THE ROLE OF THE NATIONAL METALLURGICAL LABORATORY IN RESEARCH AND DEVELOPMENT OF LIGHT METAL INDUSTRY IN INDIA

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Research in metals and their alloys now presents a wide spectrum ranging from experiments on laboratory bench scale based on speculations arising out of applied or fundamental hypotheses to organized pilot plant scale investigations and heavily financed industrial prototype trials. On one end of this spectrum lies a purely academic appeal answering human insatiable curiosity and on the other end is research in applied fields and between the two prevails a broad-based field in which the role of a young industrial laboratory, such as the National Metallurgical Laboratory, covers diverse research and development fields in ferrous and non-ferrous metals including light metals and their family of alloys; such development work in the latter fields has, of necessity, been pioneering in scope so far at the National Metallurgical Laboratory located in the Steel Town of Jamshedpur and yet considerable ground has been covered. Whilst the metallurgy of heavier metals rapidly developed with the recovery of those relatively easier to smelt, the lighter metals such as aluminium and magnesium could be produced only in the 19th century. The Second World War in its train gave a tremendous stimulus to the development of light metals and their alloys both in production targets and range of service requirements. In the case of India, if the hard core of the Second Five Year Plan has centred round the development of iron and steel industry, the industrial growth of light metals and their alloys may well claim this place of pride during the Third Five Year Plan whose outlay has now been more or less fully formulated.

The scope of research and development work on light metals and their alloys has necessarily to be linked with our indigenous resources in respect of these metals. To an Indian student of Metallurgy, the extent of such resources and limitations imposed in respect of their metallurgical characteristics, would be well known. The general position in outline in respect of indigenous resources may be reviewed as follows and as shown in the accompanying map (Fig. 1).

Bauxite

Deposits of commercial importance of bauxite are located near Katni, Bilaspur, Mandla, Sarguja and Jashpur in Madhya Pradesh and in Belgaum (Mysore), Thana, Koria and Kolhapur in Maharashtra and Gujrat States. Bauxite deposits in Ranchi and Singhbhum districts of Bihar are fairly large. The States of Kashmir, Madras (Salem) and Orissa also possess good deposits of bauxite. A rich belt of bauxite also occurs in the Vindhya mountains from where the Hindustan Aluminium Corporation Limited will receive the supply of bauxite for their 20,000 tons new smelter plant now being installed at Pipri in U.P. The production of bauxite in 1959 was 126,000 metric tons as against 139,000 metric tons in 1958. During 1959, 67,000 metric tons of bauxite were used in producing alumina...
Fig. I.

LIGHT METAL INDUSTRIES & RESOURCES - LOCATION IN INDIA

BAUXITE ••• BERYL •••••
LEPIDOLITE •••••
MAGNESITE •••
ILMENITE •••••

ALUMINUM PLANTS (EXISTING) ••••
ALUMINUM PLANTS (PROPOSED) •••••

Fig. I.
as against 55,000 metric tons in 1958. The export during 1959 was 24,000 metric tons, an increase of nearly 3,000 metric tons over the previous year. The reserves of all grades of bauxite in India have been estimated at 250 million tons. High grade bauxite deposits are estimated at 25 million tons (50% \( \text{Al}_2\text{O}_3 \) and over). Indian bauxites present certain chemical peculiarities. In North and South America, nearly all bauxites are trihydrates, i.e., with three molecules of water to one molecule of alumina. In Europe, bauxites are generally of monohydrate type, generally high in iron oxide. In Africa and Indonesia, bauxites are of trihydrate type, similar to that in America. Although Indian bauxite is mainly of trihydrate type, varying amounts of monohydrates are often found mixed with trihydrate. This lack of uniformity applies to most of the constituents of the Indian bauxite, such as, water and iron oxide (which varies from 2 to 30 per cent), titanium (which varies from 1.5 to 12 per cent), silica, etc., which also show a wide variation. In relation to American bauxites, Indian bauxites require finer grinding, better and more concentrated caustic soda solution to dissolve it, and three times more filtering capacity per unit of alumina. As the aluminium content of Indian bauxite is also less, bauxite needed per unit of aluminium is 25 per cent higher in India than in Canada or U.S.A.

**Magnesite**

In India, magnesite deposits are known to occur in the Salem district of Madras and Hassan district of Mysore. Some deposits are also reported in the Kurnool district of Andhra Pradesh, Tiruchirapalli district of Madras, Dungarpur in Rajasthan and Singbhum district of Bihar. Recently deposits of magnesite have been located at Almorah in Garhwal area in U.P. The production of magnesite was 157,957 metric tons in 1959. As much as 98 per cent was reported from Salem district of Madras State. Estimated reserves of magnesite in Salem district of Madras are 82.5 million tons at a depth of 100 ft and 330 million tons at 500 ft. The reserves of high grade magnesite in Almora district are estimated at about 4 million tons.

The present world production of magnesite is of the order of 2 million tons per annum, of which 30 per cent comes from U.S.A.

