

Aluminothermic process for the preparation of Ferromolybdenum and Ferrovandium

A. K. Suri
T. K. Mukherjee
C. K. Gupta*

ABSTRACT

The paper presents laboratory development of processes involving aluminothermic reduction, in open reactors, for the preparation of ferromolybdenum and ferrovandium starting from indigenously available resources. The coverage incorporates chemical processing that has been developed for treating the starting resources such as low grade sulphide concentrate/molybdenum scrap/molybdenum laden solution for molybdenum and vanadium bearing slag or vanadium sludge for vanadium to prepare suitable intermediates. These essentially constitute raw materials developed as alternative to the conventional ores for the two ferroalloys.

On the basis of the investigations carried out, it has been possible to project flow-sheets that warrant further exploitation for the preparation of ferromolybdenum and ferrovandium from indigenous resources.

Introduction

Ferro alloys of molybdenum and vanadium account for the major industrial scale application, as much as 80 pct or more, of these metals. The industrial method of production of ferromolybdenum is by the thermit process, where silicon and aluminium metal are used to reduce a charge mixture of molybdic oxide, and iron oxide in presence of lime or calcium fluoride. Similarly ferrovandium is produced by aluminothermic smelting of a charge composed of vanadium pentoxide, aluminium, lime/fluorspar, scrap iron and/or iron oxide. Lime or fluorspar in both cases act mainly to flux the alumina slag. In addition to metal oxide there may be an interest to use alternate sources—

oxide compounds of molybdenum or vanadium metals with iron or calcium like iron or calcium molybdate or vanadate. Chemically speaking these intermediates act as a joint carrier of the desired metal oxide (molybdic oxide or vanadium oxide) and lime or iron oxide. Their use in ferroalloy smelting would not require the extraneous addition of lime or iron oxide. In the case of ferromolybdenum an additional advantage of reducing the volatilization losses of molybdic oxide is achieved because, molybdic oxide being chemically bound would be much less prone to escape the smelting charge by volatilization. In fact molybdate or vanadate compound complexes are generated or can be generated during the processing of low grade

* The authors are with the Metallurgy Division, Bhabha Atomic Research Centre, Trombay, Bombay.

concentrates or secondary resources of these metals.

This paper deals with the chemical processes that have been developed to treat some of the indigenous resources to forms that could be used for converting these to their ferroalloys. The processes developed essentially involve taking the metal values in solution and then precipitating out desired complexes viz iron molybdate/vanadate or calcium molybdate/vanadate. These complex compounds have served as sources for producing ferroalloys of molybdenum or vanadium by open aluminothermic reduction in a refractory lined reactor.

PREPARATION OF FERROMOLYBDENUM

Resources

In India, the molybdenum resources are extremely limited. Molybdenum is occurring as a polymetal sulphide associated with copper and uranium ores in Bihar. There are also reports on the availability of molybdenum resource from the Khasi Hills (Assam), Sikkim, Madurai and Palani (Tamil Nadu), Ambamata (Gujarat), Kalan Khand (Madhya Pradesh) and in Kerala. Most of the resources are yet to be developed and utilized. The only small scale production of molybdenite that is presently taking place in the country is as a by-product of uranium ore processing at the Uranium Corporation of India Ltd. (UCIL) at Jaduguda in Bihar. In addition substantial quantity of molybdenum is being generated as a scrap and also as waste leach liquor from the electric filament lamp industry. To cite a typical example, one particular lamp industry is itself generating about 400 kg of molybdenum scrap and leach liquor containing about 2.5 tonnes of molybdenum per year. Considering the number of filament industries in the country a significant amount of molybdenum is available as an industrial waste which could be recycled.

Resource processing

The processing scheme for recovery of

molybdenum is dependent on the starting resource. The low grade concentrates are treated in a different way than the industrial wastes.

