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GOVERNMENT OF INDIA
ATOMIC ENERGY COMMISSION

PREPARATION OF COPPER-BERYLLIUM ALLOYS FROM INDIAN BERYL

by

C. M. Paul, B. P. Sharma, K. S. Subba Rao, M. G. Rajadhyaksha
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Metallurgy Division

BHABHA ATOMIC RESEARCH CENTRE
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ABSTRACT

The paper presents the results of laboratory scale investigations on the preparation of copper-beryllium and aluminium beryllium master alloys starting from Indian beryl and adopting the fluoride process. The flow-sheet involves: (1) conversion of the Be-values in beryl into water soluble sodium beryllium fluoride, (2) preparation of beryllium hydroxide by alkali treatment of aqueous Na_2BeF_4 , (3) conversion of $\text{Be}(\text{OH})_2$ to $(\text{NH}_4)_2\text{BeF}_4$ by treatment with NH_4HF_2 , (4) thermal decomposition of $(\text{NH}_4)_2\text{BeF}_4$ to BeF_2 and (5) magnesium reduction of BeF_2 (with the addition of copper/aluminium) to obtain beryllium alloys.

The method has been successfully employed for the preparation of Cu-Be master alloys containing about 8% Be and free of Mg on a 200 gm scale. An overall Be-recovery of about 80% has been achieved. Al-8% Be master alloys have also been prepared by this method.

Toxicity and health hazards associated with Be are discussed and the steps taken to ensure safe handling of Be are described.

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1. INTRODUCTION

Beryllium has come to be recognised as an important strategic material with growing applications in the fields of electronics, space-science, rocketry, nuclear power generation and aeronautics^(1,2,3). The major use of the metal is in the form of copper-beryllium alloys (0.25-2.05% Be) which have excellent mechanical and physical properties, namely superior fatigue and tensile strength, high thermal and electrical conductivity and good resistance to corrosion and wear. Copper-beryllium alloys are extensively used in the electronic, electrical and other industries for the manufacture of springs, diaphragms, electrical contacts, switches, non-sparking tools, dies, bearings, marine propellers etc.

Beryllium oxide has a high melting point (2550°C), large specific heat (0.30 cal/gm at 100°C), exceptional thermal conductivity, good thermal shock resistance and superior electrical resistance. It finds largest use in the electronic industry as a heat sink, and also in the manufacture of various ceramics.

The principal source material for beryllium is the mineral beryl ($3 \text{ BeO} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2$) which occurs in granite pegmatites and is recovered as a co-product or by-product with feldspar, mica, lithium, tantalite, columbite and cassiterite. The world production⁽⁴⁾ of beryl (equivalent to 11% BeO) has been estimated at about 8,000 tonnes in 1969, the main producer countries being Brazil, India, U.S.S.R., Argentina, South Africa, Uganda and Rwanda.

India has fairly large deposits of beryl in Bihar, Rajasthan, Andhra Pradesh and Tamil Nadu, the analysis of a typical grade being as follows:

BeO	-	11.21 %
SiO ₂	-	63.86 %
Al ₂ O ₃	-	18.60 %
Fe ₂ O ₃	-	2.29 %
CaO	-	0.45 %
MgO	-	0.28 %
P ₂ O ₅	-	0.13 %

Considering the raw material resources in the country and the present and projected indigenous requirement of Cu-Be alloys, Be-metal and beryllium compounds in the electronic and electrical industries, and space research and nuclear energy programmes, a development programme on the preparation of beryllium metal and its alloys and compounds has been undertaken at the Bhabha Atomic Research Centre, Bombay. The paper describes the development of the process flow sheet for the preparation of BeO, BeF₂ and Cu-Be and Al-Be master alloys - starting from beryl.

