

# The extraction of gold from sulphidic concentrates by roasting and cyanidation\*

by J.J. ROBINSON†

## SYNOPSIS

The refractoriness of the gold in many of the pyritic ores from the eastern Transvaal is due mainly to the association between the gold and the sulphides, but it can be due to the presence of graphitic carbonaceous material, which complicates the recovery of the gold by virtue of its adsorption activity.

In an examination of the roasting characteristics of three flotation concentrates from the Barberton area, it was found that the gold in the concentrates occurred mostly in two forms: in very fine gold particles distributed within the arsenopyrite, and in a slightly coarser form loosely associated with the pyrite. Direct cyanidation of the concentrates gave gold extractions of from 16 to 30 per cent. After the concentrates had been roasted at temperatures of 650 to 700°C for 20 minutes with rabbling, the extractions varied from 86 to over 90 per cent. Examination of the residues after cyanidation indicated that gold was present in the porous calcines or in the calcined sulphide product. This observation, together with porosity measurements, suggests that physical encapsulation of the gold particles is the main reason for the refractory behaviour of the gold in the calcines.

## SAMEVATTING

Die ekstraksievastheid van die goud in baie van die piritiese ertse afkomstig van Oos-Transvaal, is hoofsaaklik toe te skryf aan die assosiasie tussen die goud en die sulfiede, maar dit kan ook toe te skryf wees aan die aanwesigheid van grafitiese koolstofhoudende materiaal wat die herwinning van die goud deur sy adsorpsieaktiwiteit bemoeilik.

Daar is in 'n ondersoek na die roostereienskappe van drie flottasiekonsentrate afkomstig van die Barbertongebied vasgestel dat die goud in die konsentrate hoofsaaklik in twee vorms voorkom: as baie fyn goudpartikels wat in die arsenopiriet versprei is, en in 'n effens growwer vorm wat losweg met die piriet geassosieer is. Regstreekse sianidisering van die konsentrate het goudeksktraksies van 16 tot 30 persent gelever. Nadat die konsentrate 20 minute lank met roering by temperature van 650 tot 700°C gerooster is, het die eksktraksies wat van 86 tot meer as 90 persent gewissel. 'n Ondersoek van die residu's na sianidisering het getoon dat goud in die poreuse kalsiene of in die gekalsineerde sulfiedproduk aanwesig is. Hierdie waarneming, tesame met poreusheidsmetings, dui daarop dat die fisiese kapsulering van die goudpartikels die hoofrede vir die ekstraksievaste gedrag van die goud in die kalsiene is.

## Introduction

It has long been recognized that sulphide flotation concentrates from the eastern Transvaal contain relatively high concentrations of gold, a certain proportion of which is refractory even after roasting<sup>1-8</sup>.

The mode of occurrence of gold in sulphides varies considerably from fine particles completely enclosed within sulphide grains to coarser particles located in surface irregularities and fractures. Some gold, although known to be present in sulphides, cannot be resolved even by the scanning electron microscope (SEM). Two main theories have been proposed for the occurrence of this type of gold: the gold may be finely distributed throughout the sulphide mineral, or it may form a solid solution with the sulphides<sup>6</sup>. No evidence could be found for either of these theories.

The prior roasting of sulphides as a means of enhancing the extraction of gold by cyanidation has been the subject of many articles. Following a review by Nagy *et al.*<sup>6</sup> of the chemical treatments applied to the recovery of gold from refractory ores, Hansen and Laschinger<sup>3</sup> prepared

a literature survey of the roasting of sulphidic gold ores and concentrates.

As well as describing the effects of a large number of minerals, including sulphide and arsenical minerals, on the cyanidation of gold, Nagy *et al.*<sup>6</sup> described refractory behaviour caused by the presence of carbonaceous material in the ore. The effect of graphite, and of a roasting treatment to minimize its effect on gold extraction, was discussed by Raicevic and Bruce<sup>9</sup>.

Roasting of a sulphide concentrate greatly increases its porosity, thus increasing the contact between the contained gold and the cyanide leaching solution. Since gold occurs in a diversity of forms, and in association with a wide variety of other minerals, roasting produces calcines that are chemically and physically diverse, thus increasing the possible causes of refractoriness<sup>10,11</sup>. The lock-up of gold caused by the fusion or recrystallization of hematite particles was recognized by Carter and Samis<sup>12</sup> as a significant factor in the refractory behaviour of gold, while Arriagada and Osseo-Asare<sup>13,14</sup> concluded that the loss of gold in the tailing after cyanidation was largely influenced by the roasting temperature.

The electrochemical passivation of gold has been suggested as a possible reason, in some cases, for low recoveries of gold by cyanidation<sup>7,15</sup>. Filmer<sup>16</sup>, in his study of the extraction of gold from calcines, proposed that physical encapsulation of the gold might have con-

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† Metallurgy Department, Bendigo College of Advanced Education, P.O. Box 199, Bendigo, 3550 Victoria, Australia.

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tributed to incomplete extraction but that, particularly for partially roasted calcines, passivation was possibly the major factor.

The reactions that arsenopyrite undergoes during roasting are not clearly understood. In an early publication, Archibald<sup>17</sup> outlined the conditions likely to be most suitable for the roasting of arsenopyrite concentrates. In a more recent review, Jha and Kramer<sup>18</sup> described the evolution of roasting technology from Edwards-type or Wedge-type roasters to fluidized-bed roasters, and noted the wide variation in the conditions recommended for the roasting of arsenical concentrates.

As many cyanided calcines contain a considerable quantity of gold, which is retained as a refractory component, much interest has been shown in the roasting process and in the nature of the calcines. Very little direct evidence of the nature of this refractory component has been obtained.

The present study formed part of a broader investigation into the recovery of gold from refractory ores. The roasting behaviour of three flotation concentrates from the Barberton area was examined, and the gold extractions from the calcines were determined. A SEM examination of samples of concentrates, calcines, and leached calcines was also undertaken in an effort to provide some insight into the cause of the refractoriness of the gold component.

## Experimental Work

### Particle-size Distribution

A sample of each of the concentrates studied was wet-screened with a 38  $\mu\text{m}$  sieve. The material retained on the screen was further screened at 212, 150, 106, 75, and 53  $\mu\text{m}$ , and the undersize from the 38  $\mu\text{m}$  screen was classified using a Cyclosizer into six separate fractions, including an undersize or slimes fraction. The mass of each dry size fraction was noted, and a curve of cumulative size distribution was drawn for each concentrate.

Each size fraction was analysed for its gold content by fire assay, and these analyses were plotted against the mean particle size of each size fraction on the same graph as the cumulative size distribution.

### Preliminary Roasts

In an attempt to remove the impurities in the concentrates, a small amount of sample (4 g) was spread uniformly over the bottom of a flat silica dish of 75 mm diameter and roasted in a muffle furnace at controlled temperatures. During the roast, a stream of air was passed over the samples via a tube passing through the door of the furnace.

At the end of the roast, the calcine was allowed to cool, and the loss in mass was determined. The calcine was then analysed for arsenic, sulphur, and carbon, the sulphur and carbon contents being determined by the Leco method, while the arsenic was measured by atomic-absorption spectrophotometry (AAS).

### Calcining before Cyanidation

A sample of 75 g was needed for the production of a calcine of between 50 and 60 g, which was considered to be the minimum amount that could be used in a cyanide leach.

Each sample was spread thinly on a large stainless-steel tray 260 mm square, and roasted in a muffle furnace. A tube introduced through the door of the furnace permitted a flow of air over the sample.

In some of these experiments, the effect of rabbling by hand with a stainless-steel rake through the door of the furnace at 5-minute intervals was investigated.

### Cyanidation

The calcines were leached in 2-litre narrow-necked glass bottles that were rotated on rollers. The calcines were mixed with water to give a pulp of 40 per cent solids and, prior to leaching, the pulp was aerated and its pH value was adjusted to 11,0 with calcium hydroxide. Aeration was continued with further additions of calcium hydroxide until the pH value remained constant at about 11,0.

Sodium cyanide was added to the pulp in a ratio of 0,2 g of sodium cyanide to 100 g of solid. The pulp was then leached for 24 hours.

Following filtration, the solid was washed well and retained for analysis. The residual gold was determined by fire assay, and the solution was analysed by AAS for its gold content. The solution was also titrated to show the consumption of calcium hydroxide and cyanide during the leach.