Processing of low grade concentrates

The Byproduct Recovery Plant of Uranium Corporation of India Ltd. generates two grades of molybdenite concentrates analysing about 25 pct and about 40 pct Mo starting from the uranium ore that contains 0.02 pct Mo. The concentrates as obtained contain significant amounts of harmful associations like copper, nickel, silica etc. It was therefore felt necessary to prepare molybdenum intermediates like iron or calcium molybdates that by virtue of their methods of preparation would be free of all the harmful associations. For this purpose concentrates were processed in two different ways. One was based on a combination of pyro and hydro-metallurgy involving roasting-leaching-precipitation and the other was a completely hydrometallurgical route involving electro-oxidation and precipitation.

Roast-Leach Process

In this process molybdenite concentrate was first roasted at 600-650°C taking care to avoid local overheating and temperature excursions to prevent volatilization loss of molybdenic oxide and sintering of the concentrates. The roasted calcines were then leached with hot soda ash solution to solubilize molybdenum as sodium molybdate. Some copper and silicon that are co-extracted with molybdenum in the strongly alkaline leaching conditions are separated by precipitating these out by reducing the alkalinity to slightly alkaline or neutral conditions. This sodium molybdate solution served as the starting source for the precipitation of calcium or iron molybdate.

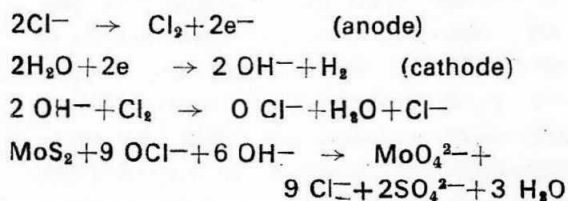
Calcium molybdate $\text{CaO} \cdot \text{MoO}_3$, was precipitated by slow addition of calcium chloride solution to the leach liquor maintained at a temperature of $90^\circ \pm 10^\circ \text{C}$ and a pH of 8-9 to obtain a product of maximum density consistent with maximum molybdenum extraction. If the mol-

ymolybdate solution was acidic, molybdenum recovery was poor. If on the other hand, the solution temperature was lower than the specified, the physical characteristics of the precipitate deteriorated. A fine precipitate, characterised by slow settling and slow filtration, resulted.

Ferrimolybdate with a general formula of $m \text{ MoO}_3 \cdot n \text{ Fe}_2\text{O}_3 \cdot 1 \text{ H}_2\text{O}$ (where m , n and 1 stand for the number of moles of MoO_3 , Fe_2O_3 and H_2O) was precipitated by adding ferric chloride solution to the acidified leach liquor maintained at a pH of 1 - 2.8. Maximum recovery of 99pct molybdenum was obtainable at a pH of 2.8 when the brick red coloured precipitate corresponding to a molybdic oxide content of 54.62 pct (after calcination at 500°C) was obtained. Yellowish green coloured precipitate obtained at a pH of 2 analysed 75.29 pct MoO_3 and corresponded to a recovery of 95 pct and the yellow coloured precipitate obtained at a pH of 1 analysed 83.91 pct MoO_3 and corresponded to a recovery of only 28 pct. Accordingly brick red coloured ferrimolybdate compound corresponding to $0.416 \text{ MoO}_3 \cdot 0.25 \text{ Fe}_2\text{O}_3$ (after calcination) was prepared and utilized for the investigations on the preparation of ferro alloys.

Electro-oxidation

The electro-oxidation process is essentially leaching of the molybdenite concentrate with sodium hypochlorite generated insitu by electrolysis of brine solution. The hypochlorite ions react with molybdenite to form molybdate ions and sulphate ions as per the reactions



The experimental procedure involves slurry-ing of the sodium chloride and the concentrate in water and carrying out the electrolysis using graphite electrodes. pH of the solution is maintained near neutral value by continuous addition

of sodium carbonate. Electrolysis is terminated when there is no further drop of pH or rather it tends to increase. At the end of electrolysis the slurry is filtered and the filtrate acidified to a pH of 2. A pre-determined quantity of calcium chloride is added to the filtrate to combine with the sulphate ion and precipitate as calcium sulphate. This step is essential before taking the solution for molybdenum recovery because any sulphur present in the solution would co-precipitate alongwith calcium molybdate. Starting from this purified filtrate the preparative process parameters for calcium and iron molybdate are exactly the same as those employed for treating the leach liquor from roast-leach process.