**Dolomite**

Dolomite deposits are known to occur in Orissa and Madhya Pradesh. In Orissa, dolomite deposits of commercial interest are found in Panposh and Birmatapur in Sundergarh district. Good deposits also occur around Gangpur, Amghat, Khakurthahal, Lanjiberna and Beldih. Dolomitic limestone deposits are found at Putada near Chaibasa in Singhbhum district. In Madhya Pradesh, dolomite deposits occur around Bhalaghat, Gwar. Rupaud in Jabalpur district and near Akaltara and Jairamnagar in Bilaspur. Other deposits of dolomite and dolomitic limestone are found in Rajasthan, Andhra Pradesh, Mysore, Bombay, Kashmir, U.P. and Punjab. The output of dolomite was 325,000 metric tons in 1959. Birmatapur and Panposh in Sundergarh district of Orissa accounted for 97 per cent of the total output. Reserves of rich dolomite deposits of Birmatapur are estimated at 252 million tons at a depth of 150 ft and 84 million tons thereof are of high grade stone. Apart from this, 5 million tons at Sambalpur and 25 million tons in Durg district have been estimated.

**Titanium and rare-earth group of metals**

In India, large quantities of ilmenite occur in the beach sands of Kerala State. The production of ilmenite during 1959 was 303,000 metric tons, 95% of the output is recorded from Kerala State. Indian reserves of ilmenite in the form of beach sand alone total between 300 and 350 million tons.

Typical analyses figures of Indian ilmenites are given below:

<table>
<thead>
<tr>
<th></th>
<th>Quilon, Travancore</th>
<th>Manavalakurichi, Travancore</th>
</tr>
</thead>
<tbody>
<tr>
<td>( \text{SiO}_2 )</td>
<td>...</td>
<td>10.40</td>
</tr>
<tr>
<td>( \text{Al}_2\text{O}_3 )</td>
<td>...</td>
<td>1.01</td>
</tr>
<tr>
<td>( \text{Fe}_2\text{O}_3 )</td>
<td>24.80</td>
<td>14.17</td>
</tr>
<tr>
<td>( \text{FeO} )</td>
<td>9.70</td>
<td>26.71</td>
</tr>
<tr>
<td>( \text{MnO} )</td>
<td>n.d.</td>
<td>0.38</td>
</tr>
<tr>
<td>( \text{MgO} )</td>
<td>n.d.</td>
<td>n.d.</td>
</tr>
<tr>
<td>( \text{CaO} )</td>
<td>n.d.</td>
<td>1.01</td>
</tr>
<tr>
<td>( \text{TiO}_2 )</td>
<td>60.30</td>
<td>53.56</td>
</tr>
<tr>
<td>( \text{Cr}_2\text{O}_3 )</td>
<td>0.14</td>
<td>n.d.</td>
</tr>
<tr>
<td>( \text{V}_2\text{O}_5 )</td>
<td>0.26</td>
<td>0.03</td>
</tr>
<tr>
<td>( \text{P}_2\text{O}_5 )</td>
<td>0.17</td>
<td>0.20</td>
</tr>
</tbody>
</table>

Rutile, the other important titanium mineral, is derived from Travancore beach sands and also found in Punjab. Kadavur in Trichinopoly district, Madras, Singhbhum and Dhalbhum districts in Bihar. Titaniferous magnete is found in Singbhum and Mayurbhanj districts containing up to 28% \( \text{TiO}_2 \). The aluminium and ferruginous laterite of India contain high amounts of titanium. If aluminium is extracted from the aluminium laterite, the sludge obtained during refining should provide a potential source of titanium. The most important deposits of titanium minerals worked in India lie in Travancore, on the South-west coast, in five stretches along the coast, viz., Nindakara (north of Quilon), Anjengo-Varkala (south of Quilon), Kovilam (south of Trivandrum), Muttam-Pudur (near Colachel) and Cape Comorin-Lipurum (on the eastern coast of Tinnevelly district). These beach sands of Travancore are a mixture of mineral sands with a specific gravity varying from 2.3 to 4.9 of monazite and contain quartz, monazite, rutile, zircon, garnet and ilmenite. These sands are derived through weathering of igneous rocks and in some regions have yielded laterites, while in the other, sandstones, as at
Varkala. The mineralogical composition of the beach sands is given below:

<table>
<thead>
<tr>
<th></th>
<th>Manavalakurichi</th>
<th>Nindakara</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zirconite</td>
<td>75-80</td>
<td>80</td>
</tr>
<tr>
<td>Zircon</td>
<td>4-6</td>
<td>4-6</td>
</tr>
<tr>
<td>Silimanite</td>
<td>2-4</td>
<td>3-5</td>
</tr>
<tr>
<td>Rutile</td>
<td>3-5</td>
<td>4-5</td>
</tr>
<tr>
<td>Garnet</td>
<td>3-5</td>
<td>less than 0.5</td>
</tr>
<tr>
<td>Silica</td>
<td>5-7</td>
<td>4-5</td>
</tr>
<tr>
<td>Monazite</td>
<td>about 1</td>
<td>0.5 to 1</td>
</tr>
<tr>
<td>Other minerals</td>
<td>less than 0.1</td>
<td></td>
</tr>
</tbody>
</table>

