

LECTURE NOTES
On
Non Ferrous Extractive Metallurgy

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CHAPTER 1

Non-ferrous mineral resources and industries in India

1.0. Minerals and ores

A *mineral* is a naturally occurring inorganic compound of one or more metals in association with nonmetals and having a fixed composition. It has well - defined physical and chemical properties. Minerals those containing metals are called metalliferous minerals and those do not contain any metal are called non-metalliferous minerals.

Examples of metalliferous minerals are:

Chalcopyrite ($\text{Cu}_2\text{S} \cdot \text{FeS}_3$)

Sphalerite (ZnS)

Cassiterite (SnC_2)

Examples of non-metalliferous minerals are:

Calcite (CaCO_3)

Fluorspar (CaF_2)

Quartz (SiO_2)

Salt (NaCl)

An *ore* is a naturally occurring mineral or combination of minerals from which one or more metals may be economically extracted . The chief factors which determine whether a particular mineral is an ore of the metal are:

(i) The % age of metal content

(ii) The cost of extraction

(iii) The market price.

The cost of extraction and market price vary time to time and also from place to place. Therefore, a mineral which may be an ore of the metal at one place, may not be an ore of different place of or time or vice- versa..

The economy of extraction generally depends on fundamental factors such as:

1. Percentage of valuable metal in the ore
2. The nature of the mineral in the ore.
3. Percentage of impurities in the ore.
4. The physical condition of the ore.
5. Location and magnitude of the ore deposits.
6. The proximity to transport facilities. and
7. The market value of the metal.

Important minerals or ores of few non ferrous metals with their chemical formulae are given below:

1. **Aluminium (Al) :**

Bauxite
($\text{Al}_2\text{O}_3 \cdot x\text{H}_2\text{O}$)
Orthoclase feldspar
($\text{K}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2 \cdot 2\text{H}_2\text{O}$)
Muscovite (mica)
($\text{K}_2\text{O} \cdot 3\text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2 \cdot 2\text{H}_2\text{O}$)
Kaolinite
($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$)
Leucite
($\text{K}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 4\text{SiO}_2$)
Cyanite
($\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$)
Sillimanite
($\text{Al}_2\text{O}_3 \cdot \text{SiO}_2$)
Corundum
(Al_2O_3)
Cryolite
(Na_3AlF_6)
Aluminite
($\text{Al}_2\text{O}_3 \cdot \text{SO}_3 \cdot 9\text{H}_2\text{O}$)

2. Copper (Cu):

Sulphides

Bornite
($\text{FeS} \cdot 2\text{Cu}_2\text{S} \cdot \text{CuS}$)

Chalcocite
(Cu_2S)

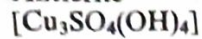
Chalcopyrite
(CuFeS_2)

Covellite
(CuS)

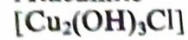
Enargite
($3\text{Cu}_2\text{S} \cdot \text{As}_2\text{S}_5$)

Oxides, Hydroxides, and Carbonates

Antlerite



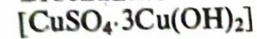
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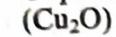
Azurite



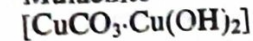
Brochantite



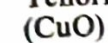
Cuprite



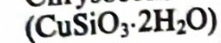
Malachite



Tenorite

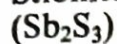


Chrysocolla

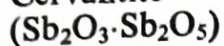


3. Antimony (Sb) :

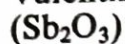
Stibnite



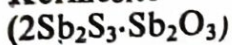
Cervantite



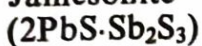
Valentinite



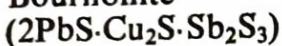
Kermesite



Jamesonite

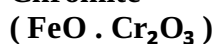


Bournonite



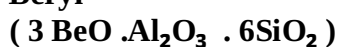
4. Chromium (Cr) :

Chromite



5. Beryllium (Be) :

Beryl



6. Barium (Ba) :

Barite (heavy spar)
(BaSO₄)

Bromlite
[(Ba, Ca)CO₃]

Witherite
(BaCO₃)

7. Thorium (Th) :

Oxide

Thorianite
[(Th, U)O₂]

Niobates, Tantalates, and Titanates

Brannerite
[(U, Ca, Fe, Y, Th)₃Ti₅O₁₆]

Eschynite
[(Ce, Ca, Fe₂, Th)(Ti, Nb)₂O₆]

Euxenite (see under rare earths)

Phosphates and Silicates

Xenotime
(YPO₄)

Allanite
[(Ca, Ce, Th)₂(Al, Fe, Mg)₃Si₃O₁₂(OH)]

Cheralite
[(Ce, Ca, Pr, Nd, Th, Ca, U)(PO₄, SiO₄)]

Thorite
(ThSiO₄)

Pilbarite
(PbO·UO₃·ThO₂·2SiO₂·4H₂O)

Monazite
[(Ce, La, Y, Th)PO₄]

8. Tin (Sn) :

Cassiterite
(SnO₂)

Stannite
(Cu₂S·FeS·SnS₂)

Franckeit
(5PbS·2SnS₂·Sb₂S₃)

10. Silver (Ag) :

Argentite
(Ag₂S)

Pyrargyrite
(Ag₃SbS₃)

Proustite
(Ag₃AsS₃)

Nessite
(Ag₂Te)

Cerargyrite
(AgCl)

Iodyrite
(AgI)

11. Zinc (Zn) :

Sphalerite
(ZnS)

Zincite
(ZnO)

Franklinite
[ZnO(Fe, Mn)₂O₃]

Smithsonite
(ZnCO₃)

Willemite
(Zn₂SiO₄)

Calamine
[Zn₂(OH)₂SiO₃]

Hemimorphite
[Zn₄Si₂O₇(OH)₂·H₂O]

12. Tungsten (W) :

Wolframite
[(Fe, Mn)WO₄]

Ferberite
(FeWO₄)

Hubernite
(MnWO₄)

Scheelite
(CaWO₄)

Cuproscheelite
[(Ca, Cu)WO₄]

Stolzite
(PbWO₄)

13. Magnesium (Mg) :

Dolomite
(MgCO₃·CaCO₃)

Magnesite
(MgCO₃)

Carnallite
(MgCl₂·KCl·6H₂O)

Brucite
[Mg(OH)₂]

Olivine
[(Mg, Fe)₂SiO₄]

14. Manganese (Mn) :

Pyrolusite
(MnO₂)

Psilomelane
(MnO₂·H₂O·K₂BaO₂)

Manganite
[Mn₂O₃·H₂O or MnO(OH)]

Braunite
(3Mn₂O₃·MnSiO₃)

Dialogite
(MnCO₃)

Rhodonite
(MnSiO₃)

15. Lead (Pb) :

Galena
(PbS)

Cerussite
(PbCO₃)

Anglesite
(PbSO₄)

Bonlangierite
(Pb₅Sb₄S₁₁)

Pyromorphite
[Pb₅(PO₄)₃Cl]

Mimetite
[Pb₅(AsO₄)₃Cl]

Vanadinite
[Pb₅(VO₄)₃Cl]

16. Titanium (Ti)

Ilmenite
[FeTiO₃]
(with Mg sometimes replacing Fe)

Rutile
(TiO₂)

Senaite
[(Fe·Mn·Pb)O·TiO₂]

17. Uranium (U) :

Pitchblende
(U₃O₈)

Uraninite
(UO₂, UO₃)

Carnotite
(K₂O·2UO₃·V₂O₅·xH₂O)

Autumite
($\text{CaO} \cdot 2\text{UO}_3 \cdot \text{P}_2\text{O}_5 \cdot x\text{H}_2\text{O}$)

Uranophane
($\text{CaO} \cdot 2\text{UO}_3 \cdot 2\text{SiO}_2 \cdot 7\text{H}_2\text{O}$)

Davidite
[(Fe, Ce, U)(Ti, Fe, V, Cr)₃(O·OH)₇]

Tyuyamunite
($\text{CaO} \cdot 2\text{UO}_3 \cdot \text{V}_2\text{O}_5 \cdot x\text{H}_2\text{O}$)

Brannerite
[(U, Ca, Fe, Y, Th)₃Ti₅O₁₆]

Schroëckingerite
($3\text{CaO} \cdot \text{Na}_2\text{O} \cdot \text{UO}_3 \cdot 3\text{CO}_2 \cdot \text{SO}_3 \cdot 10\text{H}_2\text{O}$)

1.1 Non ferrous mineral resources of India :

The mineral resources of India are broadly classified into three categories such as:

1. *Adequate to abundant* ; Minerals containing Al, Be, Cr, Fe, Mn, Mg, Ti, Zr, Th and rare earth metals.
2. *Inadequate* : Minerals containing Cu, Au, C or graphite, Pb, V, Zn, Ni, Cd, U and Sn.
3. *Poor to so far not known* : Minerals containing Sb, Bi, B, Co, Hg, Mo, Nb, Ta, P, Ce, S, Sr, Te, Ag and W .

If properly exploited, minerals in category (1) meet the domestic demand and surplus for export, minerals in category (2) are insufficient to meet the domestic demand and minerals in category (3) are not suitable for commercial exploitation.

Some important non ferrous mineral resources of India are described briefly below:

Aluminium

The chief minerals of aluminium are Bauxite, Cyanite, Sillimanite, Cryolite, Corundum, Aluminite etc. Bauxite is the main ore of aluminium . The total reserves of bauxite in India (as on 2019-20) was about 21,824,000 tonnes.

Places of deposits - Odisha (in districts of Kalahandi, Sambalpur, Bolangir, Koraput)

- Jharkhand (Lohardagga areas)
- Gujarat (in Bhavanagar and Jam nagar areas)
- Chhattisgarh (in Amarkantak area).
- Madhya Pradesh (in Jabalpur and Balaghat area).

Maharashtra (in Ratnagiri, Satara, Thane Kolaba, Pune, Kolhapur)

The share of bauxite reserves is

Odisha (71 %), Gujarat (10 %), Chhattisgarh (7.18 %), Jharkhand (6.5 %), MP (3.14 %), Maharashtra (2.73 %)

Copper

The important minerals of copper are Chalcocite, Chalcopyrite, Covellite, Bornite, Cuprite, Malachite etc. The total reserves of copper ore in India (2015) is 1511.50 MT. The major copper ore deposits are found in

- Jharkhand (Singhbhum district)
- M.P. (Balaghat)
- Rajasthan (Jhunjhunu, Alwar)
- A.P.(Guntur)
- Karnataka (Chitradurga, Hassan dist)
- Tamilnadu (South Arcot dist)

Gold

The minerals of gold are Sylvanite, Calaverite etc. The resources of gold ore of India as on 2015 were 527.96 MT.

The major areas of gold ore are in the states of Karnataka, Bihar, Rajasthan, West Bengal, Kerala, Telengana, Madhya Pradesh, and Andhra Pradesh .

Silver

India has no reserves of silver ores. Small quantities of silver are recovered as a by-product during the refining of gold, copper and lead. Major coming from the lead smelter(Tundoo) , Bihar and the gold mines in Karnataka.

The chief minerals of silver are Argentite, Nersite, Iodyrite, Proustite etc.

Lead and Zinc

The chief minerals of lead are galena, cersusite, anglesite etc. And minerals of zinc are sphalerite, zincite, smithsonite, willemite etc. The total reserves of lead and zinc ores as in 2015 were about 749.46 MT. The major resources are from Rajasthan, Bihar, Maharashtra, West Bengal, Odisha, Sikkim, Tamilnadu, Meghalaya, Gujarat and Madhya Pradesh.

Tin

The chief minerals of tin are cassiterite, stannite etc. Tin is not extracted on a commercial scale in India. Present domestic demands are met by imports.

Nickel

Minerals of nickel are nicolite, pentlandite etc.

The total reserves of nickel ore in India (2015) - 189 MT.

Nickel ores are found in the states of Odisha, Jharkhand, Nagaland and Karnataka., of which the share of Odisha is around 92 % .

Chromium

The chief ore of chromium is chromite. Resources of chromite ore of India - 344 MT. Of which Odisha produces 96 %. The other states producing chromite ore are Manipur, Nagaland, Karnataka, Jharkhand, Maharashtra, Tamilnadu, Telengana and Andhra Pradesh.

Manganese

The chief minerals of manganese are pyrolusite, psilomelane, manganite, braunite etc. Its reserve in India as on 2015, was around 496 MT. The main places of deposits are Odisha, Karnataka, Madhya Pradesh, Goa, Maharashtra, Telengana and Jharkhand states in India.

1.2 Ferrous and Non ferrous Industries in India

Ferrous industries in India :

India's crude steel production in 2019 was around 111.4 MT. The steel plants in India are of two types such as

1. Mini steel plants - They have electric furnaces, use steel scrap, sponge iron, and produce carbon and alloy steels. There are more than 650 mini steel plants in India.

2. Integrated steel plants- They handle everything in one complex starting from raw materials to finished products. There are more than 50 iron and steel industries in India. The following table shows name and location of major iron and steel industries in India.

Table 1

<u>Name</u>	<u>Location.</u>	<u>Operator</u>
1. Alloy steel plant	Durgapur, W.B	SAIL
2. Bhilai Steel plant.	Bhilai, Chhattisgarh	SAIL
3. Bokaro Steel plant	Bokaro, Jharkhand.	SAIL
4. Durgapur Steel Plant.	Durgapur, West Bengal.	SAIL
5. IISCO	Asansol, West Bengal	SAIL
6. Chandrapur Ferro-alloys.	Chandrapur, Maharashtra	SAIL
7. ESSAR Steel India Ltd.	Hazira, Gujarat.	Arcelor, Mittal
8. Jindal Steel and Power Ltd.	Raigarh, Chhattisgarh	J S P
9. Jindal Steel and Power Ltd.	Angul, Odisha.	JSP
10. JSW Steel.	Hospet, Bellary, Karnataka.	JSP Steel
11. JSP Steel	Tarapur, Baisar, Maharashtra	JSP Steel
12. JSP Steel.	Dharamtar, Maharashtra	JSP Steel
13. Rourkela Steel Plant.	Rourkela, Odisha.	SAIL
14. Salem Steel Plant.	Salem, Tamilnadu.	SAIL
15. Tata Steel Ltd (TISCO).	Jamshedpur, Jharkhand	Tata Steel
16. Tata Steel Ltd.	Kalinga Nagar, Odisha.	Tata Steel
17. Visakhapatnam Steel Plant.	Visakhapatnam, Andhra Pradesh.	RINL
18. Visvesvaraya Iron and Steel Plant	Bhadrabati, Karnataka.	SAIL

State-wise production (Total- 111.4MT)

1. Odisha - 18.6 MT
2. Jharkhand - 17.0 MT
3. Chhattisgarh - 12.8 MT
4. Karnataka - 12.7 MT
5. Maharashtra - 8.7 MT
6. Gujarat - 8.2 MT
7. West Bengal - 7.3 MT

Top 10 steel producing countries (as on 2019)

- | | |
|-----------------|-----------|
| 1. China. | 1001.3 MT |
| 2. India. | 111.4 MT |
| 3. Japan. | 99.3 MT |
| 4. USA. | 87.8 MT |
| 5. Russia. | 71.6 MT |
| 6. South Korea. | 71.4 MT |
| 7. Germany | 39.6 MT |
| 8. Turkey. | 33.7 MT |
| 9. Brazil. | 32.6 MT |
| 10. Iran. | 25.6 MT |

Global production - 1864 MT

India is the largest producer of DRI(36.86 MT)

Non ferrous Industries in India

Non ferrous metal industries are one of the key sectors of Indian economy contributing significantly to the nation's GDP. The following are some of the non ferrous industries in India:

Aluminium industries in India

- ❶ India is the second largest aluminium producer, China being the first.
- ❷ Aluminium production - 3.68 MT per year (2020).
- ❸ 5 % of the total aluminum production in the world.
- ❹ India is the second in Asia.

Companies under the Indian Aluminium Industry are:

1. Hindustan Aluminium Company (HINDALCO)
2. Indian Aluminium Company Ltd.(INDALCO)
3. National Aluminium Company Ltd. (NALCO)
4. Bharat Aluminium Corporation Ltd.(BALCO)
5. Madras Aluminium Corporation Ltd. (MALCO)
6. Vedant Aluminium Ltd.

1. Hindustan Aluminium (HINDALCO)

It is the Asia's largest integrated primary producer of aluminium. It has 39 % of the market share. It is located at Renukoot in Uttar Pradesh by Aditya Birla Group flagship Company. It produces aluminium ingots, extrusions, rolled products , wheels, foils, wires, rods etc.

2. Indian Aluminium Company Ltd.(INDALCO)

It is located at Hirakud (Odisha), Alwaye (Kerala) and Belgaum (Karnataka).

3. National Aluminium Company Ltd. (NALCO)

- ❶ Established on January 7, 1981
- ❷ Operated in 1987
- ❸ Earned profits since 1987
- ❹ In 2020-21, net turnover- 8869 crores, net profit - 1300 crores.
- ❺ Alumina refinery located at Damanjodi (Koraput), Odisha.
- ❻ Aluminium smelter at Angul, Odisha.

4. Bharat Aluminium Company Ltd (BALCO)

It was incorporated in 1965 as a public sector undertaking. It was the first public sector undertaking in India which started production in 1974. In 2001, the government of India sold it to Vedant Ltd., Vedant Resources, government of India (Sterlite Industries Ltd.) Balco has two working units, one in Korba (Chhattisgarh) and the other at Bidhanbag (West Bengal). Total aluminium production is about 345000 TPA.

5. Madras Aluminium Company Ltd. (MALCO)

It was incorporated in Tamilnadu in 1961, started production in 1965. Aluminium production is about 29500 TPA.

6. Vedanta Aluminium Limited.

It is the largest producer of aluminium, producing 2.3 MTPA. The company has an aluminium smelter at Jharsuguda (Odisha), one alumina refinery of 2 MTPA at Langigarh in Kalahandi (Odisha).

Copper industries in India

The major copper industries in India include:

1. Hindustan Copper Ltd.(HCL)

2. Bhagyanagar India Ltd.(BIL)

3. Arcotech Ltd.

1. Hindustan Copper Ltd (HCL)

It is a public sector undertaking and was incorporated on Nov. 1967. HCL's mines and plants are spread across five operating units, one each in the states of Rajasthan, Madhya Pradesh, Jharkhand, Maharashtra and Gujarat as named below.:

(i) Khetri Copper Complex (KCC) at Ketrinagar, Rajasthan.

(ii) Indian Copper Complex (ICC) at Ghatsila, Jharkhand.

(iii) Malanjkhand Copper Project (MCP) at Malanjkhand, Madhya Pradesh.

(iv) Taloja Copper Project (TCP) at Taloja, Maharashtra.

(v) Gujarat Copper Project (GCP) at Jhagadiha, Gujarat.

2. Bhagyanagar India Ltd.(BIL)

BIL is among the oldest and largest company, incorporated in 1985, providing copper products in India.

3. Arcotech Limited

It was incorporated in 1981 for manufacturing non ferrous engineering products.

Madhya Pradesh is the largest producer of copper in India - 8700000 TPA.

Copper production in India - 15300000 TPA

Rajasthan - 6200000TPA

Copper production in India is 2 % of the world copper production. Now there are three more companies producing copper, they are

(i) Hindalco Industries Ltd, Gujarat.

(ii) Sterlite Industries Ltd, Tamilnadu

(iii) Jagadiha Copper Ltd, Gujarat

Zinc and Lead industries in India

The main zinc production companies in India are:

1. Hindustan Zinc Ltd.(HZL) in Rajasthan and Uttarakhand, incorporated in 1966. It is the largest in India and second in world.

2. Mewat Zinc Ltd (MZL) in Delhi and incorporated in 1991.

3. Sunrise Zinc Ltd (SZL) , in Goa, incorporated in 1973 ,a public limited company.

4. Rose Zinc Ltd.(RZL) at Udaipur, Rajasthan and incorporated in 1990.

5. Ambuja Zinc Ltd (AZL) at Patna, Bihar and incorporated in 1987.

6. Sukinda Nickel Extraction Project , Jajpur, Odisha.

Nickel industries in India

The following are the main nickel producing companies in India.:

1. Hindustan Copper Ltd., Ghatasila, Jharkhand, produces LME grade of nickel.

2. Sterlite Industries Ltd, Thotukodi, Tamilnadu.

3. NiComet,Goa . It produces nickel and cobalt and largest in India.

4. Jhagadiha Copper Ltd , Gujarat.

5. Sukinda Nickel Extraction Project, Jajpur, Odisha.

Silver industries in India

Hindustan Zinc Limited is the only integrated silver producer in India and produces about 95 % of India's prime Silver

Chromium industries in India

The main chromium industry is the Otto- Chemie pvt. Ltd at Mumbai, Maharashtra.

Magnesium industries in India

1. National Metallurgical Laboratory (NML) Jamshedpur

2. Central Electrochemical Research Institute, Korai kudi, Tamilnadu.

Extraction of metals from oxide ores

2.1 Extraction of Aluminium

2.1.1. Properties of Aluminium:

- (i) It has excellent ductility.
- (ii) It has good corrosion resistance and non toxic properties.
- (iii) It has high reflectivity and low emissivity.
- (iv) It has excellent electrical conductivity ($Ag > Cu > Au > Al$).
- (v) It has attractive mechanical properties in general and high strength -to-weight ratio.

2.1.2. Uses of Aluminium :

- (i) It is used as a strong deoxidiser in steel making.
- (ii) It is used in electrical conductors.
- (iii) It is used in transport industries and structural applications.
- (iv) It is used in roofing systems and thermal insulations.
- (v) It is used in packaging industries, decorative and protective surfaces.
- (vi) It is used in impact extrusion of vessels and containers.
- (vii) It is used in kitchenware, chemical and milk processing industries.

2.1.3. Aluminium ores :

The most common minerals of aluminium are

1. Gibbsite ($Al_2O_3 \cdot 3H_2O$) , $Al_2O_3 \rightarrow 65.4\%$
2. Diaspore ($Al_2O_3 \cdot H_2O$) , $Al_2O_3 \rightarrow 85.4\%$

Bauxite is the mixture of Gibbsite and Diaspore, contains varying amounts of impurities like Fe_2O_3 , TiO_2 and SiO_2 . The bauxite ores found in India normally contain 58-67 % Al_2O_3 , 5-10 % TiO_2 and 0.5- 2.0 % SiO_2 . The ores found in Kashmir contain around 75 % Al_2O_3 , essentially no TiO_2 and a high amount of silica as an impurity(>5 %). This high silica content of these ores renders the Kashmir deposits unsuitable for purification by the **Bayer** process . In this process, during leaching stage, silica forms a compound $Na_2O \cdot Al_2O_3 \cdot 3SiO_2$, which in turn, forms an insoluble residue called *red mud* by reacting with other impurities in bauxite such as Fe_2O_3 , and TiO_2 . This causes a serious loss of alumina . For this reason , ores containing more than 5 % Silica are considered unsuitable.



Bauxite

2.1.4. Extraction of aluminium - concepts

Extraction of aluminium by carbothermic reduction of Al_2O_3 is not commercially adopted because;

1. It requires a high temperature (2000 °C)
2. At this temperature, undesirable aluminium carbide is formed.
3. Refractories required for this process are expensive and not readily available.

Extraction of aluminium by electrolysis of aluminium salts in an aqueous medium is not feasible.

The universally adopted process for the extraction of aluminium is the **Hall- Heroult** process ,in which alumina (Al_2O_3) dissolved in cryolite($3\text{NaF} \cdot \text{AlF}_3$) is electrolyzed. Here alumina (obtained from the Bayer process) in pure form is employed as the electrolyte in electrolytic cell.

2.1.4 Bayer process for alumina production

In the Bayer process, high purity alumina (Al_2O_3) is obtained from bauxite.The following figure shows the flowsheet of the Bayer process.

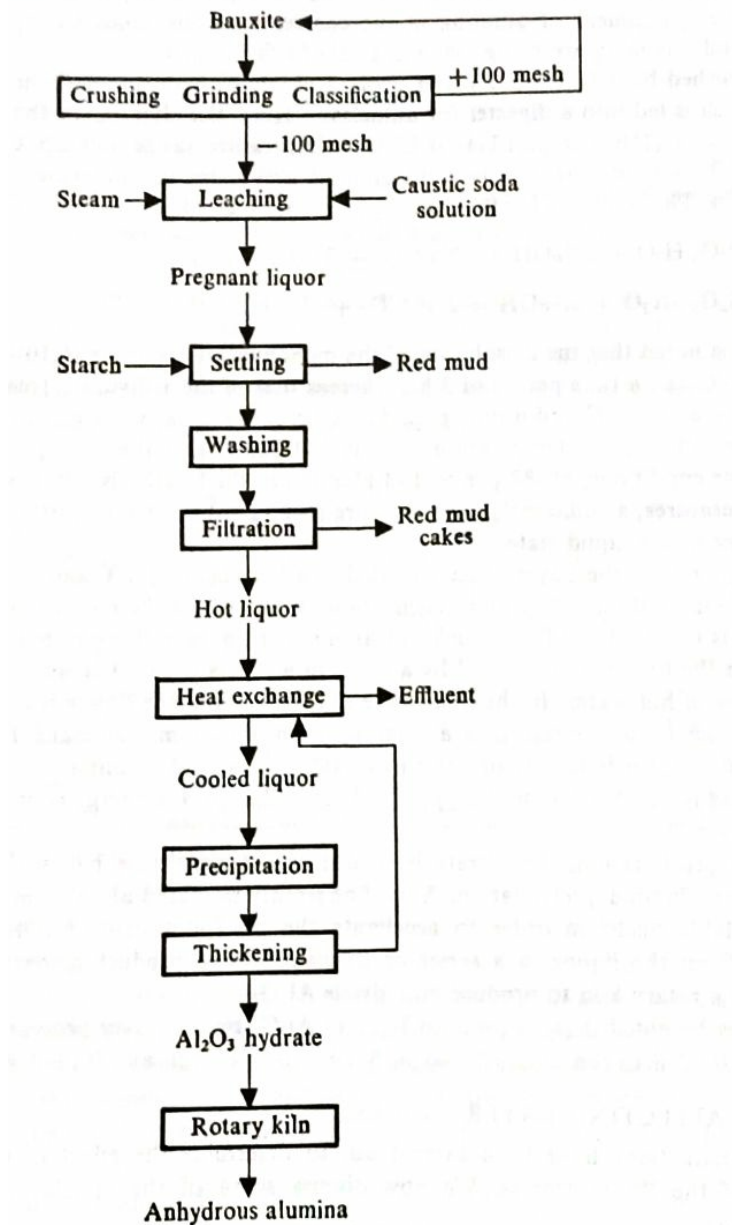


Fig.2.1 Flowsheet for Bayer process.

In this process, bauxite is initially leached by NaOH under high pressure (25 atm) and high temperature (220 °C) to form soluble sodium aluminate, from which $\text{Al}(\text{OH})_3$ is subsequently precipitated. When $\text{Al}(\text{OH})_3$ is calcined, Al_2O_3 is obtained.

The bauxite obtained from the mines is crushed and ground to a very fine size (-100 #) in Jaw crushers and hammer mills. The crushed bauxite is once again ground along with caustic soda (NaOH) in a ball mill to yield a slurry, which is fed into a digester or autoclave. Here the alumina in the bauxite is dissolved in caustic soda in the temperature range 150-220°C under a pressure of 5-25 atm. This treatment dissolves the alumina and leave the impurities as suspended solids in the solution. The reactions being,



The dissolution of the monohydrate (reaction -1) requires a temperature of 230°C and a time period of 3 hours, whereas that of the trihydrate (reaction -2) requires a temperature of 180°C and a time period of 1 hour. Since the bauxite is a mixture of monohydrate and trihydrate, the temperature used is 180-220°C and the time period allowed is 2.5 hrs. Under these conditions, 86-88 % of alumina in the bauxite is taken into solution.

The liquor from the Bayer process is cooled to below 100°C and is completely depressurized . It is then taken to the settling and clarification section where the red mud is deposited in the settling tank and removed. Additives such as starch speed of the settling of red mud. Any remaining red mud in the liquid is eliminated by a series of washers where it is confronted with a counter current flow of hot water. In the final stage of clarification, the liquor is filtered through a series of filters. The filtrate obtained is a clear solution of sodium aluminate. The heat in the liquor is tapped by heat exchangers for energy conservation and for cooling the liquor. During precipitation, the filtrate is cooled and very fine freshly prepared aluminium hydroxide is added to make available nuclei in order to accelerate the precipitation of $\text{Al}(\text{OH})_3$. The precipitate is separated from the liquor in a series of thickeners. The product is finally calcined in a rotary kiln to produce anhydrous Al_2O_3 .