No loss of gold was assumed to occur during roasting, and the loss in mass was used in the adjustment of the head-assay value of the concentrate to the assay value of the calcine.

### SEM and Electron-microprobe Analyses

The concentrates, together with some calcines and cyanided residues, were examined on a Hitachi S450 SEM coupled to a Princeton Gamma-tech (PGT) energy-dispersive system for micro-analysis.

The samples were mounted in resin, polished, and examined by optical microscopy. Relevant areas were marked and, after they had been coated with carbon, these areas were examined with the SEM at low magnification.

Initially, the SEM was set for maximum contrast in the back-scattered electron image and, with the intensity set low, the topographical features were only just visible. Because of its high atomic number, gold appeared on the screen as bright flashes, which, with experience, could be readily recognized. Further areas were then scanned slowly at a magnification of 1000 to 5000 at low intensity.

## Results and Discussion

### Chemical and Mineralogical Composition

The chemical analyses of the three flotation concentrates are given in Table I, which shows the differences in the gold, sulphur, arsenic, and carbon contents. Table II presents the mineralogical contents of these concentrates, and major differences in the pyrite and arsenopyrite contents are evident.

### Particle-size and Gold Distributions

The gold distributions and the cumulative particle-size distributions are plotted in Fig. 1 for concentrates A, B, and C. It should be noted that, in this diagram, the gold-distribution values for the  $-38 \mu\text{m}$  fraction of each concentrate are disproportionately high in the coarser fractions. This is because the particles in these size ranges were

TABLE I  
ANALYSES OF THE FLOTATION CONCENTRATES

Concentrate	Au μg/g	S %	Total C %	Org. C %	As %	Fe %
A	43,3	17,0	1,5	1,3	12,1	29,1
B	75,2	16,1	2,4	0,6	16,7	23,3
C	32,3	23,7	5,6	4,2	1,77	27,5

TABLE II  
APPROXIMATE MINERAL COMPOSITION OF THE FLOTATION CONCENTRATES

Mineral	Mass of minerals in concentrate, %		
	A	B	C
Pyrite	22,0	14,8	39,2
Arsenopyrite	26,5	36,2	4,0
Chalcopyrite	<0,1	0,1	<0,1
Pyrrhotite	<0,1	<0,1	0,9
Galena	<0,1	<0,1	0,5
Sphalerite	<0,1	<0,1	0,1
Quartz	<25,0	<15,0	20,0
Chlorite	<15,0	<6,0	11,0
Sericite	5,0	<12,0	9,0
Ankerite	} <2,0	n.d.	<6
Calcite		n.d.	<4
Graphite		1,3	0,6

n.d. Not determined

classified hydraulically and not strictly sorted according to size.

All three concentrates exhibited a wide size distribution. A comparison of the three curves of cumulative mass distribution revealed that concentrate A contained more fines than concentrate B or C.

The pattern of distribution of the gold differed for the three concentrates. When the gold concentration in each size fraction was compared with the overall analysis of the concentrate, samples A and C exhibited similar distributions, while sample B showed a distinct shift in the gold distribution from the coarser fractions to the finer ones. Since concentrate B contained a relatively high proportion of arsenopyrite, this effect may be related to the gold association within the arsenopyrite.

Optical microscopy revealed some free grains of gold in the three concentrates, most being observed in sample C. Gold grains occurred within sulphide grains and also in association with the gangue. In sample C the gold was mainly associated with pyrite, and, to a lesser extent, pyrrhotite, while in samples A and B the gold was observed in association with arsenopyrite. Gold was thought to occur largely in a finely distributed form within the arsenopyrite grains. (This is discussed later.)

#### Removal of Sulphur, Arsenic, and Carbon

Prior to cyanidation of the calcine, several roasts were conducted in a muffle furnace on small samples of the concentrate so that the optimum conditions for the removal of sulphur, arsenic, and carbon could be determined.

The effect of temperature was examined as follows. Each sample was roasted for 30 minutes at temperatures of 500, 550, 600, 650, and 700°C, and the amount of sulphur, arsenic, and carbon removed and the loss in mass were measured. The results of these tests are presented in Fig. 2.

For concentrate A, the curves for loss in mass and the removal of sulphur and arsenic indicate no further losses after about 600°C. Higher temperatures of about 700°C were required for the almost-complete removal of carbon.

Similar trends for the elimination of sulphur, arsenic, and carbon were shown by concentrate B, although the loss in mass was much higher than for concentrate A. Again, 600°C appeared to be the optimum temperature for maximum loss in mass and maximum removal of sulphur and arsenic. The results again indicated that, for a roasting time of 30 minutes, higher temperatures would be needed for the maximum removal of carbon.

Concentrate C exhibited somewhat different behaviour in these tests from that of concentrates A and B. The optimum temperature for the almost-complete removal of sulphur and carbon appeared to be 650°C whereas, even at 700°C, the removal of arsenic was only 75 per cent complete. The residual arsenic content, however, was still relatively low at 0,4 per cent since the initial arsenic content of this concentrate was low (1,77 per cent). The curve for mass loss showed little change at temperatures above 600°C.

The effect of the duration of roasting was examined in a series of roasts in which the time of roasting was varied while the temperature was kept constant. From the previous roasts, 600°C appeared to be the optimum temperature at 30 minutes' duration, and this temperature was chosen for the next set of tests.

Roasting times of 5, 10, 20, and 30 minutes were chosen, and the results for the three concentrates are presented in Fig. 3.

For concentrate A, the mass loss and the removal of sulphur and arsenic reached high values after only 10 minutes of roasting whereas, after 10 to 30 minutes, these values increased only slightly. The rate of removal of carbon was lower and, after 30 minutes, a loss of 87 per cent was achieved.

For concentrate B, as with concentrate A, the loss in mass and the removal of sulphur and arsenic were fairly rapid and, after 10 minutes, near-maximum losses were attained. The removal of carbon was again slow, and only about 60 per cent was removed after 30 minutes.

For concentrate C, the removal of sulphur and carbon was rapid during the first 10 minutes, and increased only slightly after that. Again, this concentrate showed a lower rate of arsenic removal and, after 30 minutes, 77 per cent had been removed. The maximum loss in mass was attained after 10 minutes.

#### Larger-scale Roasts and Cyanidation

Larger-scale roasts were conducted in which 75 g samples of each concentrate were calcined in a large furnace at 600, 650, 700, and 750°C, with and without rabbling, for durations of 20 minutes, and the losses in mass were recorded.

The results are shown in Fig. 4, from which it is apparent that rabbling significantly increased the loss in