2. PROCESS SELECTION AND FLOW SHEET

The first steps in the extraction of beryllium from its ore are (1) treatment of the ore for the preparation of Be(OH)₂ and (2) conversion of Be(OH)₂ to a suitable intermediate followed by reduction to metal or alloy. For opening up the ore, 2 methods have found industrial acceptance -

- (1) the sulphate process involving sulphuric acid digestion of thermally treated (heating and quenching) or chemically treated (fusion or sintering with alkali or alkaline earth carbonates)

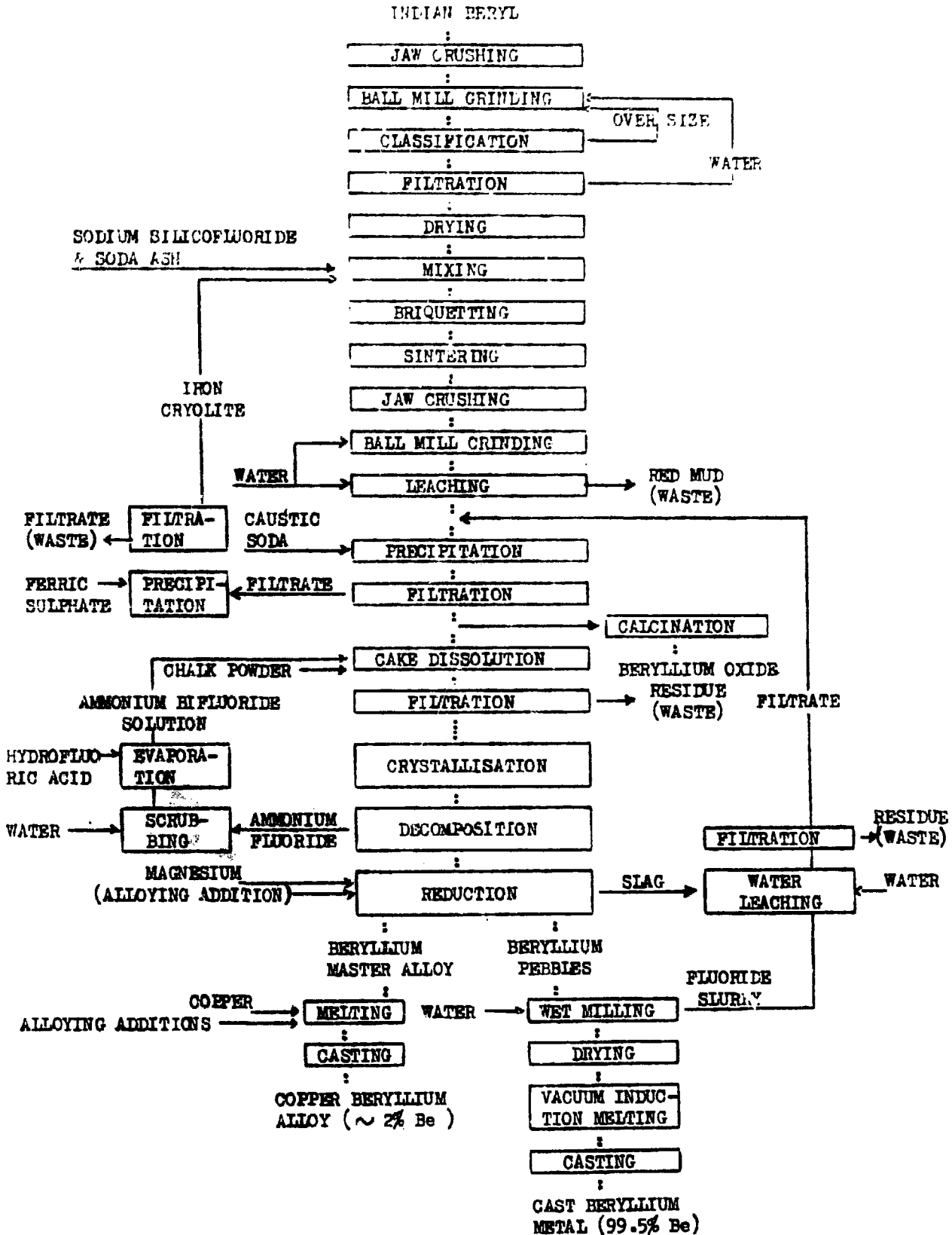


FIG.-1 : FLOW SHEET FOR THE PRODUCTION OF BERYLLIUM METAL AND ALLOYS.

beryl followed by purification of the sulphate solution and conversion to $\text{Be}(\text{OH})_2$.

& (ii) the fluoride process involving sintering of beryl with Na_2SiF_6 and Na_2CO_3 followed by extraction of the Be-values by aqueous leaching of the sinter and conversion of the extract to $\text{Be}(\text{OH})_2$.