2.1.6 Factors affecting Bayer process

The following are the important factors that affect the Bayer process:

1. The finer the bauxite, the better the digestion of alumina in leach liquor.
2. In general, wet grinding is more efficient than dry grinding.
3. Digestion is accelerated at higher temperatures.
4. For efficient calcination of alumina , the rotary kiln should be able to attain a temperature as high as 1400°C. However, if the alumina is only to be dried, a temperature of 200°C is sufficient.
5. The sensible heat of the hot pregnant liquor must recovered and used to produce steam for the digesters.

Red mud → A residue containing Fe_2O_3 , SiO_2 , TiO_2 and other impurities as well as undissolved Al_2O_3 .

2.1.7. Production of Aluminium from alumina(Hall- Heroult process)

Hall- Heroult process is based on the electrolytic decomposition of alumina dissolved in a liquid bath of cryolite. This process requires high purity alumina, cryolite, and ashless carbon electrodes. Since alumina is not a ionic compound, very

few ionic melts are capable of dissolving it to any appreciable extent. However, cryolite, a naturally mineral, can dissolve upto 15 % alumina at 1000°C. Cryolite has also been synthesized by passing HF through sodium aluminate solution, shown in the following flowsheet.

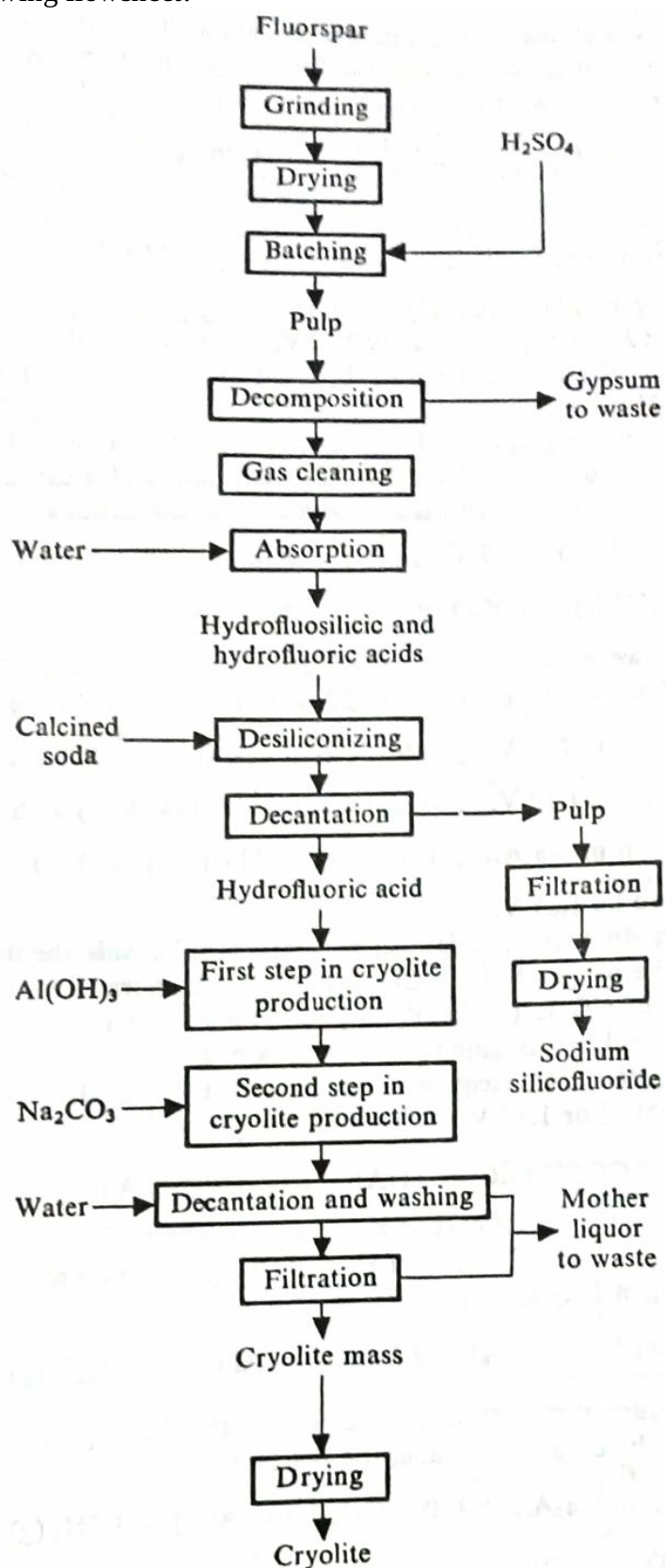
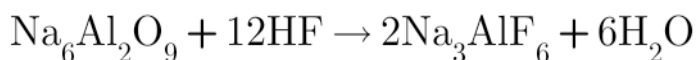
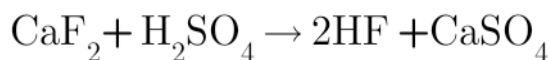


Fig.2.2. Flowsheet for production of synthetic cryolite.

The HF, being produced from naturally occurring CaF_2 . The main reactions are

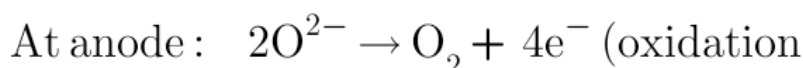
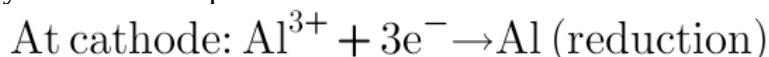


(Sodium aluminate)

Indian Aluminium industries employ a mixture of natural and synthetic cryolite.

2.1.8 Functions of cryolite in electrolysis

The extraction of aluminium is done by electrolysis, but first the aluminium oxide (Al_2O_3) must be melted so that electricity can pass through it. However, alumina has a very high melting point (2000°C), so it would be expensive to melt it. Instead, it is dissolved in molten cryolite which is an aluminium compound with a lower melting point than alumina. The use of molten cryolite as a solvent reduces some of the energy cost involved in extracting aluminium by allowing the ions in Al_2O_3 to move freely at a lower temperature.



The molten aluminium sinks to the bottom of the cell, where it is tapped off. The oxygen reacts with the carbon of the positive electrodes, forming CO_2 , so these gradually burn away. Hence, the positive electrodes have to be replaced frequently.

Electrolytic reduction cell

The figure below shows the electrolytic reduction cell for aluminium production.

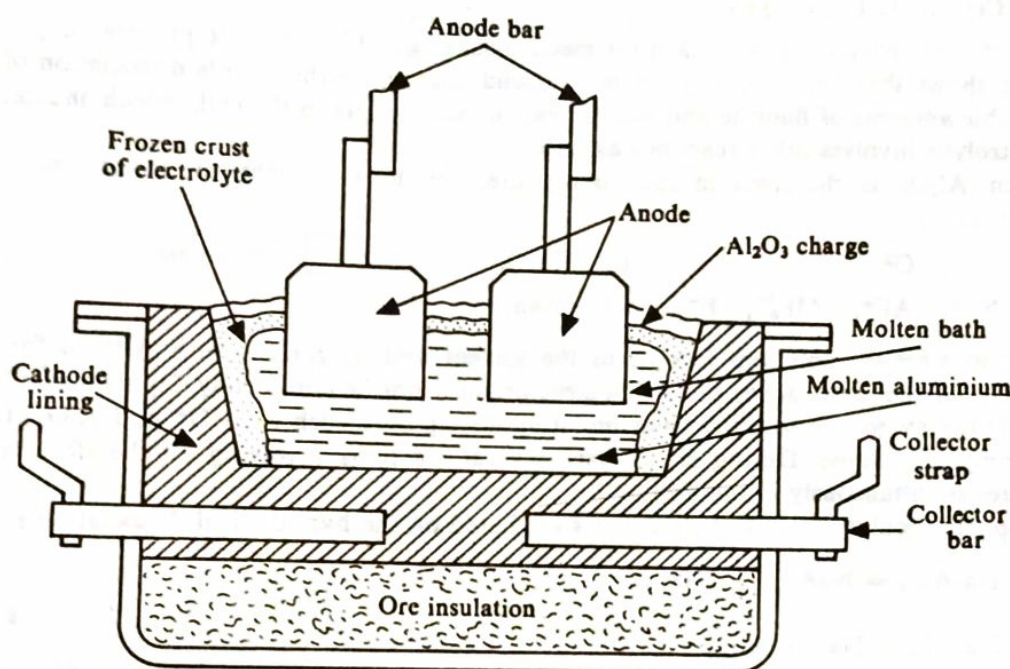


Fig.2.3 Aluminium electrolytic reduction cell.

The cell consists of a rectangular refractory lined steel box with length 5 metres, wide

2 metres and depth 1 metre. Cathode lining consists of refractory bricks faced with carbon mixed tar binder. A consumable graphite electrode serves as anode. A tap hole or outlet is there, through which metal gets collected at the bottom of the cell.

Cell operation -

During operation, the cell bath is filled with cryolite and anode is lowered into it. A current is passed through the cell circuit until the cryolite melts. Melting point of cryolite - 990°C . When the bath attains a molten state, alumina is added. The alumina decomposes to yield aluminium and oxygen. The necessary heat is supplied by the resistance offered by both the electrodes and electrolyte.

2.1.9. Anode Effect

If the alumina content of the bath (normal range: 5-10%) falls below 2%, the normal contact between the anode and the bath is interrupted by the gas film which abruptly increases the resistance. As a result, the normal operation ceases. This effect is known as **anode effect**.

Under certain conditions, the electrolytes of fused salts is associated with periodically occurring phenomena in which the terminal velocity increases to high value and current density decreases to a lower value from their normal value. This effect is also known as anode effect. In the electrolysis fused salt electrolyte, fine globules at the cathode metal get appear in electrolyte is known as metallic clouds or fogs.

2.1.10. Factors affecting electrolysis

The following are the factors that affect the process of electrolysis:

- (a) **Temperature:** The bath temperature is an important factor. An increase in temperature, the current efficiency decreases. For every 4°C rise in the bath temperature above the normal 970°C , the current efficiency falls by 1%.
- (b) **Current density:** The higher the current density, the greater the current efficiency. The current density should be maintained at a high value.
- (c) **Interpolar distance:** It is the distance from the bottom of the anode to the top of the molten metal bath. Current efficiency increases along with the interpolar distance, reaching a maximum of 90% when the distance is approximately 65 cm.
- (d) **Addition of alumina:** At values lower and higher than 4%, the current efficiency increases.

2.1.11. Refining of Aluminium (Electrolytic refining) **(Hoopes process or Three-layer process)**

The purity of metal produced by the Hall- Heroult process seldom exceeds 99.5%. This degree of purity is sufficient for most alloying purposes. However, for electrical applications, a higher degree of purity is required. To achieve this high degree of purity, an electrolytic method known as the three-layer process is employed, shown in the following fig .2.4 below.

The three-layer process of aluminium refining was developed by William Hoopes, a chemist of the Aluminium Company of America (ALCOA) in 1925. In this process, high purity aluminium, about 99.9%, can be obtained.

The process :

The cell used in this process consists of an iron tank lined with carbon at the bottom. A molten alloy of copper, impure aluminium and silicon is used as anode. It forms the

bottom layer in the cell. The density of this layer is maximum of about 4.5 gm/cm³. A dense electrolyte forms the middle layer. Its composition is 36 % Aluminium Fluoride, 30 % Cryolite, 18 % Barium Chloride and 16 % Calcium Fluoride. This layer has the density of about 2.8 gm/cm³.

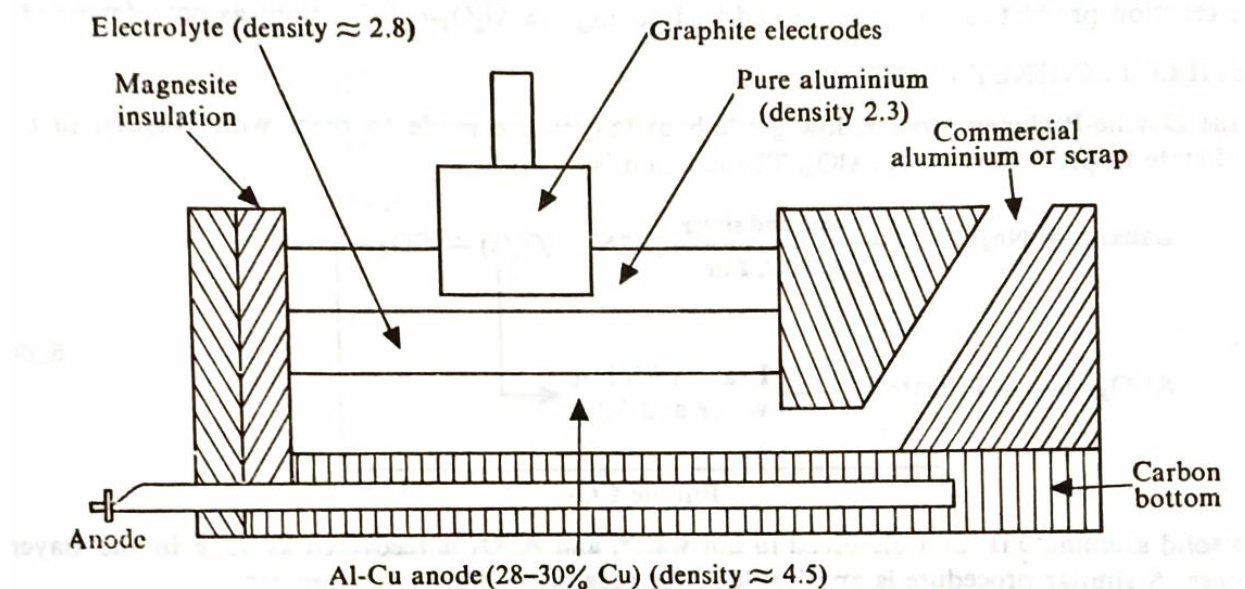
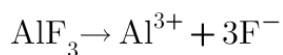
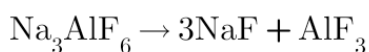


Fig.2.4 Three - layer process of aluminium refining .

The density of the electrolyte enables the aluminium purified during electrolysis to float upward to form the top layer consisting of molten aluminium having density 2.3 gm/cm³. The temperature of the operation is maintained at about 950°C. During electrolysis, Al³⁺ ions from the middle layer migrate to the upper layer, where they are reduced to aluminium by gaining 3 electrons. Equal numbers of Al³⁺ ions are produced in the lower layer. These ions migrate to the middle layer. Pure aluminium is tapped off from time to time. The three - layer process gives about 99.9 % pure aluminium. During electrolysis, the reactions being

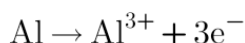
Electrochemical changes:



At the Cathode:



At the Anode:



2.2 Extraction of tin

2.2.1 Properties and uses of tin

The important properties and the uses or applications of tin are as follows

1. Tin has remarkable resistance to both corrosion and toxicity.
2. It has good mechanical properties such as ductility and malleability.
3. It has low melting point.
4. Tin is used for making collapsible tubes, foils, tin-copper wire etc.

5. Tin is used in soldering purposes.
6. It is used as coating metal exp. tin plating on steel (Cans).
7. As an alloy, it is used in white metal bearing, solder, bronze, fusible alloys etc
8. Tin is a soft metal

The usual properties of tin are:

M.P. of tin - 232°C

B.P. of tin - 2602°C

Density. - 7.265 gm/cm^3

Colour. - Silvery white metal

Latin name- Stannum

2.2.2 Important minerals of tin

The important minerals or ores of tin are as follows

- (i) Cassiterite (SnO_2)
- (ii) Stannite ($\text{Cu}_2\text{S} \cdot \text{FeS} \cdot \text{SnS}_2$)
- (iii) Franckeite ($5\text{PbS} \cdot 2\text{SnS}_2 \cdot \text{Sb}_2\text{S}_3$)

Cassiterite is the only important mineral, the others having little economic value.



Cassiterite

2.2.3 Concentration of tin ores

Naturally occurring cassiterite is usually associated with gangue materials and metallic sulphides such as galena, chalcopyrite, iron pyrite and sphalerite. The theoretical tin content of SnO_2 is 78.6 % . In reality, ores may contain as little as 1 % or even less. To upgrade these ores to about 65 % Sn, the water gravity concentration methods are employed. During such upgrading, the gangue is removed, but since the specific gravity of other metal sulphides are almost same as that of cassiterite, they can not be eliminated by ordinary gravity methods. Some tin ores are concentrated by magnetic roasting and magnetic separation methods.

2.2.4 Smelting of tin concentrate

The principle behind the smelting of tin concentrate is the chemical reduction of tin oxide by heating with carbon to produce tin metal and carbon dioxide gas. Limestone is added as a flux for the formation of slag. Smelting of tin concentrate is carried out mainly either in reverberatory furnace or in electric furnace.

1. Reverberatory furnace smelting

If tin concentrate contains iron sulphide, they are oxidised to the oxide before or during smelting. The smelting of tin concentrate is done in 3 stages. It is shown below in a flowsheet.

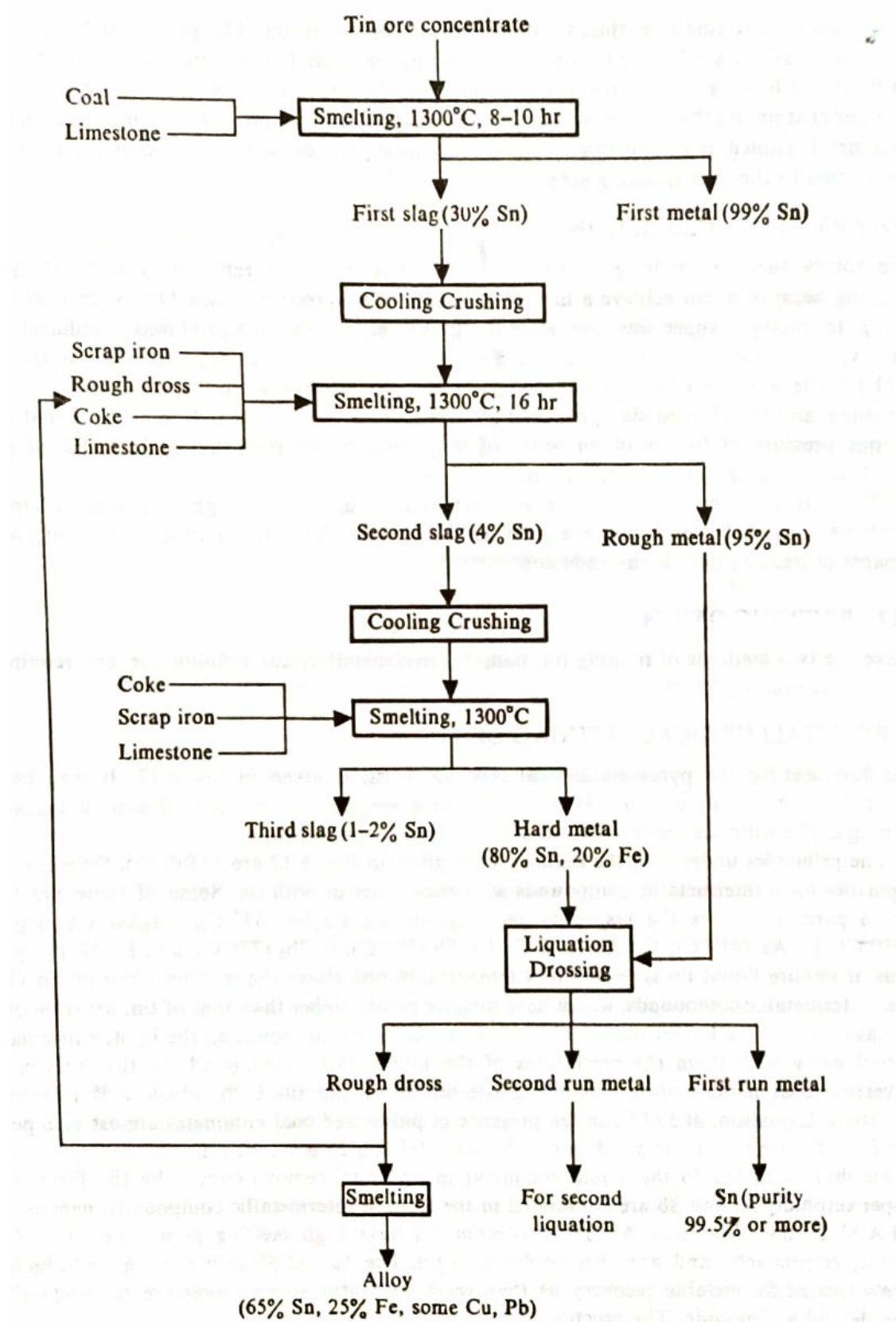


Fig. 2.5 Flowsheet for smelting of tin concentrate .

In the first stage, high quality tin (99%) is obtained by partially reducing the tin concentrate, by deliberately maintaining a high level of tin oxide in the slag and by retaining almost all the iron in the slag. This stage is subsequently reduced in two further stages ; the first stage produces tin containing 5 % iron and the second stage tin containing 20 % iron. The slag from the third stage has a very low in tin content and can be discarded.. The tin produced in the second stage and third stage is subjected to liquation refining in order to eliminate iron.

Since the tin concentrate obtained after gravity separation are fine in size , a reverberatory furnace is used for smelting purposes. In a conventional reverberatory furnace process, for the primary smelting operation, the quantity of coke and flux is carefully adjusted so as to produce an almost neutral slag (acid/basic ratio of 1). The normal composition of this slag is SiO_2 - 35 %, CaO - 30 %, FeO - 15 %, SnO_2 - 20 %. The tin in this stage is subsequently recovered from the slag. The tin oxide content in the first slag is deliberately kept high in order to obtain tin with a very high degree of purity (99 %). The slag is subsequently smelted, additional flux (limestone) , coal and iron scrap being employed as the ingredients. Iron scrap facilitates the reduction of tin oxide present in the slag.

The second slag, which contains a much smaller amount of tin than the first slag, is also smelted to recover its tin content.

2. Rotary furnace smelting

It achieves a higher efficiency of tin recovery. In rotary furnace smelting, after the first smelting, the slag produced is reduced and the tin is volatilised either in the form of SnS (when sufficient sulphur is present) or in the form of SnO (in the absence of sufficient sulphur). The tin rich fumes are transported to the first smelting stage and the cleaned slag produced after reduction is discarded.

2.2.5 Refining of tin

There are mainly two methods of refining of tin. , namely

1. Pyrometallurgical refining or Fire- refining
2. Electrolytic refining

2.2.5.1 Fire- refining of tin

In this process, the ultimate recovery of tin is more than 85 %. The fire refining process is carried out in a single container, called kettle and the entire operation takes about 40 hours.

Principle : several common impurities form intermetallic compounds with each other or with tin. Some of these are;

Ca_3Sn (675°C), FeAs (1030°C), Cu_3As (825°C), Sn_3As_2 (596°C), Fe_2As (919°C)

Cu_2Sb (585°C), FeSb_2 (726°C), and FeSn_2 (very high)

If impure liquid tin is cooled to a temperature just above the melting point of Sn (232°C), these intermetallic compounds which have melting points higher than that of tin, separate out and floats as tiny crystals. This proces is known as liquation. Liquation is a technique for separating constituents of an ore or metal or an alloy by partial melting. When the material is heated to a temperature where one of the constituents melts and the other remains solid, the liquid constituent can be drained off.

The flowsheet for the pyrometallurgical refining of tin is given below.

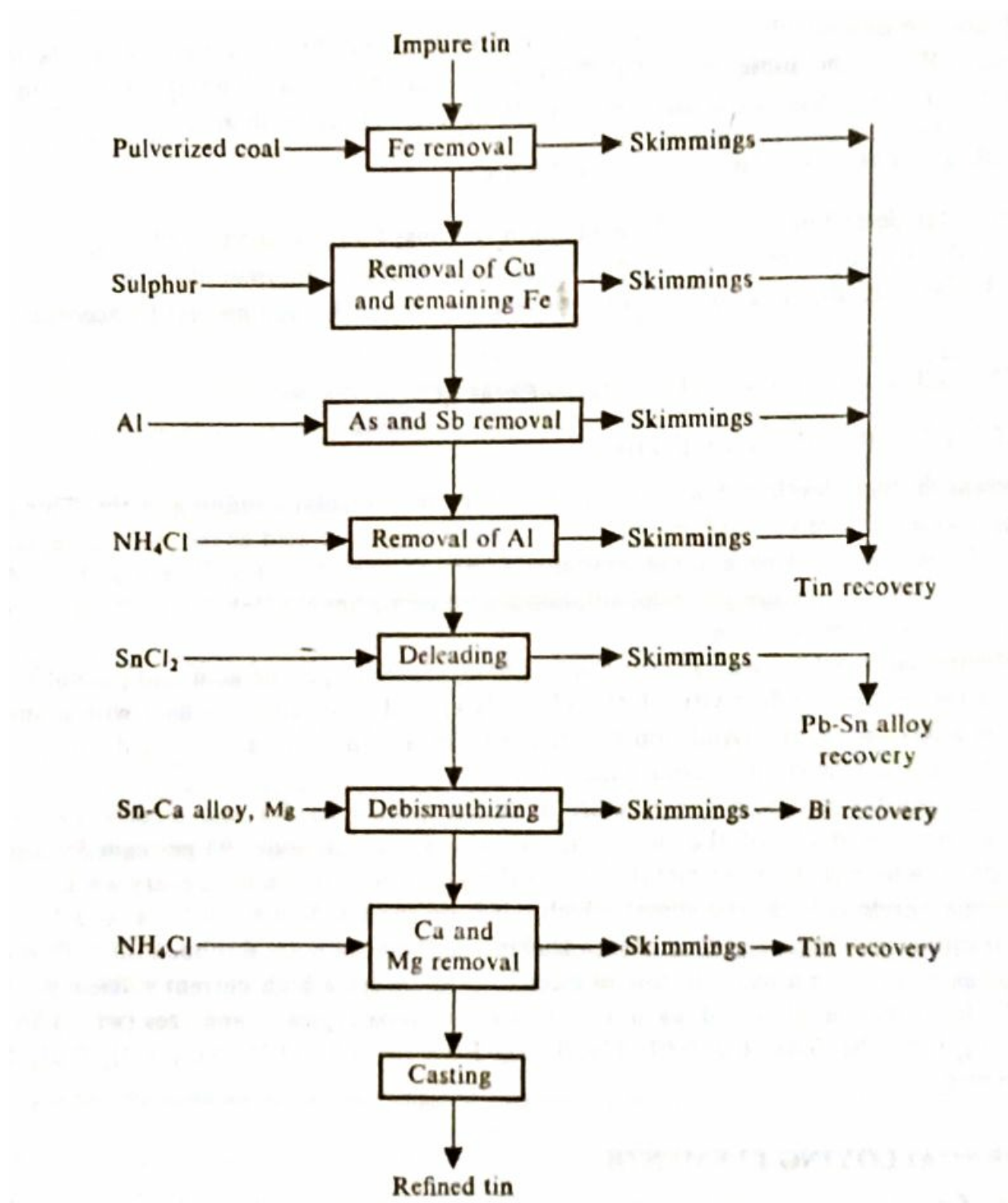
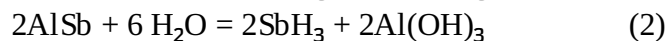
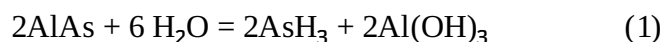


Fig.2.6 Flowsheet for pyrometallurgical refining of tin.

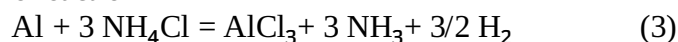
Pulverized coal is added to facilitate liquation. Light and rising particles of coal carry with them the crystallites of the intermetallic compounds to the surface.

Liquation at 550°C in the presence of pulverized coal eliminates almost 99.8% iron from the tin metal and yields a dry dross containing 25% tin.

Sulphur is added to the liquid tin metal in order to remove copper by the formation of copper sulphide. As and Sb are removed by the addition of aluminium in the form of AlAs and AlSb. These compounds have high melting points i.e 1720°C and 1070°C respectively and also insoluble in tin. The reactions are



NH₄Cl is added with tin after Al treatment to remove surplus aluminium according to the reaction



Pulverised coal facilitates the separation of the precipitate AlCl₃. Deleading of the tin by SnCl₂ is based on the reaction



Where Pb and Sn denote Pb and Sn in solution. The dross produced as a result of reaction (4) contains both PbCl₂ and some unreacted SnCl₂, which are mutually soluble. A lead- tin alloy is recovered from this PbCl₂- SnCl₂ mixture by melting it in contact with zinc as



After deleading, tin metal is treated for bismuth removal by adding Ca and Mg.

