Scholars' Mine

Masters Theses   Student Theses and Dissertations

1960

Segregation method as possible process to beneficiate oxidized lead ores

Jose Molfino Tomasio

Recommended Citation

SEGREGATION METHOD AS POSSIBLE PROCESS

TO BENEFICIATE OXIDIZED LEAD ORES

BY

JOSE M. TOMASIO

A

THESIS

submitted to the faculty of the

SCHOOL OF MINES AND METALLURGY OF THE UNIVERSITY OF MISSOURI

in partial fulfillment of the work required for the

Degree of

MASTER OF SCIENCE IN METALLURGICAL ENGINEERING

Rolla, Missouri

1960

Approved by

A. Legsdin (advisor)   _E. D. Fisher_

A. W. Schlechten   _J. C. Armstrong_

ABSTRACT

In this investigation it has been demonstrated that the Segregation Process has merit for treating oxidized lead ores.

The process comprises heating the crushed ore with salt and coal at about 750° C. to produce metallic lead attached to the carbon particles, which then is recovered by conventional flotation methods.

The quantities of sodium chloride and coal required for the best segregation of lead obtained in this work was 1.0 percent of coal and 1.0 percent of salt by weight.

Good segregation was obtained by using salt and coal as fine as 65 or 48 mesh. Segregation varied with the roasting temperature and time. At least 60 minutes was required for good segregation at 750° C.

About 3 to 4 percent moisture was required for segregation at 750° C.

Some valuable metals like silver, bismuth, precipitate along with the lead on the carbon particles. It is concluded that the process appears to have considerable merit on siliceous ores that are not amenable to conventional processing methods.

AKNOWLEDGMENT

The author wishes to thank the Missouri School of Mines and the U. S. Bureau of Mines Stations of Rolla, Missouri and Tucson, Arizona, for providing the opportunities and facilities for carrying out this investigation.

The author is grateful for the helpful advice and counsel of Professors A. W. Schlechten, and A. Legsdin of the Metallurgical Engineering Department, Professor E. D. Fisher of the Chemical Engineering Department and Professor G. C. Amstutz of the Geology Department of the Missouri School of Mines.

The author wishes to express his special gratitude to Mr. H. Kenworthy, Engineer in Charge of the Mineral Dressing Section and to Mr. W. Dressel, Engineer in Charge of the Minerals Research Unit of the Bureau of Mines in Rolla, to Mr. Carl Rampacek, Engineer in Charge of the Metallurgical Research Laboratory of the Bureau of Mines in Tucson, Arizona, and to Mr. J. Rowland, Director of the Rolla Station, for his approval and support of the program.

The samples on which testing was based were provided by the Cerro de Pasco Corporation of Peru. Their cooperation is acknowledged with sincere thanks.

Finally the author wishes to express his gratitude to the International Cooperation Administration of the United States, the Department of Education of the United States and Universidad Nacional de Ingenieria, Lima-Peru for sponsoring this investigation.

TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

INTRODUCTION

The essence of the Segregation Process is a reduction of the metal bearing constituent of an ore to the metallic state, prior to concentration. The reduced metal is then recovered by conventional flotation methods.

The process is of particular interest for low grade ores in which the metal is in an oxidized state and for which there is no satisfactory method of recovery of the metal at present.

Until now the segregation method has shown encouraging possibilities for oxidized copper ores. Attempts to apply this method to oxidized lead ores have given no satisfactory results.

The objective of this investigation was to study the possibilities of applying the segregation principles to recover lead from oxidized lead ores.

Results were produced which indicate that the segregation process can be used to recover the lead, at least, from some oxidized lead ores.

The lead-segregation process involves heating the ore with coal and a halide salt to about 750° C. to produce metallic lead, which then may be recovered by flotation with conventional lead sulfide collectors.

The investigation was performed chiefly to learn the effect

of applying the segregation process to complex Peruvian oxidized lead ores.

A typical representative sample was obtained from Cerro de Pasco Corporation (Matagente Ore Deposit, Cerro de Pasco, Peru).

During the last 40 years, the Corporation has spent much time and money in an attempt to develop a satisfactory process for recovering the values (lead, silver) from these ores.

The estimates of the ore bodies are as follows:

Matagente lead silver ore   5,000,000 tons

Pacos silver lead ores   19,000,000 tons

The average grade of a mixture of these materials was calculated as:

Silver	6.0 oz. per ton
Lead	3.25 %
Copper	0.20 %
Bismuth	0.05 %

It may be in order to point out that due to the type and intensive degree of locking of the cerussite, it was impossible to produce by conventional methods a concentrate of more than 60 to 70 percent recovery.

History of the Process

The Segregation Process, according to RAMPACEK (1959), was discovered in 1923 during experimental work in which oxide copper ores were being treated with coal to reduce the copper to the metallic state before leaching with an ammoniacal ammonium carbonate solution.

In tests of an ore from the Sagasca mine in Chile, it was observed that reduction of the crushed feed with coal at about 700^0 C. produced friable agglomerules consisting of fine flake copper, coal, and gangue instead of copper reduced in place, as had been experienced with other ores. The unusual behavior of the copper was traced to the presence of a small quantity of sodium chloride occurring in the ore. In subsequent experiments on oxidized ores, according to MOULDEN, J. C. (1928) and REY M. (1936), the presence of a halide, either occurring naturally or admixed with the ore, caused the copper to migrate from the ore particles and to segregate in the charge.

The process was developed further from 1923 to 1931, and two plants were constructed in Africa for treating oxidised copper ores. One plant was built in Southern Rhodesia by the Minerals Separation Co. and another at Katanga, in the Belgian Congo, by the Union Miniere du Haut Katanga.

The Southern Rhodesia plant had a capacity of 50 tons of ore in 24 hours. Heating and segregation were carried out in a seven-hearth, mechanically rabbled furnace. The first five hearths were

direct-fired to heat the ore to reaction temperature. Salt and coal were added to the hot ore, and the segregation was carried out on the sixth and seventh hearths. The temperature on the two segregation hearths was mantained at about 700° C. by the exothermic nature of the reaction and by indirect heating with waste gas from the upper hearths. The hot material subsequently was cooled in a rotary cooler, screened and then concentrated by flotation to recover the copper.

About 3,500 tons of ore assaying 5 percent copper were treated in the plant during a 4-month period. The recovery of copper was 87 percent.

The Katanga plant had a rated capacity of about 350 tons of ore in 24 hours; it was operated for about 4 months in 1931. A direct-fired rotary kiln was used to preheat the ore before segregation of the copper in a second rotary-reactor kiln. Cooling was accomplished in a rotary cooler attached to the reactor kiln. No data are available on operation of the plant.

The Southern Rhodesia and Katanga plants closed after short periods of operation, owing partly to mechanical operating difficulties and partly due to the low price of copper at that time.