In the present investigations, the second method - the fluoride process - was chosen for the following reasons :

- (1) Comparative cost estimates available in the literature⁽⁵⁾ on the production of $\text{Be}(\text{OH})_2$ indicate that this route is the more economical.
- (2) The process does not involve high temperature operations.
- (3) The process is more selective in that only the beryllia content of beryl is attacked, leaving behind the major constituents - Al_2O_3 and SiO_2 - which account for about 90% of the ore.

There are two methods available for the preparation of Cu-Be master alloys starting from $\text{Be}(\text{OH})_2$: (i) carbothermic reduction of BeO at temperatures above 2000°C in presence of copper in an arc furnace, and (ii) magnesiothermic reduction of anhydrous BeF_2 (in presence of copper).

The method of magnesiothermic reduction was preferred in the present investigations as :

- (1) Not only Cu-Be, but also Be-metal and Al-Be master alloys can be produced by this method,
- (2) The operation is comparatively dust-free and hence less hazardous,
- (3) The reduction is carried out at relatively lower temperatures ($1300 - 1400^\circ\text{C}$),

- (4) Cu-Be master alloys containing 8% and more of beryllium can be prepared by this method (alloys containing upto only 4% Be are obtained by the first method).

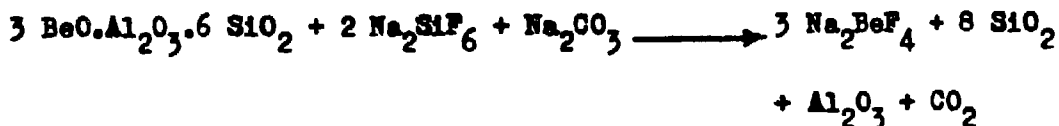
The integrated flow sheet - shown in Fig. 1 - consists of the following main operations :-

- (1) Conversion of the beryllium values in beryl to water soluble sodium beryllium fluoride,
- (2) Preparation of $\text{Be}(\text{OH})_2$ by NaOH treatment of Na_2BeF_4 solution,
- (3) Conversion of $\text{Be}(\text{OH})_2$ to $(\text{NH}_4)_2\text{BeF}_4$ by treatment with $(\text{NH}_4)\text{HF}_2$,
- (4) Thermal decomposition of $(\text{NH}_4)_2\text{BeF}_4$ to BeF_2 .
- (5) Production of Be-metal and its alloys by magnesium reduction of BeF_2 .

3. EXPERIMENTAL

(1) Conversion of the Be-values of beryl to water soluble double fluoride

The opening up of beryl by sintering with sodium silicofluoride has been the subject of quite a few investigations in the past^(6,7). The reaction proceeds according to the equation



It has been reported that the addition of Na_2CO_3 to the charge reduces the consumption of Na_2SiF_6 and prevents the loss of fluorine as SiF_4 . From a number of tests carried out in the laboratory the optimum conditions for maximum Na_2BeF_4 recovery were established as follows :

1. Charge composition

Beryl : Na_2SiF_6 : Na_2CO_3 - 11.15 : 8.1 : 0.95

2. Sintering temperature - 700°C

3. Sintering time - 4 hrs.

Beryl containing about 11% BeO (50-70 m.m. size) was first crushed in a jaw crusher to a size of about 6 m.m. and then wet ground in a ball mill to -200 mesh powder. The powder after drying was mixed with Na_2SiF_6 and Na_2CO_3 in the above ratio and compacted at a pressure of 5 tons/sq. in. into 2.5 cms dia x 2.5 cms long briquettes. The briquettes were then charged in salamander crucibles and sintered in a vertical tube electric resistance furnace. The charge was then heated at the rate of 400°C/hr and soaked at the operating temperature of 700°C for 4 hrs.

After cooling, the briquettes were taken out and wet ground in a ball mill to a size of about -200 mesh. The ground sinter (slurry) was then leached thrice, each time for $\frac{1}{2}$ hr with water at room temperature in AISI-316L stainless steel vessels keeping a fluo-salt to water ratio of 1 : 7 in order to extract the soluble beryllium values. After each leach, the solids were allowed to settle and the solution is separated by decantation and filtration.

(ii) Preparation of $\text{Be}(\text{OH})_2/\text{BeO}$

The conversion of Na_2BeF_4 into BeO involves (a) precipitating the Be-values as hydroxide using NaOH, (b) filtering and washing the hydroxide free of fluoride, and (c) drying and calcining the hydroxide.