Table - 1 presents the general conditions for the processing of low grade concentrates by roast-leach and electro-oxidation methods, and the Table - 2 presents conditions for the preparation of molybdate compounds.

Industrial wastes

Molybdenum Scrap

Molybdenum scrap can be processed by a variety of methods like oxidative roasting, electro slag refining, electrolytic refining etc. Each of the process available thus far has its own limitation. Accordingly a new approach was examined which involved dissolution of the molybdenum scrap in an acid mixture comprising of 6-8 N nitric acid and 1-3 N sulphuric acid. It was observed that the scrap dissolution rate was enhanced significantly when a mixture of nitric and sulphuric acids was used in place of only nitric acid. Molybdenum bearing acid solution is then processed for the recovery of molybdenum values. Molybdenum can be recovered either as calcium or iron molybdate or as molybdic oxide. In the case of molybdenum recovery as molybdate it is necessary first to remove all the sulphur from the molybdenum bearing solution as was practiced for electro-oxidation route. The molybdates can then be precipitated under the conditions described earlier.

TABLE—1
*General conditions for the treatment of
low grade molybdenite concentrates*

Analysis of Concentrate : Mo 24.5%, SiO ₂ 3.5%, Fe 12% Cu 7.3%, Ni 4.7% S 30.75%			
<u>Roast Leach Process</u>		<u>Electro-oxidation</u>	
1	Roasting Conditions	Amount of conc.	1 kg
	Roasting temp 600-650°C	Amount of NaCl	2 kg
	Roasting Duration 8 hours	Temperature	30-38°C
2	Leaching Condition	Vol. of water	20 lit
	Soda Ash Solution 10%	pH	6-8
	Leaching temp. 90°C	Anode current density	0.5A/in ²
		Current	100 A
		Voltage	2.5-3.5
		Duration of electrolysis	22 hrs
		Molybdenum recovery with solution	92 pct
		Precipitation of sulphates at a pH of 2 by addition of prede-termined quantity of CaCl ₂	

TABLE—2
*Conditions for the preparation of calcium and
iron molybdates from leach liquor*

	<u>Calcium Molybdate</u>	<u>Iron Molybdate</u>
Temperature	90°C	25-75°C room temp.
pH	9	2.8
Excess over stoichio- metric requirements	25-50	0
Ignition temp.	600-700°C	500°C
Molybdenum recovery, pct	99	99

Recovery of molybdic oxide from molybde-
num bearing solution can be accomplished in
two ways - one is based on the direct heating
of the solution at 95°C to precipitate molybdic
acid which on ignition yields MoO₃. The other

method involves addition of ammonium hydro-
xide to the solution to precipitate ammonium
molybdate which on thermal decomposition
yields molybdic oxide.

Molybdenum recovered either as molybdic
oxide or as molybdate of calcium or iron can be
used for composing the charge for the prepara-
tion of ferromolybdenum.

*Molybdenum Leach Liquor from
Electric Filament Lamp Industry*

The leach liquor obtainable from the electric
filament lamp industry analyses 125 gm/lit. of
Mo, 6 N HNO₃ and 11-14 N H₂SO₄. This solu-
tion is essentially similar to that generated by
acid dissolution of molybdenum scrap described
earlier except that it additionally contains large
quantities of free sulphuric acid. To recover
molybdenum as molybdate compounds it would

be necessary to neutralize the acids and also to remove all the sulphate ions by precipitation of calcium sulphate at a pH of about 2. Precipitation of large quantities of calcium sulphate infact traps some of the molybdenum values. On account of the difficulties enumerated above and relatively high molybdenum content in the solution it is desirable to treat this type of lamp industry wastes to prepare molybdic oxides instead of molybdates. Recovery of molybdic oxide could be accomplished by precipitation of ammonium molybdate and then calcining it to MoO_3 , as indicated earlier for scrap processing.