II. DISCUSSION

1. Proposed segregation reactions for lead ores

No reactions have been published which would apply to segregation of lead. Possible reactions are being proposed here for lead segregation which are in part similar to those proposed by others for copper segregation.

According to RAMPACEK (1959), the segregation of copper from oxidized copper ores takes place in three stages. Using this proposed scheme as a guide, it was assumed that the segregation reactions for oxidized lead ores could occur as follows:

A. Decomposition of sodium chloride

According to RAMPACEK (1959), sodium chloride reacts with hydrous clay minerals, such as montmorillonite and kaolinite, to liberate hydrochloric acid by the following reaction:

$$4NaCl + Al_2O_3.2SiO_2.2H_2O = 4HCl + Na_4Al_2O_3.2SiO_3 \quad (1)$$

This reaction proceeds rapidly at 700° C. in the presence of traces of water vapor resulting from progressive dehydration of silicate minerals and from moisture in the coal or coke used. MAURICE REY (1936) has, also, indicated that hydrochloric acid is formed by reaction of sodium chloride with water vapor and silica, alumina or ferric oxide. The acid was found by analysis in small amounts in the furnace gas.

Tests at the U. S. Bureau of Mines Laboratories Tucson, Arizona, demonstrated that the yield of hydrochloric acid from quartz-salt or opal-salt mixtures was no greater than that obtained by heating the salt alone in the presence of moisture. Neither hydrogen chloride gas nor chlorine was formed when mixtures of salt and quartz or dehydrated siliceous minerals were heated to 750° C. in the absence of water vapor. Similar tests of kaolin-salt mixtures yielded appreciable hydrochloric acid, owing to the release of the water of hydration upon heating to 750° C. However, when the clay was completely dehydrated by precalcining at 1,000° C., no hydrochloric acid was formed upon subsequent heating with salt at 750° C.

B. Volatilization of lead chloride

According to LYON and RALSTON (1918), the chlorides of the metals volatilize at temperatures much below their boiling points. For example, the boiling point of lead chloride has been given as 861 to 954° C. and that of cuprous chloride as 954 to 1,032° C. and yet both of these metal chlorides may be completely volatilized at temperatures of 700 to 750° C in very practical periods of time.

Gold and silver chlorides dissociate below the temperatures given above (954 to 1,032° C.) into the metals and into chlorine. In a chloridizing atmosphere, however, gold and silver chlorides probably exist and are expelled with the other metal chlorides, or oxychlorides.

The hydrochloric acid liberated in reaction 1, attacks the lead oxide minerals to form volatile lead chloride, according to reaction 2:

$$PbO + 2HCl = PbCl_2 + H_2O \qquad (2)$$

C. Precipitation

The following reactions are expected to happen:

$$C + H_2O = CO + H_2 \qquad (3)$$

$$PbCl_2 + H_2 = Pb + 2HCl \qquad (4)$$

Reaction 3 is the so-called water-gas reaction. This reaction takes place when steam is decomposed in its passage over red hot coke. Reaction 5, seems to be the principal one for reduction of lead chloride to metal.

$$2PbCl_2 + C + 2H_2O = 2Pb + 4HCl + CO_2 \qquad (5)$$

The kinetics of the reactions are all important and it can be shown that there is competition between reduction in place, that is, reduction of solid PbO to metallic lead by CO and H_2 and, reduction from the gas phase $PbCl_2$ giving segregation.

It is therefore, necessary to slow down the rate of reduction in place and to increase the rate of the segregation reaction. To accomplish this, it is necessary to have a weakly reducing atmosphere comprised principally of carbon dioxide with small amount of CO and H_2. Under these conditions the volatile lead chlorides are reduced to metallic lead on the carbon particles.

It should be noted that in the reactor the conditions are only slightly reducing and the exit gas contains mostly CO_2 and H_2O and only a small amount of CO and H_2. Ferric oxide is largely reduced to

magnetic oxide but wustite (FeO) is not formed.

MAURICE REY considers the probability that the concentration of the metal on coal is due to the fact that the coal particles are the nuclei where the reducing gases CO and H_2 are produced in the furnace, according to reaction 3.

2. Factors affecting the process

A. Size of the ore

The ore was ground to minus 10 mesh, following the recommendations of the Minerals Separation patents for copper ores. No batch segregation tests were made to determine the effect of feed size on segregation of lead.

After the processing the ore was screened on a 65-mesh screen. The oversize contained a very small amount of lead compared to the original percentage. This migration of the lead from the coarse ore to the fine coal has given the process its name of segregation.

B. Preheating of the ore

Roasting in an oxidizing atmosphere is an essential step in the segregation of lead oxide ores from Cerro de Pasco (Matagente).

Time and temperature are of paramount importance. REY, states that with some copper ores, overheating of the ore to 850° to 900° C. prior to segregation has a harmful effect on recovery. Also with lead ores the matter seems to be important and measures have to be taken to

heat the ore very carefully without overheating.

A large number of tests were performed with lead ores without favorable results, until possible poisoning of the charge was suspected. A decrease in the activity of the coal in segregation has been observed from the days of the earliest studies of the application of the process to complex lead ores, and the causes of this decay in activity is now being assumed, by the author, to be due to poisoning of the coal.

Decay in activity is due to poisoning, as reported by GRIFFITH and MARSH (1957), if it is the result of small quantities of impurities present. Poisoning is usually caused by preferential adsorption of the poison, which is held to the active surfaces by strong bonds, and the adhesion is often irreversible.

It is presumed by the author, that the action of segregation of lead will not proceed once adsorption equilibrium is established with poisons.

Good segregation was achieved after roasting the oxidized material at 700° C. for about an hour. Higher temperatures result in volatilization of lead. After roasting, the lead carbonate has been converted to PbO, which is suitable for chloridizing roasting.

C. Salt

Several chlorides can be used as a source of chlorine, but the simplest and most economical is sodium chloride, which is easily decomposed under conditions in the laboratory furnace and thus speeds

up chloridizing.

It should be ground to 48 or 65 mesh and fed to the reactor mixed with coal. Intimate contact of the salt and ore particles is not obligatory for good segregation, as the chloridizing reaction occurs in the gaseous state. There was no benefit in using more than 1.0 percent of the chloride by weight on the ore tested.

According to GAUDIN (1957), apparently the chloride is reduced on contact with carbon (e.g. coke or coal), permitting the chlorine to repeat the transfer of the metal, many times if necessary.

D. Coal

The best results were obtained with 1.0 percent of coal ground to minus 48 mesh and heated in a helium atmosphere. Coals with high volatile matter contents are not suitable since they upset the process by giving too much reduction of the mineral in place.

E. The reaction period

With a temperature of 750° C., a retention time of 60 minutes in the revolving reactor seems ample and the reaction is complete.