The analyses of pure monazite (India, Travancore) are given below:

<table>
<thead>
<tr>
<th></th>
<th>India, Travancore</th>
</tr>
</thead>
<tbody>
<tr>
<td>Thoria ThO₂</td>
<td>8.1</td>
</tr>
<tr>
<td>Ceria Ce₂O₃</td>
<td>30.6</td>
</tr>
<tr>
<td>Lanthanum oxide, La₂O₃</td>
<td>15.7</td>
</tr>
<tr>
<td>Praseodymium oxide, Pr₂O₃</td>
<td>2.9</td>
</tr>
<tr>
<td>Neodymium oxide, Nd₂O₃</td>
<td>10.5</td>
</tr>
<tr>
<td>Europium, Gadolinium and terbium oxides</td>
<td>0.7</td>
</tr>
<tr>
<td>Yttrium oxide, Y₂O₃</td>
<td>0.4</td>
</tr>
<tr>
<td>Dysprosium, holmium, erbium, ytterbium and lutecium oxide</td>
<td>0.1</td>
</tr>
<tr>
<td>Lime, CaO</td>
<td>1.0</td>
</tr>
<tr>
<td>Alumina Al₂O₃</td>
<td></td>
</tr>
<tr>
<td>Iron oxide, Fe₂O₃</td>
<td></td>
</tr>
<tr>
<td>Uranium oxide, U₃O₈</td>
<td>0.3</td>
</tr>
<tr>
<td>Phosphoric acid, P₃O₅</td>
<td>26.2</td>
</tr>
<tr>
<td>Silica, SiO₂</td>
<td>2.4</td>
</tr>
<tr>
<td>Loss on ignition, H₂O</td>
<td>-</td>
</tr>
</tbody>
</table>

**Beryl**

In India, beryl is found in Ajmer-Merwara (Rajasthan). It is also found in the mica mines of Hazaribagh and Nellore districts. The mica deposits of Udaipur also yield certain quantity of beryl.

India was once the world's only source of beryl supply. Production in India was 1.15 thousand metric tons in 1957.

**Zircon**

In India, zirconium ore is obtained as a by-product in ilmenite concentration process. Separate statistics of production are not available. India is one of the largest producers of zirconium in the world.

Naturally therefore, in consideration of our indigenous resources of light metals, investigations at the National Metallurgical Laboratory have related to and are projected to cover the treatment of Indian ores of light metals through suitable pyro- and electro-metallurgical techniques. Extensive work for the treatment and handling of bauxite ore for the Rihand Project is being planned. Thus the general theme in logical sequence would relate to extraction processes based on pyro- and electro-metallurgical technique of these metals, to be followed by methods of production of their optimum alloys, in some cases incorporating micro-additions of rare-earth group of metals in which Indian resources are abundant and ending with studies of fabrication characteristics and properties of such alloys. Whilst epoch-making discoveries are not claimed in these fields at the National Metallurgical Laboratory, progress in diverse fields has been very steady and rewarding, whilst much more remains to be accomplished in many fields. One of our main aims would be to further strengthen close connections between research and development activities of National Metallurgical Laboratory and Indian light metal industries now coming into their own during the Third Five Year Plan. In this connection, it may not be out of place to mention that whilst firm collaborative associations have been established between research, development and pilot-plant activities of the National Metallurgical Laboratory and the Indian ferrous metallurgical industries, the same is now taking shape in so far as light metal industry in India is concerned. Under the impact of our Five Year Plans these collaborative contacts will be further consolidated and expanded to mutual technical advantage. At the same time, there is little doubt that in the midst of a mass of data on different problems that confront us needing allotment of priorities, treatment on the basis of “omnium gatherum” will result in an unmanageable collection of items. As such, efforts at the Laboratory have followed the old adage choosing limited objectives in the present context of our industrial development. At the same time, it will be recognised that some of our mineral wealth is relatively of poor grade in respect of impurity contents and as such processes standard elsewhere cannot be adopted ad hoc under Indian conditions. At the same time, these objectives open new horizons before us in exploiting Indian resources of light metals to the maximum national advantage. It is on such basis that projected research and development work has accordingly been aligned with a view to developing potential uses of rare-earth group of metals in different metallurgical fields, utilisation of titanium values contained in Indian ilmenite and titaniferous magnetites, etc. It has to be conceded that the scope is as vast as it is complex both from applied as also fundamental angles—in the latter field. Concentrated attention will be devoted to dispersion-strengthened as also precipitation-hardened light alloys and study of age-hardening mechanism in these alloys. It will also be the aim to avoid duplication of work on light metals and alloys of nuclear and atomic interest and applications which will be left to the purview of research organisations of the Indian Atomic Energy Commission. Work on purification and production of semi-conductor elements such as germanium, selenium, silicon, etc. may be left for immediate studies to our sister laboratories of the Council of Scientific and Industrial Research such as the National Physical Laboratory and the National Chemical Laboratory. Studies of pyro- and electro-metallurgical extraction techniques on indigenous ores for light metals and alloys will loom largely in the immediate programme of our development work. Some of the work in these directions undertaken at the National Metallurgical Laboratory may be now outlined.
Titanium