Aluminothermic Process

Aluminothermic smeltings for the preparation of ferroalloys has been conducted in magnesite lined steel crucibles that could handle few grammes to few kilogrammes batch charges. Experimental studies were usually carried out on 100 g molybdate batches. The charge for smelting calcium molybdate was composed with iron oxide and aluminium and that of ferrimolybdate with aluminium. Weighed amounts of charge ingredients are first intimately mixed and then charged to the refractory lined vessels. The reaction was triggered by allowing a small amount of potassium permanganate and one spoon of glycerin placed at the top of the charge. After a short incubation period the reaction between glycerin and potassium permanganate takes place which generates adequate heat to initiate the desired thermit reaction. The reacted mass is allowed to cool to room temperature and the alloy button was separated

from the slag. The reduction reactions for calcium and ferrimolybdate can be represented as

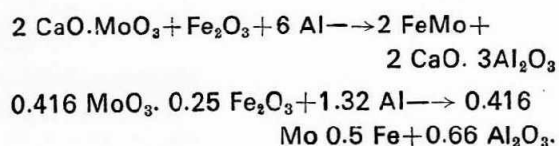


Table - 3 presents data on the aluminothermic smelting of calcium and iron molybdates.

PREPARATION OF FERROVANADIUM

Resources

Indian vanadium resources can be broadly grouped under two categories-primary resources and secondary resources. Titaniferous magnetites occurring at Singhbhum (Bihar), Masanikere (Karnataka), Khursipur and Bhandara (Maharashtra) and Mayurbhanj (Orissa) fall in the first category whereas the Indian bauxites come under the category of secondary resources. The primary sources can be treated in two ways either directly for the recovery of vanadium followed by that of iron and/or titanium or for the recovery of first iron by smelting and then for vanadium from the slag. During the caustic digestion of Indian bauxites about 30 pct of its vanadium content goes in solution whereas 70 pct remains behind in the red mud. During precipitation of aluminium hydroxide, after caustic digestion, dissolved vanadium does not coprecipitate alongwith alumina, therefore vanadium concentration gradually builds up in the caustic liquor during recycling. When the concentration of vanadium is built up to objection-

TABLE-3
Aluminothermic Reduction of Calcium and Ferrimolybdate

Charge composition, kg						Pct. Al excess over stoichiometric	Alloy yield %
$\text{CaO} \cdot \text{MoO}_3$	Fe_2O_3	MoO_3	Fe_2O_3	NaNO_3	Al		
0.1	—	—	0.068	0.01	0.055	0	96
—	0.1	—	—	—	0.0432	20	90

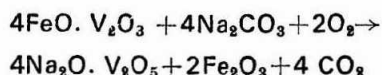
ably high level, the leach liquor is slowly cooled to precipitate vanadium as vanadium sludge. At present the aluminium industry recovers vanadium as sludge from the leach liquor only and no effort is being made to treat the red mud for the recovery of vanadium. The vanadium bearing magnetites and vanadium sludge taken together account for a rich vanadium resource and for its self sufficiency in the country. But these have not so far been developed into large scale use.

Resource processing

Processing of vanadium slag

Vanadium bearing slag analysing 16.5% V_2O_5 , 50% FeO, 16% SiO_2 , 6% Cr_2O_3 and 8% MnO was obtained from the pilot plant of M/s. Visvesvaraya Iron and Steel Ltd. (VISL) during three stage processing of titaniferous magnetite ore containing about 1 pct V_2O_5 . In the first stage, the ore is carbothermically smelted to yield vanadium bearing pig iron. In the second stage the metal is blown in a converter to generate a slag containing about 14 pct V_2O_5 and 57 pct iron oxide. In the third stage the slag is reduced once again with carbon to generate enriched vanadium bearing slag.