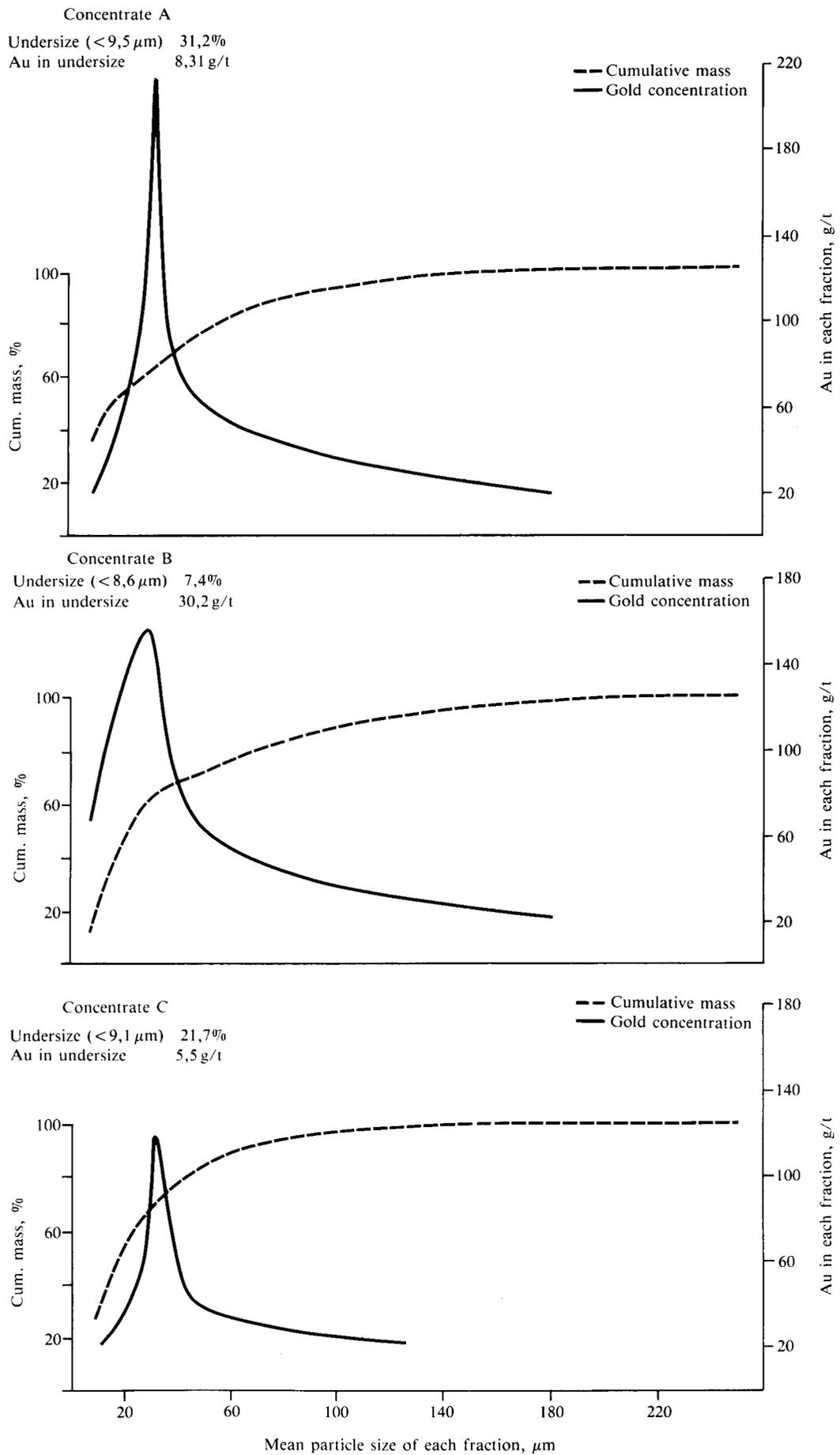
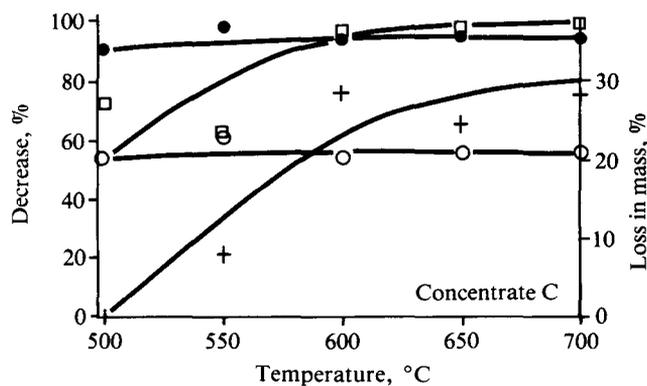
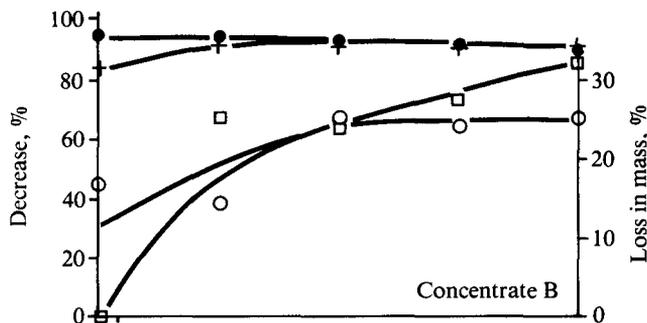
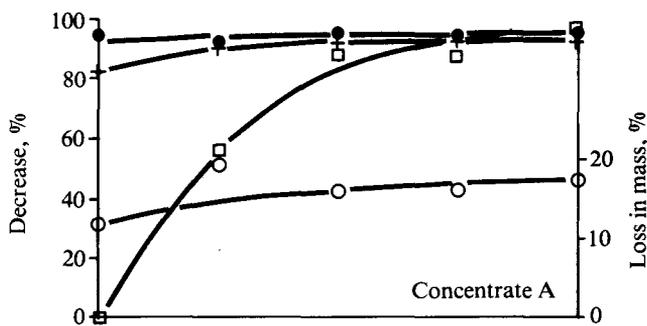
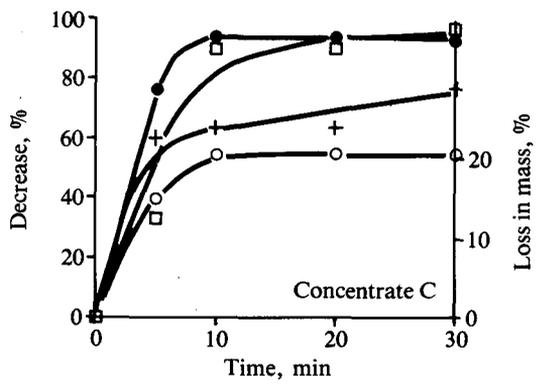
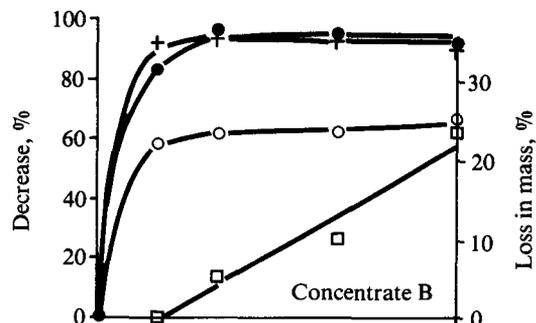
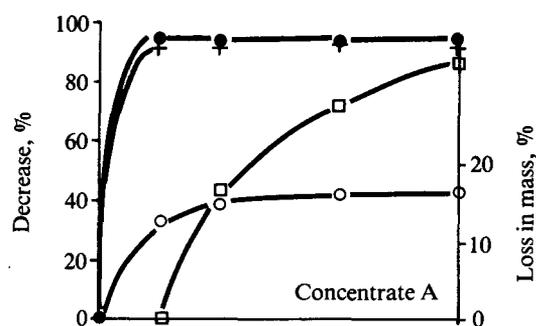


Fig. 1—Particle-size distributions and gold values for the concentrates



● Sulphur      □ Carbon  
+ Arsenic      ○ Sample mass

Fig. 2—Removal of sulphur, arsenic, and carbon during roasting of the concentrates for 30 minutes at different temperatures



● Sulphur      □ Carbon  
+ Arsenic      ○ Sample mass

Fig. 3—Effect of time on the loss in mass and the removal of sulphur, arsenic, and carbon during roasting at 600°C

mass for concentrate C, while having little effect on the loss in mass for concentrates A and B. Observation of the calcines during roasting tended to support this conclusion.

The concentrates had been spread uniformly within the container to a depth of 2 to 3 mm. Before being roasted, concentrates A and B were a dark silver-grey in colour, while concentrate C was darker (owing, most probably, to the different ratios of arsenopyrite to pyrite). Upon being roasted, concentrates A and B tended to become a uniform red-brown colour throughout. However, concentrate C, when not rabbled, was red-brown on the surface, but retained the dark unroasted colour, even at shallow depths.

The reason for this difference in roasting behaviour could be attributed to the mineralogical difference, i.e.

the high pyrite content of concentrate C.

A comparison of the losses in mass incurred by the larger samples (Fig. 4) with those from the tests on the smaller samples (Fig. 2) indicates little difference, suggesting that roasts conducted under identical conditions in the two furnaces would produce similar results.

It was argued that, since sulphur, arsenic, and carbon are generally related to refractoriness in gold ores, the removal of these impurities would eliminate, or at least reduce, the refractoriness of the concentrates during cyanidation. The preliminary roasts, the results of which are illustrated in Figs. 2 and 3, had indicated that temperatures from about 600 to 700°C with roasting times from 10 to 30 minutes were necessary for the almost-complete removal of sulphur, arsenic, and carbon from the three concentrates.