2.2.5.2 Electrolytic refining of tin

In this process, the electrolyte for the electrolysis consists of a mixture of sulphuric acid and phenol sulphonic acid, This method is applicable when tin contains large amounts impurities such as Pb, Sn, Sb and As. The cell operates at room temperature at a cell voltage of 0.3 and a current efficiency of 85 per cent. The anode is made of impure metal, the cathode is the pure metal sheets. The purity of tin obtained by this method is 99.95 %.

Extraction of metals from sulphide ores

3.1 Pyrometallurgical Extraction of Copper

3.1.1 Properties and uses of copper

The most important properties and common uses or applications of copper are as follows:

- (i) Copper has high electrical and thermal conductivities.
- (ii) It has adequate mechanical properties such as strength, hardness etc.
- (iii) It has high resistance to corrosion.
- (iv) It is soft, malleable and ductile.
- (v) It is used in power transmission lines, wiring, cables, and busbars.
- (vi) It is used in electrical applications such as high conducting wires and motors.
- (vii) It is used as electrodes, heat exchangers, spark plugs, cooking utensils, architectural applications

3.1.2 Minerals or ores of copper:

Some common minerals or ores of copper are;

Chalcopyrite, CuFeS_2

Chalcocite, Cu_2S

Bornite, Cu_5FeS_4

Covellite, CuS

Enargite, CuAsS_4

Malachite, $\text{CuCO}_3 \cdot \text{Cu(OH)}_2$

Azurite, $2\text{CuCO}_3 \cdot \text{Cu(OH)}_2$

Cuprite, Cu_2O

Chrysocolla, $\text{CuSiO}_3 \cdot 2\text{H}_2\text{O}$

Among these minerals, only the chalcopyrite is commercially important ore. The copper content of the ore varies from 0.5 to 2.0 per cent.



3.1.3 Extraction of copper from sulphide ores

The steps involved in the extraction of copper from sulphide ores namely concentration, roasting, smelting, converting and refining are described below. The flowsheet of pyrometallurgical extraction of copper is shown in fig.3.1. The left side of the flowsheet indicates the **Conventional route** whereas the right side the **Newer route**

3.1.3.1 Conventional route

1. Concentration

A naturally occurring sulphide ore normally contains 0.5 - 2.0 % copper. The ore is subjected to crushing and grinding to liberate the sulphide grains from the gangue. Next, the ground ore is subjected to froth floatation. During froth floatation, the pH is controlled by the addition of CaO and a xanthate reagent is used as collector.

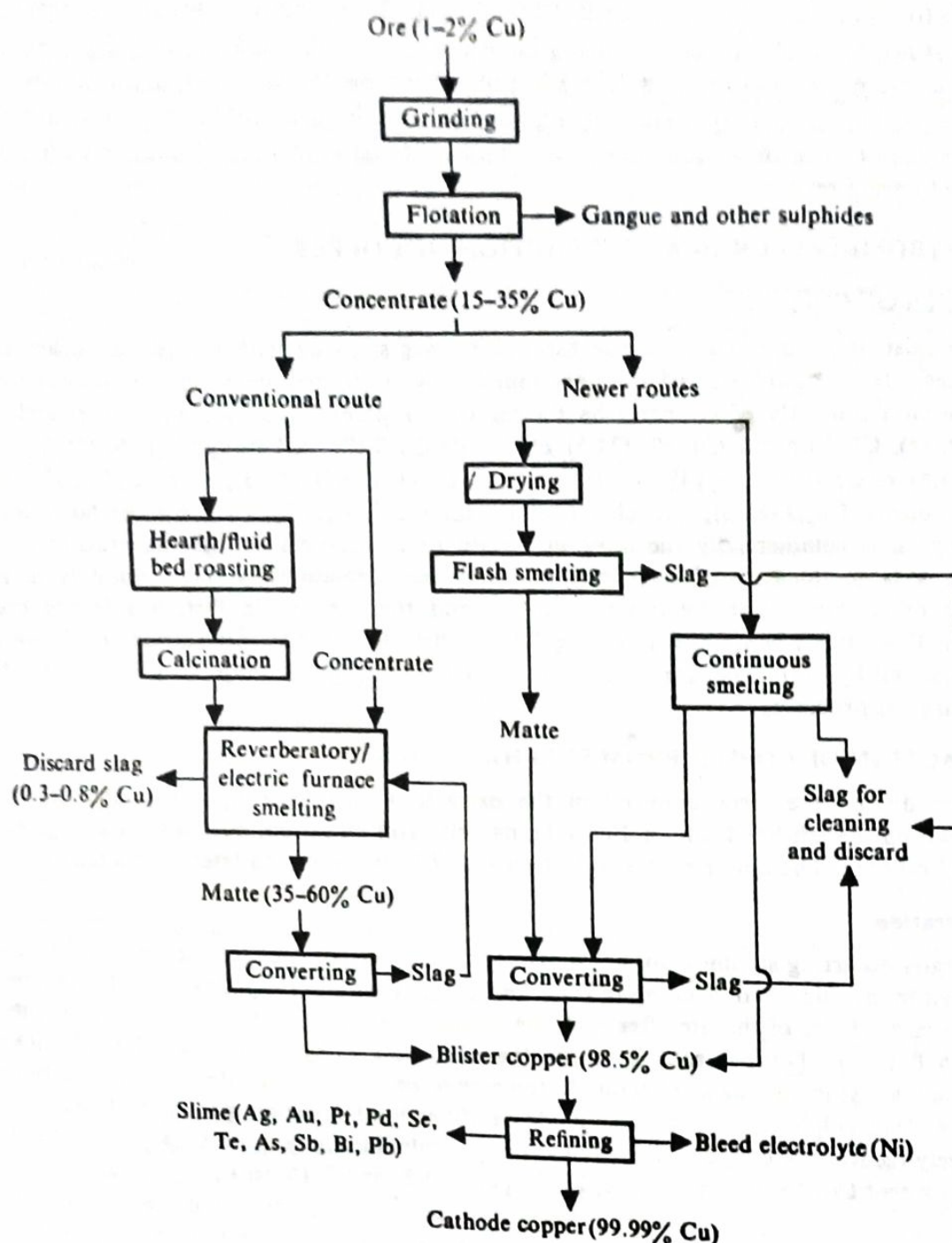


Fig.3.1 Flowsheet of pyrometallurgical extraction of copper.
The copper sulphide concentrate thus produced contains 15 -35 % Cu, 15 -35 %Fe, 25 - 35 % S and 3 - 15 % gangue. The fig.3.2 below shows the froth flotation machines.

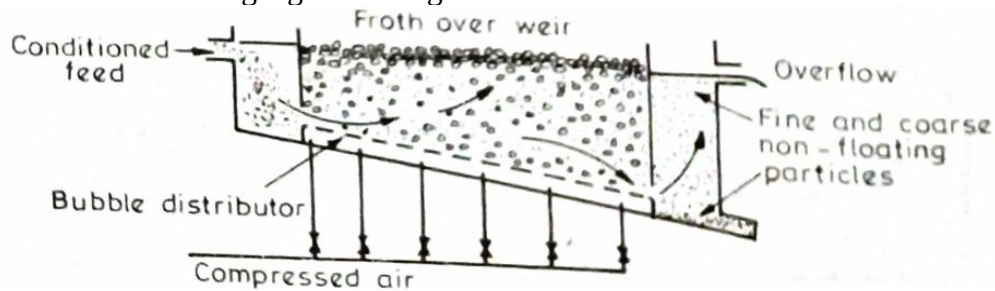


Fig.3.2. Concentration of ore in froth flotation method.

2. Roasting

The purpose of roasting is to oxidise the iron sulphide present in the copper sulphide concentrate in order to facilitate its removal in the form of slag in the next stage (smelting). The extent of roasting determines the copper grade of the matte i.e the percentage of copper in the matte, produced in the smelting stage. For concentrates with a high iron sulphide content and a low copper sulphide content (< 25 % Cu) roasting is necessary, but for high grade copper concentrates (> 30 % Cu), roasting is not necessary.

In conventional plants, roasting is carried out in multiple hearth roasters with the feed traveling from one hearth to the other (shown in fig. 3.3) . The temperature of each hearth is gradually increased by fuel firing so that the charge gets heated up from the room temperature to about 550°C. In the newer plants, fluidised bed roasters (shown in fig.3.4.), operating at about 550°C, are employed.

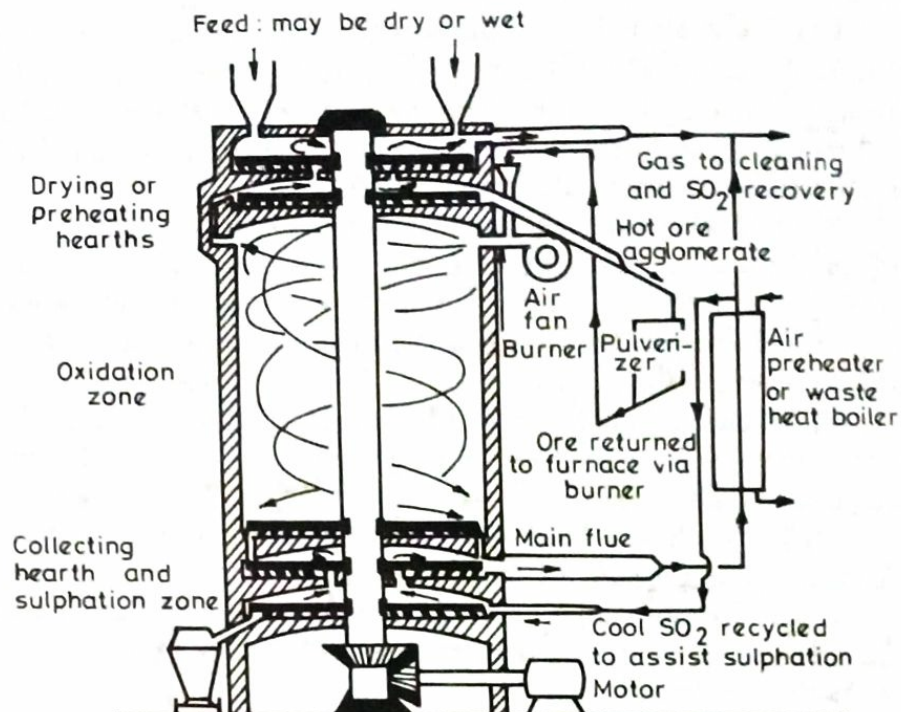
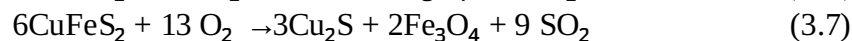
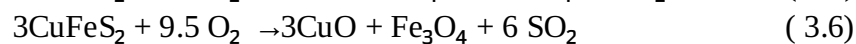
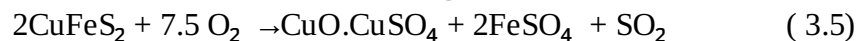
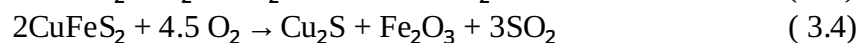
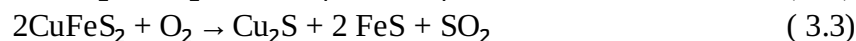
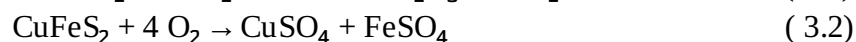
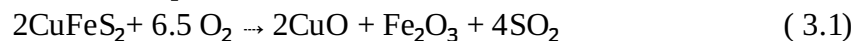


Fig.3.3 Multiple hearth roaster

During roasting, several reactions take place simultaneously. The roasted calcine consists of sulphides of copper and iron, oxides of iron, mixed sulphates copper and

iron. The important reactions are as follows:



The roasted calcine is transferred to the smelting furnace in the hot condition for easy separation and the subsequent recovery of copper.

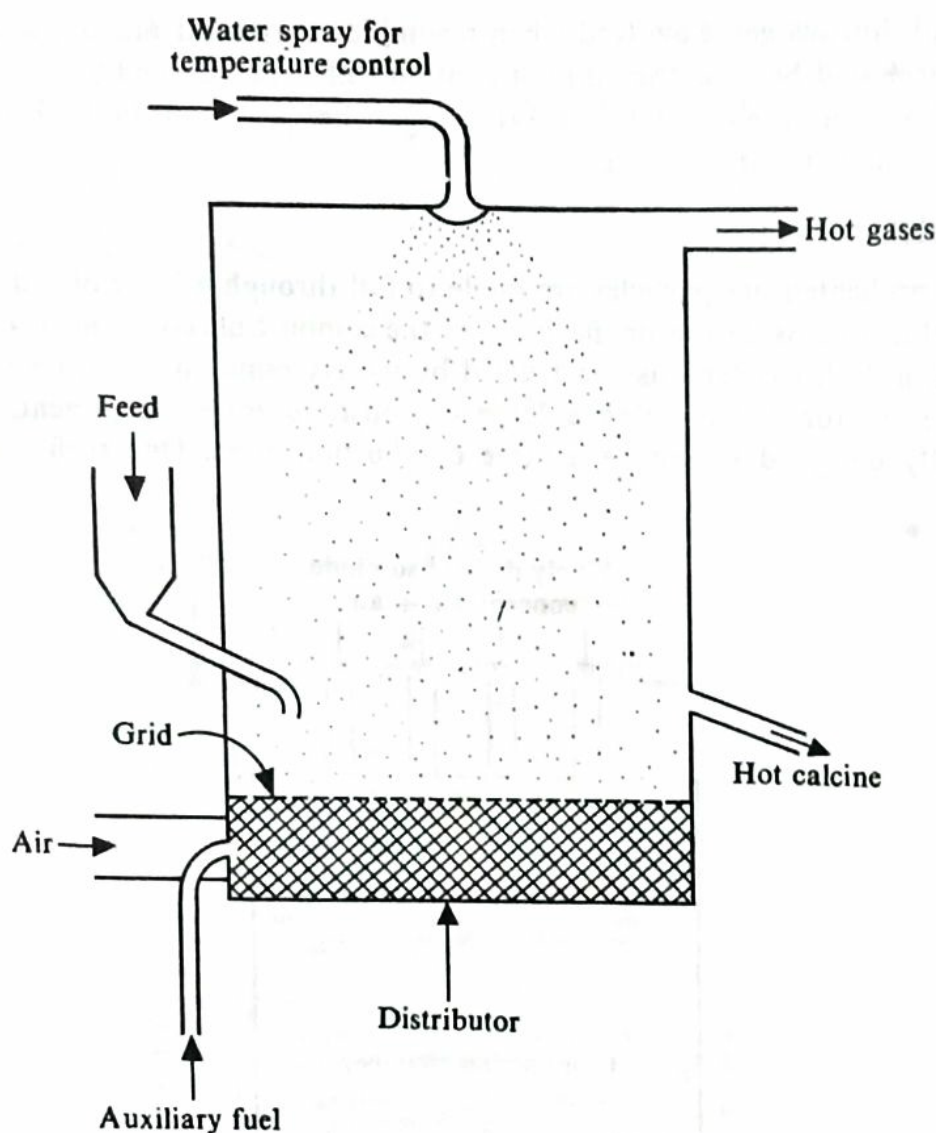


Fig.3.4 Fluidised bed roaster

3. Smelting

Smelting seeks to separate the metal sulphide in an ore, concentrate or calcine from the gangue. This can be achieved by smelting the charge with a suitable flux at a temperature of about 1250°C.

During smelting, two layers of liquid are formed, namely;

i) An upper slag layer (gangue and flux)

Sp. gr.- 2.8 to 3.8 gm/cm³

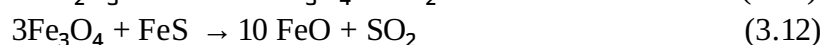
ii) Lower matte layer (metal sulphides)

Sp.gr. -5.0 to 5.5 gm/cm³

During smelting, both the copper sulphide minerals and the slag melt. Other exchange reactions take place between the oxides and sulphates of copper and the iron sulphide present. The reactions being

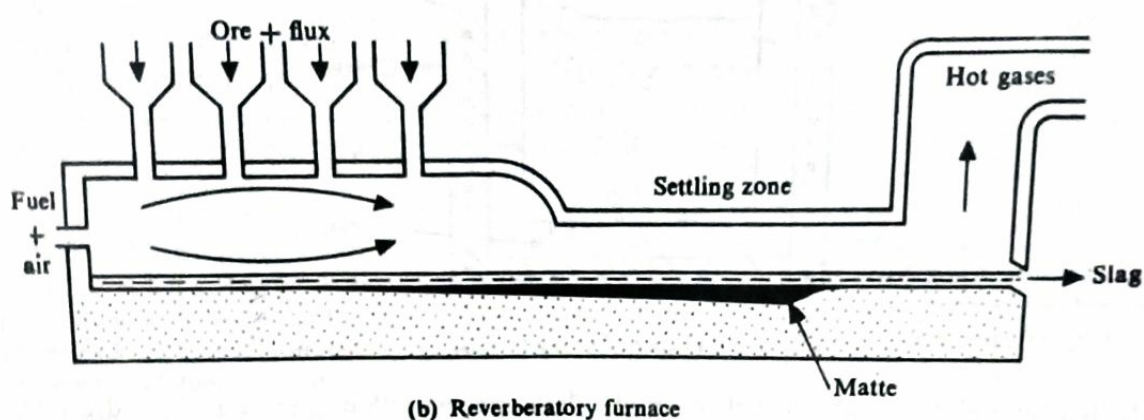
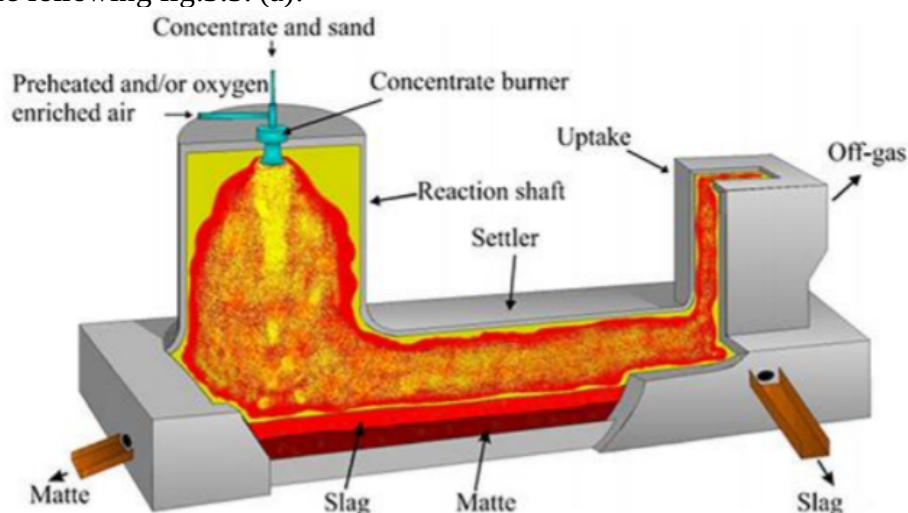


The unoxidised iron sulphide reduces the higher oxides of iron to ferrous oxide as per the reactions,



The resultant FeO and Fe₃O₄ react with flux (silica and lime) to form a slag.

The smelting operation is carried out in reverberatory furnaces fired with either coal or oil, shown in the fig.3.5 (b) below. It is also carried out in electric furnace , shown in the following fig.3.5. (a).



(b) Reverberatory furnace

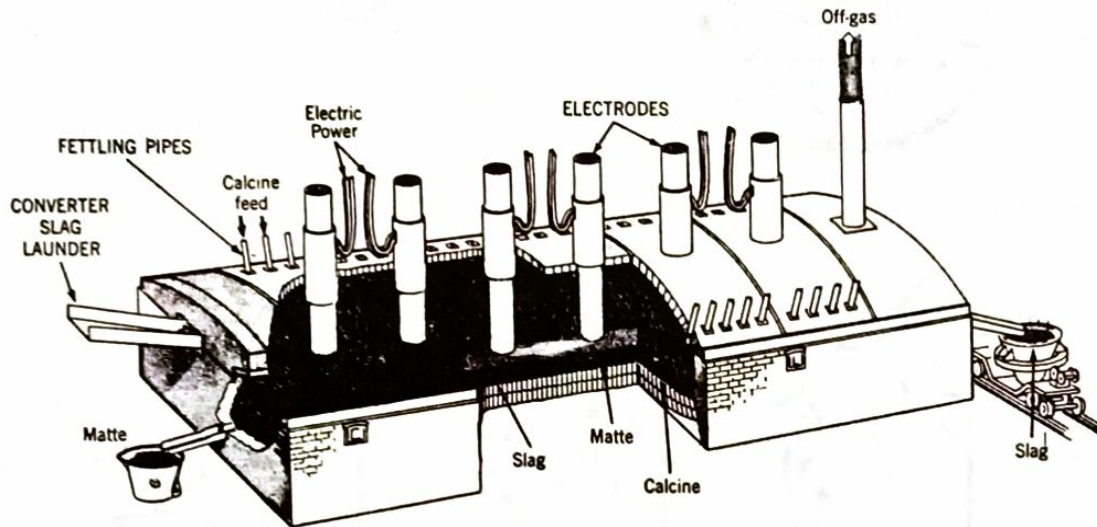
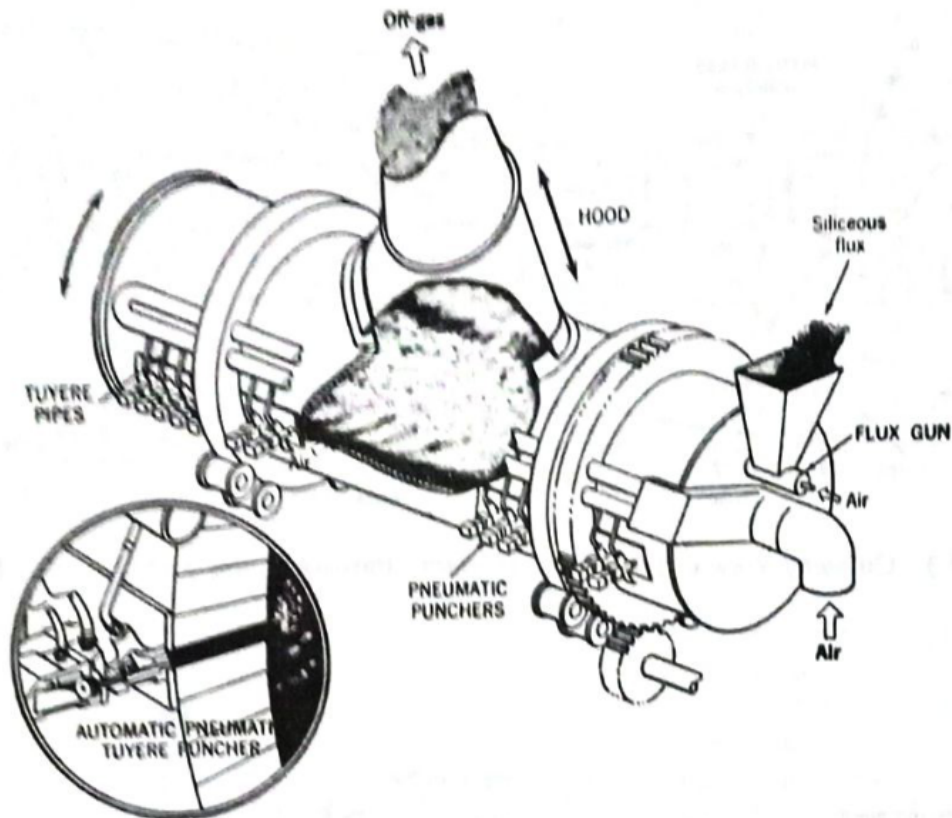


Fig.3.5 (a) Cutaway view of submerged arc electric furnace.

4. Converting

The purpose of converting is to remove iron, sulphur and other impurities from matte. Converting is carried out in a side blown converter, which is a cylindrical vessel, 4 m in diameter and 9 m in length. It is lined with chrome - magnesite refractory bricks, as shown in the following figure. In operation, the molten matte is charged into the converter and air or oxygen - enriched air is injected into the molten matte through tuyeres. The product of the converter is *blister* copper and slag.



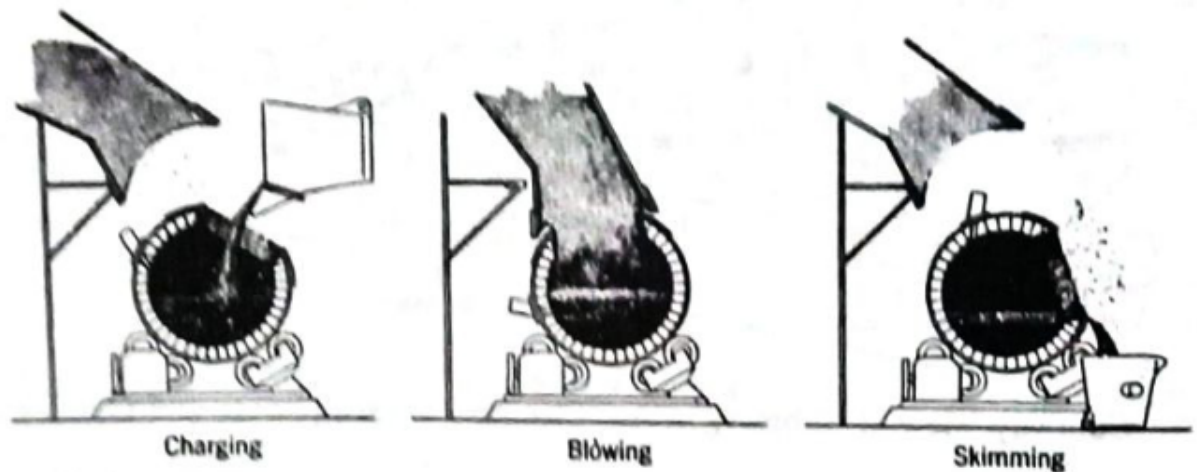


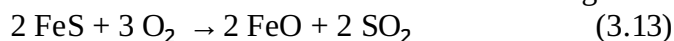
Fig. 3.6 Converter operation

Hence there is two distinct stages in converter process, namely;

1. Slagging stage, and
2. Blister formation stage.

1. Slagging stage

In slagging stage, the iron sulphide is oxidised and the oxide is slagged out by the addition of siliceous flux. The reactions being

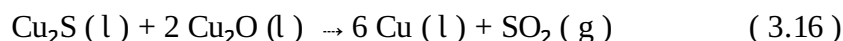
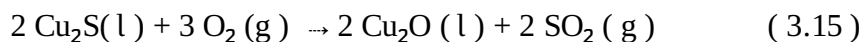


Part of FeO is also oxidised to magnetite. Slagging is carried out in two stages - by first adding freshly obtained matte to the converter and blowing air. Next, the supernatant slag formed is skimmed off by tilting the converter, since the molten slag and matte are immiscible. The composition of slag formed is :

Cu - 2.0 - 9.0 %, Fe - 40.0 -50 .0 %,SiO₂ - 20.0 - 30.0 %, & CaO + MgO - 1.0 -5.0 %

2. Blister formation stage

After slagging has been completed my , the converter essentially contains Cu₂S, which is called white metal because of its appearance. In blister formation stage, this Cu₂S is oxidised to form copper by the combination of the following reactions



The overall reaction is



The blister copper produced has 98.5 % Cu, 0.02 -0.05 % S and 0.2 - 0.5 % dissolved oxygen

3.1.4 Refining

It is the final step in copper extraction. The purposes of refining are to obtain the metal in purer form and to recover the valuable precious metals from the anode slime.

The refining of the blister copper is done by

- (i) Fire refining or poling and
- (ii) Electrolytic refining

(i) Fire refining or poling

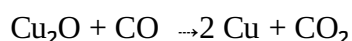
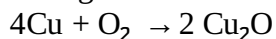
The main purpose of fire refining is to remove sulphur from liquid blister copper as SO_2 by oxidation with air and to eliminate oxygen by introducing hydrocarbons. Copper of around 99.7 % purity is obtained and the fire refined copper is known as tough pitch. Fire refining is carried out in a reverberatory furnace or in a rotary type refining furnace. The fuel used is fuel oil or natural gas pulverized coal. In the refining furnace, the surface of the blister copper is oxidised at frequent intervals. The furnace doors are kept open in order to allow a constant mild blast of air, shown in the following figure.