The addition of titanium to the family of light metals has drawn much attention by virtue of its attractive properties such as excellent corrosion resistance, high strength weight ratio and retention of strength at moderately elevated temperatures. As a first step connected with the recovery of titanium, investigations were taken up for the chlorination of ilmenite. A process for the preferential chlorination of iron from ilmenite has been worked out. The process consists in the removal of almost the entire amount of iron by selective chlorination with HCl gas at 900-1,000°C leaving a titania-rich residue that can be chlorinated with chlorine and a reducing agent to produce titanium tetra-chloride. It is expected that the process may offer scope for reduction in the cost of titanium tetra-chloride thus produced.

A systematic study on the production of titanium sponge by reduction of titanium tetra-chloride with magnesium was taken up and a unit producing one lb of metal was set up. The effect of rate of addition of titanium tetra-chloride to the magnesium and influence of using more magnesium than theoretical needs were studied. In order to stop even slight hydrolysis of titanium tetra-chloride from the distillation assembly to the reduction set-up, the distillation assembly was made a part of the main reduction unit. The apparatus worked satisfactorily. Titanium was recovered from the reaction mass by acid leaching. It is now proposed to extend this work using sodium as a reducing agent and to set up a larger assembly capable of producing 10-15 lb of titanium per batch. It is expected that the requirements of our research development programme may be thus met from this unit.

Besides the chlorination of ilmenite and the production of titanium sponge, systematic studies have also been undertaken on organo-metallic salts of titanium and titanium tetra-iodide. It has been observed that the titanium oxalate is unstable and from a solution of titanico acid and oxalic acid, a precipitate of titanyl oxalate is thrown down on addition of absolute alcohol. Various factors like temperature of solution, volume solution, acid, etc. have been studied. It has been observed that on heating titanyl oxalate in an inert atmosphere, sub-oxides of titanium are formed.

Titanium iodide is of interest for the production of pure titanium metal by the decomposition of the tetra-iodide. It has been observed that the titanium oxalate is unstable and from a solution of titanio acid and oxalic acid, a precipitate of titanyl oxalate is thrown down on addition of absolute alcohol. Various factors like temperature of solution, volume solution, acid, etc. have been studied. It has been observed that on heating titanyl oxalate in an inert atmosphere, sub-oxides of titanium are formed.

Titanium iodide is of interest for the production of pure titanium metal by the decomposition of the tetra-iodide. In fact the dissociation of titanium iodide has been used for purifying the crude titanium sponge. A process for the preparation of titanium tetra-iodide using titanium dioxide as a raw material has been developed which consists in heating aluminium powder, iodine and titanium dioxide in a sealed, evacuated glass bomb. The process gives a mixture of titanium tetra-iodide and unreacted aluminium iodide and after subsequent purification, the titanium iodide could be used for making pure metal by Van Arkel's process. The process for making titanium iodide as a raw material for producing high purity titanium may be considered an improvement since much cheaper raw material, i.e. titanium dioxide, is used instead of crude sponge.

Alumino-thermic reaction for the production of aluminium-titanium alloys

Reduction of titanium dioxide by aluminium in presence of energisers yielding aluminium-titanium alloys has also been studied. It was observed that the reduction of titanium dioxide with aluminium by using preheated reactants in presence of cryolite produces an aluminium-titanium alloy containing 19% titanium. It was not possible to attain higher titanium concentrations. Use of energisers such as barium peroxide or potassium chlorate were made for the attainment of higher temperature and to increase the titanium content in the alloy. Theoretical calculations were made to get an idea of the amount of energiser necessary to obtain suitable temperature. A study of factors such as variation of aluminium and barium peroxide additions and admixture of various amounts of calcium oxide and particle size of aluminium and rutile was made to ascertain the optimum conditions for the reduction of rutile. Presence of 55% barium peroxide of the charge and admixture of 10% calcium oxide were found to favour the reaction. Results of the experiments with decreasing amounts of aluminium with a view to increase the titanium content of alloy showed that an alloy containing 62% titanium can be prepared when aluminium addition was 70% of the rutile. Further decrease of aluminium in the charge to below 65% of rutile resulted in a slag without any separation of the alloy. Effect of different particle size of aluminium and rutile indicated that optimum results were obtained with -140+170 BSS mesh size.

Removal of aluminium from aluminium-titanium alloys by catalytic distillation

Gross's process for the production and refining of aluminium by sub-halide formation may show the possibility in as much as an alumino-thermic alloy of titanium and aluminium may be used as a raw material for the production of titanium by removing the aluminium content of the titanium alloy by catalytic distillation: 2 Al (in the alloy) + AlCl₃ → 3AlCl.