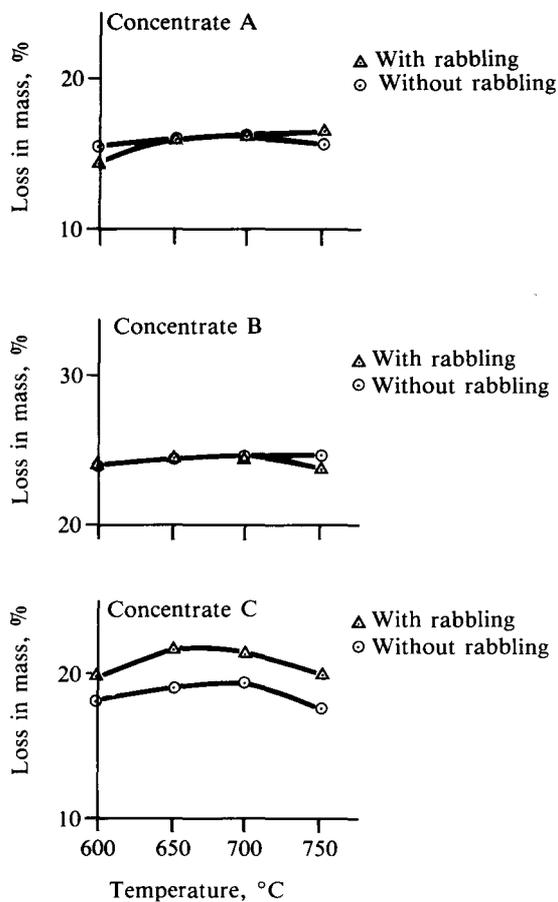


Fig. 4—Effect of rabbling on the loss in mass during roasting for 20 minutes at different temperatures

Larger quantities of the concentrates (75 g samples) were therefore roasted at temperatures of 600, 650, 700, and 750°C. At each temperature, the concentrate was roasted for 10 and 20 minutes without rabbling, and for 20 and 30 minutes with rabbling. The gold extractions that resulted from the cyanidation of these calcines are presented in Fig. 5.

Numerous factors affected the cyanidation stage and, since there were variations within and between the calcines, it was difficult to ensure consistent conditions from one cyanidation test to another. Although there are anomalies in these results, some trends were evident.

The gold extraction from concentrate A when roasted for 10 minutes was relatively low for the four temperatures investigated, while that after a roasting time of 20 minutes was higher. The differences between the gold extractions after 20-minute roasts without rabbling, and after 30-minute roasts with rabbling, were insignificant. Rabbling appeared to cause only a marginal improvement in gold extraction, and a maximum gold extraction of about 87 to 88 per cent was achieved by roasting with rabbling for 20 minutes at temperatures of 650 and 700°C.

Similar trends were displayed by the calcines from concentrate B. The roast conducted at 650°C for 20 minutes with rabbling gave a gold extraction of 86 per cent.

For concentrate C, there was a pronounced difference between the gold extractions from the rabbled and non-rabbled samples, particularly in the 20-minute roasts.

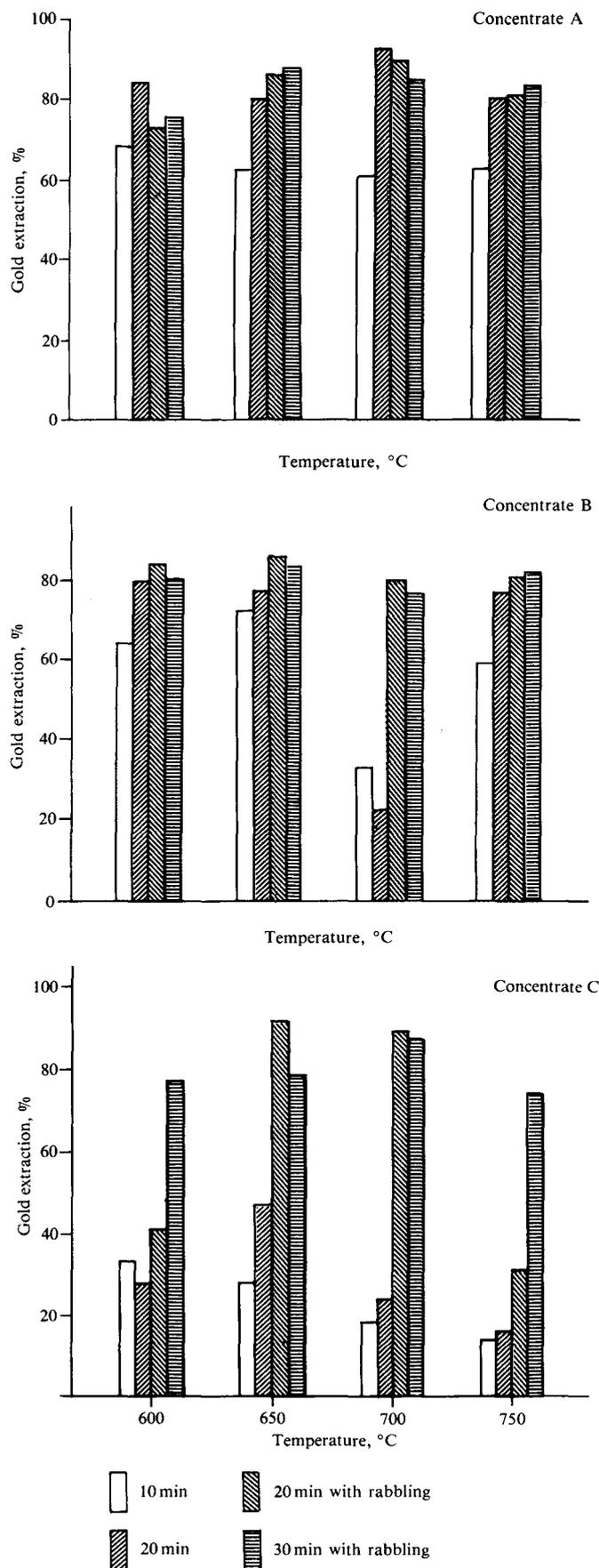


Fig. 5—Gold extraction from the concentrates after roasting under different conditions

When these results are considered together with those illustrated in Fig. 4, where the loss in mass is shown to be markedly affected by rabbling, it can be concluded that the poor extraction of gold from the non-rabbed samples may be the result of poor roasting conditions within the sample. Better contact between gas and solid during roasting produced a calcine more amenable to cyanidation. Again, roasting for 10 minutes was insufficient to give reasonably high gold extractions. The maximum gold extractions of between 90 and 94 per cent were obtained from this concentrate when it was roasted at temperatures of 650 and 700°C for 20 minutes with rabbling. A decrease in gold extraction occurred when roasting was conducted at 750°C, but there is some uncertainty about the significance of this decrease.

In a determination of the effect of washing the calcines prior to cyanidation, some duplicate experiments were conducted in which concentrates A, B, and C were roasted at 650°C for 20 minutes with rabbling. The resulting calcines were cooled in the usual manner, washed with water, and then filtered, dried, and cyanided.

The results, which are presented in Table III, show that a significant loss in mass was apparent after the calcines had been washed. The wash solutions were analysed semi-quantitatively, and were found to contain essentially magnesium, calcium, and sulphate ions, with traces of aluminium, iron, manganese, and zinc. The gold extraction was relatively unaffected by the intermediate washing step.

TABLE III  
GOLD EXTRACTION AFTER THE CALCINES HAD BEEN WASHED

Concentrate	Loss in mass after roasting %	Loss in mass after roasting, washing, and drying %	Gold extraction %
A	15,9	18,4	86,8
B	24,2	25,8	88,0
C	21,0	23,6	85,8

Samples of the concentrates were also leached directly in an attempt to determine the gold that could be extracted without any previous treatment of the concentrate. Direct leaching was conducted in two ways. Firstly, the normal cyanidation procedure, i.e. leaching, filtration, and analysis, was used, the cyanidation leach being performed in duplicate. Secondly, a carbon-in-leach (CIL) procedure was used in which the concentrate was leached in the presence of activated carbon. After the leach, the coarse activated carbon was screened from the residue, which was then analysed for any undissolved gold. The results of these tests are presented in Table IV.

The following observations were made from these results.

- There is a good agreement between the duplicate tests performed on concentrates A and B by normal cyanidation.
- For concentrates A and B, the gold extractions obtained by normal cyanidation and by CIL were very similar.

TABLE IV  
COMPARISON OF GOLD EXTRACTIONS WITHOUT ROASTING

Concentrate	Gold extracted, %	
	By normal cyanidation	By CIL
A	30,9 30,8	32,8
B	16,6 16,7	16,2
C	24,5 13,2	43,8

- Concentrate C showed a marked difference between the results of the duplicate tests on normal cyanidation, and a substantial increase in gold extraction from CIL.