Fig. 3.7. Fire refining of copper.

As the result of this slow oxidation process, impurities such as S, Fe, Se, and Zn are oxidised and the solid oxides rise to the top where they are skimmed off. The time taken for the whole period of refining is 12 - 16 hours. Normally the process is stopped when the level of Cu_2O in the bulk of the metal reaches 6 % . At this stage, almost all sulphur is eliminated from the metal.

Once the oxidation is complete, the oxidised copper is reduced to metallic copper by reducing gases such as hydrocarbons of the wood. (Poles of green wood are stirred during reduction. The reactions being



(ii) Electrolytic refining

Fire- refined copper can be further refined by electrolysis. An electrolytic refining tank or cell is made of either concrete or wood and is well insulated (shown in the figure). Each tank is 3 - 5 m long, 1 - 1.1 m wide and 1 - 1.3 m deep.

Fire- refined copper is cast in the form of an anode. The cathode is made of a pure copper sheet . The electrolyte consists of copper sulphate solution with some addition agents such as glue and alcohol. The electrolysis is carried out at a temperature of 50 - 60°C using current density of about 200 A/m^2 . The purity is 99.98 %. During electrolysis, copper is transferred from the crude anode to the pure cathode. Impurities

(1) Cu^{2+} ions (from copper sulphate solution) go to the cathode (negative electrode), where they are reduced to copper, which gets deposited on the cathode.

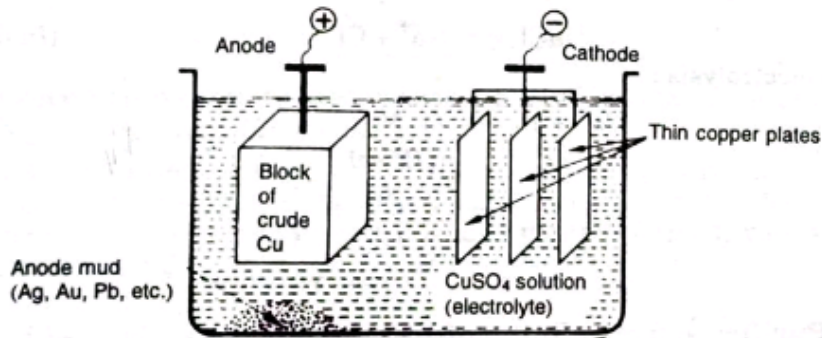
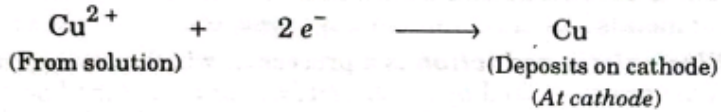
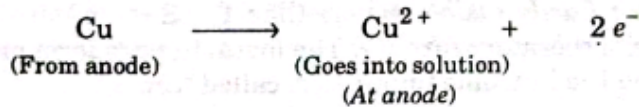


Fig. 4. Electrolytic refining of copper.

(2) Copper (of impure anode) forms copper ions, and these go into solution of electrolyte.



Thus, the net result is transfer of pure copper from anode to the cathode. Impurities like zinc, iron, etc., go into solution ; while noble impurities like silver, gold, etc., are left behind as anode mud.

such as Fe, Ni, Co, Se, Te go into solution and the precious metals collect below the anode as anode slime. The approximate composition of anode slime is as follows:

Cu = 10 - 40 %

$$A_g = 8500 - 14000 \text{ gm/ton}$$

Au = 250 - 1700 gm/ton

Pb = 2 - 20 %

$$A_s = 0.5 - 5 \%$$

Te = 0 - 4 %

Ni = 0 - 25 %

S = 2 - 10 %

Fe = 0.5 - 2 %

and Se = 0 - 25 %

3.1.5 Recovery of precious metals and chalcogenides from anode slime

The following figure shows the flowsheet for the recovery of precious metals and chalcogenides from the anode slime.

To remove Cu, the slime is first treated with a dilute acid and sulphated using H_2SO_4 at about 200°C followed by leaching. The copper solution contains tellurium which is cemented with active copper and the copper telluride formed is sent for tellurium recovery. The decoppered slime is next roasted at about 700°C by indirect heating. The selenium is volatilised and scrubbed in water to obtain a strong selenic acid solution from which Se is recovered by gaseous reduction with SO_2 . The decopperised selenium free slime is then smelted along with soda ash, silica and return slags as fluxes to produce a silver - rich alloy.

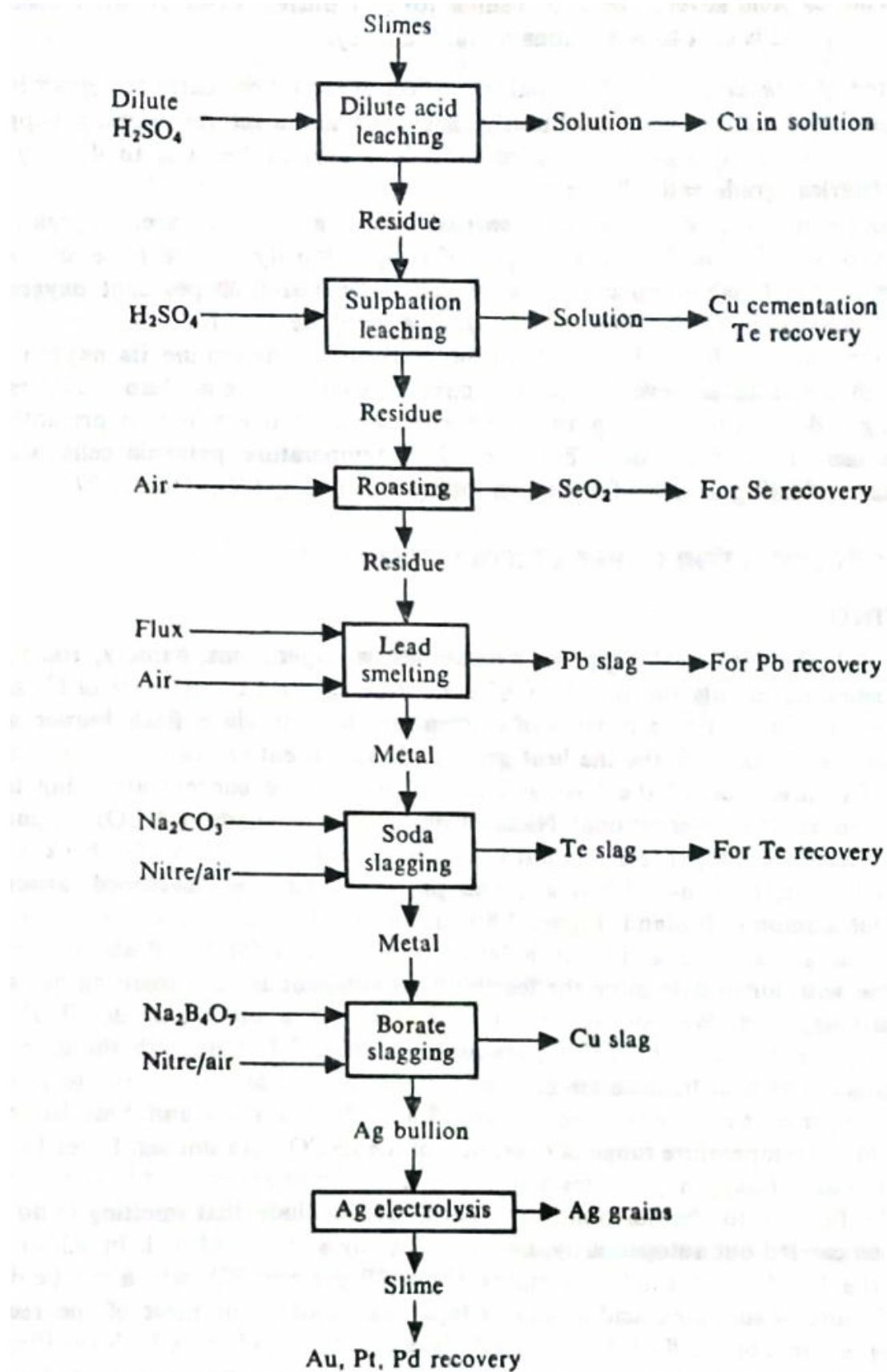


Fig.3.9 Recovery of precious metals and chalcogenides from anode slime .

This alloy is oxidised to transfer the lead into the slag. During this stage, As, Bi and Sb are eliminated. If the slag rich in Pb, then it is sent to a lead refinery. The silver - rich alloy, after lead removal, is oxidised in the presence of soda ash to remove any residual tellurium and further oxidised in the presence of borax to remove any residual copper. At this stage, the alloy contains more than 90 % Pb, the remainder essentially being Au, Pt and Pd.

3.1.3.2 Newer route

Flash smelting :

Flash smelting is a process that combines the operations of flash roasting and smelting. The object is to burn the concentrate with oxygen or preheated air blown in with the concentrate. Sufficient heat is generated to form a matte and a slag which settle into the hearth. The following figure shows the flash smelter.

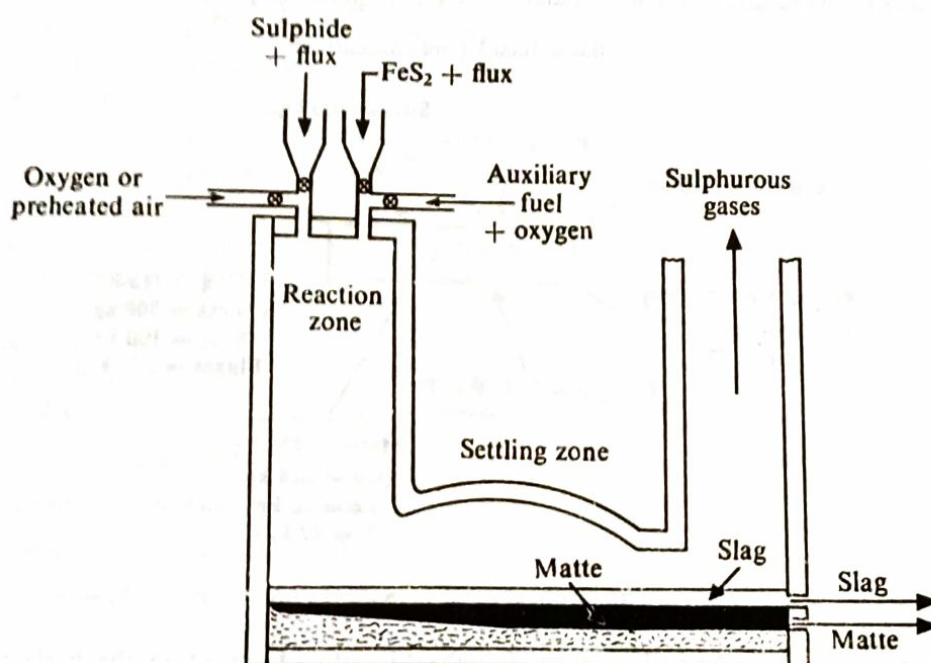
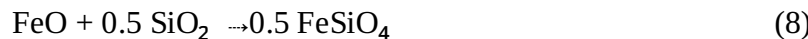
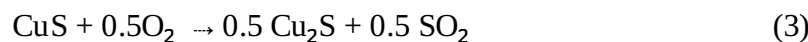
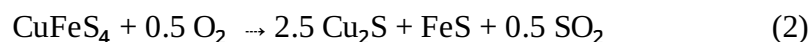
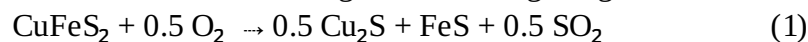


Fig.3.10. Flash smelting furnace

In flash smelting, enriched hot air or pure oxygen or a mixture of oxygen and hot air is blown via a flash burner into the dried concentrate of copper and flux. The main reactions that occur during flash smelting are given below:



The gases coming out of the furnace are very rich in SO_2 due to high combustion rate and can be used for the manufacture of sulphuric acid. The matte produced in flash smelting contains 45 -65 % Cu depending upon the quantity of fuel used and the degree of oxygen enrichment employed. The slag has a copper content of 0.8 - 1.5 %. The reactions in the flash smelting furnaces produce copper matte, iron oxides and sulphur dioxide. The reacted particles fall into a bath at the bottom of the furnace, whereas the iron oxide reacts with fluxes such as silica and limestone to form slag. In most cases, the slag can be discarded. The sulphur dioxide produced by flash smelting is captured in a sulphuric acid plant to manufacture sulphuric acid.

3.2 Pyrometallurgical extraction of Lead

3.2.1 Minerals of lead

In nature lead exists in the form of sulphides, oxides, or chlorides. The minerals of lead are

1. Galena- PbS
2. Cerrusite- PbCO_3
3. Anglesite- PbSO_4
4. Bonlangerite- $\text{Pb}_5\text{Sb}_4\text{S}_{11}$
5. Pyromorphite- $\text{Pb}_5(\text{PO}_4)_3\text{Cl}$
6. Mimetite- $\text{Pb}_5(\text{AsO}_4)_3\text{Cl}$
7. Vanadinite- $\text{Pb}_5(\text{VO}_4)_3\text{Cl}$

Among these the most common ore is galena (PbS).



Galena

3.2.2 Properties and uses of lead

The important properties and main uses of lead are as follows:

- (a) It is dull, silvery -grey metal.
- (b) It is soft and easily worked into sheets.
- (c) It has high corrosion resistance.
- (d) It is very malleable, ductile and poor conductor of electricity.
- (e) Lead is used mainly in the manufacture of batteries, cables, pigments, and flexible sheets and pipes (as shown in fig.3.11.)
- (f) Lead alloys are used in the manufacture of solders, printing metals and bearings.
- (g) It is used as a composite material in structural engineering.
- (h) It is used in architecture for roofing and stained glass windows.

3.2.3 Extraction of lead from lead ore

The various steps involved in the treatment of lead ore for its extraction are as follows:

1. Concentration
2. Roasting and sintering
3. Smelting
4. Refining

These steps are discussed in detail below:

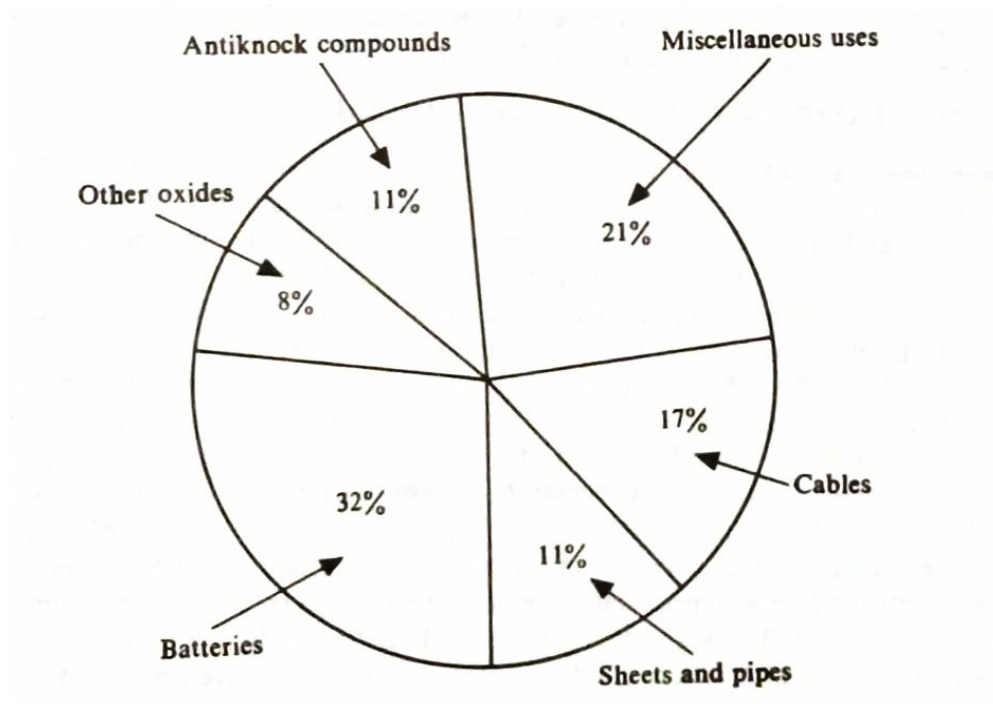


Fig. 3.11 Various uses of Lead

1. Concentration of the lead ore

The most common ores contain galena (PbS) associated with ZnS , FeS , CuS , PbCO_3 , and several precious metals. The lead sulphide along with the precious metals is first separated from the other sulphides and is concentrated by differential flotation method. This concentrate may contain 60 - 80 % lead. The process of flotation is shown in the figure

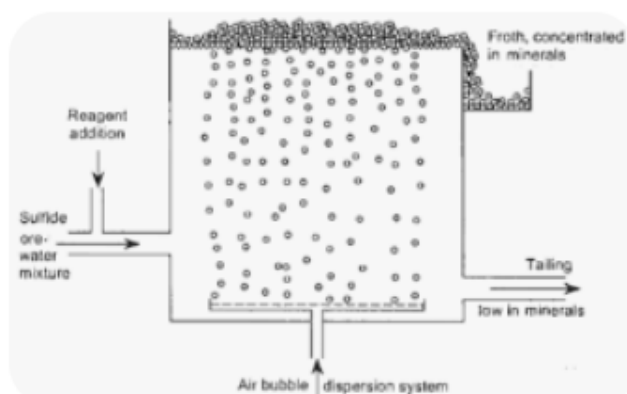


Fig. 3.12. Froth flotation process of lead ore

2. Roasting and Sintering

The concentrate after the differential flotation is roasted in Dwight Lloyd sintering machine to form its oxide. During roasting the temperature is controlled. In roasting, most of the sulphur is eliminated and produces a sinter of adequate strength and porosity for the subsequent blast furnace operation. Roasting operation is carried out at a temperature of about 800°C. The following figure shows the Dwight Lloyd sintering machine.

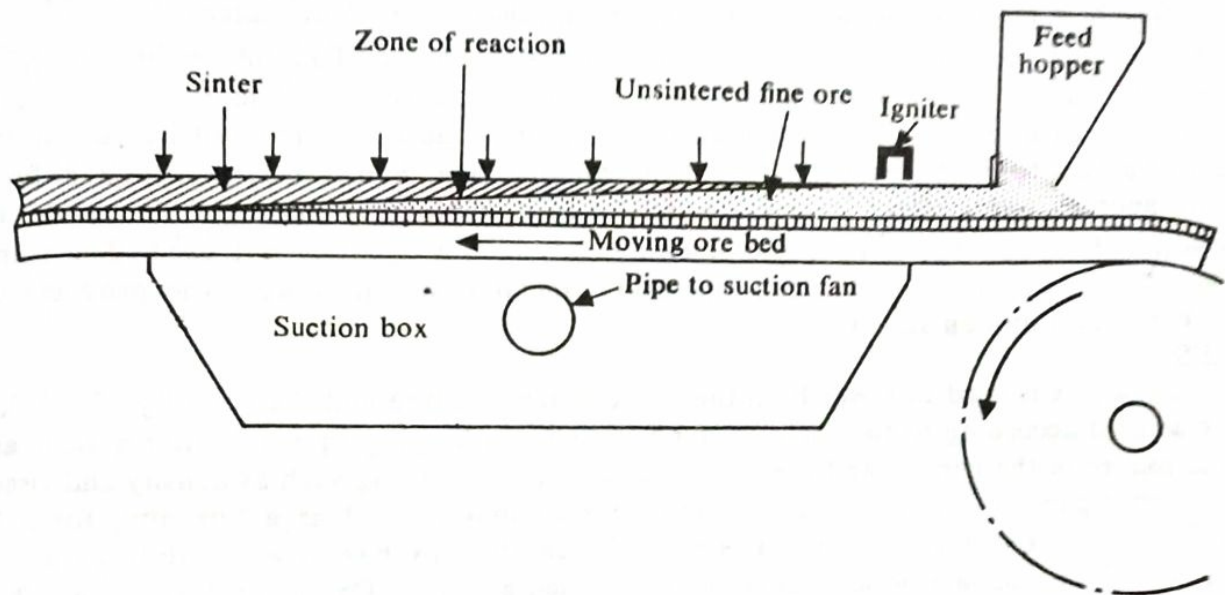
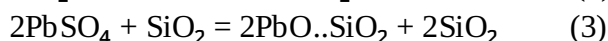
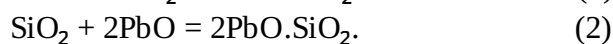


Fig.3.13 Dwight Lloyd sintering machine

The reactions that take place during roasting and sintering are given below:



A Dwight Lloyd sintering machine is shown in the above figure. In this machine, both roasting and sintering are carried out simultaneously. It consists of an endless steel belt capable of moving on rollers. The concentrated ore, mixed with a little coke and lime is slowly dropped on the belt. When the belt moves, it carries the ore and passes under a fire box where the ore is ignited. Burning of ore is then subjected to a downward movement of the air by means of a suction box under the belt. The ore continues to move on burning till it topples as the belt turns over the roller.

3. Smelting

The sinter produced by the sintering machine is reduced to the metal in a lead blast furnace (shown in the figure). The lead blast furnace for smelting is a small furnace with total height of around 8 m, top diameter 3 m, crucible depth 3/4 m and stack height 5 m. The stack is a vertical shaft. The temperature at the bosh is around 1200°C. The bosh is water cooled to keep the temperature low. A cold blast of air is blown in through 15 - 20 tuyeres around the bosh to oxidise the carbon charged with the sinter which produces the required heat. At the top of the furnace, either bag

houses or electrostatic precipitators are provided to recover the lead fumes from the outgoing gases

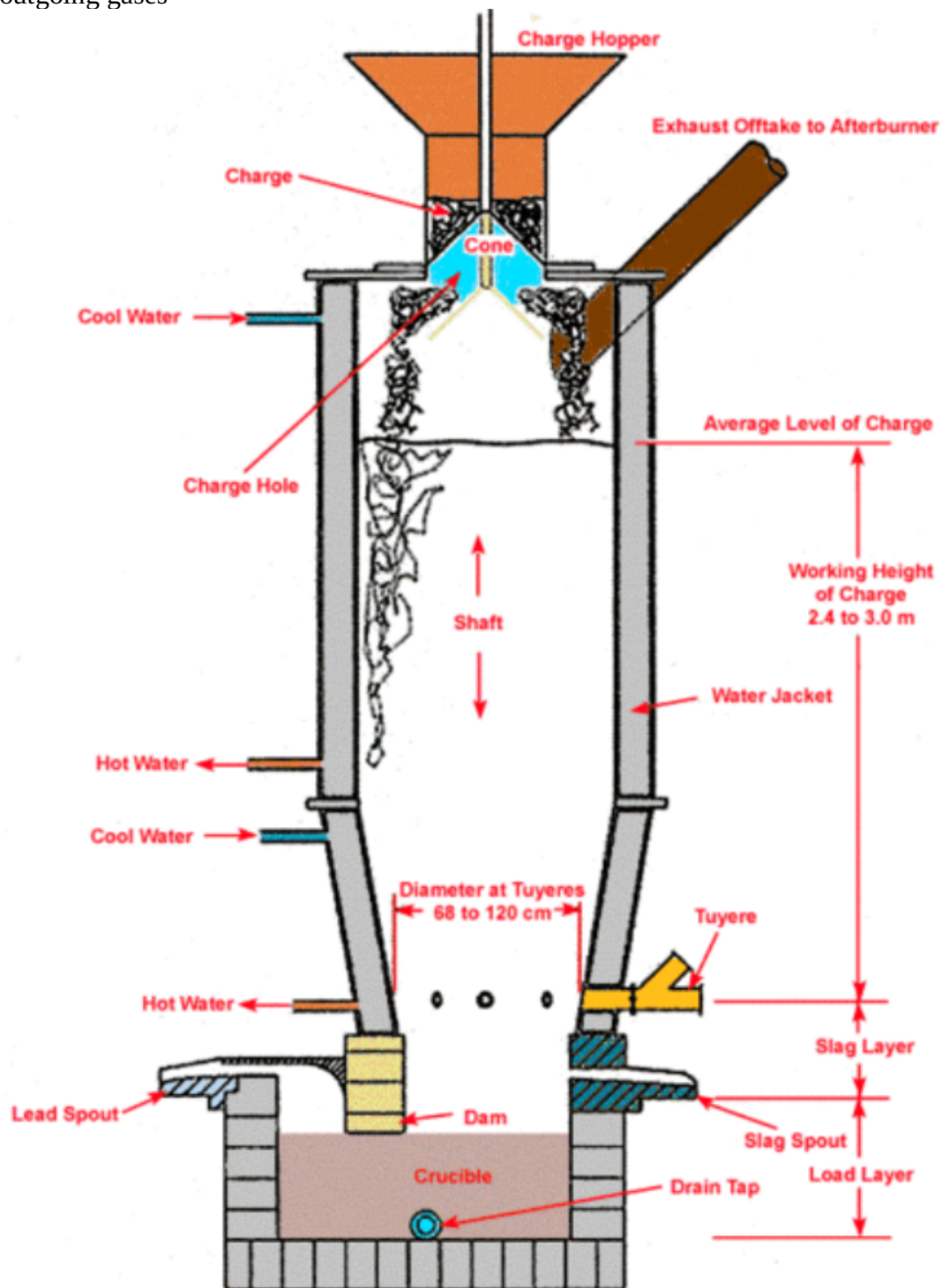


Fig. 3.14 Blast furnace for smelting lead
The following figure shows the flowsheet for the lead smelting in blast furnace.

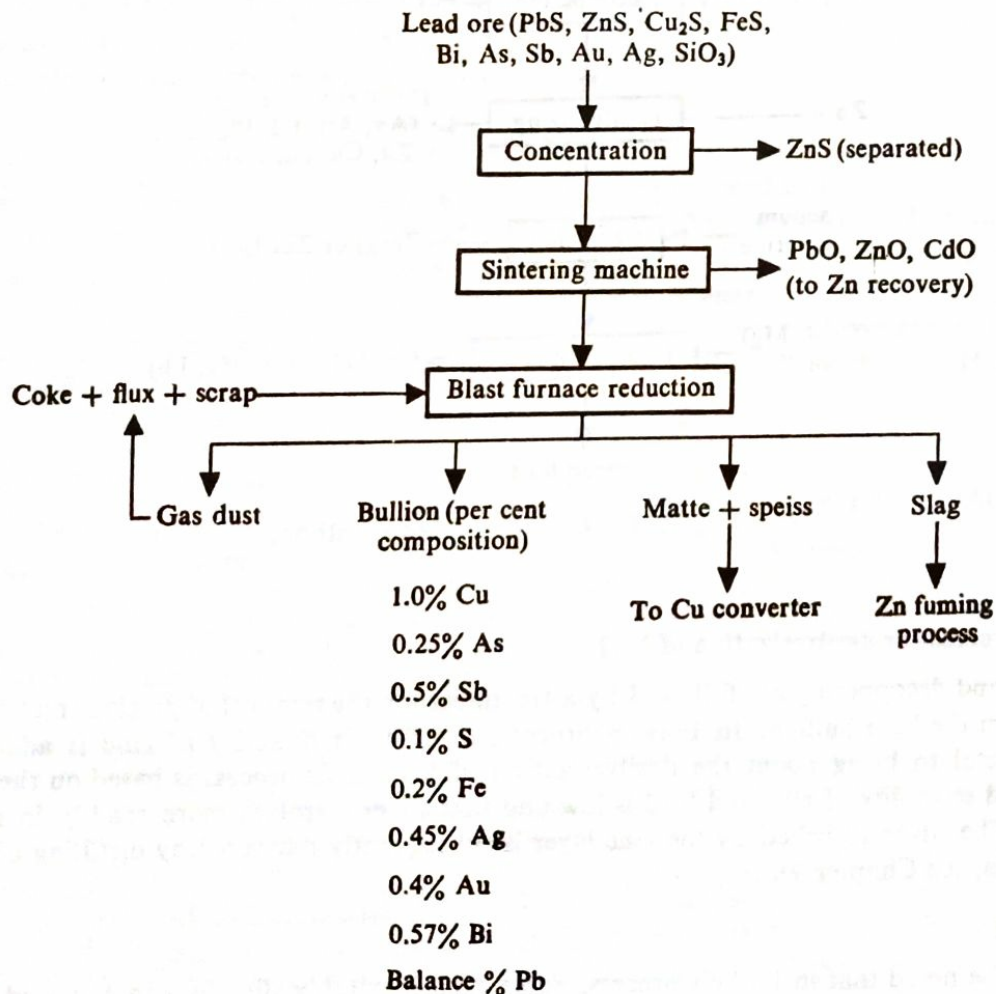


Fig.3.15 Flowsheet for lead smelting in blast furnace.