During initial investigations the hydroxide was prepared by the addition of stoichiometric amount of NaOH into a cold Na_2BeF_4 solution containing about 4-5 gms of BeO/litre. The hydroxide obtained by this method was slimy and difficult to filter.

It has been reported⁽⁸⁾ that the hydroxide precipitated in a hot alkaline medium is granular and easily filterable. This has been confirmed in the laboratory scale investigations. For best results the entire quantity of NaOH (20% solution) was taken in an AISI 316 stainless steel vessel and treated with 20% of the stoichiometric amount of Na_2BeF_4 solution (4-5 gms of BeO/litre) required for the reaction $\text{Na}_2\text{BeF}_4 + 2 \text{NaOH} \longrightarrow \text{Be}(\text{OH})_2 + 4 \text{NaF}$. The resultant alkaline solution was heated to about 95-100°C and granular $\text{Be}(\text{OH})_2$ was precipitated from the solution, under continuous agitation, by the progressive addition of the remaining 80% of the double fluoride solution. The slurry was then allowed to settle and the hydroxide was repulped and filtered to remove last traces of fluorides. The dried hydroxide was calcined at 900-1000°C in beryllia lined salamander (clay bonded graphite) crucible.

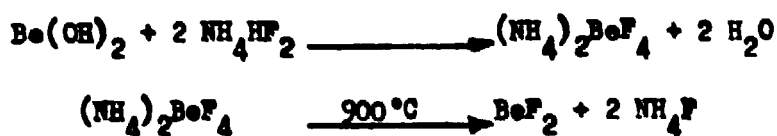
The filtrate from the hydroxide filtration contains valuable fluorine (as NaF) which is recovered and recycled in the process. It is mentioned in the literature that fluorine can be recovered as iron cryolite by the reaction



In the present work the filtrate (NaF-25 gm/litre) was acidified to a pH of 4 with HCl and treated at room temperature with stoichiometric amount of FeCl_3 (30 wt% aqueous solution). The fluorine present in the solution was recovered as insoluble Na_3FeF_6 .

(iii) Preparation of anhydrous BeF₂ from Be(OH)₂

Owing to the low volatility, high hygroscopicity and hydrolytic nature of BeF₂, its preparation in a pure and anhydrous form by the fluorination or hydrofluorination of the oxide is difficult. The accepted method for the preparation of BeF₂ in the anhydrous and pure form from Be(OH)₂ is based on the following reactions



The hydroxide cake was transferred into high density polythene vessels and agitated with stoichiometric amount of a 50% solution of NH₄HF₂ for the formation of (NH₄)₂BeF₄. The (NH₄)₂BeF₄ solution formed was filtered and evaporated on water bath in high density polythene vessels to crystallise out the double fluoride salt. The crystals were filtered out from the mother liquor, and dried before being fed to the subsequent decomposition stage.

The decomposition of (NH₄)₂BeF₄ is an intricate step in the flow sheet for Be-metal production on account of the corrosive nature of the molten salt and its tendency to excessive frothing during decomposition. In the earlier preliminary investigations when gram quantities of the double fluoride were decomposed in batches in platinum dish, it was observed that the fluoride frothed excessively during decomposition resulting in the over-flow of the material.

In order to overcome this problem, a graphite assembly was developed for the later investigations. The design of the decomposition

assembly was such as to permit the continuous removal of the fluoride from the hot zone, in order to eliminate frothing due to bubbling of NH_4F vapours through molten BeF_2 . The assembly consisted essentially of an induction heated graphite tube (3" I.D., 1' long) kept inclined to the horizontal. The double fluoride was fed at the top end of the tube and the molten BeF_2 flowing out through the other end was cast in a graphite mould. The by-product NH_4F was collected in a water cooled AISI-316 stainless steel condenser connected at the top end of the graphite tube.

(iv) Preparation of beryllium and its alloys
by the magnesium reduction of BeF_2

Possible reducing agents for BeF_2 are sodium, potassium, calcium and magnesium. Out of these, sodium and potassium are more difficult to handle, while calcium combines with Be to form a stable intermetallic (CaBe_{13}). Magnesium is however the most suitable metal for the reduction of BeF_2 (9,10,11,12).