The results relate to the mineralogical constitution of the concentrates, particularly to the association of the gold within each concentrate. The low overall extractions confirm that a large proportion of the gold was refractory, owing either to passivation or to the fact that a large proportion of the gold was locked within sulphide particles and was inaccessible to the cyanide.

Concentrate B, which had a high arsenopyrite and a low pyrite content, gave the lowest gold recovery. Since the gold had been observed to be intimately associated with arsenopyrite, and the gold was coarser and more loosely bound when associated with pyrite, this result would be expected.

The gold extraction was higher for concentrate A than for concentrate B. This would be predicted from the lower ratio of arsenopyrite to pyrite in concentrate A.

It may be noted that concentrate C contained the highest proportion of 'organic' or graphitic carbon. During normal cyanidation, gold tends to be adsorbed onto this carbon, and is consequently lost to the residue. This phenomenon, known as preg-robbing, is less likely to occur in CIL, since the gold is adsorbed preferentially onto the activated carbon. This explains why the recoveries from this concentrate were lower for normal cyanidation (24,5 and 13,2 per cent) than for CIL (43,8 per cent).

The relatively coarse nature of the gold associated with the pyrite could possibly lead to variations in gold content between portions of the same concentrate and, together with the possible variability in the preg-robbing properties of the graphite in concentrate C, may account for the difference in values obtained in the duplicate tests on concentrate C by normal cyanidation.

#### SEM Investigations

Samples of the concentrates, calcines, and leached calcines were examined with the SEM. The photomicrographs in Figs. 6 to 22 illustrate some of the observations made in a study on the behaviour of gold during roasting and cyanidation.

Several gold grains approximately 20 µm in diameter were observed in each concentrate. Many arsenopyrite grains were easily recognized from the rhombic sections and, although there were free grains of arsenopyrite, many were present as composites (Fig. 6).

Apart from the few coarser particles of gold that were observed within the concentrates, varying proportions of

gold were detected by the SEM within the arsenopyrite, but these could not be resolved as individual gold particles. It is possible that the gold existed as fine particles dispersed throughout the grains (particles smaller than the resolution size of the microscope) or in solid solution. Further work is needed to establish the mode of occurrence of this gold.

The number and variability of the minerals in the concentrates, together with their diverse manner of decomposition when roasted, led to calcines composed of a wide variety of mineral products. The rhombic cross-section typical of arsenopyrite persisted after roasting. Fig. 7 illustrates such a particle. These calcined grains of arsenopyrite were predominantly enclosed by a rim of denser, less porous material. Arsenopyrite contained within composite particles was still capable of undergoing decomposition (Fig. 8).

Although the gold in the arsenopyrite could not be resolved as individual particles, many fine particles of gold (approximately  $0.2\ \mu\text{m}$  in diameter) were observed within the rim material and in the porous interior of the calcined products (Figs. 7, 9, 10). It is possible that this gold became visible after roasting as a result of the diffusion and growth of gold particles during the decomposition of the arsenopyrite. The sulphide minerals that were present in the concentrates had decomposed to form porous calcines composed mostly of hematite. Although many of the sulphide particles formed an outer rim during decomposition (Figs. 7, 8, 10), some particles had no rims (Fig. 11).

Fig. 12 illustrates the occurrence of a relatively large grain of gold, approximately  $20\ \mu\text{m}$  in diameter, which is associated with some decomposed sulphide and decomposed carbonate gangue. In the enlarged view of this section (Fig. 13), the typical porosity of the hematite calcine products can be seen. This particle originated from concentrate A, which had been roasted at  $650^\circ\text{C}$  for 30 minutes.

It has been widely reported that pyrite and arsenopyrite both proceed via a pyrrhotite stage during roasting to magnetite and finally to hematite<sup>14,18</sup>, and in the present investigation pyrrhotite was observed in samples that had been incompletely roasted. Fig. 14 is a photomicrograph of an incompletely roasted pyrite particle from concentrate C, which had been roasted at  $600^\circ\text{C}$  for 5 minutes. At that stage of roasting, porous pyrrhotite had developed through to the outer surface while a central core of unreacted pyrite remained. The photograph shows that the porosity resulted from a series of channels radiating from the unreacted core to the outer surface. No rim formation was evident.

Compared with the particle illustrated in Fig. 14, which was from the same calcine sample, the particle shown in Fig. 15 is in a more advanced state of oxidation. Originally consisting of pyrite, the particle was found to be composed of pyrrhotite, and the porosity had increased and had become less directional. A grain of gold approximately  $10\ \mu\text{m}$  in diameter was also present within this grain.

Fig. 16 is an enlarged view of another particle from the same calcine. This particle had been oxidized to a greater degree than those in Figs. 14 and 15, but oxidation was still incomplete. The material had the typical porous appearance of hematitic calcine, although it con-

sisted predominantly of a network of pyrrhotite with some iron oxide (probably magnetite) around the pores. A comparison of these three micrographs (Figs. 14 to 16) appears to indicate that the porosity of the particle in Fig. 15 is at an intermediate stage between that in Fig. 14 and that in Fig. 16. No rims formed with this type of transformation and, from the observations made on the calcine particles, it appeared that rim formation could be restricted to particles that had previously consisted of arsenopyrite, and could therefore be related to the decomposition mechanism of arsenopyrite. Similar micrographs on the decomposition of pyrite have been presented by Monteiro<sup>19</sup>.

Numerous SEM observations on the calcined material after cyanidation revealed a number of important features. No coarse gold (i.e. with a particle diameter greater than  $10\ \mu\text{m}$ ) was observed. However, much finer gold was present, particularly in calcined arsenopyrite grains, as shown in Figs. 17 to 22.

Much of this fine gold, which was not leached by the cyanide, appeared to be encapsulated within the pore walls of the calcine, as shown in Figs. 17 to 20. The particles in which this gold was observed consisted of arsenopyrite before roasting, and were samples from concentrate B (Figs. 17, 18), concentrate C (Fig. 19), and concentrate A (Fig. 20). These calcines had been roasted at  $650^\circ\text{C}$  for 30 minutes.

It would be difficult to estimate the total quantity of this gold component in the cyanided residues. However, it does represent refractory gold, since it is enclosed by the iron oxide product, and contacting with cyanide solution would be difficult.

The gold particle in Fig. 21 was located within a cavity and, since the sample had been sectioned, it is possible that this particle had been isolated from the cyanide solution. Encapsulation of this type would also contribute towards the refractory gold in the residue.

Within a number of composite particles (such as that shown in Fig. 22, in which a surface of the porous calcined sulphide material was exposed and another surface was covered by gangue minerals), a larger number of the fine gold particles were located in the decomposed sulphide material adjacent to the gangue than near the exposed surface. This suggests that the penetration of the cyanide through the porous hematitic material may be limited, and may be prevented particularly by certain gangue minerals. Gold undissolved by this means would represent another component of the overall refractory fraction.

#### Surface Area and Pore Size

Calcines obtained from the roasts conducted at  $650^\circ\text{C}$  for 30 minutes were subjected to surface-area measurement by both the B.E.T. and the mercury-porosimeter methods. The results are shown in Table V.