3. Electron microscope studies

A sample of segregated copper from Tucson, Arizona, was studied under the electron microscope as a new approach to the understanding of the attachment of the metal to the carbon.

According to WYCKOFF (1949), the preparation of samples suitable for examination under the electron microscope is dominated by the extreme opacity of all matter to electrons. What is to be observed must be very thin and must be of such a character as not to be destroyed in the vacuum of the instrument.

Suspensions of segregated copper - some of them dispersed with ultrasonic waves - were examined after depositing them on a very thin membrane. A metallic grid having about 200 meshes per inch was used as a membrane support.

Membranes of collodion were quickly and easily made and were satisfactory for routine work at low and moderate magnifications.
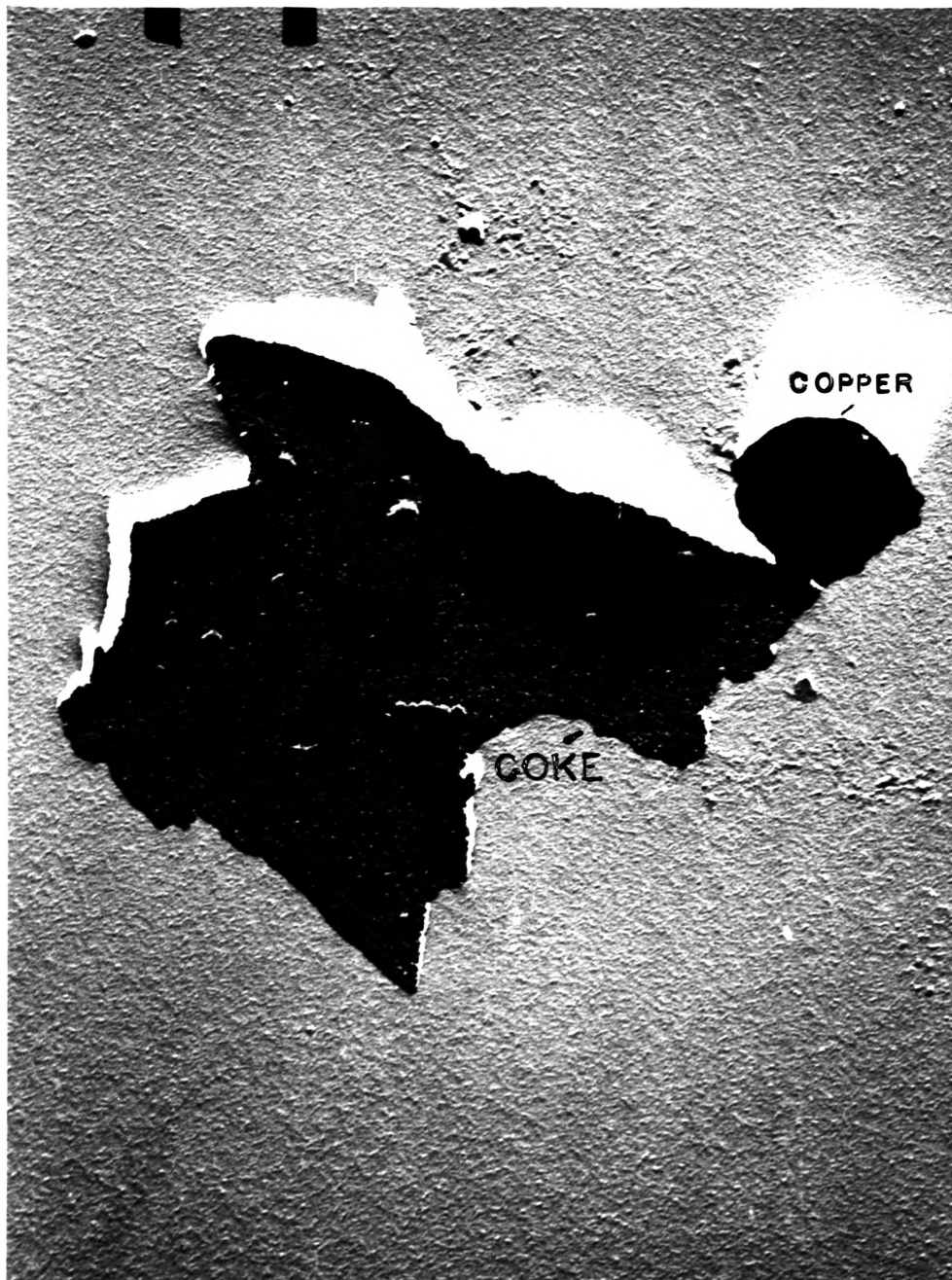
The appearance of minute detail can be enhanced by the deposition of heavy atoms on the surface of a preparation. If the deposit is made obliquely it will be of varying thickness over different parts, and the varying opacities that result will greatly enhance contrast in the image it produces. The technique developed to take advantage of this fact has been called shadow-casting.

Shadowing was made through a vacuum evaporation of an extremely thin layer of chromium. If a sufficiently good vacuum exists in the bell jar at the moment of this vaporization, the metal atoms will travel in straight lines in all directions from the filament and some of them will be deposited on the sample.

Figures 1, 2, 3, and 4 are electron micrographs at different

magnification showing very interesting features as to size, shape, and growth characteristic of particles.

Electron microscope studies of the results of copper segregation have been submited here, as a possible aid for the understanding of the deposition of lead in lead segregation.

Figure 1. Electron micrograph showing attachment
of a spherical particle of metallic Cu
to one of the corners of the coke.
Total magnification: 12,500
Dispersion: None
Shadowing : Chromium

Figure 2. Same as figure 1, but at higher
magnification.
Total magnification: 22,500

Figure 3. Electron micrograph showing copper
particles with typical dendritic or
skeletal shapes as a result of rapid
crystal growth
Total magnification: 4,375
Dispersion: None
Shadowing : None

Figure 4. Same as figure 3, but at higher
magnification.
Total magnification: 12,500

III. LABORATORY INVESTIGATIONS

Physical nature of the Cerro de Pasco ore (Matagente)

Introductory remarks

The Matagente ore deposit was formed by the oxidation of sulfides originally present in the lead-zinc veins. Leaching by, and movements of groundwater produced Pacos, spanish term for oxidized caps of ore bodies. This now represents a body of lead oxide ore about 1,600' long, 20 to 600' wide and about 150' deep. Below the Pacos, the grade of the lead and zinc is too low to justify its exploitation, at least not in bulk, since the primary ore is contained only in veins. In the Pacos, the lead minerals are cerussite, anglesite, and plumbo-jarosite, according to AMSTUTZ and WARD (1956).

The area of the Matagente ore deposit was the scene of the greatest activity in the days when this was a silver-producing camp, as reported by MILLER and SINGEWALD (1919), and by the C. de P. Staff (1950).