Aluminium trichloride reacts with aluminium in the Al-Ti alloy, forming the monochloride which may distil out and re-form as aluminium and trichloride at lower temperatures leaving behind the unreacted titanium. Attempts are also being made to study the disproportionate kinetics of lower halides of titanium to study the feasibility of producing titanium from scrap and alloys. An assembly has been set up to study the reaction between titanium tetra-chloride vapours at various partial pressures and titanium metal kept at various temperatures using argon as a carrier gas for titanium tetra-chloride.

Work on the production of aluminium powder of high purity for different needs has progressed
successfully and detailed plans are being worked out to set up a larger unit. Investigations have also been taken up to produce aluminium powder suitable for aluminothermic reactions by granulating and blowing. It has been observed that the powder produced by blowing the molten metal flowing through gravity yields the powder suitable for aluminothermic reactions. A systematic study on the effect of rate of flow of metal, temperature of molten metal, the pressure of air, etc. is under way.

Magnesium

In this era of "missiles", weight saving in alloys poses a problem of utmost importance. Magnesium and its alloy have largely contributed towards fabrication of light structural components for achievement of speed and fuel economy. Existing processes for the extraction of magnesium can be broadly classified into two groups, namely, electrolytic and direct reduction processes. The electrolytic processes are fundamentally similar except in the nature of the cell feed and the cell characteristics. The fused salt technique employs anhydrous magnesium chloride as its cell feed.

Direct reduction processes generally employ reductants such as carbon, calcium carbide, ferro-silicon, aluminium-silicon, aluminium, etc. Among the direct reduction processes, two basic processes, namely, Hansgrig's carbo-thermic process and Pidgeon's silico-thermic processes were placed on a commercial scale during World War II. In the carbo-thermic process, the reaction between a mixture of magnesium oxide and carbon is carried out at 1,700°C in an arc furnace when carbon monoxide and magnesium volatilise into a condensation chamber. The volatile products must be shock-cooled to avoid a reversal reaction which would result in the formation of carbon and magnesium oxide as follows:

\[ \text{MgO} + \text{C} \rightarrow \text{CO} + \text{Mg} \]

The magnesium so obtained is in a powder form and is carefully remelted for further use. Silico-thermic process consists in the reduction of calcined dolomite with ferro-silicon under a vacuum with the following reaction taking place:

\[ 2 \left( \text{CaO} \cdot \text{MgO} \right) + \text{Si} = (\text{CaO})_2 \cdot \text{SiO}_2 + 2 \text{Mg} \]

Increasing interest is now being evinced in the development of suitable direct reduction processes in view of the operational advantages of these processes. A few direct reduction processes run on a continuous basis are under development in Germany and in U.S.A. A recent development in the field of silico-thermal reduction of dolomite which shows promise in extraction metallurgy of magnesium is the "Magnetherm process" patented by Le Magnesium Thermique Magnetherm, France. The process consists in the electro-thermal reduction of calcined dolomite with ferro-silicon under a vacuum in the middle of a liquid pool constituting lime, silica and alumina. In the light of the modern developments towards direct reduction processes and the inherent advantages embodied therein, studies were undertaken at the National Metallurgical Laboratory for the development of silico-thermal process utilising indigenous resources. Advantages offered by Pidgeon's process are that the raw materials do not require elaborate processing and the magnesium metal obtained is of high purity. In respect of raw materials also, India has vast resources of dolomite spread over its various States and the best known occurrences are in Orissa with reserves of over 252 millions tons. Laboratory scale studies were undertaken at the National Metallurgical Laboratory and the factors so far studied include temperature of reduction, effect of particle size of reactants, effect of catalysts on the reaction kinetics, briquetting pressure, grade of ferro-silicon, condensation characteristics of magnesium vapours, etc. On the basis of these studies, a small unit was designed and fabricated at the National Metallurgical Laboratory. A 6" dia. heat resisting alloy retort was employed embodying a condensation chamber with arrangements for deflecting alkalies separately into a different zone to eliminate fire hazards. Magnesium metal obtained in a massive bright crystalline form analysed as follows:

Cd  ...  traces
Mn  ...  0·025%
Cu  ...  0·002%
Al  ...  0·003%
Zn  ...  0·003%
Mg  ...  Remainder

A pilot plant capable of producing 25 lb of magnesium metal per day is now being established. A flow-sheet of the process is given below:

Dolomite
\( \text{Calcination} \rightarrow \text{Grinder} \)
\( (\text{CaO} \cdot \text{MgO}) \)
\( \text{Ferro-silicon} \)
\( \text{Catalyst} \)
\( \text{Mixer} \)
\( \text{Briquetting} \)
\( \text{Vacuum furnace} \)
\( \text{Residue} \)
\( \text{Magnesium crystals} \)

The pilot plant will cover an area of 2,400 sq ft constituting the following principal equipment:
1. Kiln for dolomite calcination,
2. Grinders for size reduction of calcined dolomite, ferro-silicon and catalyst,
3. Briquetting machine,
4. Vacuum furnaces with accessories.