For each sample, the total surface area of the particles in the calcine was measured by the B.E.T. gas-adsorption method, whereas only the total volume of the macropores present (i.e. the pores with a diameter greater than 3 nm) was measured by the mercury-intrusion method. The surface area of the macropores was determined from the intruded volumes on the assumption that the pores were

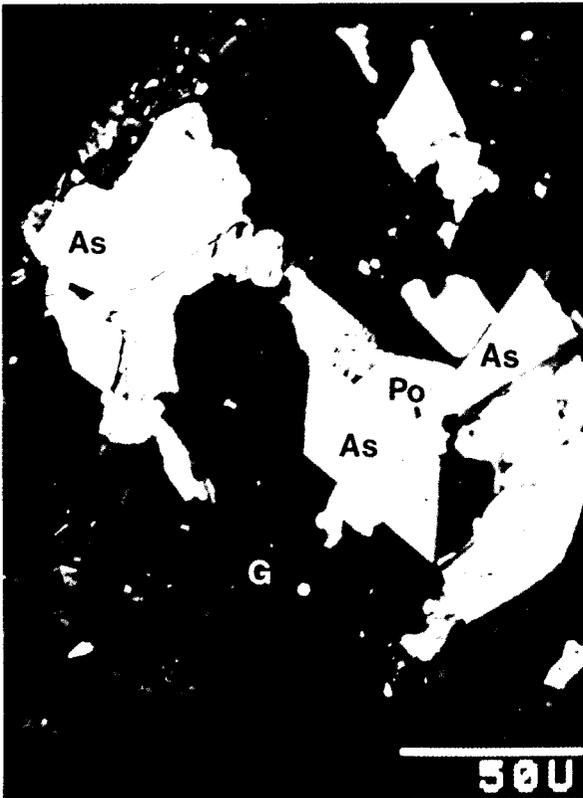


Fig. 6—Composite grain in unroasted concentrate A, with typical rhombic sections of arsenopyrite (As) associated with pyrrhotite (Po) in gangue (G)



Fig. 8—Composite particle with calcined grains of arsenopyrite, now hematite (He), in gangue matrix (G) from concentrate A roasted at 650°C for 30 minutes



Fig. 7—Typical calcined arsenopyrite grain, now consisting of hematite (He) with small grains of gold (Au) present. Most calcined arsenopyrite grains exhibited the rim formation evident here. This particle, from a sample of concentrate A, was roasted at 650°C for 30 minutes

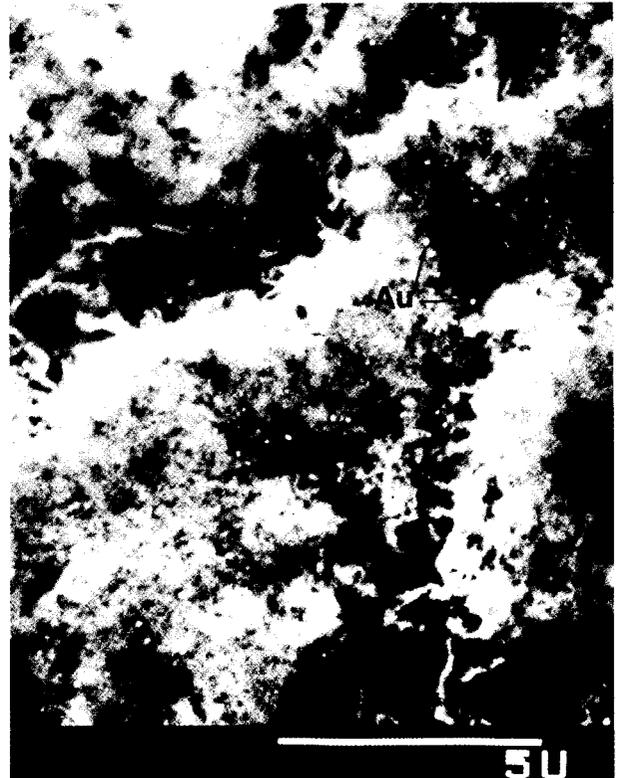


Fig. 9—An enlarged view of a calcined arsenopyrite grain exhibiting numerous small gold particles, which appear as bright specks. The grain originated from concentrate B, which was roasted at 600°C for 30 minutes

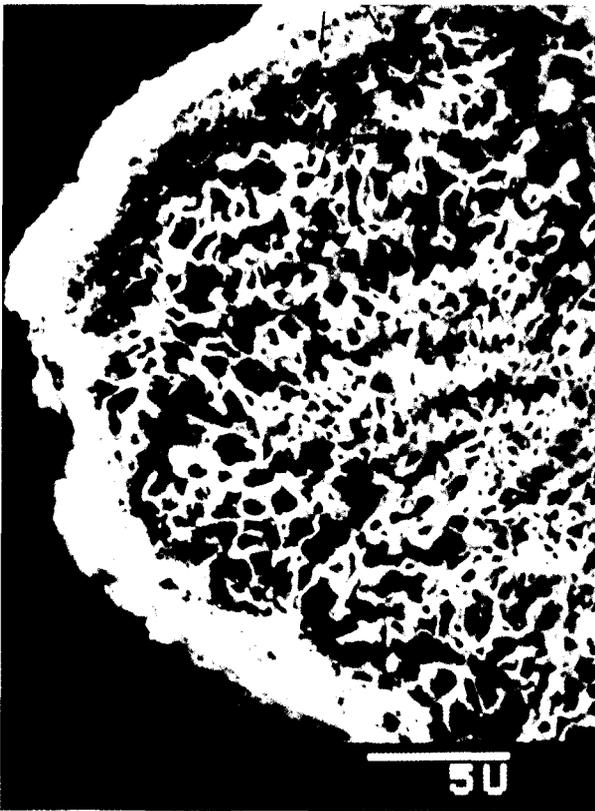


Fig. 10—Particle from calcined concentrate A roasted at 550°C for 30 minutes. This particle displayed a rim and fine, evenly distributed gold (Au)



Fig. 12—A relatively large grain of gold (Au) of approximately 20  $\mu\text{m}$  diameter within a composite particle, which is made up of decomposed sulphide and some decomposed carbonate gangue. The calcine was produced by the roasting of concentrate A at 650°C for 30 minutes



Fig. 11—Sulphide grain showing porous hematite (He) without rim formation from calcined concentrate A roasted at 650°C for 30 minutes

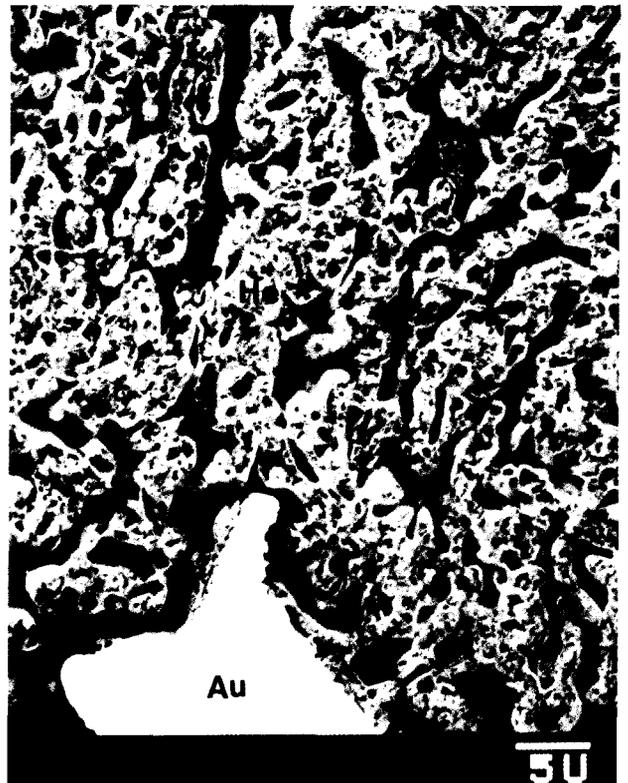


Fig. 13—An enlarged section of Fig. 12, showing the porosity of the hematite (He)



Fig. 14—Decomposing pyrite grain, originally from concentrate C roasted at 600°C for 5 minutes. Porous pyrrhotite (Po) is developing with channels through to the outer surface. No rim has formed



Fig. 16—Partly decomposed pyrite grain from the calcine sample shown in Fig. 14

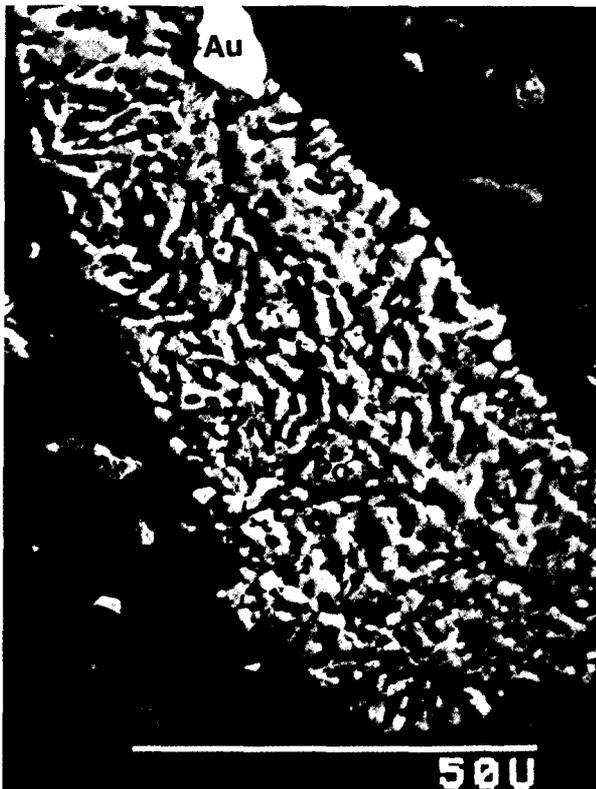


Fig. 15—A photomicrograph of the calcine sample in Fig. 14 showing a gold grain (Au) approximately 10 μm in diameter located in a porous grain of pyrrhotite (Po) altered from pyrite

TABLE V  
SURFACE-AREA MEASUREMENT BY DIFFERENT METHODS

Sample	Surface area, m <sup>2</sup> /g	
	B.E.T.	Porosimeter
Calcine A	2,93	3,48
Calcine B	2,24	2,82
Calcine C	2,74	3,09

cylindrical, and it is these calculated surface areas that are shown in Table V.