The lead oxide ore zone is at the same time a great mass of oxidized argentiferous material known as the pacos ores, the richest portion of which was worked first by means of open cuts and latter also by shallow underground mining. The huge caves left unsupported have since collapsed and increased the size of the open cuts, leaving a series of immense pits, or "tajos abiertos", as evidence of the great amount of material removed in those days.

The silver content of the Pacos material left behind varies

greatly, but the copper content is almost uniformly negligible. It is an excellent example of enrichment in silver and lead, as well as, leaching of copper in the zone of oxidation. The oxidized ores, which were Cerro de Pasco's richest material for over a century, are presently of little or no importance in the mining activity of the district. Interesting information on metallurgical processes applied in early times in this area is contained in the book of BARGALLO (1955).

At the beginning of the present research, work was carried out to furnish mineralogical data, fundamental to the understanding of the behavior of the different minerals present. Understanding of the mineralogy and physical relationships of the components of this complex ore was, fundamental to the consideration of the problems involved in its treatment. Much valuable information was gained from the following textbooks and reports on oxidized ore deposits: ANDERSON (1930), BLANCHARD and BOSWELL (1954), COOKE (1913), EDWARDS (1954), EMMONS (1917), JOHNSON (1959), KERR (1959).

Collecting of the above data has required isolation of individual minerals under the microscope for chemical analysis, microchemical tests of individual grains, microscopic examination of etching reactions on polished sections, petrographic identifications, detailed microscopic counts of a large number of sized and briquetted particles, and finally correlation of these counts with the chemical analyses throughout.

Mineralogical composition

The sample is an oxidized ore, consisting of cerussite with a small amount of galena in a gangue composed essentially of quartz,

clay minerals, chlorite, -limonite, and manganese oxides, with lesser amounts of barite, siderite,chrysocolla and malachite.

According to AMSTUTZ and WARD (1956) and DE LAS CASAS (1953), there is also present hydrocerussite, anglesite, pyromorphite, caledonite, plumbojaresite, smithsonite, hydrozincite, calamine, hemimorphite, etc. Whereas these authors stated that the cerussite is coated with a rythmic deposition of limonite, the author has found that some of the cerussite is also intimately associated with the quartz as fracture filling. Cerussite occasionally surrounds quartz grains.

Extensive study of particles under high magnification furnishes no evidence of the manner of occurrence of the silver content. Hence, it may be assumed that it occurs as a disseminated silver mineral too minute for microscopic detection or, as a solid-solution in the galena.

Another possible explanation, by plotting the silver content against Al_2O_3, PbO, SiO_2, and Fe, AMSTUTZ (1956) found that silver is symbathic with Al_2O_3. From this he concluded that silver is present as submicroscopic particles in the clay fraction.

Figures 5 and 6, show crystals of cerussite partly coated by limonite. This coating is considered by AMSTUTZ and WARD (1956) to be the major obstacle to a successful concentration by gravity or by sulfidization.

Figure 5. Particles of cerussite coated with various
thin layers of limonite (yellowish and brown-
is colors). The cerussite has a transparent
glassy to whitish appearance. Limonite specks
pepper over the free surface of the cerussite.
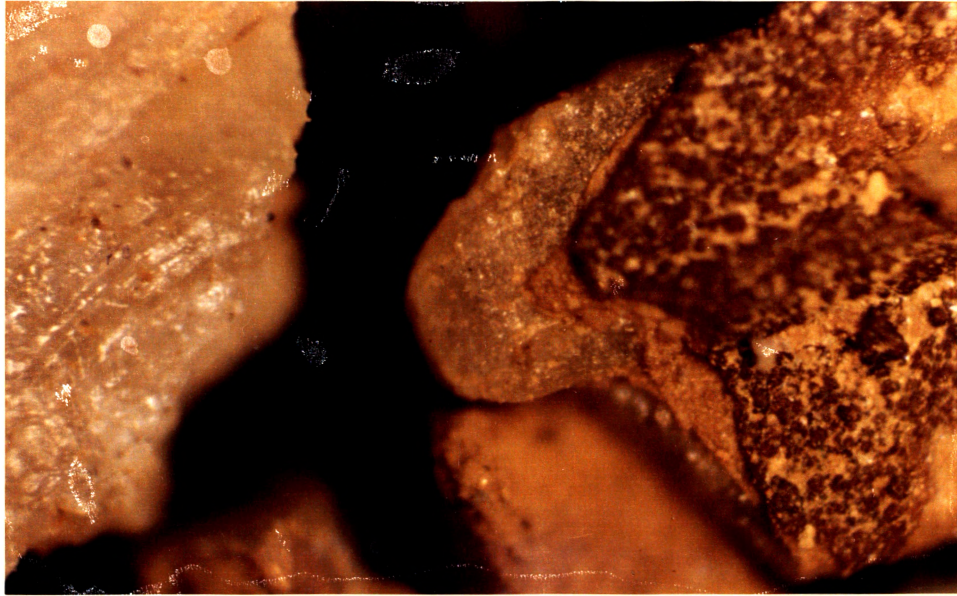Mesh size: 80 to 100

Figure 6. Same as figure 5

Chemical composition

Typical analyses of oxidized lead ores from Matagante, Cerro de Pasco:

Copper	.32 %
Zinc	3.60 %
Insoluble	35.00 %
Lead	3.00 %
Iron	25.70 %
Gold	.02 oz. per ton
Silver	6.00 oz. per ton

Batch - tests

Equipment

Segregation tests were made in small rotating retorts. Figure 7 is a view of the retort and of the auxiliary equipment. Each retort consisted of a cylindrical drum 9 inches long and 6-3/4 inches in diameter, inside dimensions. One end of the cylinder was cast as an integral part of the retort, and on the other end was a removable lid fastened to the cylinder with bolts. Hollow end trunnions, about 4 inches long and having 1-inch outside and ½-inch inside diameters, served as axes of rotation. The retorts were supported by trunnions on pillow blocks and were driven at 2 r.p.m. The units were heated externally by one gas-fired burner. During operation a thermocouple was inserted into one of the open trunnions, and the other trunnion served as a vent for the reaction gases.