Besides magnesium production, it is also proposed to incorporate a magnesium powder making unit in this pilot plant. Magnesium metal has been successfully powdered and different grades having bulk densities to rigid specifications have been prepared. It has
been observed that during the process of powder-making the oxidation of the surface to an extent could not be ordinarily avoided. Therefore, powdering under an inert atmosphere to eliminate surface oxidation has to be undertaken. Development of magnesium production in the country should go a long way in meeting the ever-increasing demands for this light metal for multitudinous types of light alloys.

Work has also been taken up for the production of magnesium from sea-water magnesium chloride. Messrs Tata Chemicals Limited have been supplying us the crude magnesium chloride for this purpose. The investigation has been divided into two parts:

1. Dehydration of the hydrated magnesium chloride.
2. Electrolysis of dehydrated magnesium chloride in the electrolytic cell designed in the laboratory.

Dehydration is carried out in shelf drier fitted with stirring rakes fabricated in the laboratory. Lumps of hydrated magnesium chloride containing about 6-7 molecules of water are broken up into small pieces and passed through a range of slowly rising temperature in a current of hot air. The dried product contains about 1-5 mols of water per molecule of magnesium chloride.

Several experimental cells have been tried. The cell body is used as cathode with ceramic hood to prevent action of chlorine on the metal produced. Initial difficulties have been to a large extent overcome.

Work is now directed to modification of cell design to make continuous running of the cell possible for electrolytic magnesium production.

Aluminium-magnesium alloys

Alloys of aluminium with magnesium are characterised by their high resistance to corrosion, superior high temperature properties and good machineability. These are extensively used for structural members in aircraft and marine vessels, brake shoes, etc. But Al-Mg alloys with magnesium content higher than about 6% are not normally hot-workable, with the result that the benefit of these excellent properties of the higher magnesium content alloys is not available in the wrought form. Several authors have observed that incorporation of cerium or misch metal in aluminium and magnesium and their alloys results in grain refinement and improvement in their elevated temperature properties. No tangible work has, however, it is understood, been reported on the alloys of the Al-Mg system. In view of expected and current requirements of these alloys and utilisation of indigenous aluminium and magnesium, it was felt that high magnesium binary Al-Mg alloys could be made readily hot-workable through making use of well-established effects of addition of rare-earth group of metals to these difficult alloys. A systematic study was, therefore, initiated on the effects of the addition of cerium misch-metal to binary Al-Mg alloys containing magnesium contents of 7-10%.

Cerium misch-metal was added to these alloys up to 5% and its effects on changes in their microstructure, tensile strength and workability have been studied under controlled conditions. It has been seen that microstructure of both as cast and wrought specimens undergoes considerable changes with additions of misch-metal. The initial Al-Mg phase, present in the untreated alloys, is replaced by a new cerium containing phase which could be due to the formation of either Al₃Ce or Mg₃Ce (or both). With higher misch-metal contents the cerium phase predominates.

The tensile strength of these alloys was reduced at first till the misch-metal content rises to 2-3% after which it again increased (as per Table I given below). The hot-workability of all the Al-Mg alloys (7-9% Mg) was distinctly improved by additions of misch-metal, while in the case of Al-10% Mg alloy, considerable improvement was observed in physical properties only when 2-3% misch-metal was added. The tensile strength of wrought alloys was also distinctly improved by increase in misch-metal contents as also hardness values apart from appreciable improvement in hot-workability.

<table>
<thead>
<tr>
<th>Serial No.</th>
<th>% Misch-metal added</th>
<th>As cast Mg-7%</th>
<th>Mg-8%</th>
<th>Mg-9%</th>
<th>Mg-10%</th>
<th>Wrought Mg-7%</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0</td>
<td>9.5</td>
<td>10.0</td>
<td>10.5</td>
<td>10.6</td>
<td>23.5</td>
</tr>
<tr>
<td>2</td>
<td>1</td>
<td>7.7</td>
<td>9.1</td>
<td>8.9</td>
<td>7.7</td>
<td>25.4</td>
</tr>
<tr>
<td>3</td>
<td>2</td>
<td>6.2</td>
<td>8.5</td>
<td>7.9</td>
<td>6.6</td>
<td>26.7</td>
</tr>
<tr>
<td>4</td>
<td>3</td>
<td>5.2</td>
<td>8.4</td>
<td>7.9</td>
<td>6.9</td>
<td>28.0</td>
</tr>
<tr>
<td>5</td>
<td>4</td>
<td>4.6</td>
<td>8.7</td>
<td>8.1</td>
<td>8.3</td>
<td>29.0</td>
</tr>
<tr>
<td>6</td>
<td>5</td>
<td>8.05</td>
<td>9.6</td>
<td>8.7</td>
<td>9.8</td>
<td>30.0</td>
</tr>
</tbody>
</table>

It is proposed to complete the work on the present series of alloys by studying their corrosion resistance (salt spray and atmospheric), as also other physical properties, such as, fatigue and high temperature creep resistance, etc. Fundamental studies will also be carried out on the crystal structure of the new phases observed, for their identification, and on the ageing characteristics of these alloys.