The charge for the blast furnace consists of

- (i) Sinter
- (ii) Coke (10 %)
- (iii) Limestone (5 - 10 %)
- (iv) Iron scrap (1 %) and
- (v) Quartz

A lead blast furnace produces 200 - 600 tons of lead per day. Limestone and quartz are added to form the slag. The normal composition of the slag is

SiO₂ : 18 - 35 %

Fe : 30 -38 %

CaO : 19 - 20 %

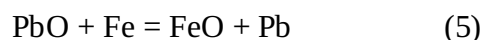
Pb : 2 - 4 %

The use of iron scrap serve the following functions:

1. It reduces any residual PbS left unroasted in the sintering machine.
2. It combines with silica to form 2FeO.SiO₂, which lowers the melting point of the slag.
3. It keeps lead losses low.

The smelting reactions being





The smelting process produces four distinct layers as

First layer - Slag (sp. gr. is kept around 3.6)

Second layer - Matte (containing Cu and other elements, sp. gr. - 5.0

Third layer - Speiss (if ' As ' is present in the charge

FeAs_4 + impurities, sp. gr.- 6.0)

Fourth layer - Lead, called base bullion ,(sp.gr. - 11)

3.2.4 Refining of lead

The refining of lead involves the following

(i) Treatment of the base bullion

(ii) Electrolytic refining of lead

(i) Treatment of base bullion:

The lead - rich layer produced in the blast furnace, called as base bullion, is rich in several other valuable elements. Each of these elements is recovered during a series of steps as shown in the following flowsheet.

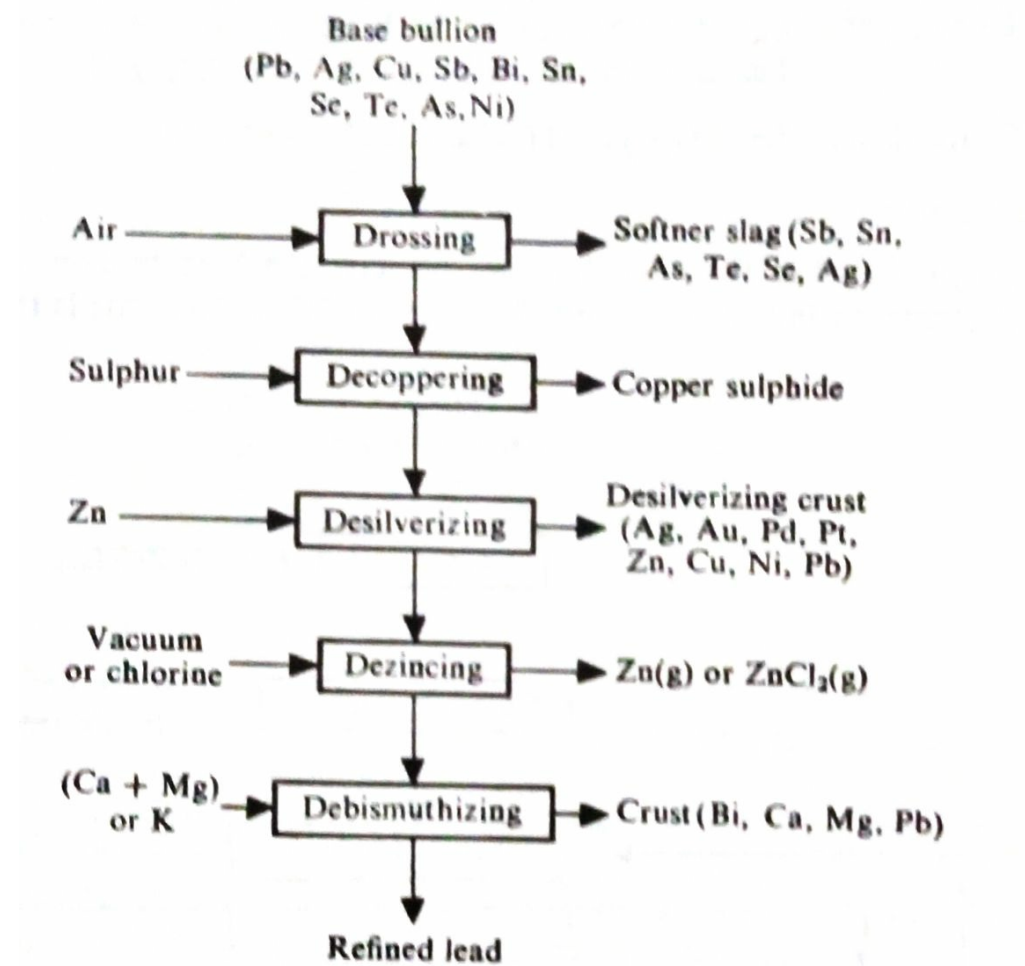


Fig.3.16 Flowsheet for refining of lead bullion.

The various steps are described in detail below

1. Drossing : The base bullion is first subjected to drossing at around 350°C . Drossing is a combination of liquation and oxidation and is usually carried out in a reverberatory furnace. At this temperature, many metals get oxidised and skimmed off. Subsequently the temperature is raised to 500 - 550°C and sulphur is added to eliminate copper in the form of copper sulphide.

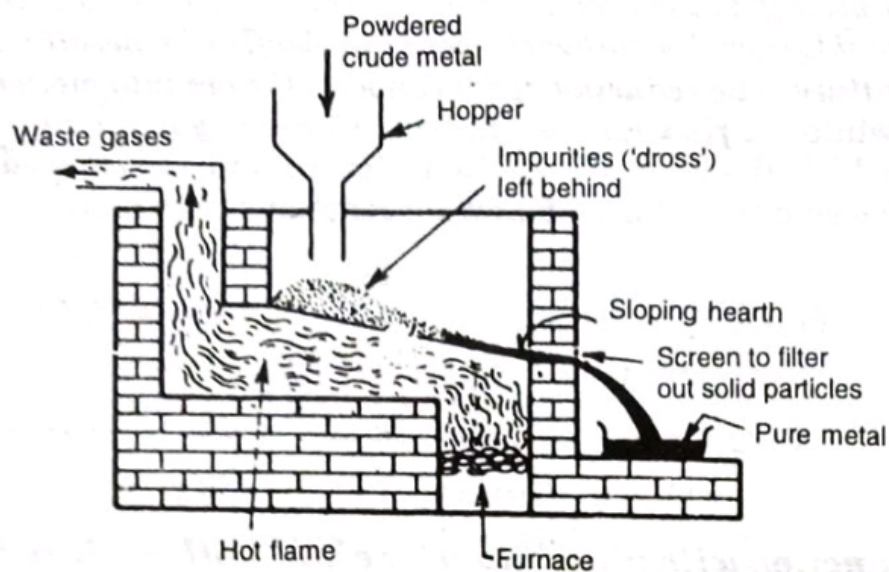


Fig.3.17 Liquation process

2. Desilverising (Parke's process): After drossing, it is treated for the removal of precious metals mainly, silver, from the base bullion. In Parke's process , sufficient quantity of zinc is added to the molten metal to bring about the desilverisation of lead. This process is based on the fact that, the mutual solubility of zinc and lead is low and silver dissolves more readily in zinc than lead. The silver absorbed by the zinc layer is then recovered by distilling off the zinc.

3. Dezincing : In Parke's process, some zinc inevitably dissolves in the lead and vice - versa (lead dissolves 0.6 % zinc and zinc dissolves 1.6 % lead in molten state.) The lead containing zinc is subjected to cupellation, where zinc is removed by oxidation. Zinc can also be separated either by the application of a vacuum or by preferential chlorination with chlorine.

4. Debismuthizing : In the base bullion, if bismuth is present in a large quantity, it can be separated by the addition of an alloy calcium and magnesium which forms an insoluble intermetallic compound with bismuth.

(ii) Electrolytic refining of lead :

The Betts electrolytic process is an industrial process for purification of lead from base bullion , shown in the figure.

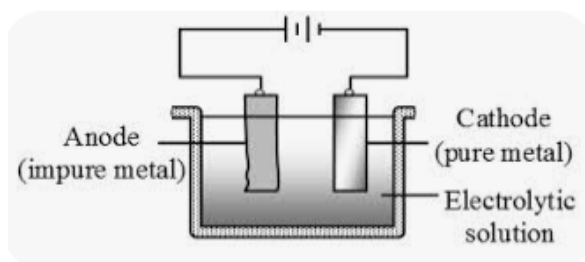


Fig. 3.18 Electrolytic refining process of lead.

This process is used when high purity lead is required, especially for bismuth free lead. The electrolyte for this process is a mixture of lead fluorosilicate and hexafluorosilicic acid operating at 45°C. Cathodes are thin sheets of pure lead and anodes are cast from the impure lead to be purified. A potential of 0.5 V is applied. At the anode, lead dissolves, as do metal impurities that are less noble than lead. Impurities that are more noble than lead such as silver, gold, and bismuth flake from

the anode as it dissolves and settle to the bottom of the vessel as anode mud, pure metallic lead deposits on the cathode, with less noble metals remaining in solution.

3.3 Pyrometallurgical and Hydrometallurgical Extraction of Zinc

3.3.1. Minerals of zinc

Some of the important naturally occurring minerals of zinc are

- (i) Sphalerite or zinc blend - ZnS
- (ii) Zincite - ZnO
- (iii) Franklenite - $\text{ZnO}(\text{Fe, Mn})_2\text{O}_3$
- (iv) Calamine - $\text{Zn}_2(\text{OH})_2\text{SiO}_3$
- (v) Smithsonite - ZnCO_3
- (vi) Hemimorphite - $\text{Zn}_2\text{SiO}_4 \cdot \text{H}_2\text{O}$

Among these , sphalerite is the most common mineral. Zinc ores are usually associated with several compounds, mainly sulphides of Pb, Cu and Cd. Both pyrometallurgical and hydrometallurgical processes of extraction are employed.



Sphalerite

3.3.2 Properties and uses of zinc

1. At room temperature, it remains brittle with a crystalline state.
2. It has good formability.
3. It is used as a protective coating for steel (galvanizing), because it resists atmospheric corrosion by forming an impervious basic zinc carbonate layer.
4. It is used in the fabrication of alloys such as Cu-Zn brasses.
5. It is used in the manufacture of paints and pigments.
6. It is used in the production of die casting.
7. It is used in the rubber industries, concrete manufacturing, and in paints.
8. Rolled zinc plates are used in dry cell batteries.

The principal ore of zinc used for its extraction is sphalerite or zinc blend (ZnS). It is associated with sulphides Pb, Cu and Cd. Hence differential flotation can be used to separate the sulphides of Pb, Cu and Cd from Zn. This produces a concentrate of 50 - 60 % Zn. Both pyrometallurgical and hydrometallurgical techniques are employed in the extraction of zinc. For low grade ores, generally hydrometallurgical process is employed.

3.3.3. Pyrometallurgical extraction of Zinc

The various steps involved in the pyrometallurgical extraction of zinc are described below:

1. Crushing and grinding of the ore : The ore is initially crushed to about 1.9 cm. It is then combined with water and ground to less than 0.1 mm in a ball mill. The finely

ground particles and water form a slurry that flows from the mill to the flotation cell or tank

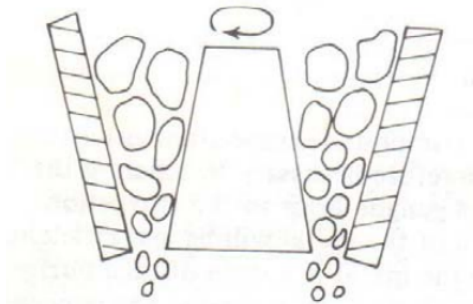


Fig.3.19. primary and secondary crushing

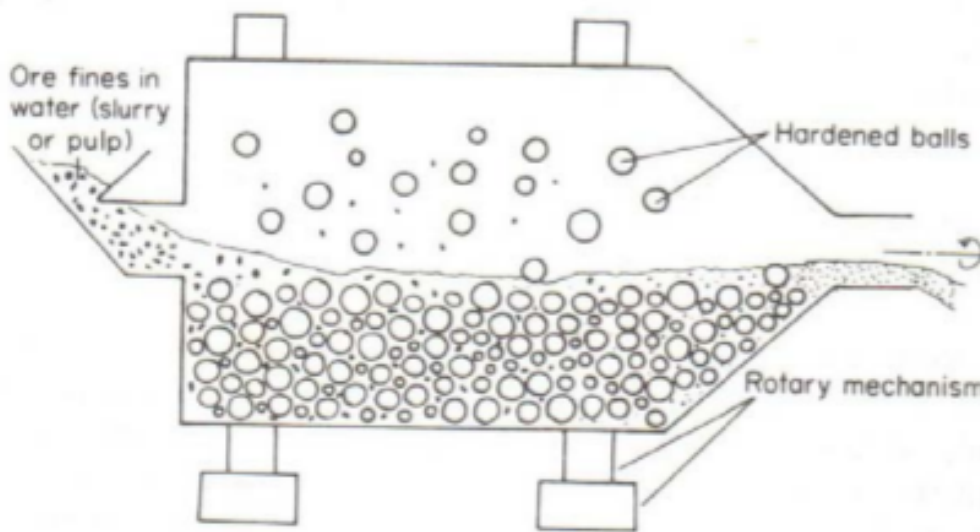


Fig.3.20. Grinding in ball mill

2. Flotation : As zinc blende is a sulphide ore containing about 3 -15 % Zn and associated with other sulphides, differential flotation can be employed to separate zinc sulphide from other sulphides.

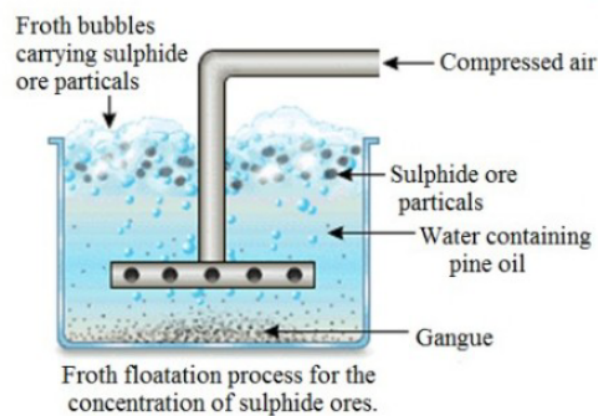


Fig.3.21

The basic principle behind the froth flotation process is the difference in the wetting ability of the ore and the suspended impurities. Pineoil is added as collector and

frothers used are cresols or aniline. The oil along the ore floats on the surface of water. A froth is generated by blowing air through the mixture. The froth is skimmed off to recover the concentrated ore. After flotation the concentrate contains 50-60%Zn

3. Roasting and sintering : After froth flotation, the next step in the extraction of zinc is roasting. Roasting is a process of heating a sulphide ore to a very high temperature in the presence of air. It is carried out in fluidised bed roaster or multiple hearth roaster at a temperature of around 800°C.

In roasting, the sulphide ore is converted into its oxide as per the reaction



The zinc calcine (ZnO) obtained contains 1 - 4 % S. The sulphur dioxide produced is used in the manufacture of sulphuric acid. The following figure (3.22.) shows the process in fluidised bed roasting.

The calcine obtained as the result of roasting is subsequently sintered in Dwight Lloyd Sintering machine at a temperature of around 1200 - 1400°C to eliminate residual sulphur, cadmium and lead as shown in fig.3.23. The following is the typical compositions of the concentrate, the roaster calcine and the sintered product for a given plant.

Concentrate : Zn (56.5%), Pb(1.07%), Cd(0.49%), S(31.24%)

Roaster calcine : Zn(62.0%), Pb(0.54%), Cd(0.63%), S(4.7%)

Sinter : Zn(66.2%), Pb(0.24%), Cd(0.02%), S(0.01%)

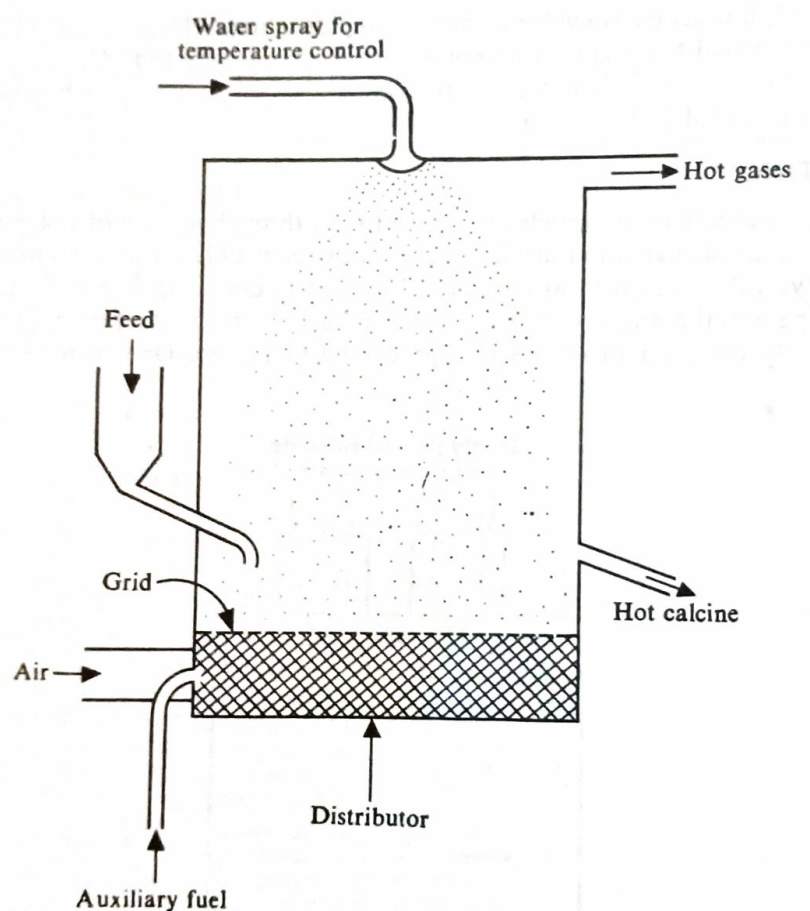


Fig.3.22. Fluidised bed Roaster.

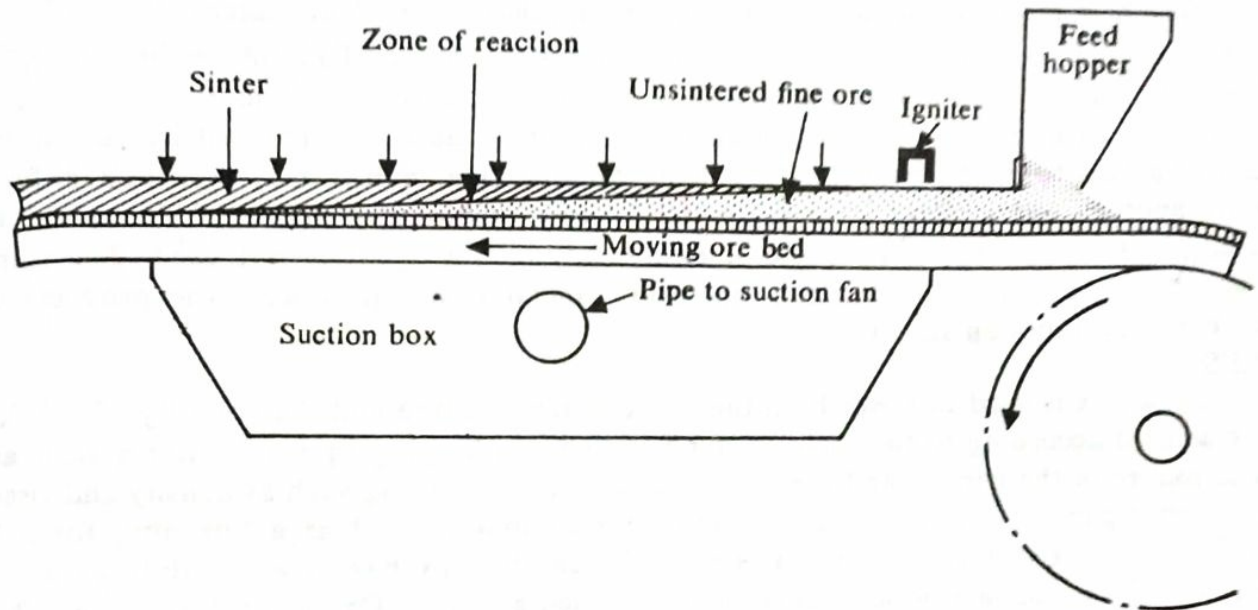


Fig.3.23. Dwight Lloyd Sintering machine.

4. Reduction : Reduction of ZnO to zinc metal can be achieved by the following processes;

- (a) Horizontal Retort reduction process
- (b) Vertical Retort reduction process
- (c) Electrothermic reduction process
- (d) Electrolytic reduction process
- (e) Imperial smelting process

4.(a) Horizontal Retort reduction process

In this process, 2 m- long retorts, each having a diameter of 0.25 m and thickness of 5 - 6 m, are lined up in a gas - fired furnace in batches of several dozens at a time. These retorts are generally made of clay and last for about 35 operations.

The retorts are first charged with a loose mixture of ZnO and an excess of coke breeze and then heated to a maximum temperature of about 1400°C to volatile the zinc which is subsequently condensed. Each retort can yield about 50 Kg of zinc per day . The following figure shows the horizontal retort process.

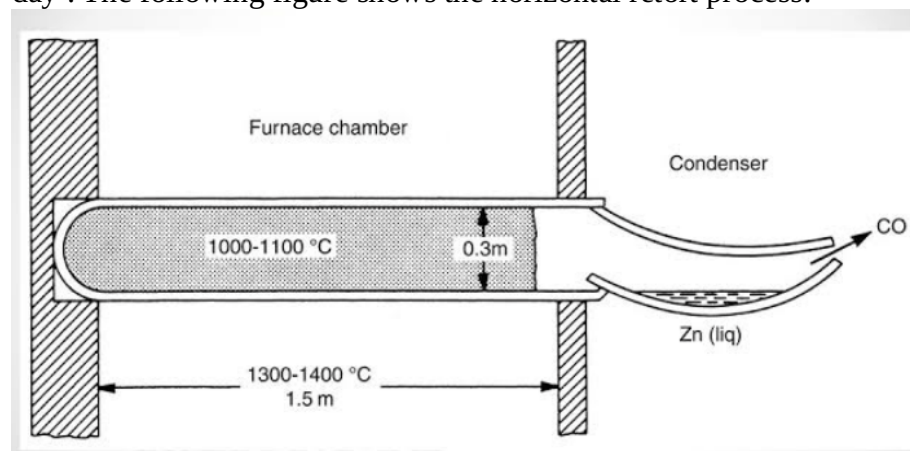


Fig.3.24 Horizontal Retort reduction process.

4.(b) Vertical Retort reduction process

Vertical retorts are usually made of silicon carbide because it has a conductivity that is about five times higher than that of clay. A vertical retort has much thicker walls, is much larger in size (height about 10m, diameter of 0.7m and thickness 30cm) and has a much higher capacity than that of a horizontal retort type.

The following fig.3.24 shows the vertical retort process.

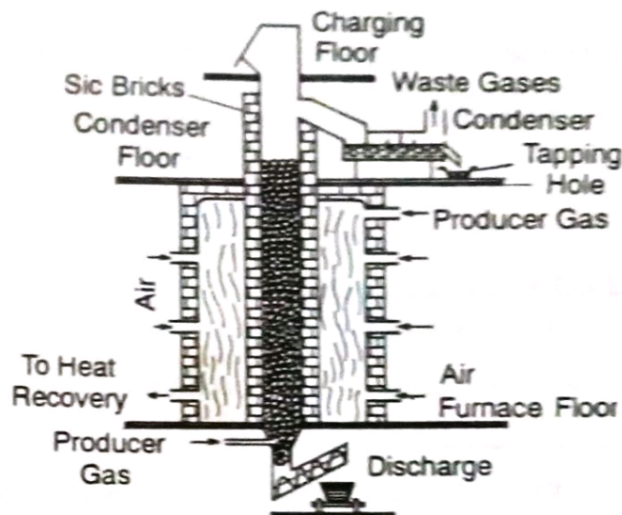
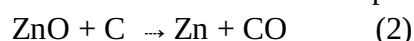


Fig.3.25 Vertical retort reduction process.

The furnace is heated by producer gas externally. Carbon monoxide and zinc vapours which are formed during the reaction, are carried to the condenser by a mild current of producer gas introduced from the bottom. Zn vapours condense in the condenser to form zinc spelter and CO is again led to the furnace. Molten zinc is periodically tapped off from the condenser from time to time into a receiver. The fresh charge is added through the charging door and the waste residue in the retort gradually moves out downwards which is withdrawn by an automatic screw conveyor fitted at the bottom by a roll discharge mechanism. The most important advantage of this method is that the formation of CO is prevented to a greater . As a result, loss of zinc due to the formation of zinc oxide is prevented. The reaction being



4(c). Electrothermic reduction process

In this case, a vertical retort can also be used. The retort is normally 15 m high with an internal diameter of 2.4 m and produces about 100 tons of zinc per day. In this process, electrodes of graphite are introduced through the silicon carbide walls at two places - near the bottom of the retort and at a zone about 9 m from the bottom. The charge in this zone forms the resistance and the electrical energy supplied provides the heat necessary for the reduction of the charge.

The gas or vapour liberated due to the reduction of the charge contains

40 - 45 % Zn, 45.0 % CO₂, 5 - 8 % H₂, 5 - 10 %N₂, traces - CO.

This vapour or gas is bubbled through an U - tube arrangement whose exit is maintained under a vacuum to enable the suction of retort gases through the molten zinc. The zinc recovery for these processes is about 95 %. This zinc is called spelter. The residues of the retort process contain 3 - 4 %zinc, iron, gangue, copper, and

precious metals. The following figure shows the flowsheet of extraction of zinc by reduction of ZnO by carbon.

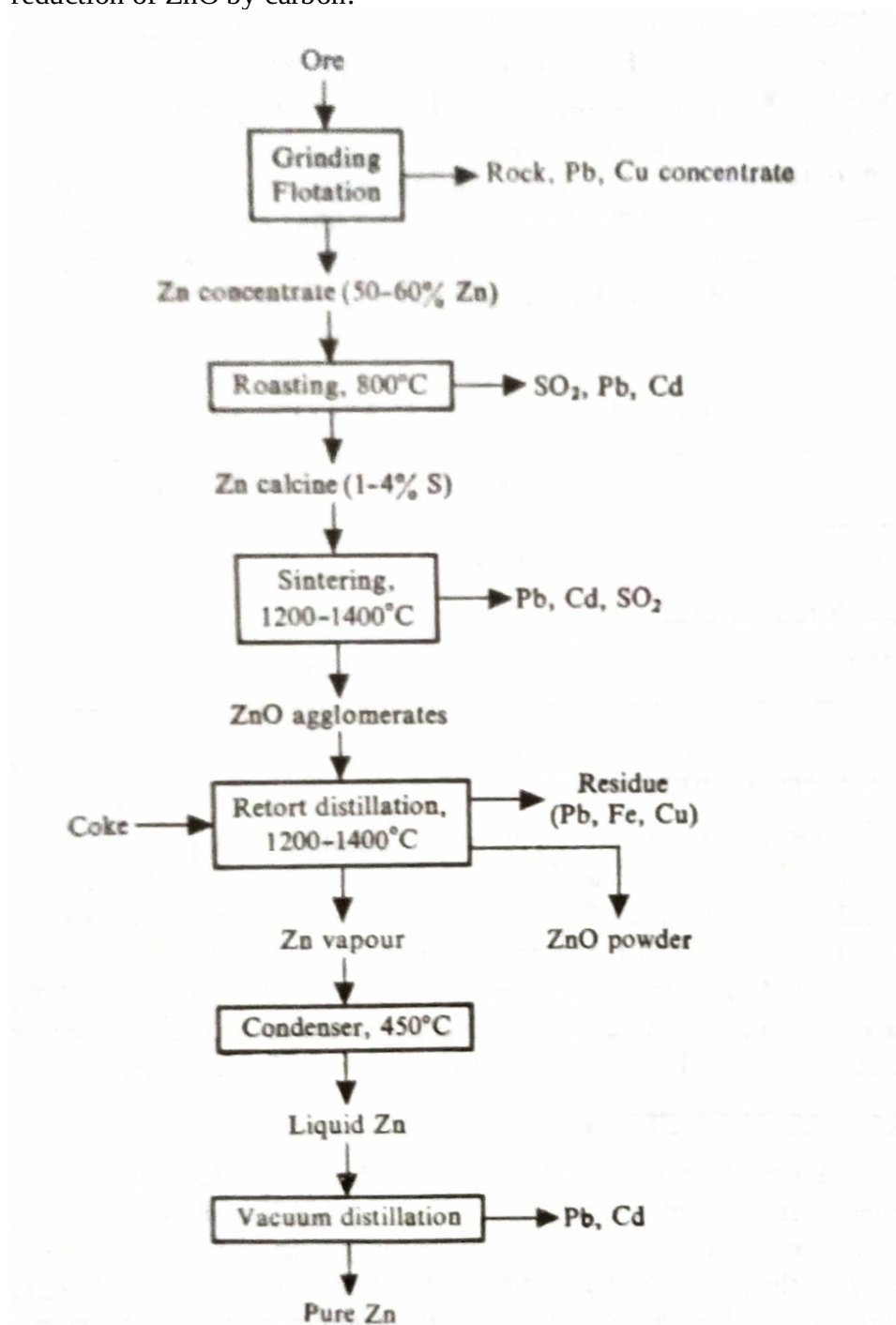


Fig.3.26 Extraction of zinc by reduction of ZnO by carbon.