Description of procedures

Tests on the dry-crushed ore, at first, were made following the same procedure used in copper segregation. These tests, however,
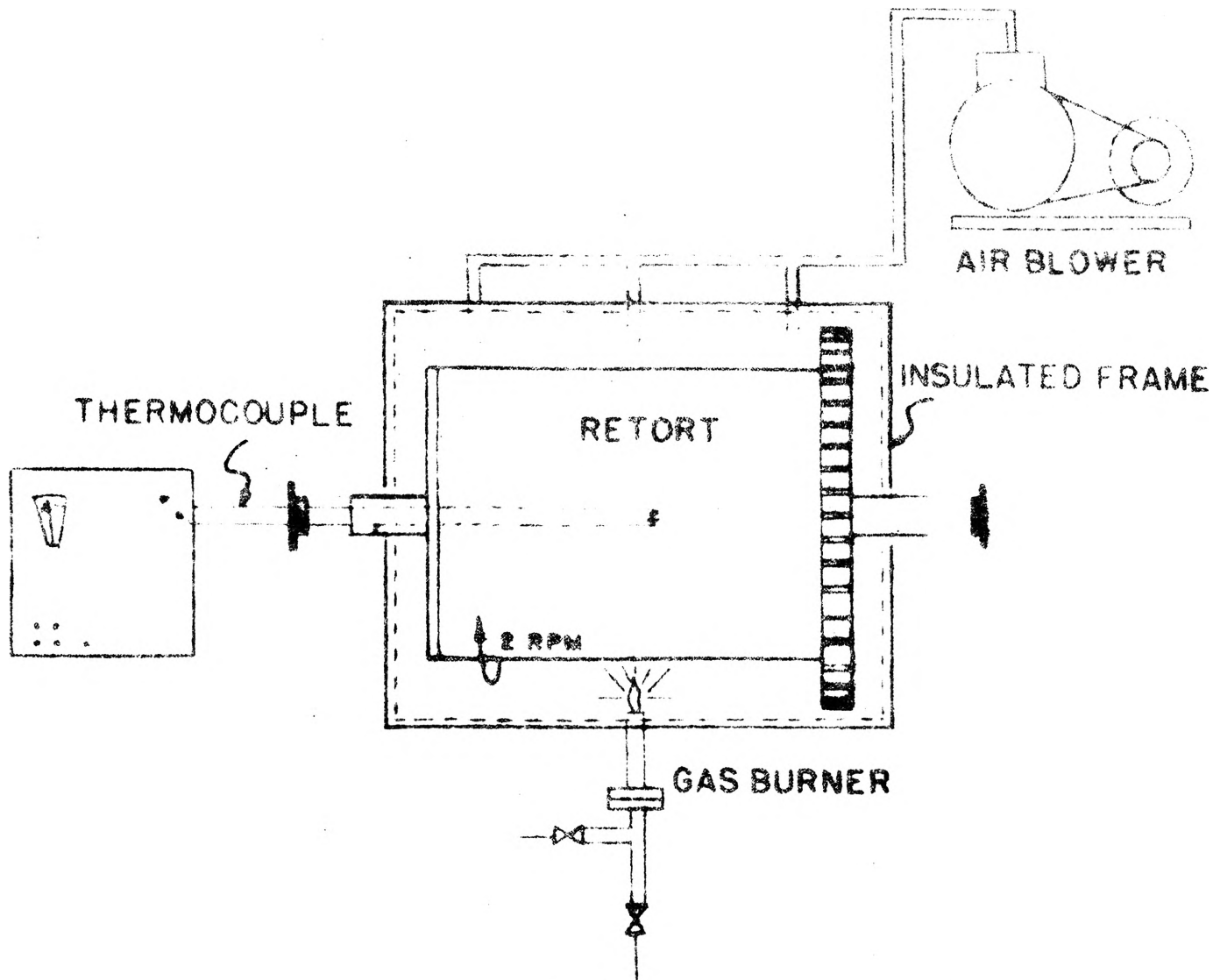
Figure 7. Roasting Retort — Not to scale

AIR BLOWER

THERMOCOUPLE

RETORT

INSULATED FRAME

2 RPM

GAS BURNER

gave no segregation of lead.

For the segregation of copper from oxidized copper ores, two procedures have given results. In one procedure the crushed, minus 10-mesh, ore is premixed cold with salt, coal or coke and a small amount of water and this charge is heated in a closed container at 750° C for one hour.

The second procedure, the two-stage method, differs from the first in that the crushed ore alone is first preheated in a retort to 750° C. Then, the desired quantity of salt and coal or coke are admixed to the hot ore and the mixture is now heated for one hour at 750° C to segregate the copper.

In both procedures the hot retort with the calcine is first rapidly cooled to 200° C. by blowing air on the retort. The calcine is then removed and is allowed to cool to room temperature.

The segregated copper is then recovered from the calcine by conventional copper flotation methods.

Some forty very similar, one and two-stage procedures were tried out on the Cerro de Pasco oxidized lead ore. All results were negative. No segregated lead could be observed under the binocular microscope.

The procedure was then modified by replacing the coal or coke by specially pre-treated bituminous coal. The coal was ground to

minus 48-mesh and then heated in helium atmosphere for 30 minutes
at a temperature of 300° C. This pre-treated coal was then used to
prepare the charge which was then handled as described above. The
results still were negative.

Experimental work on a synthetic mixture

Since initial tests with the oxidized Peruvian lead ore showed
no segregation, for this reason, additional charges were made of Syn-
thetic mixture.

A series of tests with a mixture of cerussite, silica, dolo-
mite, salt, coal and a small amount of water were carried out under
the conditions of the segregation procedure.

As in all segregation tests performed, the total weight was
250 grams. The composition of the charge used was as follows:

Silica	224.0 grs.
Cerussite	16.0 "
Dolomite	5.0 "
Salt	2.5 "
Coal	2.5 "

Microscopic study of samples roasted at temperatures as high
as 750° C., indicated the formation of lead glass, which was the main
reason for failure to obtain segregated lead. It was decided to work
with lower temperatures.

Tests at temperatures of 700° C., produced successfully segre-

gation. The surface of the coal grains carefully observed under the m microscope showed the presence of small metallic lead globules.

From the results of tests with synthetic mixture, it was demonstrated that lead segregation is experimentally possible. Therefore, additional tests were carried out using now the oxidized lead ore from Cerro de Pasco (Matagente) instead of the synthetic ore.

Experimental work on Cerro de Pasco ore (Matagente)

Test No. 46

A further modification in the segregation procedure was made. It consisted of roasting the minus 10-mesh ore in an oxidizing atmosphere at a temperature not higher than 700° C. for one hour. After this period of time, the mixture of -48 mesh helium pre-treated coal and salt was added and the operation continued for another hour, increasing the temperature from 700° to 750° C. In this case also segregated lead was obtained.

After cooling the retort with air on the outside, a preconcentration of the calcine was made by magnetic separation. A large amount of magnetic material was rejected by this means. The presence of magnetic material indicated that mild reducing conditions prevailed in the retort, as ferric oxide was largely reduced to magnetic oxide but wustite (FeO) was not formed.

By sink-float separation, the segregated lead was recovered from the non-magnetic material.

Microphotographs of different grains were taken to show the metallic lead coating on the surface of the coal particles.

Pellets as shown in figure 12 were also present. It is interesting to note the typical eutectic formation characteristic of alloys.