Magnesium in the form of chips (25 mm x 25 mm x 25 mm) was taken in a graphite crucible and the base metal (Cu or Al) - also in the form of chips - (25 mm x 25 mm x 3.5 mm) was placed over it. Anhydrous BeF_2 was then charged over the metals and the crucible was finally covered with a graphite lid. The crucible containing the charge was then heated in a 12 k.w. induction furnace and held at 1100°C for $\frac{1}{2}$ hour for the completion of the reduction reaction. The temperature is finally raised to $1300-1400^\circ\text{C}$ and held for another $\frac{1}{2}$ hour to increase the fluidity of the MgF_2 slag and effect slag-metal separation. During this period the charge was kept agitated for 15 minutes with a graphite stirrer and then cast in a cold

graphite mould. After separating the alloy, unreacted BeF_2 in the slag was leached out with water and recovered for recycling. The alloys after remelting were cast and their microstructures examined.

Similar experiments were also carried out for the preparation of Al-Be alloys.

In the electronic and electrical industries Cu-Be alloys containing about 2% Be (with minor alloying additions like Co, Ni, Sn, Zn etc) are used in the form of foils, wires and rods. The master alloy was therefore diluted with the required amount of copper in an induction furnace. The diluted alloy was then cast in graphite moulds and hot rolled at 790°C after encasing in mild steel. The alloy was then unsheathed and further cold rolled to size with intermittent solution treatments at 790°C for 1 hour.

4. RESULTS AND DISCUSSION

(a) Sintering of beryl

In a series of sintering experiments (on a scale of 1.25 gms of contained BeO), the influence of time and temperature on the recovery of beryllium values of beryl as water soluble Na_2BeF_4 was examined (Table 1).

It will be observed from the Table that a maximum recovery of 96% was obtained when the sintering was carried out at 700°C for 4 hours. With the increase in sintering temperature, the maximum recovery came down owing probably to the formation of insoluble Be-compounds and also the decomposition of silicofluoride resulting in the loss of fluorine as SiF_4 . On a scale of 50 gm of contained BeO however marginal decrease in the recoveries was observed - 94.5% and 92.5% for 700°C (4 hours) and 750°C (2 hours) respectively. •



(a)



(b)



(c)



(d)

Plate 1 - Photomicrographs of as cast master alloys of Cu-Be and Al-Be etched in aqueous 1-3 wt% FeCl_3 solution

(a) Cu-3.9% Be; B.F.; x 200

(b) Cu-3% Be; B.F.; x 560

(c) Cu-24.2% Be and 1.25% Mg; B.F.; x 100

(d) Al-9.52% Be and 0.24% Mg; B.F.; x 560

(b) Preparation of anhydrous BeF₂

Easily filtrable granular Be(OH)₂ could be prepared by the progressive addition of the double fluoride (Na₂BeF₄) solution into a hot alkaline solution containing stoichiometric amount of NaOH and Na₂BeO₂ formed by the addition of 20% of the total double fluoride solution. The recovery was 97%.

Fluorine present as NaF in the hydroxide filtrate could be recovered as Na₃FeF₆ at a recovery of about 90%.

Decomposition of (NH₄)₂BeF₄ was studied on both batch and continuous process. Batch operations were rendered difficult on account of excessive frothing during decomposition. Continuous decomposition of (NH₄)₂BeF₄ was however found to work satisfactorily and with the graphite assembly set-up for the purpose it was possible to obtain BeF₂ at the rate of 500 gm/hr at a recovery of 97%.

(c) Preparation of Be-alloys

In the preparation of Cu-Be alloys, the influence of excess BeF₂ in the charge on the residual Mg-content in the final alloy was investigated. The residual Mg-content in the alloy was found to decrease from 2% to traces (Table 2) when the BeF₂ excess was varied from 6% to about 25% in the charge.

Under the same conditions with the BeF₂ content of the charge fixed at 25% excess of the stoichiometric requirement, as the beryllium loading in the final alloy was increased from about 8% to 23% (on the basis of Mg-charged) Be-recovery was found to decrease from 85.8 to 77.6% (Table 3). Residual Mg-content in the alloys containing more than 6% Be has been found to be around 0.5%. It can be seen that the Be-recovery in

the preparation of Cu-8% Be alloy is only 85.8% even though the alloy is almost free of residual Mg. The following possible reasons can be attributed to this - (1) oxidation and volatilisation of Mg during reduction, (2) reduced Be remaining in the slag without getting dissolved in molten copper and (3) carbide formation (by reaction with crucible material) and oxidation of reduced Be.