Aluminising of ferrous materials

Work under this head, although not falling distinctly under light metals and alloys, is referred to here in outline on the possible opening for indigenous aluminium. Aluminising of ferrous materials is aimed at replacing galvanising which involves import of about 30,000 tons of zinc for protection of ferrous materials against atmospheric corrosion and also to impart a certain measure of oxidation resistance and resistance to sulphurous atmospheres.

Of the various methods of coating steel with aluminium, the hot-dip process is the most versatile in its scope and applications. It consists in degreasing the steel base with either alkaline or solvent degreasers,
pickling in sulphuric or hydrochloric acid, washing, drying and suitably fluxing prior to immersion in a bath of molten aluminium.

Work done at the National Metallurgical Laboratory relates to the use of different types of fluxes prior to aluminising. Pilot plant trials of the patented process are now in progress. Initially work on hot-dip aluminising of wire has been undertaken. Fig. 2 shows a general view of the Pilot Plant established at the National Metallurgical Laboratory.

Pole line hardware for Posts and Telegraphs Department was aluminised and given field tests. Aluminised hardware is superior to galvanised one with respect to its atmospheric corrosion resistance. Several miles of aluminised steel wire has been supplied to the General Manager, Posts and Telegraphs workshops, Calcutta, for field trial on telephone lines. Fig. 3 shows coils of aluminised wire. Tests carried out at British Iron and Steel Research Association's Coating Research Laboratory in U.K. on our aluminised wire samples are highly encouraging. Their report indicates:

(a) effective preparation and preflushing as there is no sign of failure in alloy layer;
(b) wiping leaves a thin coating without decreasing lustre;
(c) coating thickness of 0'6–1'5 mils is obtained;
(d) good wrapping properties are observed.

**Coating weight**

Representative 12" samples were tested for coating weights as per ASTM A 428-58T Stripping test wherein a substantial weight of coating is dissolved in 20% sodium hydroxide solution at 90°C and finally in conc. hydrochloric acid in one to three cycles. Wiped coatings had 220–350 mgms/ft of aluminium on 10 SWG wire. Coatings obtained by use of sizing die had a coating weight of 330–400 mgms/ft.

**Salt spray corrosion test**

Accelerated corrosion test as per ASTM B117-54T shows that an increase in coating weight from 220 mgms/ft to 350 mgms/ft in wiped coatings decreases corrosion rate from 65 mgms/ft/120 hrs to 15 mgms/ft/120 hrs. Corrosion rate of coatings obtained by use of sizing die is only around 10 mgms/ft/20 hours.

**Soundness tests**

Coatings behaved well in Preece test withstanding five dips and in 15% HNO₃ test wherein very little gas was evolved in 5–6 hours.

**Electrical resistance**

Good aluminised steel wire has an electrical resistance around 24 ohms/mile and will be suitable for telephone lines.

Work on joint design and soldering of aluminised wire is in hand to enable utilisation of this product by Posts and Telegraphs Department.
Mechanical properties

Tensile test results on M.S. wire in the uncoated, coated and drawn conditions are given below:

<table>
<thead>
<tr>
<th>Sample</th>
<th>Dia. in.</th>
<th>Max. stress T.S.I.</th>
<th>Elongation%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Uncoated annealed</td>
<td>0.125</td>
<td>30.93</td>
<td>18.75</td>
</tr>
<tr>
<td>M.S. wire</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Hot-dip aluminised wire</td>
<td>0.123</td>
<td>33.82</td>
<td>12.5</td>
</tr>
<tr>
<td>Cold-drawn aluminised</td>
<td>0.116</td>
<td>46.46</td>
<td>6.25</td>
</tr>
</tbody>
</table>

Al-Alloy bearing

Considerable work has so far been reported on solid aluminium bearings from many parts of the world including U.S.S.R. and U.S.A. Typical aluminium bearing alloys developed in General Motors Corporation (by Alfred W. Schulchter) consist of 0.5-5% Si; 0.2-2% Cd; 0.1-0.15% Zn, and the rest Al plus the usual impurities. The alloy is hardened at 482-565°C for 8-15 hrs. Bearings of the alloy are ductile and stable against shocks, need no supporting shell, do not seize, and are stable against oil corrosion.

From a study of the ternary diagram of Zn-Al-Cu an approximate composition of 60:34:6 was selected which was supposed to consist of two constituents, CuAl₂ an intermetallic compound and a solid solution of zinc and aluminium. A study was made on melting in different kinds of furnaces, castability in sand and metal mould, thermal treatment, anti-frictional property, wearing property, thermal conductivity and microstructure. Bonding characteristics with gun-metal and steel were also studied to find out its suitability for backed bearing.

Electric pot furnace was found very suitable for melting this alloy. The charge was covered with graphite powder to protect the surface from the atmospheric oxidation. After the alloy had melted the molten bath was scavenged with dry nitrogen gas.