The porosimeter measurements did not show the presence of any pores less than 10 nm in diameter in any of the samples. For all three samples, larger measurements of surface area were obtained by the porosimeter method than by the B.E.T. method, although the relative surface areas of the three samples were the same for each method (i.e. the lowest surface area was obtained on calcine B, and the highest on calcine A). These relative values do not reflect any correlation with the mineralogical composition, particularly with the pyrite-to-arsenopyrite ratios. However, they do bear some relationship to the particle-size distribution in each original concentrate. The percentage undersize by mass in each concentrate had been recorded previously, and was listed as 31,2 for concentrate A, 7,5 for concentrate B, and 21,7 for concentrate C. These are of the same relative order as the measurements of surface area, which may suggest that

Figs. 17 to 20—These photomicrographs of cyanided calcine residues reveal a refractory gold component. The calcines were those obtained by the roasting of concentrate B (Figs. 17, 18), concentrate C (Fig. 19), and concentrate A (Fig. 20) at 650 °C for 30 minutes. The gold particles, visible as bright specks, were contained within the pore walls of the hematite (He).

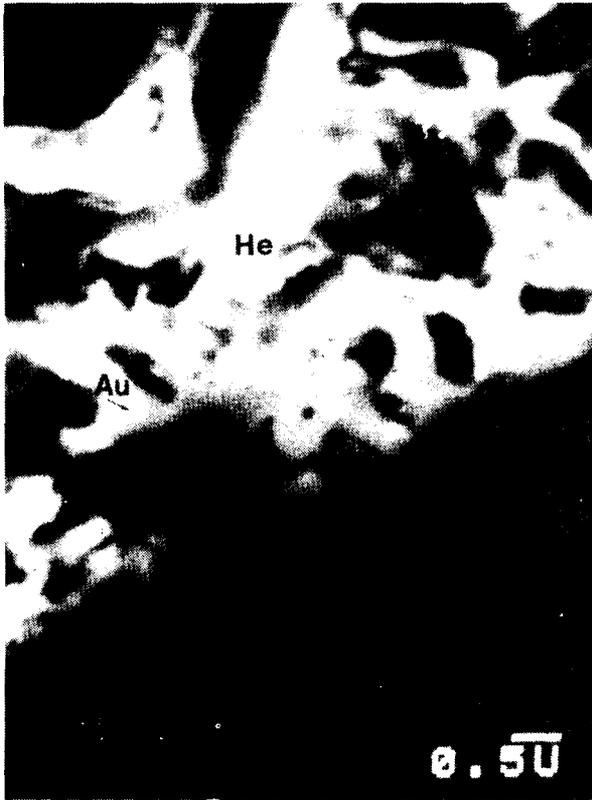


Fig. 17

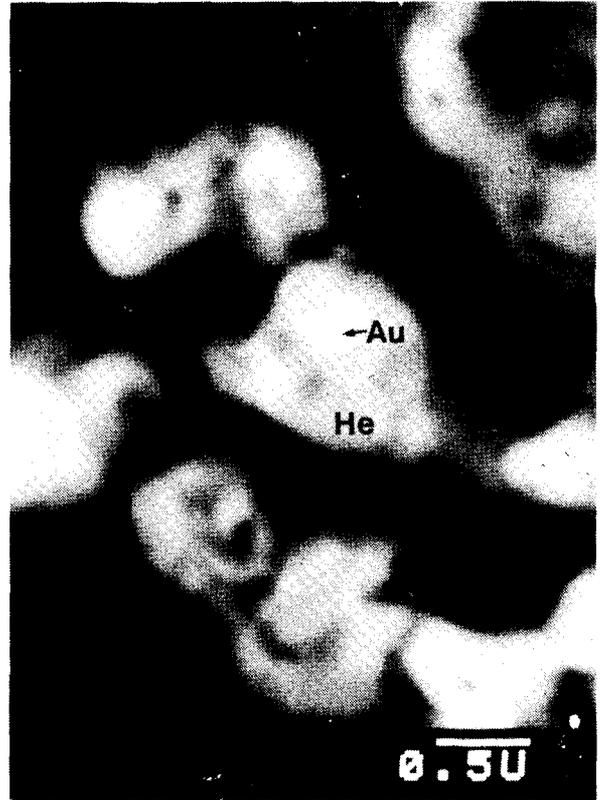


Fig. 19

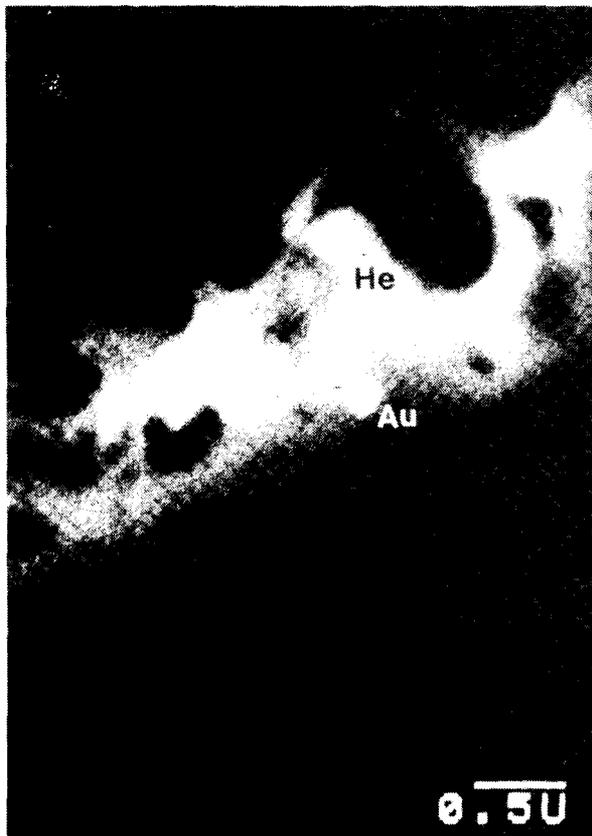


Fig. 18

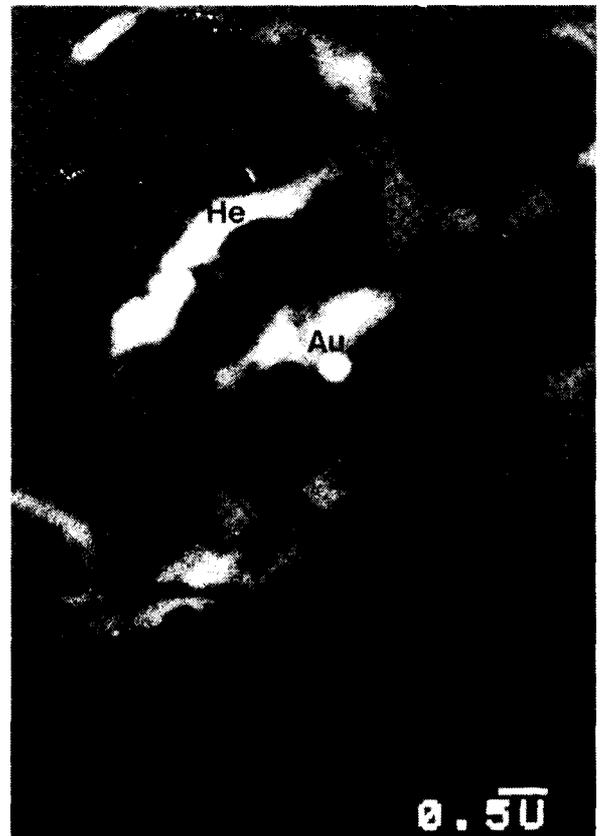


Fig. 20

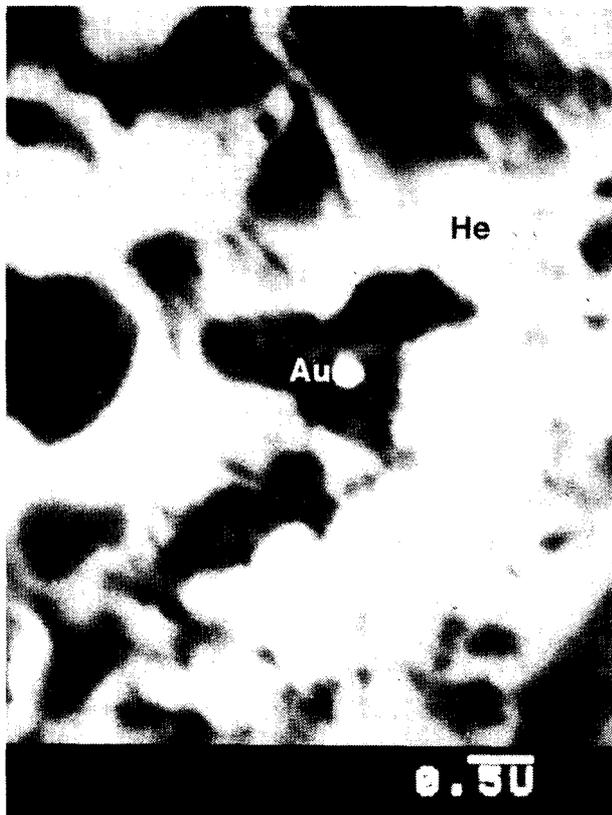


Fig. 21—Calcine obtained by the roasting of concentrate B at 650°C for 30 minutes, showing a gold particle (Au) within a cavity

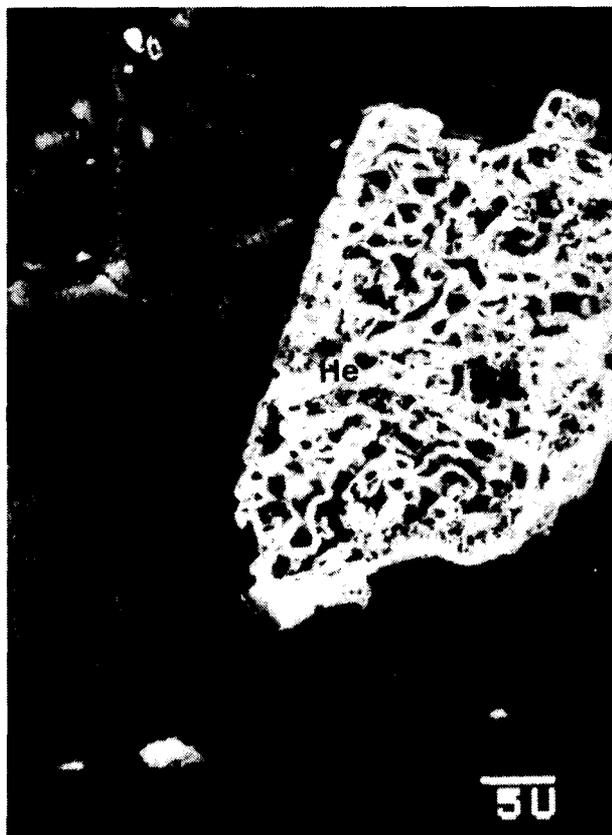


Fig. 22—Composite particle, originally from concentrate A, which had been roasted at 650°C for 30 minutes and then cyanided

the surface area of the calcines were not influenced to any large extent by the pyrite-arsenopyrite composition but by the initial particle-size distribution of the concentrate.

The fact that the surface areas measured by porosimeter were greater than those obtained by the B.E.T. method indicates that only macropores were present, and that the pores may not be truly cylindrical but may have a somewhat spherical shape.

The absence of any micropores within the calcines further supports the proposal that the fine gold observed within the cyanided residue was encapsulated within the iron oxide, which would have made it difficult for the cyanide solution to reach this gold.

### Conclusions

There appeared to be two major associations of gold in the concentrates: as fine particles distributed within arsenopyrite, and as a coarser form associated mainly with pyrite. The relative proportion of these two associations varied with the ratio of arsenopyrite to pyrite.

Direct cyanidation of the three concentrates yielded relatively low extractions of gold: 30,8, 16,6, and 18,8 per cent for concentrates A, B, and C respectively. The low value for concentrate B reflects the high proportion of gold locked within the arsenopyrite grains. Carbon-leach tests indicated a marked increase in gold extraction for concentrate C (increased to 43,8 per cent extraction), while the results for concentrates A and B were similar to those obtained by normal cyanidation. This marked increase for concentrate C suggests that the low extraction obtained by direct cyanidation can be partly attributed to the adsorption of soluble gold by the graphite in this concentrate. This was to be expected, since concentrate C contained the highest percentage of graphite (4,2 per cent, compared with 1,3 and 0,6 per cent for A and B respectively).