After recovering the unreacted BeF_2 from the slag by aqueous leaching the Be-recoveries based on beryl and BeF_2 were found to be about 80% and 90% respectively in the preparation of 8% Be master alloys.

The higher beryllium recovery observed in the preparation of the 8% alloy can be attributed to the better slag-metal separation resulting from the larger difference in the densities of the alloy and the slag. It can also be observed from the photo-micrographs (Plate 1) that the 8% alloy is more uniform in composition as compared to the 23% alloy. This is possibly due to the eutectoid reaction that takes place in this composition range. In view of the above, the preparation of 8% Be-master alloy appears to be more attractive for commercial exploitation.

By employing similar techniques it was possible to prepare Al-Be master alloys containing about 8% Be and 1.2% residual magnesium on a 100 gm scale at a recovery of 90% (on the basis of Mg charged) by the reduction of BeF_2 (taken 25% excess) with Mg in presence of Al at 1100-1400°C.

The possibility of preparation of copper-beryllium alloys at much lower temperature with the incorporation of magnesium chloride flux in the charge was examined and it was possible to obtain alloys analysing about 8% Be and free of magnesium at a recovery of 84.5% by reduction of a

charge containing 25% excess BeF and MgCl₂ (MgF₂ : MgCl₂ : : 1:1) at 1000°C for ½ hour. In this case the selective recovery of unreacted BeF₂ from the slag by water leaching is not possible as both MgCl₂ and BeF₂ are soluble in water. However, Be can be selectively precipitated as Be(OH)₂ and recovered, by treatment of the leach liquor with NH₄OH.

In the preparation of Al-Be alloys with a charge containing 100 gms of Al, MgCl₂ flux, (MgF₂ : MgCl₂ : : 1:1), 25% excess BeF₂ and Mg adjusted to give 9% Be loading in the final alloy, as the reduction temperature was increased from 1000°C to 1300°C the Be-loading increased from 4% to about 8% and the Be-recovery (on the basis of Mg-charged) in the alloy from 41 to 90.5%. This is attributed to the increase in the liquid solubility of Be in Al with temperature.

(d) Fabrication of Cu-Be alloy strips

A 12 mm thick cast alloy containing about 1.6% Be and 0.3% Co was rolled to a final thickness of 0.216 mm. The alloy encased in mild steel was first hot rolled at 790°C to a thickness of 3 mm, unheated and further cold rolled to the final thickness. During cold work when the hardness rose to 200 V.P.N., it was solution treated (790°C for 1 hour) prior to further cold working. The sheet on age-hardening gave a hardness of 270 V.P.N.

5. TOXICITY AND HANDLING OF BERYLLIUM

Beryllium and its compounds are extremely toxic and if deposited in the body, especially in lungs, cause extensive local damage. When deposited in lungs they cause reduction in respiratory capacity and

general debility (berylliosis) terminating in tuberculosis or death in severe exposures. In case of skin contamination severe skin ulcers and granuloma are observed particularly on hands and face.

In view of this, adequate precautions^(13,14,15) have been taken in the laboratory while handling beryllium and beryllium compounds. The laboratory consists of two rooms (each 20' x 20' floor area and 12' height) for the handling of beryllium, and a change room (10' x 20' floor area and 12' height) all located at one end of the top most floor (3rd floor) of a building. The walls and roof of the laboratory are epoxy painted to minimise accumulation of powder and the floors are covered with PVC tiles to facilitate easy decontamination. All operations involving handling of beryllium compounds* have been carried out under fumehoods and inside glove boxes connected to a common ventilation systems. The laboratory is equipped with 6 laboratory type fume hoods (4½' x 2½' area 3½' height) and a special walking type fume hood (10' x 5' area x 10' height) all having glass shutters, a canopy hood (2' x 2') and a glove box (3' x 5' area x 3' height). The change room accommodates a washing machine (for the laundry of work clothes) an emergency shower and racks and store-wells for keeping protective wears and clothings.

