in the head sample or starting material which reports to the concentrate and is calculated using grade and yield data gathered during processing trials. Graphite *grade* is generally determined by the graphitic carbon content of a concentrate. *Yield* is the proportion of the total weight of the starting material contained in the concentrate. An example of a grade and recovery calculation for a flake graphite is given in Appendix 6. An assessment of the economic potential of samples from a deposit will require an indication of the likely processing scheme, including grade and recovery data over a range of flake sizes. The results of various use-related tests may also be required.

Graphite is relatively easy to separate from its host rock, though it is more difficult to produce high grades (90% and above), and many producers process their graphite several times to eliminate impurities. Graphite with very high carbon contents (96 - 99%) must be chemically and thermally treated to reduce the level of impurities. A flow chart illustrating a suggested laboratory processing scheme is given in Figure 4. A typical commercial processing scheme is given in Figure 5.

Determination of flake size will indicate the size at which the graphite may become liberated from the host rock. This is important as it will minimise the amount of crushing and grinding required prior to mineral separation. Flakes in the size range 250 µm to 1 mm will command the highest prices, with fine graphite flakes (down to 125 µm) also in some demand. An excess of graphitic fines will reduce the flake size and therefore the value of the final product. Also fine graphite will coat other minerals, which may then act as graphite during froth flotation and be recovered with the graphite concentrate - thereby reducing the grade of the product. Mica will often occur interlayered with graphite which may be difficult to remove during preparation. Fine material (such as clay and lateritic soil) may coat the graphite. These minerals will complicate graphite processing.

5.1 Preparation of graphite-bearing rock for beneficiation

The concentration of graphite from graphite-bearing material is dependant on the graphite flake morphology and liberation size. As graphite is resistant to weathering it may often be pre-concentrated in graphite-bearing soil, but with graphite-bearing rock the graphite needs to be liberated. Roller mills and cone grinders (or high-speed impact

Flake graphite

mills in larger-scale operations) produce a differential grinding effect that tends to concentrate the graphite in the coarser size fractions. Granular materials, such as quartz and feldspar, are ground into the fines whereas graphite flakes slip through relatively unscathed. However this may be countered by graphite being ground by contact with the granular material during grinding to produce an even distribution of graphite. Care should be taken not to over grind the graphite as it will tend to smear out over the other minerals present.

5.2 Graphite concentration

Examination of the size fractions by binocular microscope indicates if the correct liberation size was chosen, as essentially all the graphite flakes will be free of the host rock. If a large proportion of graphite is 'locked' into the host rock then attempts at separation will produce poor results and the larger size fractions may need to be ground further. If this is required, the next largest sieve diameter is used as the new liberation size. For example, if the original liberation size chosen was 2 mm, then 1 mm is selected as the new liberation size (and so on down the sieve series 2 mm, 1 mm, 500 μm, 250 μm and 125 μm). The process of grinding and microscopic examination is repeated until the correct liberation size is achieved. A procedure for grinding and screening prior to graphite concentration is given in Appendix 4.

5.2.1 Froth flotation

Graphite is generally concentrated from crushed rock by froth flotation. Historically, it was the first ore to be concentrated using flotation. Graphite is a naturally hydrophobic mineral and will float with little encouragement. Despite its natural floatability, the separation of feldspar, quartz, mica and carbonate gangue is normally improved by the addition of reagents such as kerosene (which coats the graphite) and pine oil (which stabilises the froth). Selectivity may be improved by adjusting the pH with caustic soda to the 7.5 - 8.5 range (Crozier, 1990).

Sized material is essential for efficient froth flotation. Material less than 125 μm will not concentrate effectively. Laboratory flotation is normally carried out as a batch process in glass cells using a mixer capable of injecting a stream of bubbles into the sample. Appendix 4 outlines the method of graphite flotation used in BGS laboratories. This procedure produces a concentrate (material that floats) and a tailing (material that sinks). A second flotation is carried out to 'clean' the first graphite concentrate, as well as to remove any mica and material smeared with graphite during preparation. The two-part flotation process results in three products: a concentrate (from second flotation),

Flake graphite

middling (material that sank during the second flotation) and tailing (material that sank during the first flotation). Only small amounts of reagents are required; 3-4 drops of each are usually sufficient after which no more froth will appear, and it is important not to add too much initially. Froth flotation generally works best on a 'starvation diet' of reagents. Graphite concentrates grading up to 95% can be produced by froth flotation in the laboratory; grades higher than this can only be achieved by chemically leaching out the impurities from the concentrate.