The slag is treated with soda ash under oxidising conditions in a rotary furnace to convert the contained vanadium into soluble sodium salts as per reaction



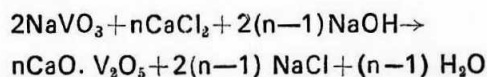
The roasted calcines are leached with hot water to solubilize sodium vanadate and the leach liquor processed for the precipitation of red cake (sodium hexavanadate) or calcium vanadate or ferrous vanadate. Like molybdate compounds these vanadates will act as joint carriers of vanadium oxide and lime or iron oxide. Accordingly it may not be necessary to make extraneous addition of lime or iron oxide when composing the charge for smelting of ferroalloys.

In the preliminary studies on the treatment of slags, effect of roasting temperature, amount of soda ash and soaking time on vanadium recovery showed that it was possible to obtain a maximum recovery of about 85 pct employing a treatment temperature of 800°C, and a soaking duration of 2 hours with a charge composed of slag and soda ash (20 pct by weight of the slag). Higher temperature resulted in reducing the recovery values due to the formation of insoluble vanado-silicate complexes. A sharp improvement in recovery value to 98 pct was obtained when a small quantity of potassium chlorate (5 pct of the slag charge) was added. This improvement is mainly due to the fact that the addition of an oxidant would convert all the vanadium (to its pentavalent form which would easily get extracted on water leaching. To sum up the optimum roasting conditions for vanadium recovery of 98 pct are—soda ash 20 pct by weight of the slag, potassium chlorate 5 pct by weight, temperature 800°C, duration 2 hours.

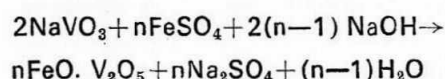
Precipitation of red cake (sodium hexavanadate) could be accomplished by acidifying the solution to a pH of 2-3, adding sodium chlorate and boiling the solution for 2-3 hours with continuous stirring. Since red cake or V_2O_5 are partially soluble in water as well as in acidic solutions, complete vanadium recovery cannot be accomplished in this step. Almost quantitative vanadium recovery is possible only by precipitating it in the form of insoluble compounds like calcium or iron vanadates.

Calcium vanadate was prepared by adding calcium chloride solution to the sodium vanadate leach liquor maintained at 90°C. Depending on the amount of calcium chloride added and the pH of the solution it was possible to precipitate either $2CaO \cdot V_2O_5$ or $4CaO \cdot V_2O_5$. The former was usually obtained from the neutral solution (pH 7) whereas the latter from a highly alkaline solution of pH 12. Vanadium recovery as calcium vanadate corresponded to about 99 pct when the amount of calcium chloride used was about 50 pct excess over stoichiometric

requirements. The precipitation reaction could be presented as



Ferrous vanadate was prepared by the addition of ferrous sulphate solution to the vanadium leach liquor. It was possible to obtain ferrous vanadate compound with a FeO to V_2O_5 mole ratio of 2 and 3 by adding essentially 125 pct of the stoichiometric amounts of FeSO_4 required as per the reaction



at a pH of 7 - 8. Precipitation of $4\text{FeO} \cdot \text{V}_2\text{O}_5$ on the other hand required higher pH of 12.

Vanadium recovered either as red cake or as calcium or iron vanadate serves as the starting material for the manufacture of ferrovanadium by the thermit process.