Spectrographic analysis of tests 46 and 47 segregated material showed lead as the major constituent. It also indicated that other constituents were present in minor amount (pages 34 and 35). The reason for this is not fully understood but is presumed by the author that, these metals also precipitate and are preferentially adsorbed during the reaction.

In test 47 and following tests, a standard flotation procedure was used to recover the lead from the segregated charges. The non-magnetic calcines were wet-ground to minus 100-mesh in a laboratory rod mill. The metallic lead then was floated, using potassium amyl xanthate as the collector and cresylic acid as the frother.

A concentrate product of 42.0 % lead was obtained with a recovery of 50.5 %. This low recovery is assumed to be due to loss of lead during reaction operation. Some of the lead chloride can escape from the retort since the thermocouple is not tightly fixed and also some losses of lead may occur in the large amount of magnetic material rejected. Some ten additional similar tests gave approximately the same lead segregation results determined by microscopic examination. Additional research is necessary to establish better conditions for an increase in recovery.
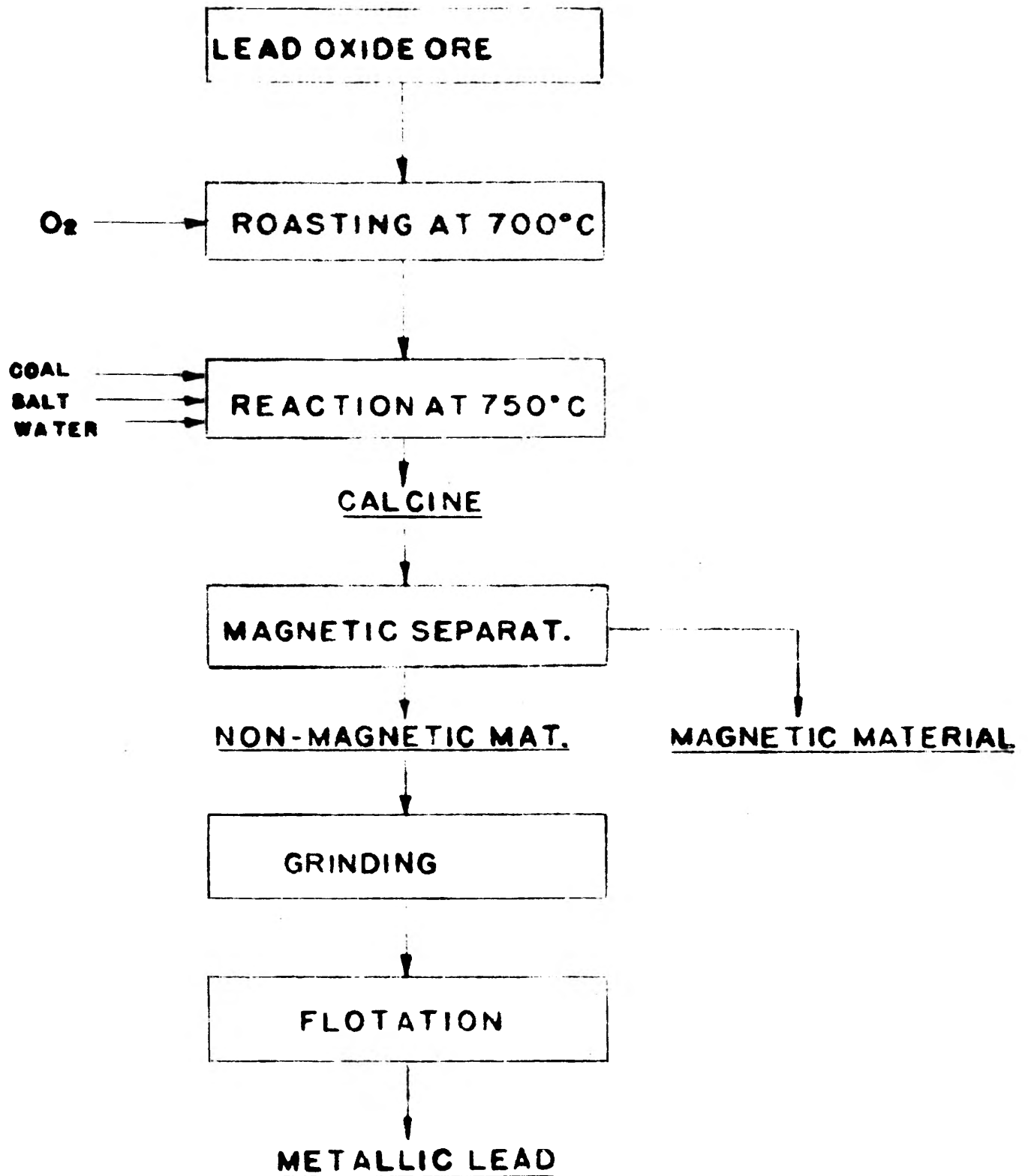
Figure No. 8 .— Flow sheet of the treatment of Cerro de
Pasco ore (Matagente) by the segregation
process in this investigation.

REPORT OF QUALITATIVE SPECTROGRAPHIC ANALYSIS

Sample ___Test No. 46 - segregated lead___ Date ___2-16-60___

Element	Code	Element	Code	Element	Code	Element	Code
Ag	m	Eu	•	Mn	Tr	Si	M
Al	m			Mo	•	Sm	•
As	Tr	Fe	m			Sn	Tr
Au	•			Na	•	Sr	Tr
		Ga	Tr	Nd	•		
B	Tr	Gd	•	Ni	Tr	Ta	•
Ba	Tr	Ge	•			Tb	•
Be	•			Os	•	Te	•
Bi	m	Hf	•			Th	•
		Hg	•	P	•	Ti	Tr
Ca	m	Ho	•	Pb	M	Tl	•
Cb	•			Pd	•	Tm	•
Cd	Tr	In	•	Pr	•		
Ce	•	Ir	•	Pt	•	U	•
Co	•						
Cr	Tr	K	Tr	Rb	•	V	Tr
Cs	•			Re	•		
Cu	m	La	•	Rh	•	W	•
		Li	•	Ru	•	Y	•
Dy	•	Lu	•			Yb	•
				Sb	Tr	Zn	m
Er	•	Mg	m	Sc	•	Zr	•

CODE: (blank) — Not determined
  • ~~(blank)~~ — Not detected
  tr — Trace constituent
  m — Minor constituent
  M — Major constituent