5.2.2 Air classification

Industrial-scale froth flotation concentration of graphite requires large amounts of water and may not be an appropriate process for mines situated in arid areas. Air classification can separate graphite flakes from their host material by exploiting their low density and platy nature. Using either 'zigzag' air classifiers or air tables, closely-sized material can be upgraded by effectively 'winnowing out' the graphite. A schematic diagram of a zigzag classifier is shown in Figure 6. Air classification requires different operating conditions for different size fractions, as larger flakes require a higher air flow to separate them and vice versa. An outline procedure for air classification of flake-graphite-bearing material is given in Appendix 5. Pre-concentrates grading up to 70% graphite and recoveries of 70% graphite can be achieved, though the presence of mica or other minerals with a platy habit may prejudice the effective pre-concentration of graphite using this method. Where other platy minerals are absent, air classification may halve the total weight of material required to be processed by froth flotation, with the attendant savings in water and reagents.

5.3 Assessment of products

The effectiveness of the laboratory processing trials is measured by calculation of the grade and recovery of the graphite products. Grades are generally determined using the LOI technique described above and recoveries calculated using the method set out in Appendix 6. Recovery values will serve to indicate the efficiency of the beneficiation scheme adopted and the 'amenability' of the graphite-bearing rock to processing. The relatively high economic value of graphite products may mean that low rates of recovery can sometimes be tolerated. Data on graphitic carbon content (or grade) of laboratory concentrates from a specific size range may be compared directly to commercial specifications.

Chemical analysis of the ash produced by LOI tests on concentrates will indicate if undesirable oxides are present. These include major elements such as SiO_2, Al_2O_3, MgO, CaO and Fe_2O_3. Trace metals such as

Flake graphite

copper, cobalt, tin and arsenic are undesirable in graphite used for alkaline batteries and carbon brushes. Appendix 7 gives the chemical analysis of the Lac Knife graphite from Canada.

5.3.1 Use-related testing

A number of tests related to specific applications of flake graphite can be carried out on concentrates from laboratory processing trials. Use-related tests for graphite used in the manufacture of crucibles include 'apparent density', 'flakiness' and 'toughness'. 'Apparent density' is largely controlled by the thickness of the flakes, assuming a close size distribution. A value of 45 g per 100 cm³ is considered good and anything over 58 is poor. This simple bulk density value is determined by the weight of graphite required to fill a 100 cm³ container. 'Flakiness' is determined by placing 100 g of graphite (greater than 250 µm) into a 250 ml measuring cylinder. The cylinder is then tapped until the material is completely packed down. The volume which the material occupies is known as the flakiness index, which for crucible grade flake should not be less than 129 ml. 'Toughness' is determined by grinding a sample of graphite of a fixed size distribution under standard conditions and then determining the amount of less than 75 µm material produced.

Flake graphite
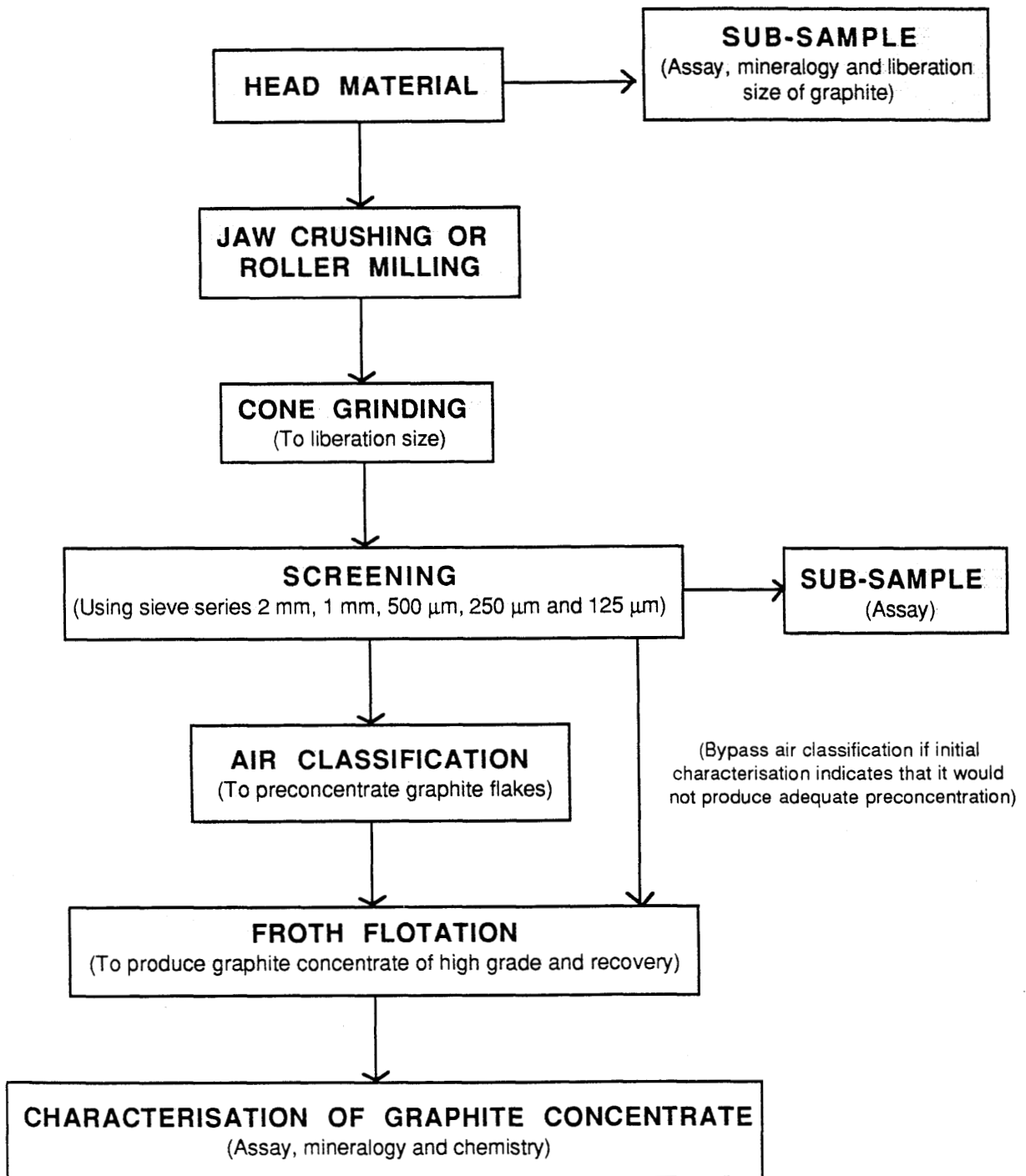
Figure 4. Suggested flowsheet for laboratory processing of flake graphite.