For the muffle-furnace roasting (for 20 minutes with rabbling at temperatures of 650 and 700°C) of the three concentrates followed by cyanidation of the calcine, the maximum gold extractions obtained were 87 to 88 per cent for concentrate A, 86 per cent for concentrate B, and 90 to 94 per cent for concentrate C. The loss in mass shown by the calcine of concentrate C, unlike that of concentrate A or B, indicated a marked dependence on rabbling. This effect correlated with the gold extractions, having the greatest effect on the gold extraction from concentrate C. The calcines contained a water-soluble component composed largely of magnesium and calcium sulphates, and the removal of this component did not significantly alter the gold extraction from the calcines.

The gold that was detected in different arsenopyrite grains but could not be resolved as individual particles became visible after roasting as fine, distributed particles of approximately 0,2  $\mu\text{m}$  diameter. The number of these particles varied from one calcined grain to another, indicating a variation in gold concentration between the arsenopyrite grains.

After cyanidation of the calcines, no coarse particles of gold (i.e. greater than 10  $\mu\text{m}$  in diameter) were observed. Many fine particles of gold were still present in the calcined arsenopyrite grains, and these were thought to constitute the major refractory component in the

cyanided calcine.

The SEM study confirmed that most of the fine gold particles that remained after cyanidation were located in the calcined sulphide product: enclosed within the pore walls, sealed within the ends of pores, or located near gangue minerals in composite particles. These gangue minerals, because of their lower porosity, may have prevented the cyanide solution from penetrating the calcined sulphides.

Surface-area measurements indicated the absence of micropores. The penetration of the cyanide to the fine gold particles would therefore be limited. This would suggest that physical encapsulation of gold particles was the main reason for the refractory behaviour of the gold in the calcines.

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## Automation in mineral processing

The 6th Symposium on Automation in Mining, Mineral, and Metal Processing is to be held in Buenos Aires from 4th to 8th September, 1989. The Symposium is being sponsored by IFAC (the International Federation of Automatic Control), and is being organized by AADECA (the Asociación Argentina de Control Automático). The working language of the Symposium will be English.

The purpose of the Symposium is to present, analyse, and discuss the latest developments of automation in the mining, mineral, and metal processing industries, including land and off-shore oil prospecting and exploration. Papers are invited on automation in the following or related fields, which promise new contributions to automation application and development:

- Mining and Mineral Processing
- Metal Processing
- Ceramic Processing and Cements
- Measurement and Analysis
- Production Control and Management

- Systems and Equipment
- Control Theory
- Mechanical Automation
- Artificial Intelligence and Expert Systems.

Abstracts of 250 to 350 words should be sent to  
Prof. Ing. J.P. Weisz  
AADECA  
6th MMM - 1989  
Callao 220-1°-B  
Buenos Aires - 1022  
Rep. Argentina.

Abstracts should be submitted by 30th May, 1988. The other deadline dates are as follows:

Preliminary acceptance: 31st August, 1988.  
Full papers submission: 30th October, 1988.  
Final acceptance: 28th February, 1989.

Submission of definitive texts: 30th April, 1989.

Enquiries in connection with the Symposium should be directed to Ing. J. Paiuk at the above address. Telephone: (541) 45-6746 and 46-4487. Telex: 17349-CASBA-AR.