REPORT OF QUALITATIVE SPECTROGRAPHIC ANALYSIS

Sample ___ bar No. 67 - retorted lead ___ Date __3-16-60__

Element		Element		Element		Element	
Ag	m	Eu	•	Mn	tr	Si	M
Al	m			Mo	•	Sm	•
As	tr	Fe	m			Sn	tr
Au	•			Na	•	Sr	tr
		Ga	tr	Nd	•		
B	tr	Gd	•	Ni	•	Ta	•
Ba	tr	Ge	•			Tb	•
Be	•			Os	•	Te	•
Bi	tr	Hf	•			Th	•
		Hg	•	P	•	Ti	tr
Ca	m	Ho	•	Pb	M	Tl	•
Cb	•			Pd	•	Tm	•
Cd	tr	In	•	Pr	•	U	•
Ce	•	Ir	•	Pt	•		
Co	•					V	tr
Cr	•	K	tr	Rb	•		
Cs	•			Re	•	W	•
Cu	m	La	•	Rh	•	Y	•
		Li	•	Ru	•	Yb	•
Dy	•	Lu	•	Sb	tr	Zn	m
Er	•	Mg	m	Sc	•	Zr	•

CODE: (blank) — Not determined
• (blank) — Not detected
tr — Trace constituent
m — Minor constituent
M — Major constituent

Figure 9. Coal particle coated completely with
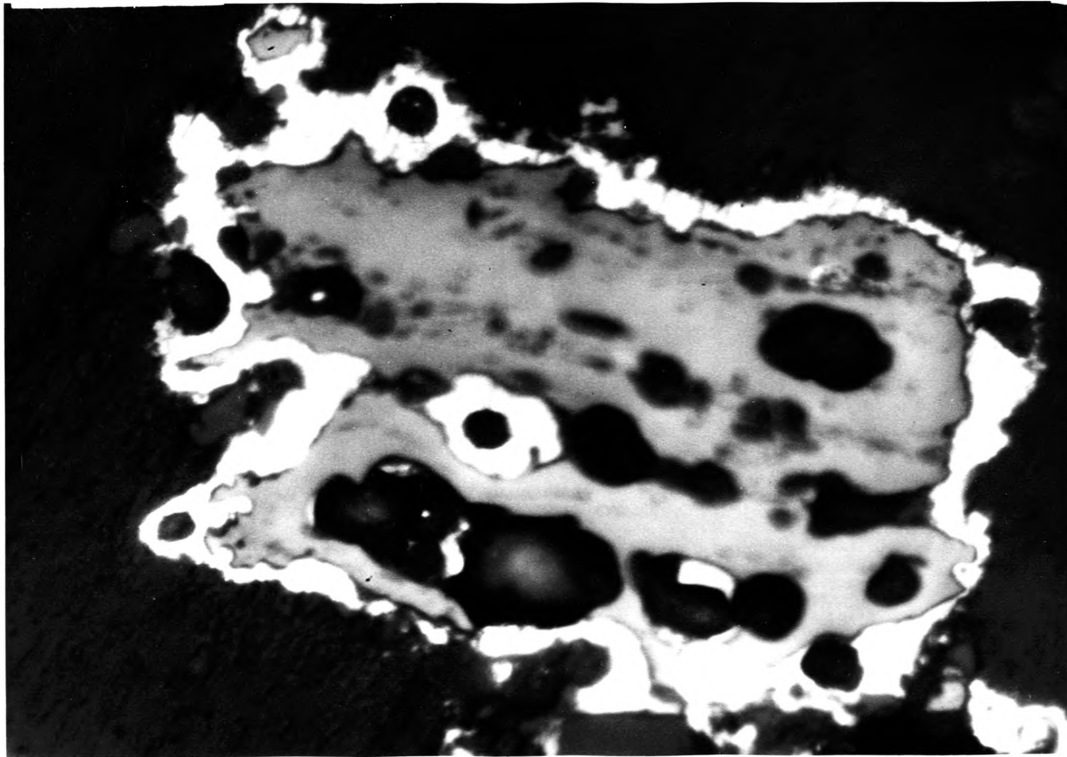metallic lead.
Magnification: 550x

Figure 10. Coal particle with a thick film of
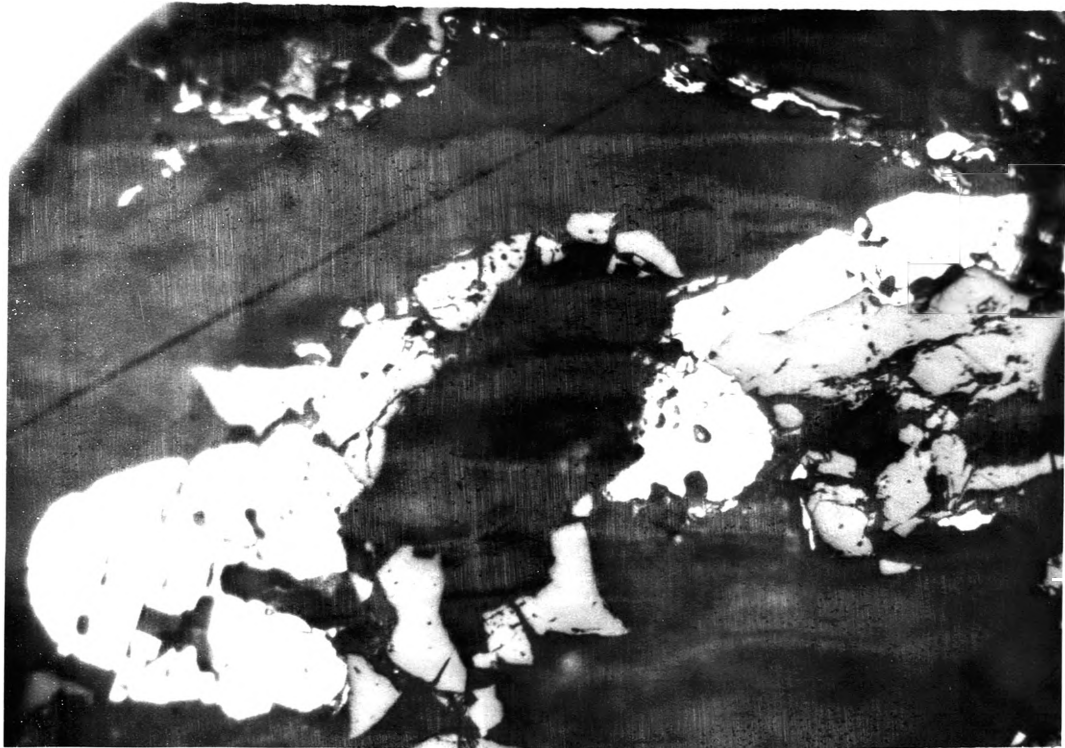metallic lead.
Magnification: 400x