Flake graphite

Run of mill ore

Jaw crusher

Cone crusher

Fine ore bin

Rod mill

Rougher flotation cells

Classifier

Tailings to dam

Rod mill

Cleaner flotation cells

Centrifuges

Drier

Screens

Bagging

Figure 5. Process flowsheet at Lynx graphite mine, Zimbabwe (Muchemwa, 1987).

Flake graphite

Figure 6. Schematic diagram of a zig-zag air classifier.

Flake graphite

6. CASE HISTORIES

Laboratory evaluation of contrasting flake graphite-bearing rocks from Mozambique and Zambia are described briefly below.

6.1 Mozambique

Three samples of graphite-bearing schist from Ancuabe, Mozambique were evaluated. These consisted of fresh core (11% graphite); weathered rock (13% graphite) and eluvial soil (9% graphite). They all contained flake graphite (0.5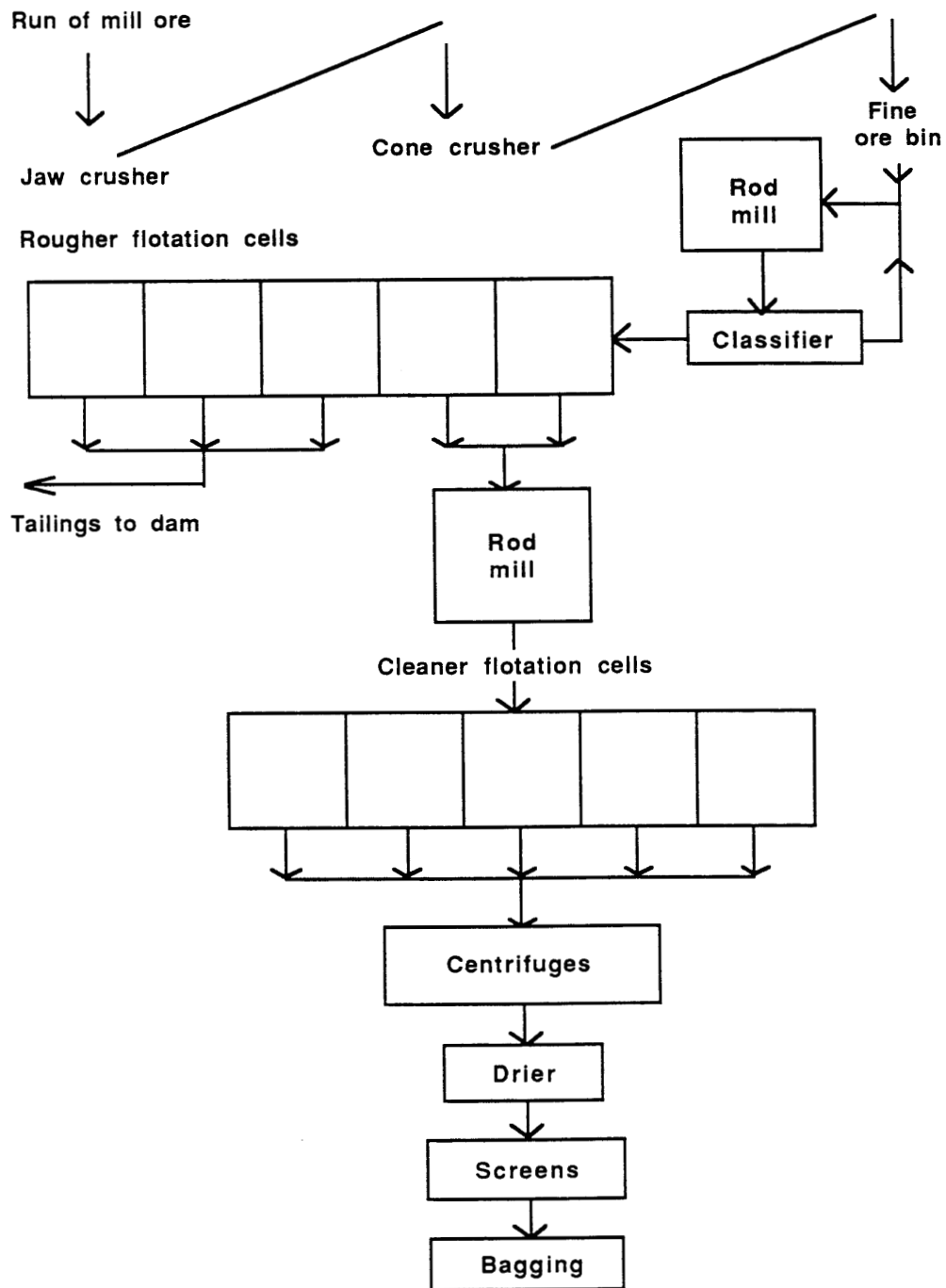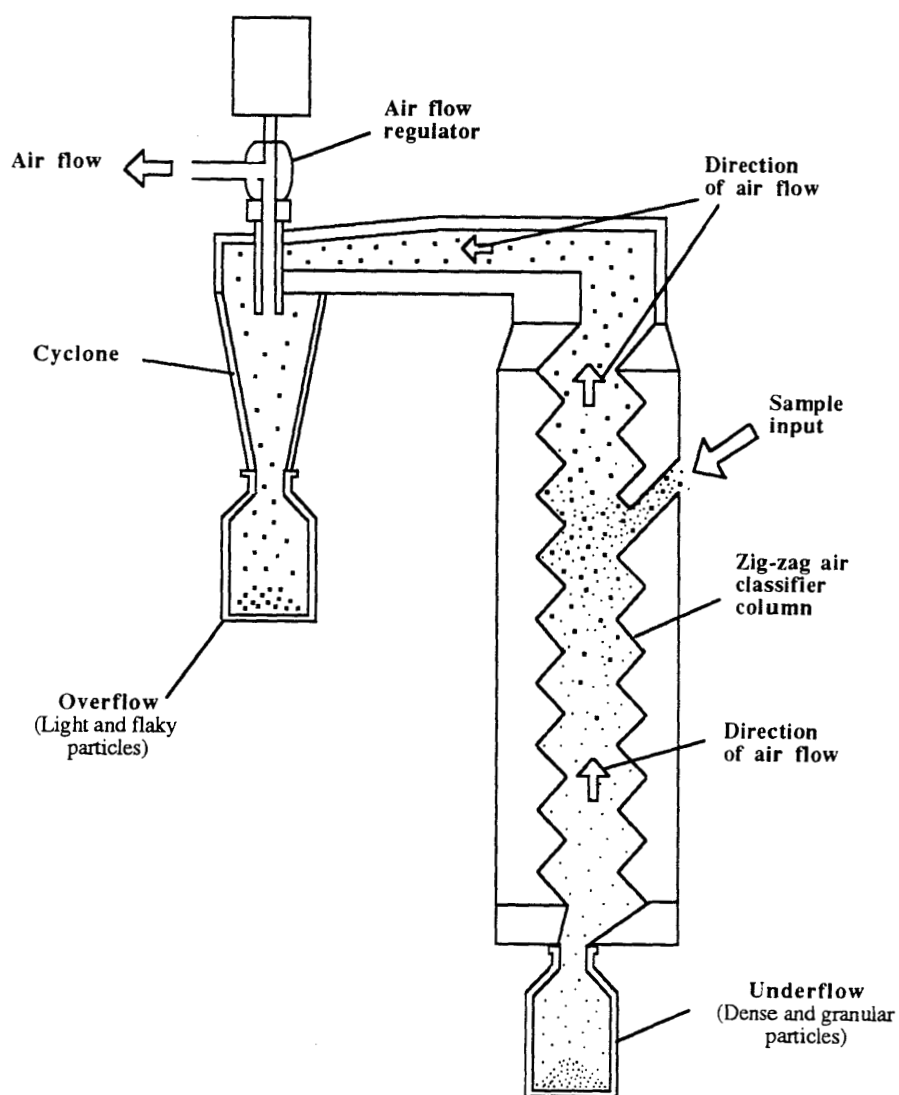-2 mm in size), with varying amounts of quartz and feldspar; the weathered sample was iron stained and the eluvial samples contained a large proportion of iron-oxide-rich soil. The samples were processed down to 1 mm, which was taken as the liberation size. Graphite-rich >1 mm fractions were produced during stage cone grinding, as a direct result of preferential concentration. The >125 μm size fractions were pre-concentrated using 'zigzag' air classification and the air classified concentrates further upgraded using froth flotation. The results are given in Table 4.