The laboratory is connected to a ventilation system (located on the terrace) which provides an air-velocity greater than 150 ft/min at all hood openings and the laboratory air is passed through a system of roughing and absolute filters assembled in a filter house, before letting out into the atmosphere. The air discharge rate is 250 m³/min

* Of the compounds of beryllium, beryl is non-toxic and hence a facility existing outside the laboratory (in the Ore Dressing Section of the Metallurgy Division) has been made use of for the crushing and grinding of beryl.

which has been achieved by means of a centrifugal blower having a discharge head of 150 mm of water. This also provides about 30 air changes per hour in the laboratory.

Samples of in-plant and environmental air are taken periodically to determine the concentration of beryllium. The daily average in-plant air concentration of beryllium has been maintained at less than 2 micrograms per m^3 while the environmental contamination has not been allowed to exceed 0.01 micrograms per m^3 , in accordance with Health Physics Regulations. A surface contamination tolerance level of 10 micrograms per m^2 has been adhered to. Steps have been taken to ensure that the effluents discharged into public streams do not have a beryllium concentration exceeding 1 ppm.

In view of the extreme toxicity of beryllium, the disposal of wastes from beryllium industry has assumed greater significance. In the laboratory the solid wastes sealed in polythene bags and packed in mild steel drums are disposed of by ground burial. The liquid wastes on the other hand, after the removal of Be-values by chemical treatment are discharged to common drains at safe discharge levels (1 ppm).

A thorough house keeping routine has been strictly enforced for the protection of personnel and to limit the spread of contamination. The entry and exit to the laboratory is strictly through the change room which is separated from the main laboratory by means of a barrier. All the operational staff in the laboratory are provided with over-coats, gloves, over-shoes and respirators. Vacuum cleaning and wet mopping are employed to reduce the dust level in the laboratory. Laboratory coats and over shoes are changed once in three days and laundered in the washing machine set up for the purpose,

A strict medical supervision has been kept on all working personnel. Each worker is examined for body contamination by urine analysis. An initial medical examination and six-monthly chest X-rays screenings, vital capacity tests and weight checks have been conducted on all personnel working in the laboratory.

6. CONCLUSION

The production of Cu-8% Be master alloys starting from Indian beryl has been successfully demonstrated on a laboratory scale adopting a flow-sheet that incorporates (i) opening up of beryl by the silico-fluoride route, and (ii) preparation of master alloys by the magnesiothermic reduction of BeF_2 in presence of copper. The overall beryllium recovery has been observed to be around 80%.

Cu-2% Be alloy shapes required in the electronic industry have been successfully prepared by dilution of the master alloy followed by rolling. Such shapes have been tested by the actual users and found acceptable.

7. ACKNOWLEDGEMENTS

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Table 1

INFLUENCE OF SINTERING TIME AND TEMPERATURE ON BERYLLIUM RECOVERY

Charge composition : Beryl (11.21% BeO) : Na_2SiF_6 : Na_2CO_3
: : 11.15 : 8.1 : 0.95

Scale of operation : 1.25 gm of contained BeO

Sintering time hrs →	Recovery of BeO %						
	1/2	1	1½	2	3	4	5
Sintering temperature °C ↓							
700	55.5	62.7	88.0	91.4	93.4	96.0	94.4
750	65.8	85.6	86.7	93.8	93.1	92.0	78.7
800	69.8	86.6	85.6				

Table 2

INFLUENCE OF EXCESS BeF_2 ON THE RESIDUAL Mg-CONTENT IN THE ALLOY

Conditions:

Scale of operation - 200 gm of copper

Reduction temperature - 1100°C

Duration - $\frac{1}{2}$ hour

Final temperature - 1400°C

Maximum possible Be-loading based on Mg charged %	Excess BeF_2 charged %	Alloy composition	
		Be %	Mg %
9.00	25	7.93	Traces
10.86	15	9.45	1.49
10.86	10	9.42	1.52
10.86	6	9.22	2.02

Table 3

INFLUENCE OF Be-LOADING ON Be-RECOVERY

Conditions:

Scale of operation - 200 gm of copper

BeF₂ - 25% excess over stoichiometric

Reduction temperature - 1100°C

Duration - ½ hour

Final temperature - 1400°C

Maximum possible Be-loading based on Mg-charged %	Alloy Recovery %	Alloy composition		Be-recovery based on Mg-charged %
		Be %	Mg %	
9	97.5	7.93	Traces	85.8
15	88.1	13.93	0.61	81.9
20	87.5	17.90	0.60	78.2
25	85.4	22.79	0.51	77.8