Figure 11. Metallic lead has grown beyond the
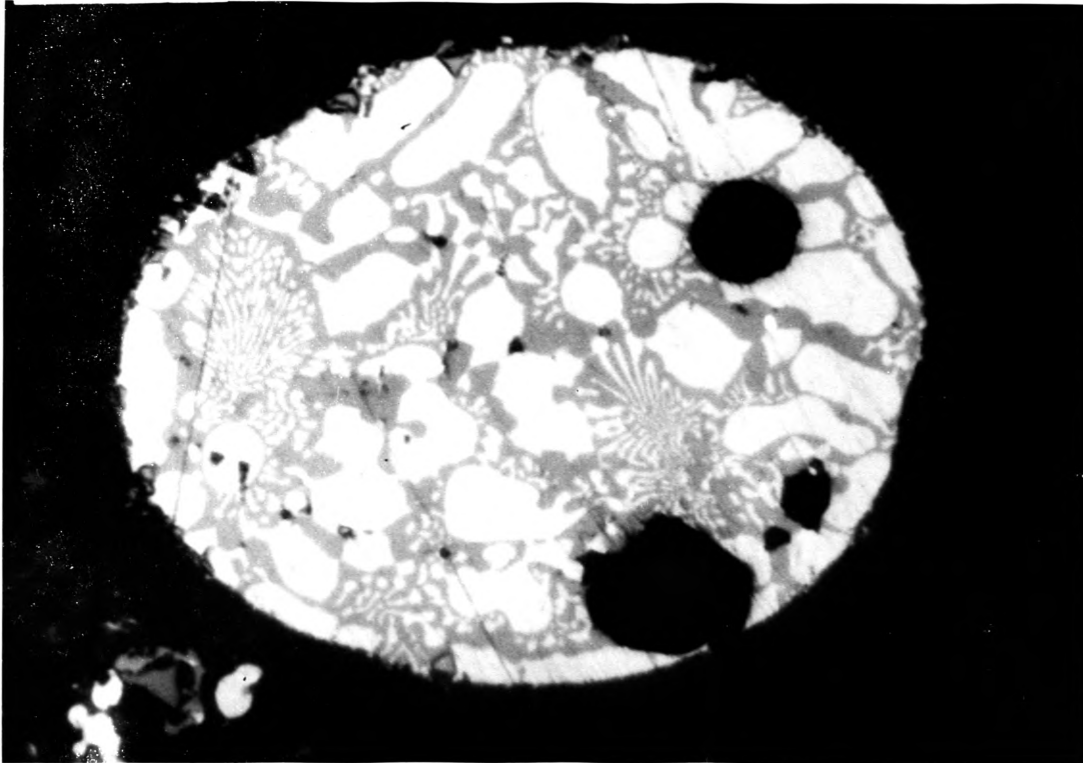limits expected.
Magnification: 250x

Figure 12. Pellet showing eutectic formation
typical of alloys.
Magnification: 550x

CONCLUSIONS

The review of the published literature showed that, to date, no process has been found which allows commercial segregation of lead.

The investigations reported in this thesis resulted in a segregation process which may be of commercial value. The tests were carried out on small-scale batches of oxidized lead ores from Cerro de Pasco (Matagente), Peru. Lead recoveries obtained were low but it is expected that with further work recoveries can be improved. It is also anticipated that further experiments should yield factors which have a negative effect on segregation of lead.

It is concluded that the process has considerable merit on oxidized siliceous ores of lead that are not amenable to conventional processing methods.

BIBLIOGRAPHY

1. AMSTUTZ, G.C. and WARD, H.J. (1956), Geologia y mineralizacion del deposito de plomo de Matagente, Cerro de Pasco, Peru. Bol. Soc. Geol., Peru 30, p. 18-31.

2. ANDERSON, A.L. (1930), The incipient oxidation of Galena, Econ. Geol. 25, p. 528-542.

3. BARGALLO, M. (1955), La mineria y la metalurgia en la America Espanola durante la epoca Colonial. Fondo de cultura economica. Mexico-Buenos Aires, 442 p.

4. BLANCHARD, R. and BOSWELL, P.F. (1934), Additional limonite types of galena and sphalerite derivation. Econ. Geol. 24, - p. 671-680.

5. CERRO DE PASCO CORPORATION, GEOLOGICAL STAFF (1950). Lead and Zinc deposits of the Cerro de Pasco Copper Corporation in Central Peru. Int. Geol. Congr., Rept. 18th. Session, 1948, Part VII. The Geology, Paragenesis and Reserves of the ores of lead and zinc. London, p. 154-186.

6. COOKE, H.C. (1913), Secondary enrichment of silver ores. Journ. Geol. 21, p. 11.

7. DE LAS CASAS, F. (1953), Matagente Report. Cerro de Pasco Corporation report, unpublished.

8. EDWARDS, A.B. (1954), Texture of the ore minerals, Melbourne: Australasian Institute of Mining and Metallurgy.

9. EMMONS, W.H. (1917), The enrichment of ore deposits. Bull. U.S. Geol. Surv. 625-630 p.

10. EMMONS, W.H. (1933), Recent Progress in studies of supergene enrichment. In: Ore deposits of the Western States, Lindgren - Vol. A.I.M.E., p. 386-418.

11. GAUDIN, A.M. (1957), Flotation, p. 467, McGraw-Hill Book Company Inc., N.Y.

12. GRIFFITH, R.H. and MARSH, J.D.F. (1957) Contact Catalysis, Oxford University Press.

13. JOHNSON, R.F. (1955), Geology of the Atacocha Mine, Dept. of Pasco, Peru. Econ. Geol. 50, pp. 249-270.

14. KERR, PAUL F. (1959) Optical Mineralogy, McGraw-Hill Book Company Inc. N.Y.

15. LYON, D.A. and RALSTON, O.C. (1918), Innovations in the Metallurgy of lead, U. S. Bureau of Mines, Bulletin 157.

16. MILLER, B.L. and SINGEWALD, J.T. (1919), The Minerals Deposits of South America, McGraw-Hill Book Co., N.Y.

17. HOULDEN, J.C. and TAPLIN, BRUCE (1928), Heat Treatment and Concentration of copper ores. U.S. Patent 1,679,337, July 31,1928.

18. RAMPACEK, CARL; MCKINNEY, W.A. and WADDLETON, P.T. (1959) Treating oxidised and mixed oxide-sulfide copper ores by the Segregation Process. U.S. Bureau of Mines Report of Investigations 5501.

19. REY, MAURICE (1936), The Segregation Process for low-grade copper oxide ore, Proc. 7th Internat. Cong. Paris. Min. Met. and Appl. Geol. Met. Sec., vol. 11-3, 1935, pp. 55-62; Rev. Met., vol. 1936 pp. 295-302.

20. WYCKOFF, W.O. (1949) Electron microscopy, Interscience Publishers, Inc., N.Y.

VITA

Jose Tomasio Molfino was born in Arequipa, Peru, on November 26, 1926. Elementary and secondary schooling was received in Peru.

He was graduated as Chemical Engineer in 1949 at San Marcos University, Lima, Peru.

Before coming to the United States he was working as a lecturer at the Engineering University in Lima. The purpose of his trip was to receive training in Mineral Dressing Metallurgy and its associated Microscopic Mineralogy.