Air classification produced concentrates with up to 73% graphite and up to 71% recoveries. Froth flotation produced concentrates with up to 90% graphite and recoveries of up to 72%. Approximately 85% of the head material was rejected by using air classification prior to froth flotation. This highly effective pre-concentration of graphite would result in considerable savings of water and reagents during the final froth flotation process.

Table 4. Concentrate grade and recovery of graphite from Mozambique.

Sample	Air classification			Froth flotation		
	Fraction Wt %	Assay Wt %	Recovery Wt %	Fraction Wt %	Assay Wt %	Recovery Wt %
Core	14.4	53.7	63.7	9.5	88.4	65.1
Weathered	13.7	73.2	71.1	11.5	90.2	72.2
Eluvial	6.5	55.3	36.7	3.2	90.5	29.3

Note: Due to lack of material the air classification concentrate and middling of the -250+125 μm fractions were combined prior to froth flotation. Consequently some of the froth flotation recovery figures are higher than those for air classification.

Flake graphite

6.2 Zambia

Three samples of graphite-bearing schist from Njoka and Petauke, Eastern Province, Zambia, were examined. The Njoka graphite samples comprised a rubbly graphite-schist (A) and a consolidated graphite-schist (B). Both consisted mainly of feldspar with a small amount of mica, graphite (16% in A and 18% in B) and quartz. The petrography of Njoka (B) is briefly described below and illustrated by the photomicrographs in Figures 7 and 8. The graphite in both Njoka samples is the fully ordered variety. The samples were crushed to <1 mm (which was taken as the liberation size) by a combination of jaw crushing, disc milling and cone grinding, and then screened down to 125 µm. Graphite-rich >1 mm fractions were produced during stage cone grinding. The >250 µm fractions were pre-concentrated by air classification and the products of air classification, together with the 250-125 µm fraction, were concentrated by froth flotation. The results are given in Table 5. Air classification produced relatively low-grade flake-graphite concentrates with very low recoveries, due to mica concentrating with the graphite. Froth flotation produced higher-grade concentrates with good recoveries. The main impurity in the concentrates was found to be mica.

The Petauke graphite schist contains mainly feldspar and mica, with smaller amounts of graphite (6%) and quartz. Material was processed in a similar fashion to that from the Njoka deposit, although the 250-125 µm fraction was pre-concentrated by air classification and the products upgraded by froth flotation. Results are also given in Table 5. Air classification produced a very low-grade flake graphite concentrate with a low recovery due to the presence of mica. Froth flotation produced a high-grade flake graphite concentrate with a very low recovery due to the mica, and further processing would be required to retrieve the graphite lost in the flotation tailings.

Table 5. Concentrate grade and recovery of graphite from Zambia.

Sample	Air classification			Froth flotation		
	Fraction Wt %	Assay Wt %	Recovery Wt %	Fraction Wt %	Assay Wt %	Recovery Wt %
Njoka A	12.9	53.1	40.8	16.0	78.2	70.1
Njoka B	11.0	70.7	41.7	23.3	73.2	73.5
Petauke	11.2	24.9	40.2	2.3	87.1	39.0

Flake graphite

6.2.1 Petrographic description of Njoka (B) graphite

Njoka (B) graphite is a quartz-albite-schist containing graphite and mica (possibly phlogopite). The quartz and feldspar form a crudely foliated mosaic with curved, slightly interlocking interfaces. The foliation is typically created by elongation of the feldspar grains. The feldspar has patchy turbid areas of (sericitic) alteration. Parallel to the quartz-feldspar lithons (lenses) is a slightly undulose schistose fabric formed of graphite flakes, intergrown with mica. The mica shows some alteration. Graphite flakes are up to 3-4 mm long, generally 0.4-2 mm, and from 0.05 mm thick.