4(d) Imperial Smelting process

In Imperial Smelting Process, the ZnO is reduced by carbon. The reduction is carried out in an Imperial Smelting blast furnace, in which the zinc that is evolved in the form of vapour, is condensed by using molten lead.

The ISP furnace is of a square - cross section and consists of a water - jacketed brick - lined shaft. Since the feed arrangement at the top of the furnace operates under a positive pressure, it becomes necessary to introduce the preheated coke and sinter through a double bell charging system. The furnace gases generated during reduction,

pass upward through the charge and are evenly divided between condensers. These condensers serve two purposes, they rapidly cool the furnace gases, resulting in the condensation of zinc contained in these gases and they help in dissolving the condensed zinc in the molten lead. The following figure shows the Imperial Smelting process.

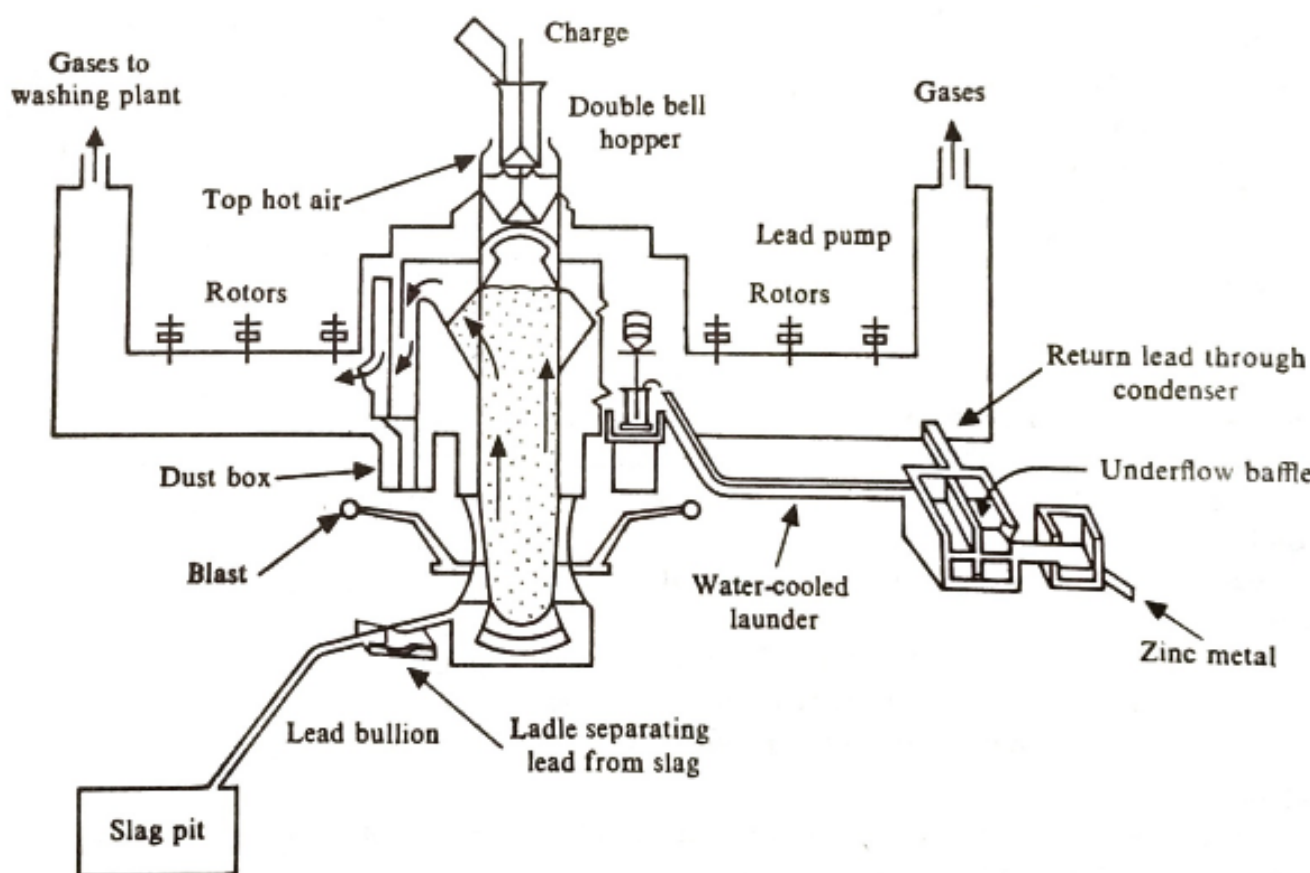
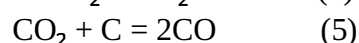
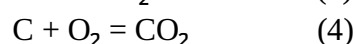
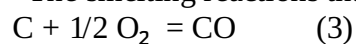


Fig.3.27 Imperial Smelting Process for reduction of ZnO.

Each condenser consists of a bath of molten lead. Rotors capable of generating an intense spray of lead droplets over the entire volume of the condenser, are immersed into the molten lead bath. When the furnace gases containing zinc pass through this spray, they are cooled very rapidly. As a result, the zinc vapour present in the furnace gases either goes directly into the solution with the lead or forms liquid zinc, which in turn, immediately dissolves in the lead.

The zinc containing lead is constantly pumped out from the condensers at a temperature of about 550°C into water cooled launders, where it is cooled to a temperature of about 440°C, as shown in the figure below. During cooling, the lead becomes saturated and any excess zinc emerges from the lead and floats in the form of vivulets on the surface of the lead, because the density of zinc is less than that of lead. The supernatant zinc is separated and the lead is continuously returned to the condensers. The zinc is collected in a holding tank where it is treated with metallic sodium in order to remove arsenic.

The smelting reactions that take place in the furnace are



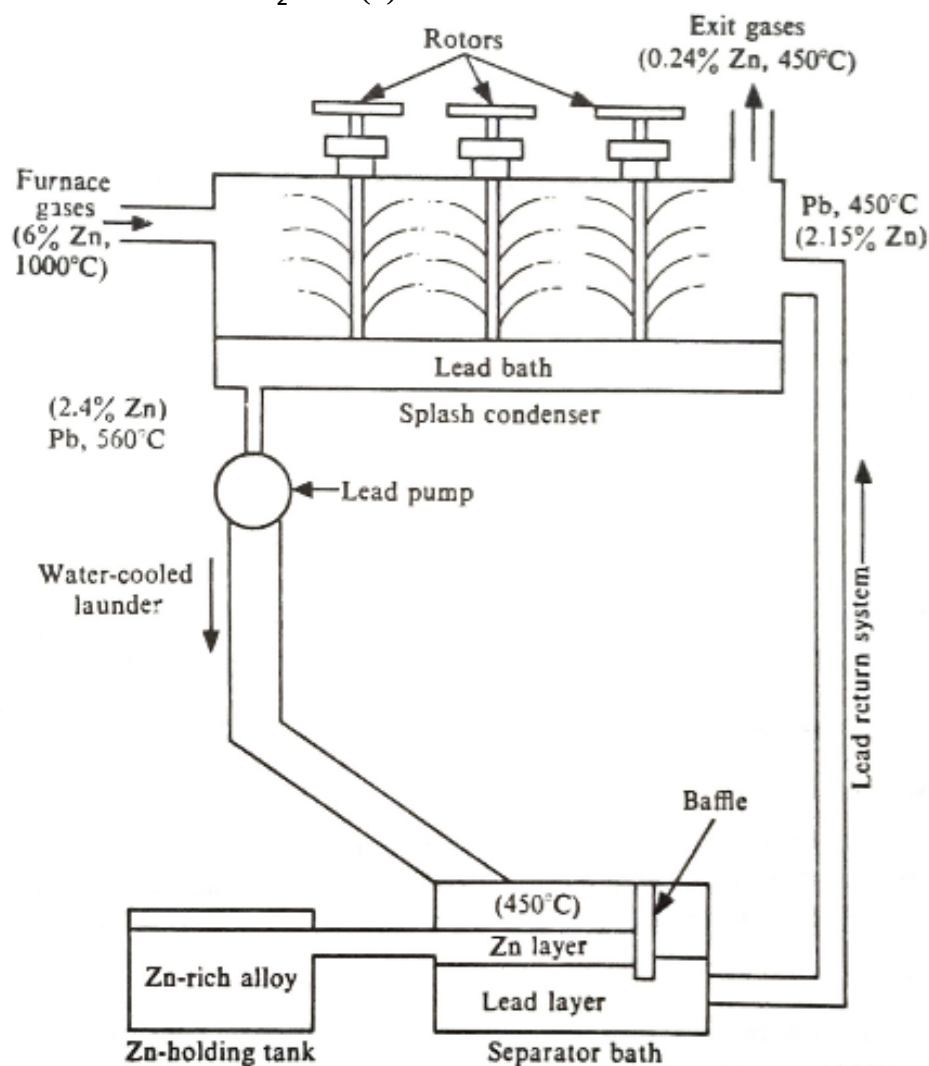
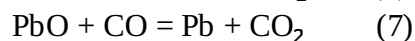
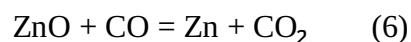


Fig.3.28. Zinc recovery system.

After the reduction has been completed, the molten slag and lead are taken out of the furnace hearth at a temperature that is approximately equal to the melting point of the slag, whereas the gases emerge from the top of the furnace at a temperature slightly higher than the equilibrium temperature of reaction (6). The latter temperature is raised to 1000°C by admitting air above the top of the furnace charge in order to generate heat by the combustion of CO.

In Imperial Smelting Process, preheated coke(800°C) and sinter are charged into the top of the furnace. The lead in the sinter, which is reduced to liquid metal, flows down to the bottom of the furnace.

Advantages of ISP

1. It is possible to smelt low grade, complex charges of zinc and lead ores and concentrates.
2. Recovery of zinc is less expensive
3. Furnace operation is fully automated.
4. The mechanism is highly robust.

3.3.4 Hydrometallurgical extraction of zinc

Hydrometallurgical processes are used for the extraction of zinc from a wide variety of ores, especially from low grade ores. These processes yield a high degree of purity and also recovering of valuable by- products. The following figure shows the flowsheet for the hydrometallurgical extraction of zinc.

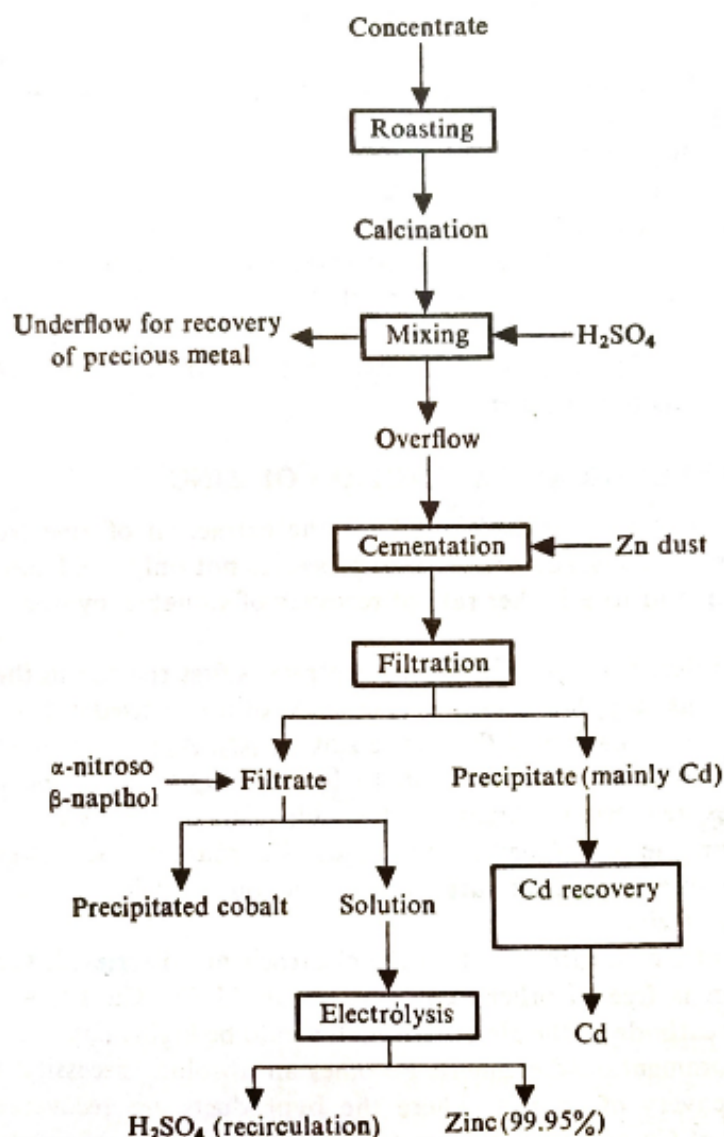


Fig.3.29. Hydrometallurgical extraction of zinc.

In this process the zinc concentrate is roasted in fluidised bed roaster. The roasted zinc concentrate is dissolved in an acid and valuable byproducts are precipitated out. The leach solution is finally electrolyzed for the recovery of zinc. The main steps are as follows:

1. Dissolution of zinc calcine in H_2SO_4
2. Precipitation of cadmium by adding zinc dust to the leach solution i.e cementation.
3. Precipitation of cobalt from leach solution by adding α - nitroso- β - naphthol.
4. Adjustment of the pH of the leach solution to about 5.3 to hydrolyze aluminium and iron and addition of MnO_2 to oxidise iron to the trivalent state and to produce a precipitate that is flocculent and that can absorb the entire quantity of As, Sb and Ge.

5. Electrolysis of the purified leach solution using a cell with a Pb- Ag alloy as anode and aluminium as cathode.

3.3.5 Refining of Zinc

Refining of zinc can be accomplished mainly by the following two processes

- (i) Electrolytic refining of zinc, and
- (ii) Refining of zinc by distillation

(i) Electrolytic refining of zinc

The electrolytic refining process consists of four steps as leaching, purification, electrolysis and melting and casting. The scheme of the process is shown in the figure.

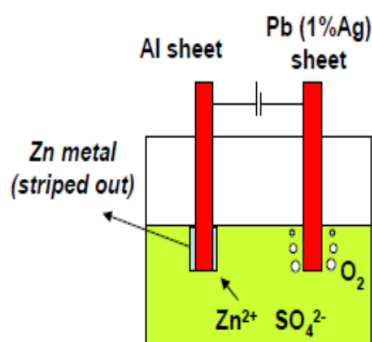
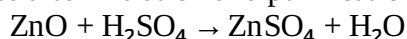
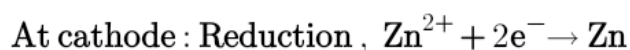
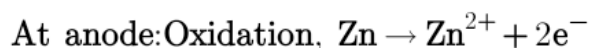


Fig. 3.30 Electrolytic refining of zinc

The crude zinc oxide is dissolved in dilute H_2SO_4 to produce a solution of zinc sulphate after filtration and purification as per the reaction;



The zinc sulphate is electrolysed using a Pb-Ag alloy as anode and an aluminium as cathode. Oxygen is released at anode and zinc is deposited on the cathode. Sulphuric acid is regenerated at the anode and can be used again. The electrode reactions being



The zinc is stripped periodically from the cathode and is melted and cast into blocks or ingots. Electrolytically refined zinc is generally 99.95 % pure. About 80 % of the world 's zinc is produced by this process.

(ii) Refining of zinc by distillation

The raw zinc from the retort or blast furnace processes contain as major impurities lead and cadmium. Of these, cadmium is more volatile and lead is less volatile than zinc. In the horizontal retort process, the first fraction of zinc distilled over contains most of the in the charge , whereas the last fraction contains 1 to 2 per cent lead.

The distillation is carried out in a system of two refluxing units, as shown in the figure below . The columns are built of superimposed trays or plates of silicon carbide, which is the material in the column walls. The lower half of the column is heated externally, whereas the upper half is well insulated.

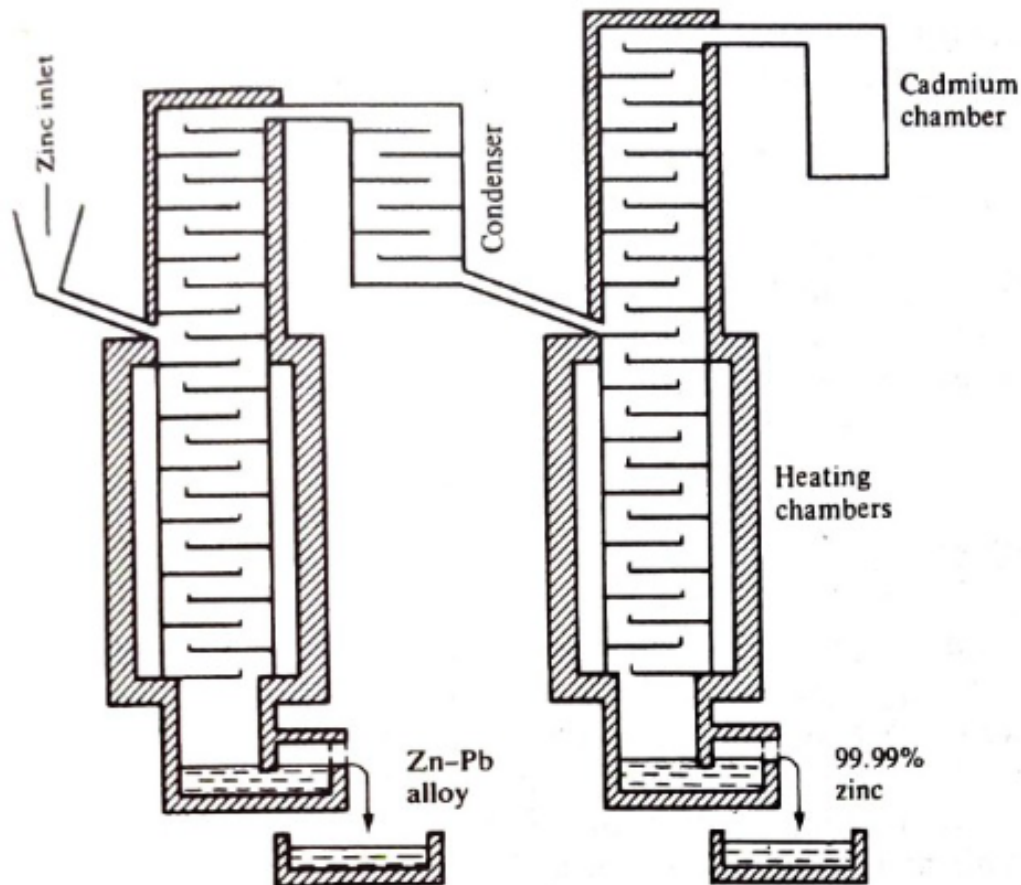


Fig.3.31 Refining of zinc by distillation .

The impure zinc is introduced in the middle of the first column. During its flow, through the lower part of the column, zinc and cadmium evaporate and the melt becomes enriched in lead. The vapour ascends to the upper part where further enrichment takes place by condensation of lead. The vapour from the first column is condensed and is passed on to the second column. Here the temperature is somewhat lower and zinc remains in the liquid state, whereas cadmium evaporates and recovered by condensation of the overflowing vapours. Zinc with purity of 99.9 % is withdrawn from the bottom of the second column, whereas from the first column a melt enriched in lead is obtained.

3.4. Extraction of Nickel

3.4.1 Minerals and Ores of Nickel :

Nickel ores occur both in oxide and sulphide forms. It occurs in nature mostly in combination with arsenic, antimony and sulphur. The important minerals of nickel are as follows:

(i) Pentlandite $[(\text{Ni Fe})_9\text{S}_8]$

It is a nickel sulphide ore, associated with copper and iron pyrites. It contains about 22 % nickel

(ii) Nicolite (Ni As) , also known as Kufper nickel.

(iii) Garnierite $[(\text{Ni Mg})\text{SiO}_3 \cdot x\text{H}_2\text{O}]$

It contains around 5 to 8 % nickel

(iv) Pyrrhotite $(\text{Ni}_2\text{FeS}_4)$

Canada is the principal nickel producer and accounts for nearly 80 % of the world production.

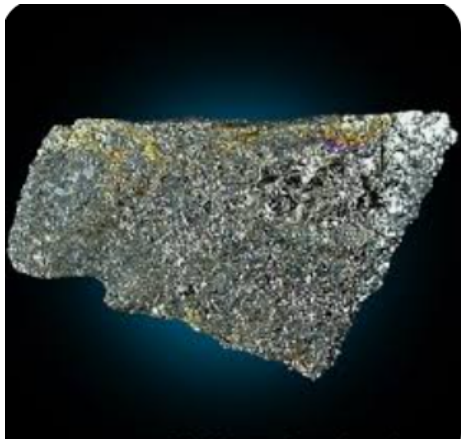


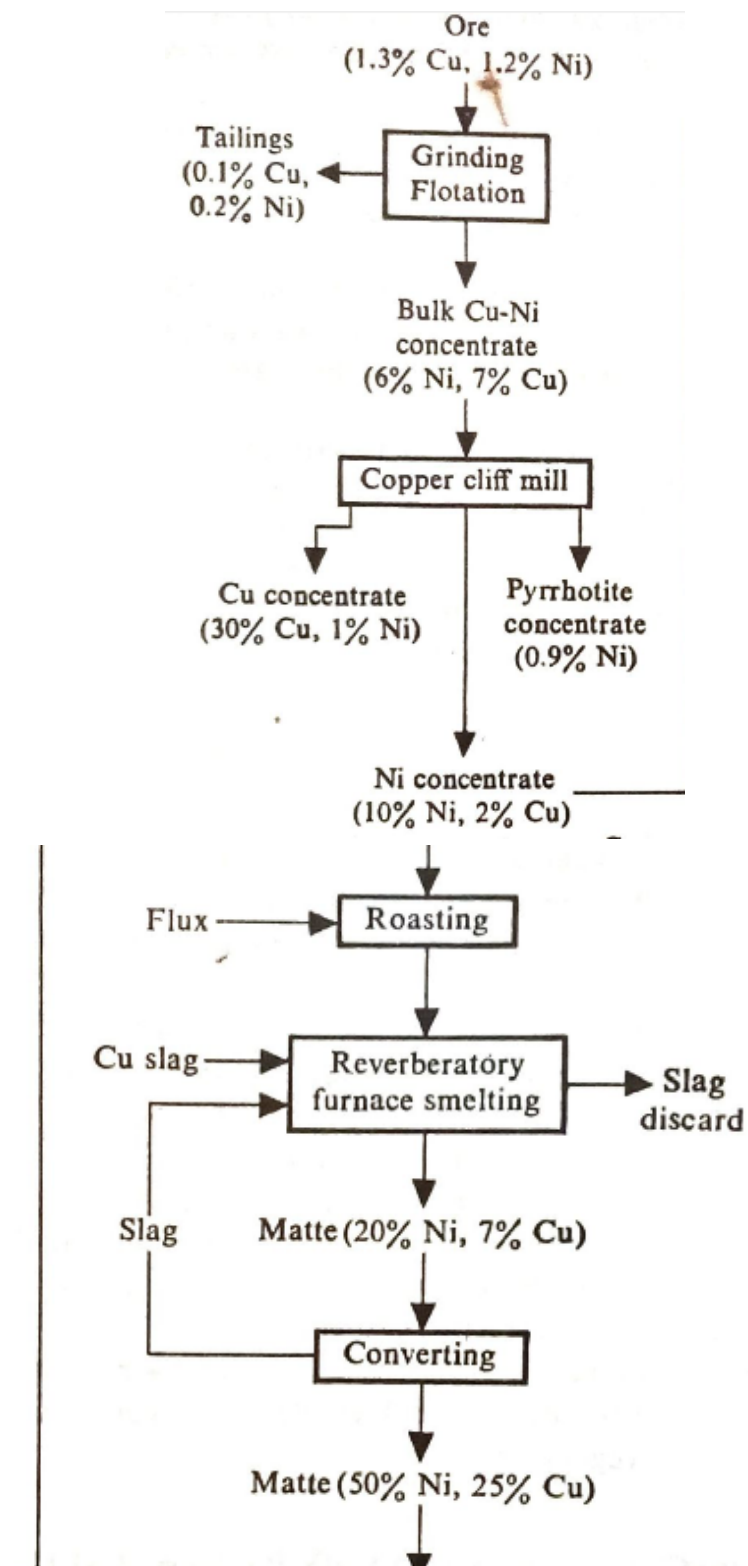
Fig. Mineral Pentaldite

3.4.2. Properties and uses of nickel

1. Nickel is a silvery white or greyish metal.
2. It is highly ductile, malleable and tenacious.
3. It is a ferromagnetic metal and resistant to oxidation under ordinary atmospheric conditions.
4. It has high toughness and elasticity.
5. It is used as a catalyst.
6. It is used for making crucibles, spatulas, tongs etc.
7. It is an important alloying element used in steel, stainless steels and other corrosion and heat resisting steels and steel castings.
8. It is used in construction of heavy guns, gears etc in underlaying cables, making armour plates, aeroplane and automobile parts.
9. Cupronickel is used in making bullet sheaths, german silver is used in domestic utensils, sterlite is used in the manufacture of high speed tool steels etc.
10. It is used in the field of chemical processing, space research, nuclear reactor engineering, oil industries etc.