Processing of Vanadium Sludge

Vanadium sludge analyses 15-20 pct V_2O_5 . The oxide in the sludge is present in the form of water soluble complex sodium salts. Simple water leaching usually takes all the vanadium content of the sludge to the solution. The solution is then processed for the recovery of vanadium either as red cake, or as calcium or iron vanadate. The processing steps for any of the said vanadium compounds are essentially the same as that employed for treating the sodium vanadate leach liquor obtainable from water-leaching of roasted calcines during the processing of vanadium bearing slags.

Table - 4 presents general conditions for the precipitation of vanadate compounds from the leach liquor obtainable either from the processing of vanadium bearing slag or sludge.

Aluminothermic process

Aluminothermic smelting of ferro-vanadium has been conducted essentially in the same

TABLE- 4
Conditions for precipitation of calcium and iron vanadates

Salt added	Amount of salt required to form the compound $2\text{FeO} \cdot \text{V}_2\text{O}_5$ or $2\text{CaO} \cdot \text{V}_2\text{O}_5$	pH	Vanadium recovery	Mole ratio n
CaCl_2	150	7	99	2
CaCl_2	200	12	99	4
FeSO_4	100	7	93	1.989
FeSO_4	150	9	99	3.04
FeSO_4	200	12	99	4.1

way as that of ferromolybdenum. The smelting charges were composed either with ferrous vanadate, aluminium and lime/fluorspar or with calcium vanadate, aluminium and iron oxide (Fe_2O_3). In both cases compounds of the type $2\text{FeO} \cdot \text{V}_2\text{O}_5$ or $2\text{CaO} \cdot \text{V}_2\text{O}_5$ were preferred because of their higher vanadium contents. The powdered charge was intimately mixed, placed in an open top magnesia lined steel reactor and triggered with a potassium permanganate-glycerine mixture. The aluminothermic reaction proceeded as per the reactions

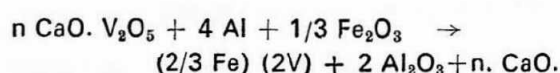
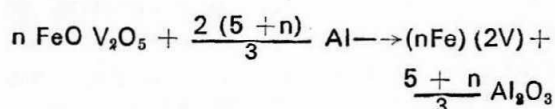


Table - 5 summarizes the conditions for conducting the thermit reaction with different vanadate compounds to produce ferrovanadium analysing about 40 pct of vanadium content. It can be seen that the alloy yields were higher from $2\text{CaO} \cdot \text{V}_2\text{O}_5$ than from $4\text{CaO} \cdot \text{V}_2\text{O}_5$, this may be due to the higher lime content of $4\text{CaO} \cdot \text{V}_2\text{O}_5$ which may be adversely influencing the yield. It may also be noted that increasing the batch size of thermit reduction has a salutary effect on the alloy yield. This is mainly attributed to improved utilization of thermal effect of aluminothermic reduction.

TABLE-5
Aluminothermic reduction of calcium and ferrous vanadate

Charge composition							Pct Al excess over stoichio- metric	Alloy yield (40%V)
2CaO. V ₂ O ₅	4CaO. V ₂ O ₅	2FeO. V ₂ O ₅	Fe ₂ O ₃	CaO	CaF	Al		
0.1	—	—	0.0711	—	—	0.0586	10	91
—	0.1	—	0.0528	—	—	0.0436	10	81
—	—	0.1	—	0.01	—	0.045	20	82
—	—	0.4	—	0.04	—	0.164	10	90

Summary

Development of raw materials for refractory metal ferroalloys is of immediate interest to the country and considerable amount of work is being carried out in various laboratories in this direction. In this paper laboratory feasibilities have been demonstrated for the utilization of the secondary resources/industrial wastes of

molybdenum and vanadium to prepare their ferroalloys and other industrially usable products. Fig. 1-3 show summarized flowsheets for the treatment of low grade molybdenite concentrate, molybdenum scrap, leach liquor from electric filament industry, treatment of vanadium slag and sludge. These flowsheets show various alternate routes that lead to production of a variety of industrial products.