Figures 7 and 8 show photomicrographs of Njoka (B) graphite in reflected and transmitted light respectively. Figure 7 shows the graphite as light grey to white elongated grains with the quartz, feldspar and mica represented by the darker grey material surrounding the graphite. Most of the graphite flakes are oriented normal to the plane of the photomicrograph, with some inclined (the light grey areas). Figure 8 shows the same area as in Figure 7 (at a slightly lower magnification) with black graphite surrounded by quartz and feldspar. Pale yellowish-brown mica is aligned parallel to the graphite.

Based on study of the thin section, a liberation size of 1 mm was selected as the most appropriate for processing of the Njoka graphite. The close association of mica with graphite is an indicator of potential problems during processing. Mica tends to concentrate with graphite during air classification and may affect recovery of graphite during froth flotation.

Flake graphite

Figure 7. Photomicrograph of Njoka (B) graphite schist (reflected light)

Figure 8. Photomicrograph of Njoka (B) graphite schist (transmitted light)

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Flake graphite

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Appendix 1: Specifications of graphite for different applications.

Application	Type	C content	Flake size	Comments
Refractories				
Magnesia graphite	F	85 - 90%	150 - 710 µm	Ash <2%, often up to 10%.
Alumina graphite	F	Min. 85%	150 - 500 µm	
Crucibles	AF	80 - 90%	+150 µm	Bulk density 48 - 54 g per 100 cm^3
Expanded graphite	F	Min. 90%	200 - 1700 µm	
Foundry additive	AFV	40 - 70%	53 - 75 µm	Sulphides and other readily fusible minerals undesirable
Foundry core and mould washes	AF	70 - 90%	~75 µm	
Brake / clutch linings	AFV	Min. 98%	<75 µm	
Bearings	FV	90 - 93%	+150 µm	
Lubricants	AFV	98 - 99%	53 - 106 µm	Free from sulphides, abrasive material and metallic contaminants.
Dry cell batteries	A	Min. 88%	85% <75 µm	No metallic impurities and S less than 0.5%
Alkaline batteries	AF	Min. 98%	5 - 75 µm	No impurities such as Cu, Co, Sb and As.
Recarburizing steel	A	98 - 99%	~5 µm	
Carbon brushes	AFV	95 - 99%	<53 µm	< 1% ash / silica. No abrasive or metallic contaminants.
Electrical	FV	93 - 95%	+150 µm	
Pencils	AF	95 - 97%	+150 µm	Free of gritty impurities
Conductive coatings	A	50 - 55%	<75 µm	May contain up to 25% silica, but free from gritty impurities.
Packing paints	FV	85 - 90%	<150 µm	
Polishes	AFV	85 - 90%	<150 µm	
Drilling mud (lubricating)	F	80%+	N/A	About 4lb per barrel of mud
Explosives (control of burning rate)	AF	65%	<150 µm	Free from sulphides and acids. Low moisture content
Nuclear reactors (moderators & reflectors)	F	93 - 95%	N/A	Free from high neutron absorbing elements, e.g. boron
Boilers (scale prevention)	F	50%+	N/A	

Key : A - amorphous; F - flake; V - vein; N/A - Not available
Table adapted from Russell (1988), Harben (1992), Goossens (1982), Muchemwa (1987) and Tron (1964).

Flake graphite

Appendix 2: Particle size and carbon content data for commercial graphite

Source		Particle size	% carbon
China	Large flake	80% > 300 μm	85 - 90%
	Medium flake	80% > 180 μm	85 - 90%
	Small flake	50% > 180 μm	80 - 90%
Madagascar	Large flake	97% >250 μm	85 - 94%
	Medium flake	97% >180 μm	80 - 92.5%
	Fine flake	95% > 180 μm (min)	75 - 90%
	Extra fine flake	N/A	70 - 90%
Norway	Large flake	>150 μm	85 - 95%
	Medium flake	>100 μm	85 - 95%
	Small flake	<75 μm	80 - 95%
Canada (Lac Knife)	Large flake	>150 μm	92 - 95%
	Medium flake	>75 μm	90 - 94%
	Small flake	<75 μm	85 - 89%
India	Large flake	180 - 600 μm	70 - 88%
	Small flake	<150 μm	70 - 88%
Zimbabwe (Lynx mine)	Flake 'A'	>160 μm	86 - 95%
	Flake powder	<160 μm	80 - 90%
Sri Lanka (vein graphite)	Large lump / lump	>10 mm	92 - 99%
	Chippy dust	<5 mm	80 - 99%
	Powders	<75 μm	70 - 99%
Mexico	Amorphous		80 - 90%
Italy	Amorphous		60 - 80%
Austria	Amorphous		66 - 99%

Table adapted from Harben (1992), Muchemwa (1987), Graffin (1983) and Bonneau (1990).