3.4.3 Pyrometallurgical extraction of nickel

The following figure shows the flowsheet used by INCO for nickel extraction from sulphide ore pentlandite. The extraction process involves the following steps:



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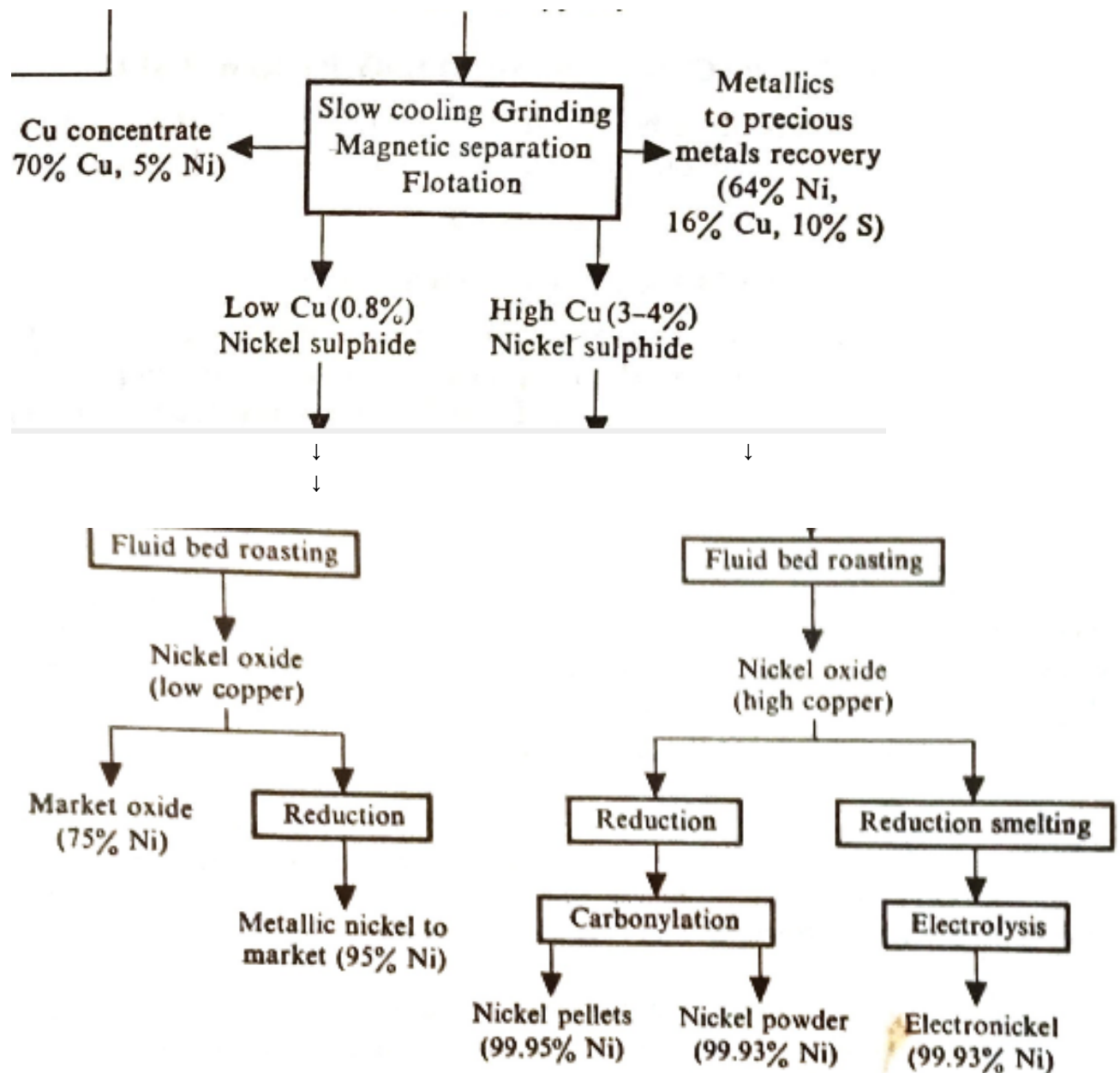
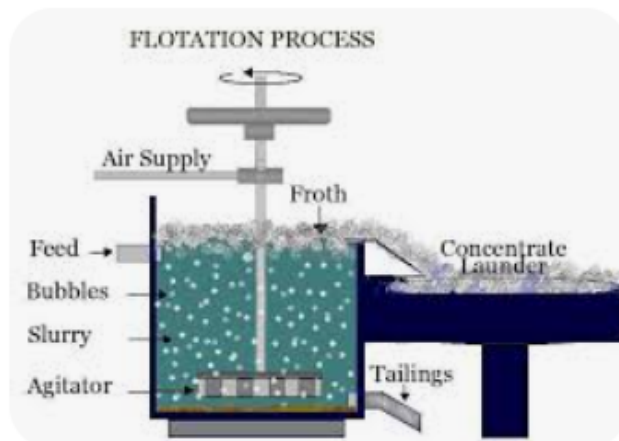


Fig. 3.33. Flowsheet for nickel extraction by INCO process.

1. Concentration :

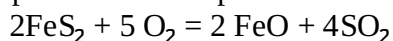
The ore containing about 1.2 % nickel is crushed and ground and subjected to concentration in froth flotation process as shown in the following figure.



The concentrate contains copper concentrate, pyrrhotite concentrate and nickel concentrate.

2. Roasting

The nickel concentrate containing about 10 % Ni is roasted either in fluidised bed roaster or multiple hearth roaster. The roasting is continued for several weeks during which time, most of the iron sulphide present in the ore is oxidised to ferrous oxide and sulphur dioxide as per the reaction



By roasting, about 40 % of sulphur is removed and the process is carried out at a temperature of 550 - 600°C.

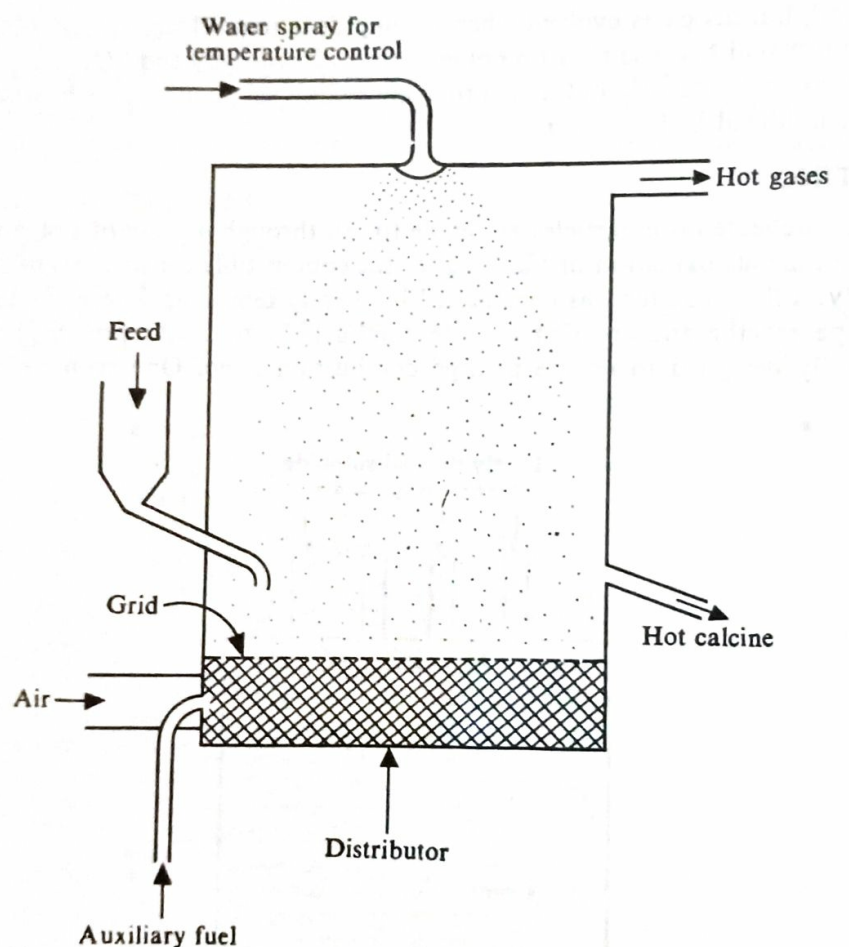


Fig.3.35. Fluidised bed roaster

3. Smelting and Converting

The roasted calcine is smelted in a reverberatory furnace to produce a matte containing 20 % Ni, 7 % Cu, 40 % Fe and 27 % S. The converter slags from both the nickel and copper converters are returned to the reverberatory furnace.

The matte produced in the smelting stage is subjected to conversion in converting stage. The converting involves in two stages. In the first stage, the matte is converted in a *pierce - smith* converter to produce a nickel - riched matte with 50 % Ni, 25 % Cu and 22 % S.

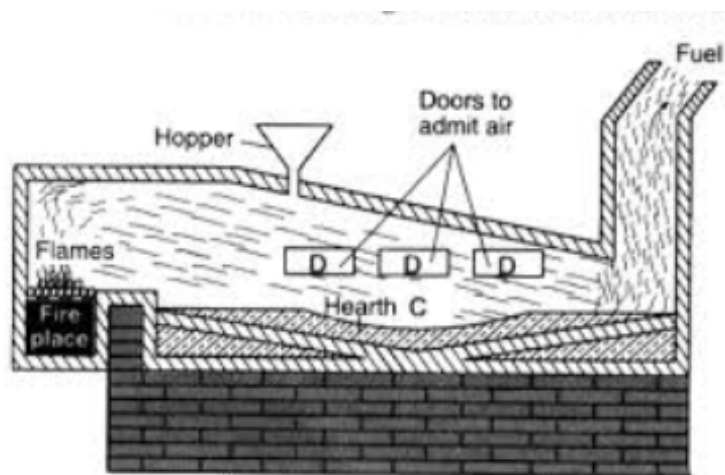


Fig.3.36 Reverberatory furnace for smelting of nickel.

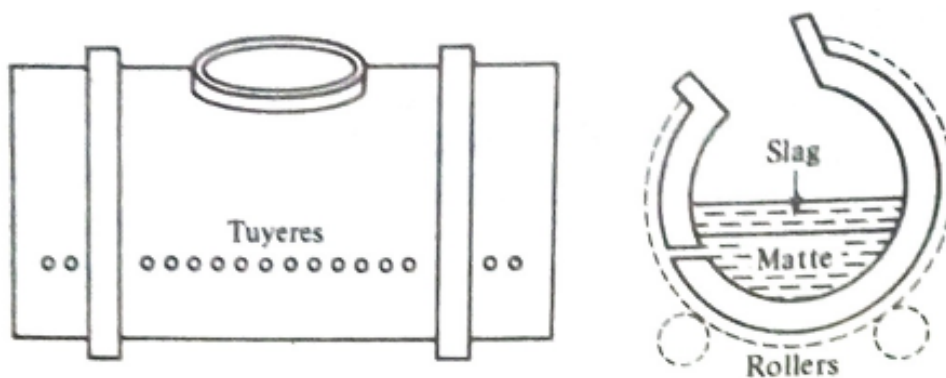


Fig. 3.37. Pierce - smith converter (schematic)

The converting is completed at about 1150°C to produce this matte. In the second stage, the converter matte is subjected to a slow cooling process, where the matte is cooled from its melting point to about 400°C in a period of 3 days. During slow cooling process, first Cu_2S precipitates and grows. At 700°C, a second phase called nickel - copper alloy phase, starts to precipitate. At 575°C, the third solid phase Ni_3S_2 starts to precipitate. The slow cooling rate provides the necessary grain growth so that in subsequent processing, the sulphides can be recovered by froth flotation.

The slow cooled matte is subjected to grinding and flotation using diphenyl guanidine (DPG) as collector and frother. Prior to flotation, the nickel - copper alloy is isolated by magnetic separation and sent for the recovery of the precious metals. The copper sulphide concentrate produced by froth flotation contains about 70 % Cu, 5 % Ni, and 20 % S and is sent to the copper circuit. The nickel sulphide is recovered as a low copper - nickel sulphide with 74 % Ni, 0.8 %Cu, 0.8 %Fe and 22 %S and as a high copper - nickel sulphide with 72 % Ni, 3-4 %Cu, 0.8 %Fe and 21 %S. Both the nickel sulphide fractions are roasted after pelletisation in fluidised bed roasters in the temperature range 1100- 1250°C to produce granular nickel oxide. The low copper - nickel is marketed as nickel oxide. The high copper - nickel oxide is sent to the refineries.

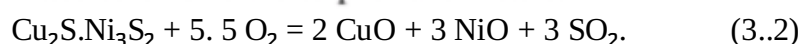
3.4.4 Refining of nickel

The sinter nickel oxide, containing 75% Ni, can be used as such in the production of steel; but for the production of metallic nickel the oxide has to be reduced and then refined. There are two methods: (1) The Mond process (or Carbonyl process) and (2) Electrolytic process.

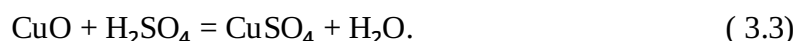
1. Mond process (Carbonyl process)

This process is based on the property that nickel possesses of combining with carbon monoxide to form a volatile compound, nickel carbonyl $[\text{Ni}(\text{CO})_4]$ which is stable below 60 °C but on heating to 180 °C breaks into the original forms.

As the nickel oxide sinter contains small amounts of sulphur and metallic elements which reduce the efficiency of the subsequent processing. So it is first given a preliminary oxidized roasting. Temperature of 750 °C is maintained and a flow of air admitted to oxidize the sulphur and metals.



Then the calcine, high copper, nickel oxide is leached with dilute sulphuric acid. The leaching removes large part of the copper as copper sulphate and left a residue of NiO.



This copper sulphate is filtered to remove the undissolved nickel oxide, the copper sulphate recovered by crystallization.

NiO is reduced to finely divide metallic nickel by an atmosphere of water gas (36% CO and 52% H₂) at 350–400 °C. To ensure efficient reduction two reducers are operated in series, the partially reduced material is conveying from the bottom of the first to the top of the second. The passage through each reducer takes about 4 h.



At the comparatively low temperature carbon monoxide plays very little part in the reduction, this is mainly carried out by the hydrogen. The result of the greater activity of hydrogen [reaction (13.4)] is to yield an exit gas which after removal of the water is very rich in carbon monoxide and thus suitable for the formation of nickel carbonyl in the next operation.

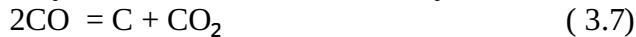
It is then cooled, passed on to volatilizer, where at a temperature of about 40–90 °C, the active nickel is exposed to a flow of carbon monoxide gas to form gaseous nickel carbonyl $[\text{Ni}(\text{CO})_4]$, which is carried out in the gas stream. The material passes through eight volatilizers in series; the process takes place in several days. This is a slow process.



This gas $\text{Ni}(\text{CO})_4$ is then passed through a tower containing nickel shots (4-6mm diameter) at 180°C and the compound nickel carbonyl is decomposed into metallic nickel and carbon monoxide, because nickel carbonyl is stable below 60°C.



Close control of temperature is necessary because at 200°C some carbon and carbon monoxide are formed by decomposing carbon monoxide in presence of nickel, which acts a catalyst. This carbon is absorbed by metallic nickel.



Decomposed metallic nickels are deposited on the nickel shots and increase in size to form nickel pellets.

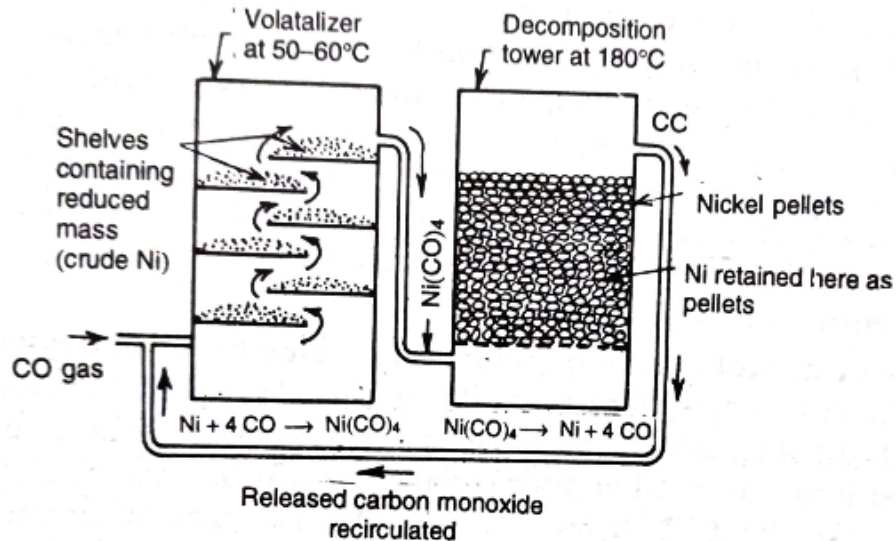


Fig. 3.38. Refining of nickel by Mond's process

2. Electrolytic refining process

Sintered nickel oxide is first reduced to metal by reduction smelting in presence of carbon and flux.



Crude nickel metal is then cast into anodes, which contain 95% Ni, 2% Cu, 0.75% Fe, and 0.7% S. Cathodes are produced in a somewhat similar to that are used in the preparation of electrolytic copper starting sheets. Thin nickel sheets are produced in starter cells.

Electrolysis is carried out in concrete tanks, solution of nickel sulphate and chloride containing about 60 gm of Ni^{2+} per litre, 95 gm of SO_4^{2-} per litre, 35 gm of Na^+ per litre, 55 gm of Cl^- per litre and 16 gm of H_3BO_3 per litre are used as electrolyte. Electrolysis is carried out at 60 °C at a current density of 170 A/m². On passage of current, nickel is dissolved from the anodes and is deposited on the cathode starting sheet. To prevent the deposition of copper and iron, the cathodes are enclosed in a canvas cover, which is tightly woven in order that a hydrostatic head of 25–50 mm is maintained over the level in the tank; thus ensuring a constant outflow of electrolyte through the canvas.

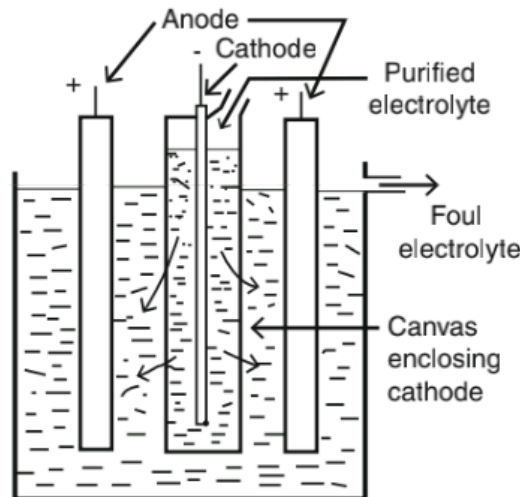


Fig. 3.39. Electrolytic refining of nickel.

Actually nickel ions, set free at the anode, are not carried out directly to the cathode but along with copper and iron ions in solution flow continuously out of the tank

as foul electrolyte. The foul electrolyte is free from copper by agitation with active nickel powder, the copper is cementing out. Vigorous agitation is done by passing air, oxidizes the iron which is precipitates as $\text{Fe}(\text{OH})_3$. Small amount of chlorine gas is passed to ensure oxidation to form precipitates of $\text{Fe}(\text{OH})_3$. Cobalt is removed as cobalt hydroxide by further chlorine gas oxidation. At this stage cobalt is removed; further removal of impurities, such as arsenic and lead, are taking placed. The purified solution (contains 0.01 gm of Co per litre, 0.001 gm of Cu per litre, 0.004 gm of Fe per litre, 0.0002 gm of As and Pb each per litre) is then passed to the cathode compartment. When deposition is complete the cathodes are withdrawn, washed and either marketed in sheet form or melted and cast into ingots and shots (99.93% Ni, 0.05% Co, 0.005% Cu, 0.002% Fe, 0.001% S, 0.0002% As and 0.001% Pb).

Extraction of metals from Halides

4.1 Extraction of Titanium

4.1.1 Properties and uses of titanium

1. It has high strength-to-weight ratio and is strong, lustrous and corrosion resistant
2. Pure titanium is ductile.
3. It has high stiffness, toughness and low density.
4. It has very low electrical and thermal conductivities.
5. It is used as a deoxidiser in steel making.
6. It is an alloying element with most metals and some nonmetals.
7. It is used in aircraft, pipes for power plants, spacecrafts, missiles, naval ships , airframes, jet engine components, chemical process industries.
8. It is used in hip and knee replacement, pace- makers, plates, screws and carnial plates for skull fractures.
9. Titanium alloys retain their strength at higher temperatures and show less creep.
10. Pure titanium is used in the form of sheets, tubes rods,bars and forgings.

4.1.2. Minerals and ores of titanium

Titanium is widely distributed and constitutes 0.44 % of earth 's crust. The most important minerals of titanium are

1. Ilmenite ($\text{FeO} \cdot \text{TiO}_2$) , contains 50-60 % TiO_2 , 9-26% FeO , 14-26 % Fe_2O_3 etc



2. Rutile (TiO_2)



3. Senaite ($\text{Fe.Mn.Pb} \text{ O} \cdot \text{TiO}_2$



4.2 Extraction of titanium from ilmenite

Extraction of titanium from ilmenite involves the following steps:

1. Treatment of ilmenite for upgradation
2. Chlorination of TiO_2 to get TiCl_4
3. Production of metallic titanium by reduction of TiCl_4
4. Production of ductile titanium

4.2.1 Treatment of ilmenite for upgradation

To obtain titanium from ilmenite, the ores must be upgraded to a titania - rich product containing over 90 % TiO_2 . Ilmenite contains titania and iron oxide in spinel or bound form, therefore can not be upgraded by physical methods. Iron oxide can be removed by preferential chemical reactions with suitable reagents. These methods are:

- (i) Reduction smelting of ilmenite with carbon and flux, resulting titania - rich slag and pig iron.
- (ii) Direct leaching of ilmenite with acids under atmospheric pressure or high pressure in the preferential dissolution of iron
- (iii) Selective halogenation of iron oxide to produce volatile iron halides.

(i) Reduction smelting of ilmenite (Sorel Process)

TiO_2 is a stable oxide and can not be easily reduced. Iron can be selectively reduced from ilmenite by carbon. Hence carbon smelting in an electric furnace produces pig iron and a high titania slag with a minimum of other oxides. The reaction is



The slag contains 70 - 90 % TiO_2 , 5 - 10 % Fe and minor quantity of other oxides like MgO and CaO.

(ii) Direct leaching of ilmenite

It is possible to selectively leach the iron oxides present in ilmenite by hot concentrated acids. These oxides can be leach faster using finer particle size, high temperatures and high pressures. Here the ilmenite is prereduced and the reduced iron is leached with dil HCl, leaving behind high grade TiO_2 as a residue.

(iii) Halogenation of ilmenite

Halogenation of ilmenite aims at forming a volatile iron halide using reagents such as chlorine, bromine, hydrochloric acid and ammonium chloride. The removal of volatile iron halides leaves a TiO_2 - rich residue. This residue is chlorinated at 800-

850°C in the presence of carbon to produce TiCl_4 .

For selective chlorination of ilmenite, iron oxides in the ilmenite are easily chlorinated in the presence of carbon by a variety of chlorinating agents. For chlorination, the mineral taken in a powdery form, is briquetted with charcoal and a binder such as asphalt, tar, starch, molasses, or ferric chloride. It is done at 550-600°C and yield a product containing 90 % TiO_2 . By increasing the temperature to 900-1000°C, a residue rich in 95-98 % TiO_2 is obtained. This TiO_2 is chlorinated to give crude TiCl_4 , which is further purified by vacuum distillation to get refined TiCl_4 . (Shown in the flowsheet)

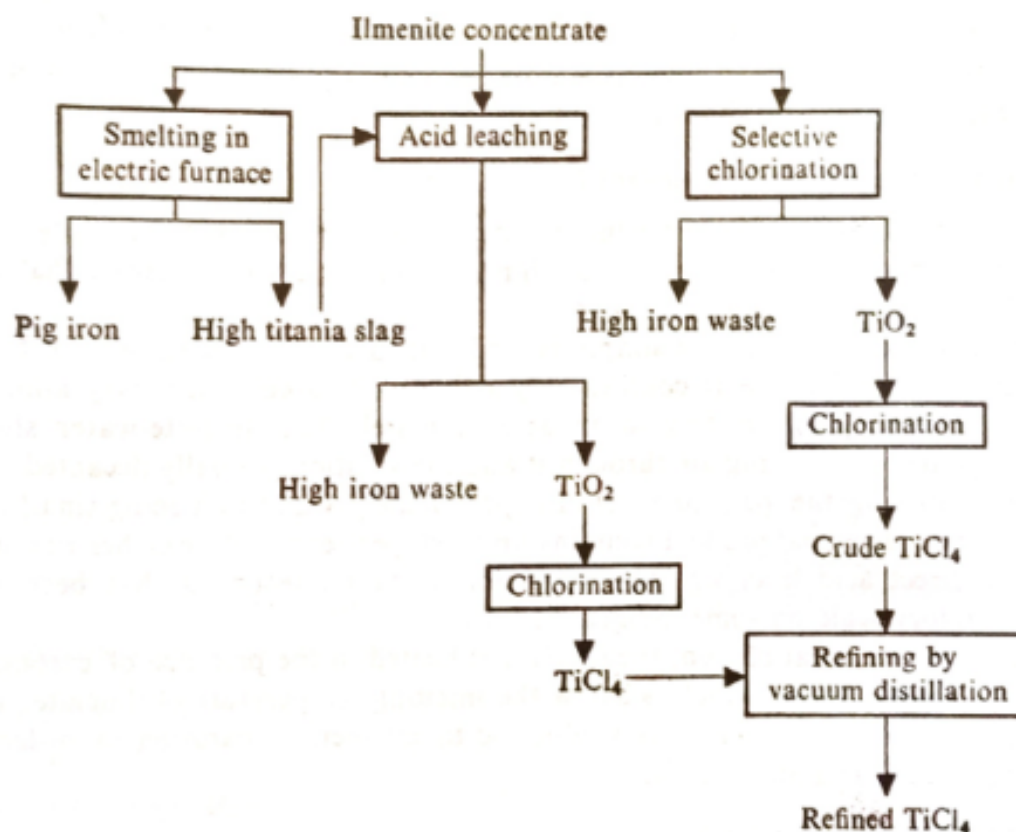
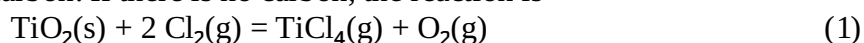


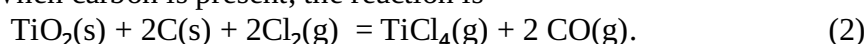
Fig.4.1.Production of Titanium Tetrachloride from Ilmenite

4.2.2. Chlorination of TiO_2 to get TiCl_4

The TiO_2 from natural rutile or upgraded ilmenite is chlorinated to produce TiCl_4 which is suitable for metal production. Chlorination is carried out in presence of carbon. If there is no carbon, the reaction is



When carbon is present, the reaction is

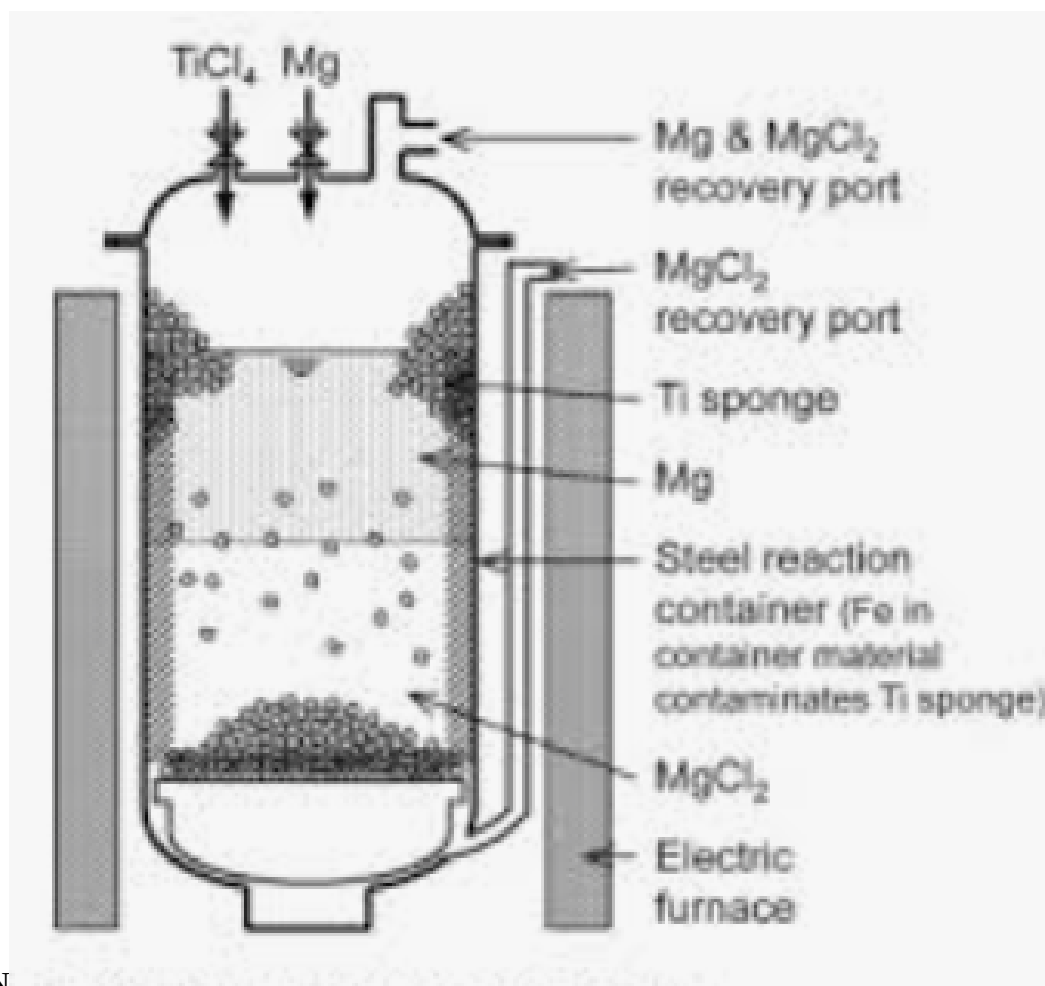


4.2.3 Production of metallic titanium by reduction of TiCl_4

Commercially titanium can be produced mainly by the following processes:

- (a) Kroll's Process
- (b) Hunter's Process

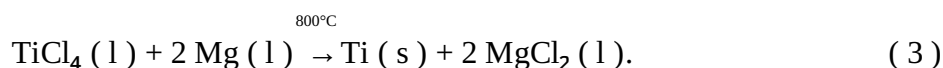
(a) Kroll's Process : In Kroll's process, titanium tetrachloride (TiCl_4) is reduced by magnesium to produce a spongy , brittle metal. Further, it is treated to get a ductile metal.(Shown in the following figure)



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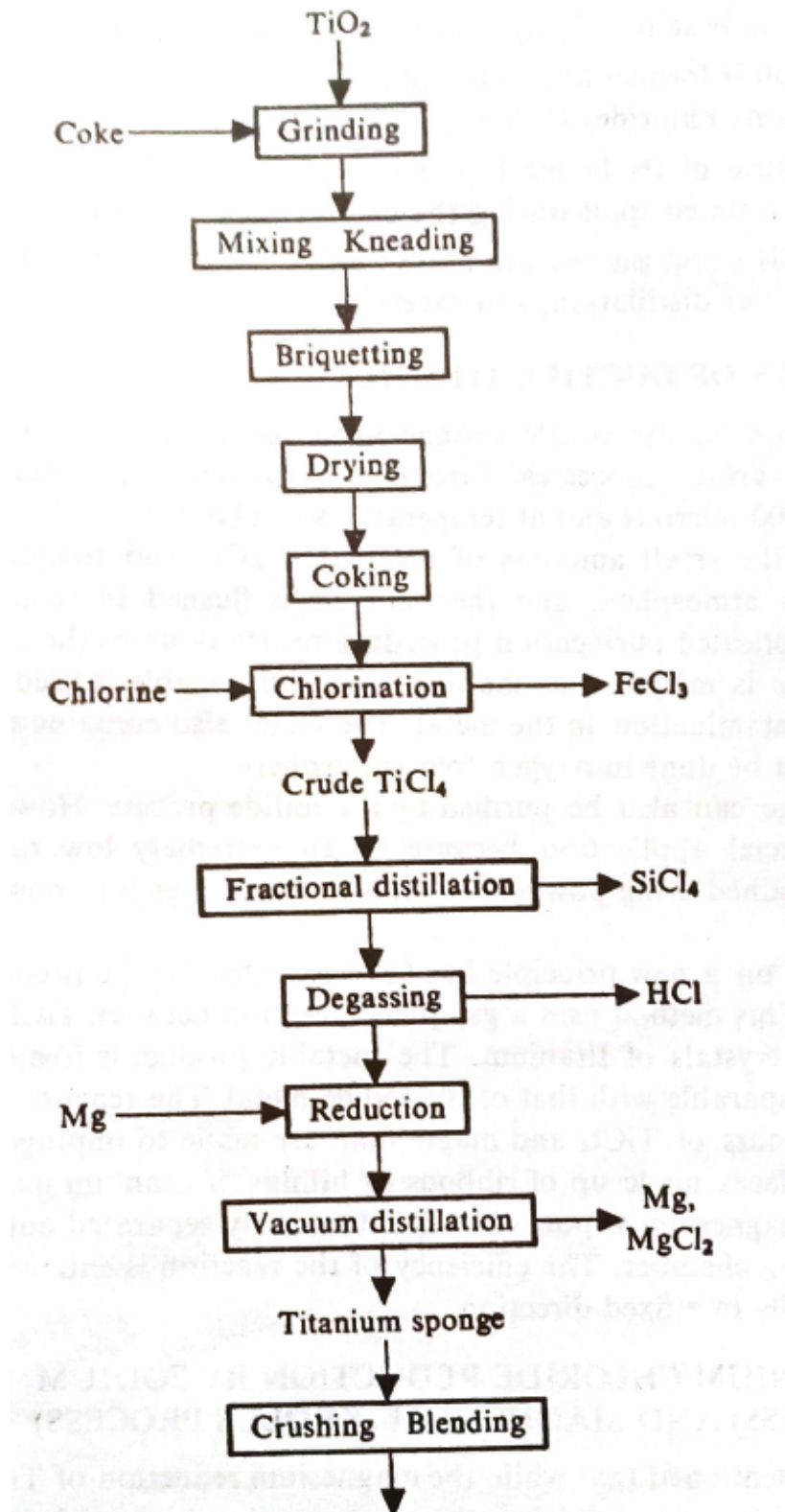
Fig.4.2 Kroll's process (schematic)

The reduction reaction in Kroll's process is written as



The reaction is carried out in a stainless steel container which has a provision for external heating by gas electricity. After reaction is over, the titanium sponge is recovered by dissolving MgCl_2 and the excess magnesium by leaching with cold dilute hydrochloric acid or distilling of MgCl_2 and the excess magnesium by vacuum heating. The residue is pure titanium sponge.

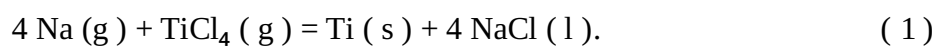
Titanium tetrachloride is introduced into the chamber in the form of a stream of drops. The following figure shows the flowsheet for the production of titanium sponge.



Graded titanium sponge.

Fig.4.3 Flowsheet for the production of titanium sponge

(b) Hunter's Process ∴ In this process, titanium tetrachloride is reduced by sodium to give directly ductile titanium. The overall reduction of the TiCl_4 by sodium is given as



The following figure shows the schematic diagram of Hunter's process

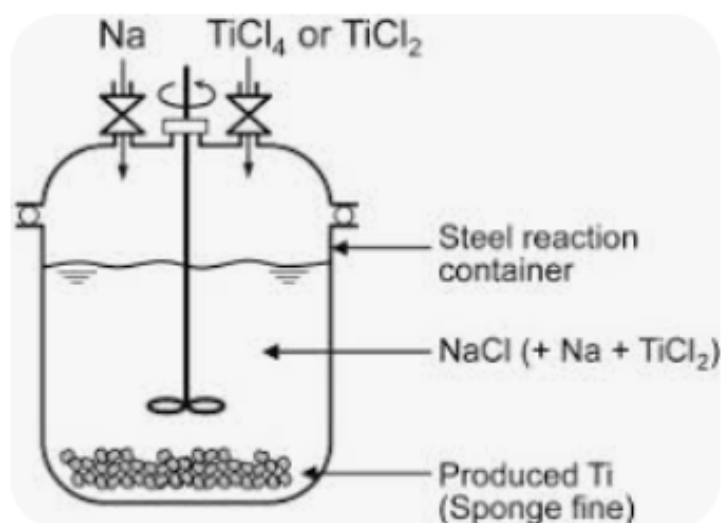
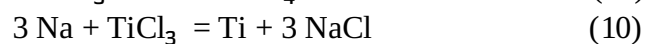
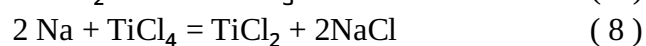
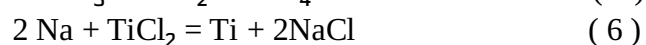
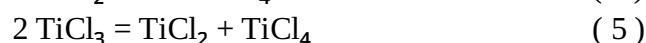
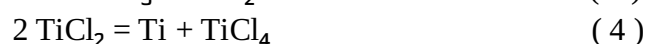
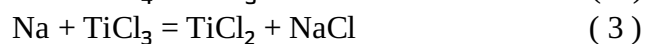
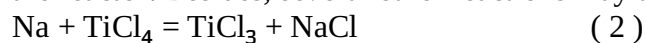
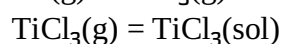
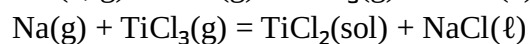
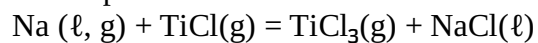


Fig.4.4 Hunter's process (schematic)

The reaction is highly exothermic and a temperature of about 1000°C is obtained in the reactor. Besides, several other reactions may take place simultaneously as follows.



The important reactions are



Extraction of Precious Metals

5.1. Extraction of precious metals

The precious metals include gold, silver and platinum group metals. These metals have a low affinity for oxygen and hence are found in nature either in the native state or in the form of easily reducible compounds. They are usually associated with large quantities of gangue materials. Therefore, their extraction requires an elaborate method for the separation of the gangue materials from the valuable metal. Such separation is often achieved by leaching the metal or its compound, followed by reduction.

5.2. Extraction of gold

5.2.1 Minerals or ores of gold:

The important minerals or ores of gold are

- (i) Sylvanite - $(\text{Au,Ag})\text{Te}_2$
- (ii) Calaverite - (AuTe_2)

5.2.1 Properties and uses of gold :

- (1) Gold is highly ductile and malleable.
- (2) It is a soft metal and has high formability.
- (3) It is used chiefly for making jewellery, dental alloys, gold coated base metal and parts of scientific instruments.
- (4) It is used as decorative purposes, manufacture of containers and pipes.
- (5) It is used in the preparation of certain medicines.

5.2.2 Process of extraction of gold

Gold is generally extracted by any one of the following processes:

- 1. The amalgamation process
- 2. The chlorination process
- 3. The Cyanidation process

Each of these processes is preceded by a preliminary concentration step. The metal is liberated from the rock mineral by crushing and grinding. Sometimes flotation process or gravity concentration process is used for its separation.

1. Amalgamation process - In this process, a slurry made up of the wet ground gold ore is allowed to flow down a slopping surface lined with copper. (Shown in the figure). The surface is continuously washed with an amalgam of silver and mercury. The amalgam takes up gold from the slurry and the metal is subsequently recovered from the amalgam by distilling the mercury. This process yields a low recovery of gold (40 -60 %).

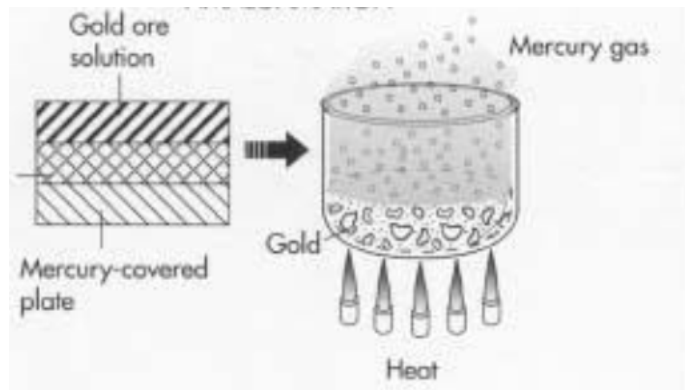
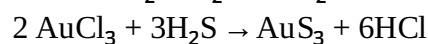
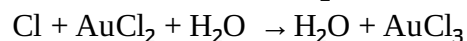
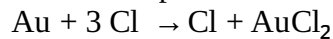
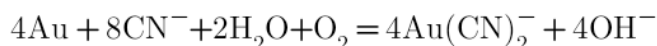
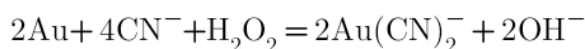
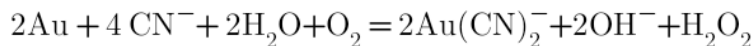


Fig. 5.1 Amalgamation process

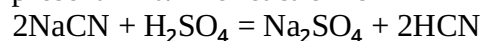
2. Chlorination process : In this process, gold ore is chlorinated at high temperatures by the presence of chlorine to form soluble gold chloride (AuCl_3), which is subsequently precipitated as sulphide by passing H_2S gas through the solution. And then the sulphide is smelted to yield the metal. The reactions are



3. Cyanidation process : In this process, gold present in the gold ore is dissolved in an NaCN solution for leaching. This cyanide leaching takes place more readily when oxygen is bubbled through the NaCN solution so as to cause intense aeration and when an additional oxidising agent such as H_2O_2 is present. A higher rate of leaching is obtained by passing oxygen under pressure. The reactions may be written as



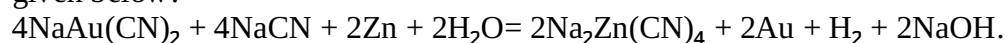
It should be noted that NaCN is a highly poisonous chemical. An even more poisonous chemical namely, HCN gas, is produced if any acid such as H_2SO_4 is present in it. The reaction is



This reaction implies the loss of the leaching agent, So an alkali is added to prevent such loss.

Gold is recovered from the cyanide solution by any one of the following methods:

(i) Cementation process : In the cementation process, gold is precipitated from the cyanide solution by adding zinc dust or wire or aluminum powder. This cemented gold is purified by heating, when zinc volatilizes. The overall cementation reaction is given below:



The following figure shows the flowsheet of extraction of gold.

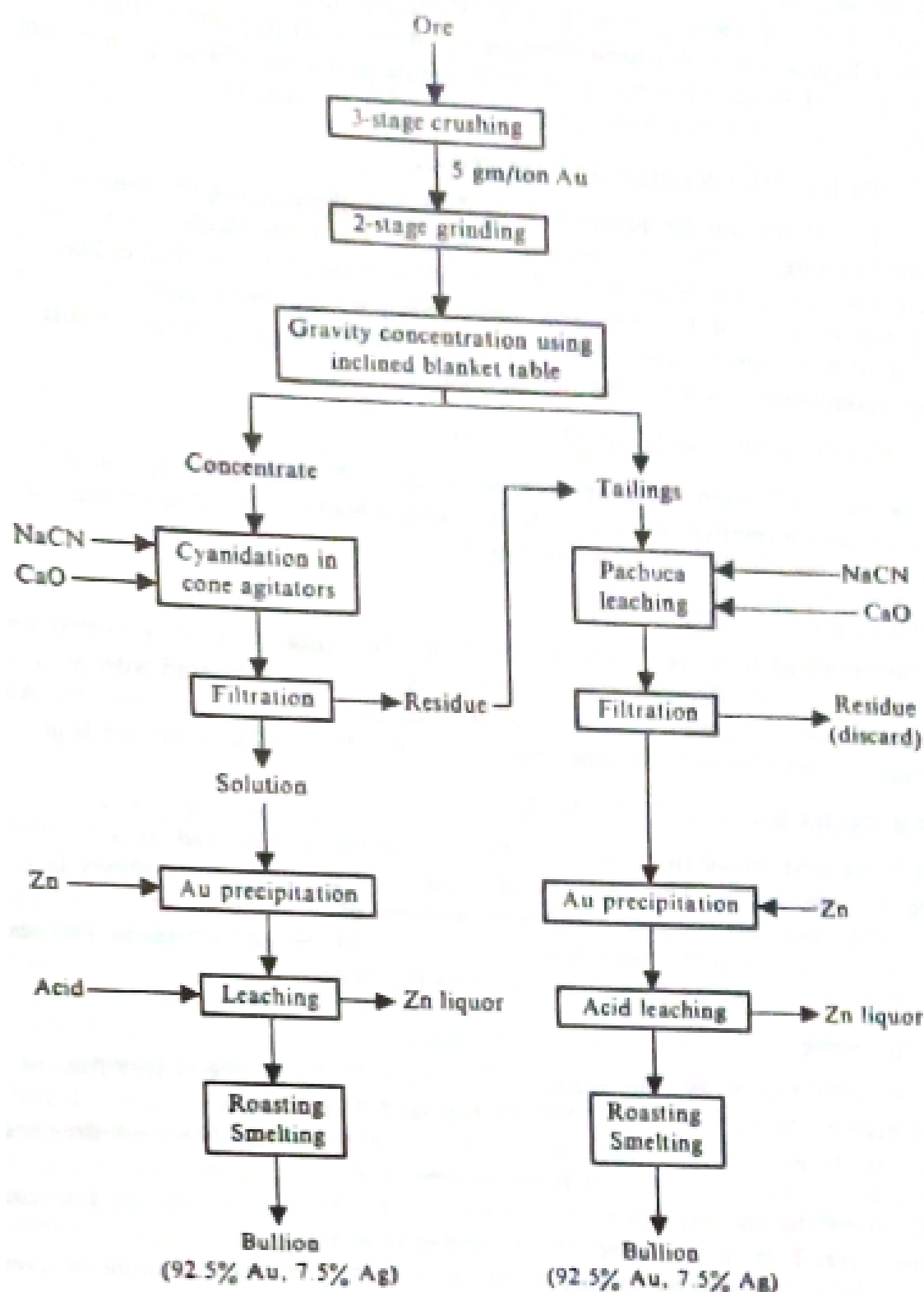
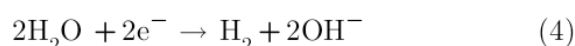
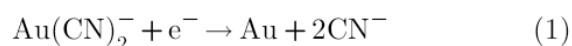


Fig.5.2 Flowsheet for the extraction of gold.

The common impurities in gold are silver, copper and platinum group metals. For purification process, this impure gold can be melted along with a flux (borax,silica or sodium carbonate) and then fire - refined. The metal can also be purified by the parting process, i.e, by leaching with hot sulphuric acid which dissolves only silver and baser metals and not gold and the platinum group metals. The platinum group

metals can be recovered by electrolyzing an aqueous solution of AuCl_3 using impure gold bullion as the anode and pure gold as cathode.

(ii) **Electrolytic process** : Gold can be extracted by electrolyzing from diluted cyanide solution. During gold winning, the following cathodic reactions should be considered.



The complex $\text{Au}(\text{CN})_2^-$ is reduced to metallic gold, under diffusion control(reaction-1) . Reactions (2) and (3) , representing oxygen reduction in alkaline solutions, are the main cathodic reactions competing gold deposition and consuming a great deal of the available current on the cathode. Reaction (4) represents the hydrogen evolution in alkaline solutions, along with gold deposition.

(iii) **Selective absorption process** : In selective absorption process, the complex gold cyanide is absorbed, at the room temperature, on a specially prepared carbon powder surface. When carbon powder is heated in the presence of water, the complex is released into aqueous phase. The metal is thus preferentially separated from the various other impurities. Subsequently, the metal can be recovered in the elemental form either by cementation or electrolysis.

CHAPTER 6

Production of Secondary Metals

6.0. Secondary metals and byproduct metals

Those metals that are recovered from scrap, are called secondary metals. Few examples of non ferrous secondary metals that are recovered from scrap are copper, lead, aluminium and zinc. Their recovery from scrap requires less energy than that required from the primary ore.

On the other hand, metals like selenium, bismuth and cadmium are called byproduct metals, since they are essentially obtained as byproducts in the extraction of other metals such as copper, lead and zinc.

6.1 Production of secondary copper

6.1.1 Sources of secondary copper :

- (i) From foundries - dross, spillages, skimmings etc.
- (ii) From fabricators - swarf, punchings, turnings, borings etc.
- (iii) From user industries - scrap cables, wire, radiators, brass, bronzes etc.

6.1.2 Recovery of copper from scrap

The following figure shows the flowsheet for the recovery of copper from scrap. First, the compacted scrap is smelted in a blast furnace along with a return converter slag, a flux and a reductant to produce a molten black copper. The slag is discarded. It contains Cu- 1 %, Sn - 0.8 %, ZnO - 10 %, CaO - 15 %, FeO - 35 %, Silica and alumina - 37 %. And the furnace gas contains zinc, tin and lead, and are sent to bag houses to recover valuable oxide fumes. The molten black copper is sent to a converter where high grade brass, bronze or gunmetal is added to it. Coke is fed into the converter as fuel. After converting, the slag contains 30-35 % Cu, 20% Zn or 15% Sn depending upon their uses in the converter. These slags are sent to the blast furnace.

In the fire-refining furnace, flux is added to the copper and air is blown upward through the mixture to oxidise the impurities. Fire-refined copper is cast into anodes, which are used in electrolysis. The anodes are submerged in a sulphuric acid solution containing copper sulphate. As copper is dissolved from the anodes, it deposits on the cathode. Then the cathode copper (99.9% Cu) is extracted and recast.

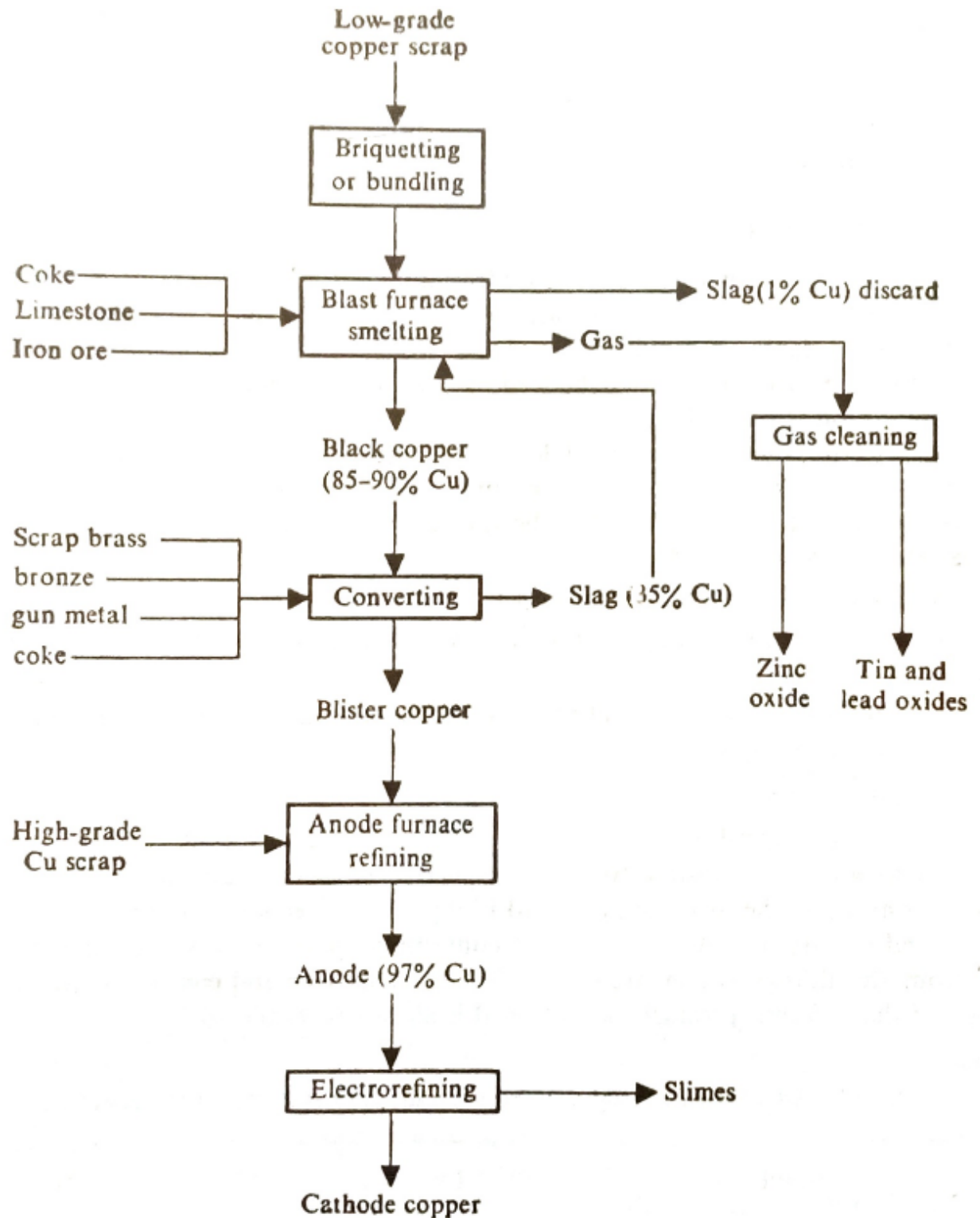


Fig.6.1 Production of secondary copper

6.2 Production of secondary lead

6.2.1 Sources of secondary lead

- (i) From lead scrap such as lead-acid battery plates
- (ii) Hard cable sheaths, type casts, babbitt alloys, solder scrap, lead pipes and sheets.

6.2.2 Recovery of lead from lead-acid battery scrap

A lead battery essentially consists of the following components as shown in the figure.

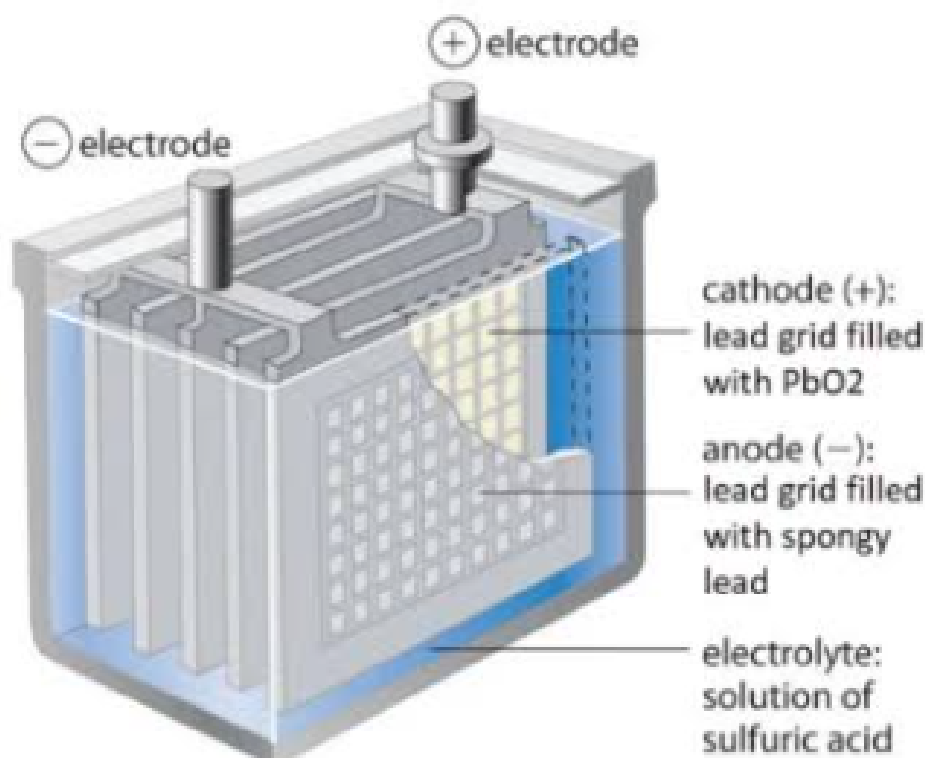


Fig.6.2 Lead- acid battery

- (i) A plastic container with an iron grip or handles.
- (ii) Grid plates and pole bridges made of lead and lead alloys containing upto 9% antimony.
- (iii) The filling mass which is essentially lead oxide or lead sulphate and free of antimony.
- (iv) Accumulator acid

The above fig.6.2. shows a lead- acid battery containing different components.

Lead can be recovered from the lead-acid battery scrap by **Fischer process**.

In this process, the acid and lead free components such as containers and separators are separated from the lead containing the metallic grids and fillings by mechanically breaking the batteries and by screening and gravity separation methods. The lead containing these grids which have antimony are separated from the fillings which are essentially antimony free and can be made into slurry. The grid metal is sent to a lead refining where kettles are used for lead and antimony recovery.. The middlings are into lead blast furnace, where the slurry is subjected to lead sulphide roasting to eliminate sulphur. The plastic materials are rejected.

6.3. Production of secondary zinc

6.3.1 Sources of secondary zinc

- (i) Scrap brass

(ii) Zinc dross (FeZn_{13}). : Dross is formed due to the reaction between molten zinc and steel as well as by the reaction of molten zinc with iron salts during the galvanising of steel. Dross contains a substantial amount of entrapped zinc.

6.3.2 Recovery of zinc from dross

Some of the important processes for the recovery of zinc from dross are given below:

1. Distillation process : This process is based on the relative instability of the intermetallic compound FeZn_{13} . When this compound is heated in a retort, the zinc vapours are distilled off and are condensed in a suitable condenser. Recovery up to 95% is achieved by this method. However, distillation process requires rather sophisticated equipment, skilled operation and general facilities for high temperature operation.

2. Aluminium process : In this process, zinc is displaced from FeZn_{13} by aluminium to form FeAl_3 . A small amount of aluminium is able to liberate a large amount of zinc. Since the melting point of FeAl_3 is higher and the density is nearly half that of zinc, the separation of zinc is easily facilitated.

Name	Location	Operator
Alloy steel plant	Durgapur, West Bengal	S.A.I.L

2.