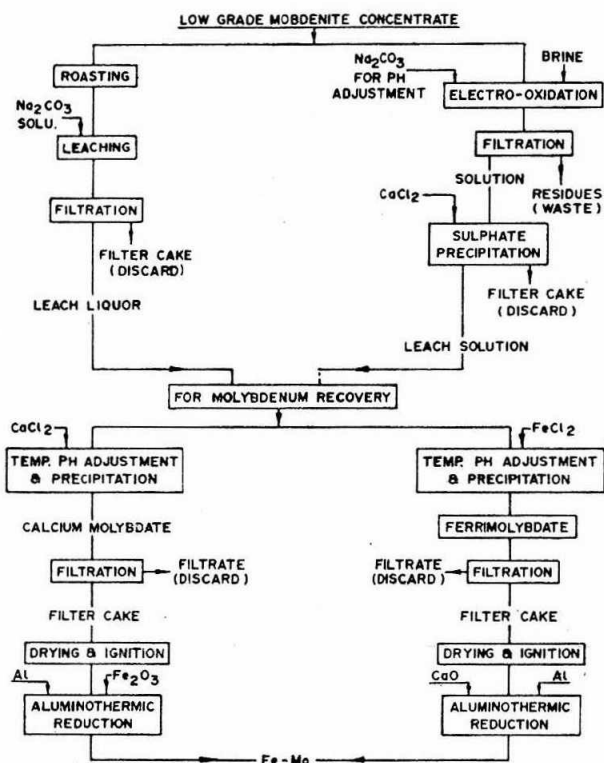


FIG. 1 - FLOW SHEET FOR THE TREATMENT OF LOW-GRADE MOLYBDENITE CONCENTRATE.

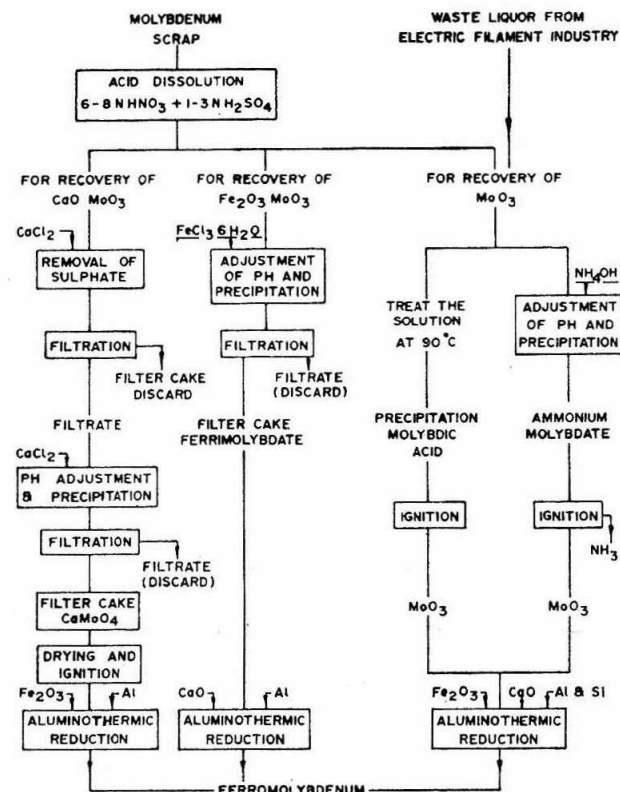


FIG. 2 - FLOW SHEET FOR THE TREATMENT OF MOLYBDENUM INDUSTRIAL WASTES.