Flake graphite

Appendix 3: Graphitic carbon assay

Assuming a representative sample (about 5-10 kg) of the graphite has been taken, the laboratory evaluation should follow two roughly similar paths. This depends on the degree of induration of the sample.

Preparation of graphite-bearing rock

1. Retain a reference sample and jaw-crush the remaining material to less than 4 mm.

2. Extract a 50g sub-sample by cone and quartering. Grind to less than 500 µm.

3. Determine particle-size distribution of crushed material by screening remainder on the sieve series 2 mm, 1 mm, 500 µm, 250 µm and 125 µm.

Preparation of graphite-bearing soils

1. Retain a reference sample and sub-sample 1 kg by cone and quartering.

2. Determine particle-size distribution of sub-sample by dry screening from 4 mm down to 125 µm. Extract small sub-samples from each size fraction and grind to less than 500 µm.

3. Screen remainder of sample at 4 mm and reduce >4 mm by jaw crushing. Extract a 50 g sub-sample by cone and quartering and grind to less than 500 µm.

Flake graphite

Appendix 3 (continued)

Assay of graphite-bearing rock

1. Extract 1 g of ground sample (<500 μm) and place in weighed silica crucible.

2. Dry overnight at 105°C and weigh accurately (to 4 decimal places).

3. Heat in furnace to 1000°C for 2 hours to completely oxidise the graphite. Cool in a desiccator and weigh.

4. Calculate graphite content from following equation;

   Percentage graphitic carbon

   $$= \frac{105°C \text{ weight} - 1000°C \text{ weight}}{105°C \text{ weight}} \times 100$$

Assay of graphite-bearing soil

1. Follow points 1-2 in the procedure for assay of graphite-bearing rock.

2. Heat to 375°C for 16 hours to oxidise and remove all the organic matter and dehydrate iron hydroxides. Cool in a desiccator and weigh.

3. Heat to 600°C for 1 hour to dehydroxylate clays and any remaining iron hydroxides. Cool in desiccator and weigh.

4. Heat in furnace to 1000°C for 2 hours to completely oxidise the graphite. Cool in desiccator and weigh.

5. Calculate graphite content from following equation;

   Percentage graphitic carbon

   $$= \frac{600°C \text{ weight} - 1000°C \text{ weight}}{105°C \text{ weight}} \times 100$$

Flake graphite

Appendix 3 (continued)

Minerals likely to interfere with graphite assay by loss-on-ignition

Mineral	Composition	Decomposition	Weight loss
Calcite	$CaCO_3$	Decarbonation at 920 - 975°C	43.97%
Dolomite	$CaMg(CO_3)_2$	Decarbonation at 720-950°C	47.7%
Kaolinite	$Al_4Si_4O_{10}(OH)_8$	Dehydroxylation at 450 - 700°C	~13%
Limonite	$FeO(OH).nH_2O$	Dehydroxylation at 350 - 400°C	>10.1%
Goethite	$FeO(OH)$	Dehydroxylation at 250 - 360°C	10.1%
Gibbsite	$Al(OH)_3$	Dehydroxylation at 240 - 560°C	34.6%
Brucite	$Mg(OH)_2$	Dehydroxylation at 400 - 450°C	30.89%
Talc*	$Mg_3Si_4O_{10}(OH)_2$	Dehydroxylation at 850 - 1000°C	4.75%
Tremolite*	$Ca_2Mg_5Si_8O_{22}(OH)_2$	Dehydroxylation at 990 - 1070°C	2.8%

Temperature ranges for carbonates apply only under dynamic heating conditions (e.g. TG analysis). Under static heating, carbonates will decompose at least 100°C lower.

* It is not possible to distinguish the weight losses from these minerals from that of graphite.

Flake graphite

Appendix 4: Sample preparation and froth flotation procedure

Sample preparation:

1. Weigh the sample prior to stage grinding using either a roller mill or cone grinder until all material passes the liberation size.

2. Dry screen the ground samples through the sieve series, beginning with the sieve immediately below the liberation size. Weigh the material retained on each sieve and calculate the size distribution.

3. Extract a representative 50 g sub-sample from each size fraction for determination of carbon content. Grind to less than 500 μm.

Froth flotation:

1. Add 200 - 500 g of sized material to the cell and fill with deionised water.

2. Add a drop of kerosene to the suspension and mix. The kerosene collector coats the graphite.

3. Add a drop of methyl isobutyl carbinol (MIBC) frother and continue mixing.

4. Inject air into the sample. This generates bubbles from the base of the cell. The graphite adheres to the MIBC-stabilised bubbles and floats to the surface forming a froth.

5. Scoop out the graphite froth.

6. Repeat steps 2-5 until no more graphite remains.

7. Following steps 2-6, refloat the initial graphite concentrate. No further addition of reagents is required.

8. Oven dry concentrates, middlings and tailings overnight at 60°C. Weigh and calculate % yield.

9. Extract a representative 50 g sub-sample from each product for determination of carbon content. Grind to less than 500 μm.

10. Determine grade using procedure set out in Appendix 3.

11. Calculate recovery using procedure set out in Appendix 6.

Flake graphite

Appendix 5: Air classification procedure

Air classification is a method of concentrating graphite flakes by using a stream of air to separate granular minerals such as quartz and feldspar. The equipment used at BGS is an Alpine zigzag air classifier, which consists of a zigzag column up through which air flows. Material treated by air classification must be closely sized and fall within the range 125 μm and 6 mm (see Appendix 4). Graphite flakes are entrained in the upward air flow, reporting to the overflow, and granular material falls through the airflow, reporting to the underflow. The aim of air classification should be to produce a graphite preconcentrate with a high grade and recovery and low yield.

1.   The conditions for the separation of flake graphite from granular material depend on the grain size, with higher air flow rates required for coarser size fractions and vice versa. Air classification of graphite-bearing material is a process of trial and error.

2.   Pass the graphite-bearing size fraction through the air-classifier at a fixed air flow rate, aiming to remove graphite flakes only into the overflow (graphite concentrate). If granular material reports to the overflow then the fraction should be recombined and re-passed at a lower air flow rate. If there appears to be an excess of graphite flakes reporting to the underflow then re-pass at a higher air flow rate.

3.   Once a graphite concentrate has been produced, the underflow material should be re-passed at a higher air flow rate in order to remove all the remaining graphite. This time granular material will report to the overflow (middling fraction) and an attempt should be made to produce an underflow as free from graphite as possible (tailing fraction).

Flake graphite

Appendix 6: Graphite grade, yield and recovery calculation.

The following is a worked example of grade, yield and recovery calculations for a series of products from laboratory beneficiation trials on a flake graphite.

Weathered graphite schist:

Head sample weight = **20 kg**
  Head sample grade = **13.2%** graphite equivalent to **2.64 kg** graphite

Sample is screened:

1mm-500μm fraction yield = **7.68 kg** or **38.4 wt %**
1mm-500μm fraction grade = **17.3%** graphite equivalent to **1.32 kg** graphite

% graphite recovered from the head sample to the 1mm-500μm fraction
$$= 1.32/2.64 \times 100$$
$$= \underline{\mathbf{50\%\ recovery}}$$

1mm-500μm fraction is air classified:

Product	yield wt (kg)	wt % of size fraction	graphite grade (%)
conc	1.35	17.6	82.5
mid	1.51	19.7	11.5
tail	4.81	62.7	2.4
total	7.68	100	

In order to calculate the % graphite recovered from the head to each product, first calculate the weight of graphite in each product from the yield and grade values:

wt graphite in conc $= 1.35/100 \times 82.5 = \mathbf{1.11\ kg}$
wt graphite in mid $= 1.51/100 \times 11.5 = \mathbf{0.17\ kg}$
wt graphite in tail $= 4.81/100 \times 2.4 \;\; = \mathbf{0.11\ kg}$

therefore the % graphite recovered from the head to each of the products is as follows:

1mm-500μm fraction concentrate $= 1.11/2.64 \times 100 = \underline{\mathbf{42.2\%\ recovery}}$
1mm-500μm fraction mid $= 0.17/2.64 \times 100 \;= \underline{\mathbf{6.4\%\ recovery}}$
1mm-500μm fraction tail $= 0.11/2.64 \times 100 \;= \underline{\mathbf{4.2\%\ recovery}}$

Flake graphite

Appendix 7: Chemistry of Lac Knife graphite, Quebec, Canada

Component	>500 μm wt %	500-150 μm wt %	<150 μm wt %
Graphitic carbon	94.1	95.5	90.2
CO_2	0.72	0.62	0.23
S	0.35	0.38	0.78
Volatiles	0.75	0.79	1.37
Ash	4.32	3.57	6.73
Ash composition			
(i) Major elements			
SiO2	47.0	44.5	46.0
Al2O3	14.1	15.0	15.3
Fe2O3	22.8	24.2	23.0
MgO	5.05	5.32	4.15
CaO	4.39	3.92	4.94
Na2O	0.55	0.51	0.84
K2O	3.26	3.58	3.20
TiO2	0.53	0.54	0.43
MnO2	0.15	0.17	0.15
P2O5	0.24	0.15	0.18
(ii) Trace elements (PPM)			
V	3,100	2,800	2,200
Mo	1,900	3,800	5,400
Cu	603	688	883
Co	133	195	33
U	230	220	120
Ni	489	495	449

Data from Bonneau & Raby (1990).