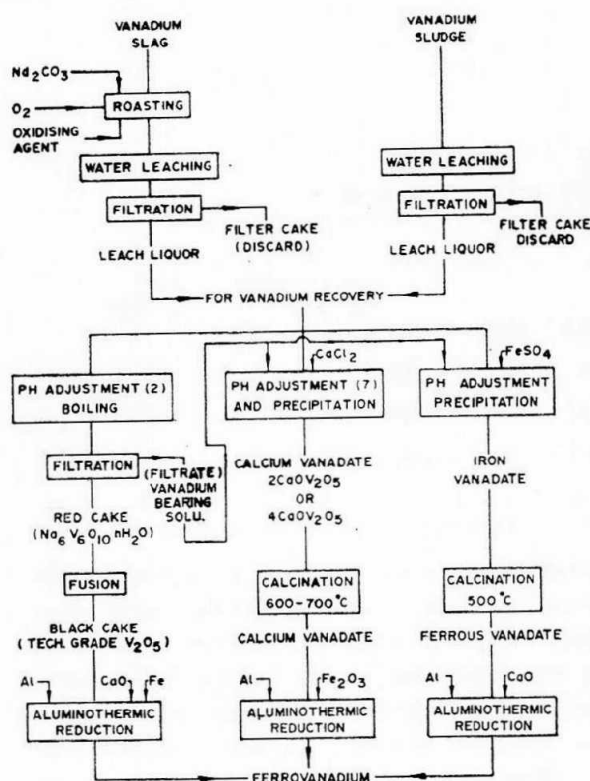


FIG.3-FLOW SHEET FOR TREATMENT OF VANADIUM RESOURCES - SLAG AND VANADIUM SLUDGE.

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Discussion

N. A. Subramanian, Paramount Sinters Pvt. Ltd., Nagpur

Q. Why is the copper and nickel content in UCIL Moly concentrate so high? In I. B. M., Moly concentrate with less than 1% copper could be produced by acid working of flotation concentrates and doing additional cleaner flotations?

A. Yes it is possible to bring down the copper percentage. BARC also worked on this technique in 1971 but later had to give up this method due to economic considerations.

Q. At I. B. M. several Moly ores from Karadikuttam (Tamil Nadu) have been investigated and also the ores from Rakha Copper Mines. In both the cases copper content in moly concentrate were lower.

A. Yes. One can obtain cleaner concentrates with high Mo and low Cu/Ni but the recovery of moly goes down significantly.

Q. Is the copper and nickel content in UCIL moly concentrates high due to insufficient liberation? Did they try finer grinding?

A. Not due to insufficient liberation but insufficient cleaner operation. Higher grade moly can be obtained with much lower recoveries.

R. Y. Sane, Paramount Sinters Pvt. Ltd., Nagpur

Q. What was the scale of experiments tried at BARC for these processes, when you have mentioned recovery of 99%? Has VISL offered any Pilot plant tests of these processes, since Vanadium bearing slag is one of their by-products.

A. The scale of operation at BARC has been from 100 gms of oxide compound to 10 kg of charge mix. 99% of recovery is in the precipitation of compounds during hydro-processing. VISL had sent a sample for trial purposes but they have not shown any interest on pilot plant trials. BARC also has not pursued the matter further.

A. K. Tripathy, Sct., RRL, Bhubaneswar

Q. What is the advantage of the process over using Red cake as starting material? How much of Aluminium is used and how much recovered?

A. The red cake is not a desirable starting material because of its high sodium content. The amount of aluminium used depends on the ferro-alloy being made and the amount of aluminium tolerable in the ferro alloy. Any excess of aluminium beyond an optimum amount for obtaining maximum recovery ends up as an addition of aluminium impurity in the ferro alloy.

A. S. Ray, Alloy Steels Plant, Durgapur

Q. What are the proportions of the various charge constituents for the production of FeV and FeMo by aluminothermic process? In which form the iron is introduced and what is the method of ignition i.e. top priming or bottom priming.

A. Normally iron is introduced in the ferro alloy as mill scale or iron ore or steel scrap. For ignition top priming using KMnO_4 and glycerine was utilised. As we are transferring the technology of producing the ferro alloys to private parties through the Technology Transfer Group of BARC, I would not like to divulge any information on charge compositions in fairness to our transferees.