The alloy was poured in different types of cast iron moulds. It produced very clean and flawless ingot surface. In sand castings also, it produced equally uniform surface, free of blow-holes. The alloy had good casting properties and could be cast in the same absolute as that of permanent mould casting.

The micro-structure of the chill-cast alloy consisted of two phases. The effects of annealing on the distribution of phases were studied and it was found that specimen annealed at 400°C showed structure similar to the usual tin-base babbitt metal. The micro-hardness of the hard phase embedded in the grey matrix of a solid solution of zinc and aluminium showed the hardness of 380 V.P.N., and the matrix having a hardness of 67 V.P.N. The overall hardness of the alloy was found to be 95 B.H.N. The alloy showed comparatively low wear and coefficient of friction under lubricated conditions.

Continuous casting of light metals and alloys

Work on continuous casting of light alloys has recently been taken up at this Laboratory. A small experimental unit was designed and fabricated in the Laboratory for casting 1 ¹/₂" products. Mechanical drawing arrangement was provided. A double-walled water cooled copper die (1 ¹/₂" I.D. by 2" long) was designed and fabricated with the exit water falling directly on the cast rod in the form of a spray. In the beginning difficulties were experienced due to water rising into the annular space around the semi-solid rod, with water circulating at a rate of 3,500 cc/min and a rod drawing rate of nearly 18"/min. When Al was poured in the graphite pot preheated to 350°C, the cast rod could be drawn continuously. Because of the inherent difficulties in the drawing arrangement, it is being redesigned for further experiments on the continuous casting of the light metal and alloys with a view to study their metallurgical characteristics, effects of rate of cooling, rate of drawing, etc.

Aluminium-silicon alloys

Alloys of aluminium-silicon have today attained considerable commercial importance in their wide use for multifarious applications and as much as 80% of the world’s production of light alloy castings has been estimated to belong to the family of these alloys. Comprehensive investigation was undertaken at the National Metallurgical Laboratory on the preparation of aluminium-silicon alloys by the alunino-thermic reduction of quartz. Based on thermo-dynamical and experimental data obtained, the reduction of quartz has been found to depend upon a number of factors, the chief of which are particle size of quartz, amount of cryolite in the charge, temperature of the reactants and holding time. Physical properties of the alloys experimentally made by this method have been determined and compared with the physical properties of corresponding alloys produced experimentally here by direct alloying of the two metals. This comparison yielded identical results in the two cases. Reference to the general economics of this process has also been made. It has finally been stressed that this method can be usefully exploited for commercial applications in countries dependent on silicon imports and rich in bauxite and quartz, such as India. The process is simple and can be easily worked without any complicated prior treatment of raw materials. The temperature necessary for alunino-thermic reduction is easily attainable and therefore does not need any costly equipment. The reduction and alloying of silicon can be completed at one stage. It is, therefore, recommended that this process may advantageously be adopted under Indian conditions wherein foreign exchange has to be spent on import of metal silicon. Aluminium-silicon alloys are used for pistons in the internal combustion engine and more recently for the cylinder block also. The high wear resistance, good casting properties, and high corrosion resistance make this alloy suitable for these purposes. Aluminium has
very little solubility for silicon and so the micro-
structure of these alloys consists of a matrix of
aluminium in which silicon is dispersed. Ordinarily
silicon exists as coarse particles and this adversely
affects the resulting properties. To improve the distri-
bution and size of silicon particles the alloy is modified
with sodium or phosphorus. Work is being done in
this Laboratory on modification of aluminium-silicon
alloys by other elements and a process has been found
out which greatly improves the size and distribution
of the silicon particles. The fluidity of the alloy was
also greatly improved at the same time. This is shown
in Figs. 4 and 5. Fig. 4 is the fluidity pattern obtained
with an alloy modified with our nucleant and Fig. 5
that obtained by the addition of phosphorus.

In this connection work on the production of
aluminium-silicon alloys by direct smelting of clays,
etc. will also be taken up along lines indicated by work
done chiefly in the U.S.A. by O. C. Fursman and
L. H. Banning at the U.S. Bureau of Mines; a wide
range of crude Al-Si alloys (Al 15-55%) were produced
by the smelting of clays or similar Al-silicate raw materials
in a pit-type 3-phase C-lined arc furnace. Hogged
fuel or wood chips were used as the major source of
C in the charge. Suggested potential uses of the crude
alloys are as hardeners for producing high Si-Al alloys,
as reducing agents in the ferrous industries and as
raw materials for aluminium production.

Beryllium

This brittle metal, difficult to extract economically—
one called the “world's number-one metallurgical
headache” owing to its high melting point coupled with
its high vapour pressure at temperature little above the
melting point and its apparent toxicity—seemed to have
reached its limit of application. Then sixteen years ago
beryllium in the atomic energy field became the new
directive for research into industrial beryllium. The
metal was seen to be of great value in reactors, the
most effective practical element of low atomic weight
which could serve as moderator capable of “slowing
down” high-velocity neutrons. Moreover, beryllium
metal is also superior to other materials as a reflector
of neutrons.

In India, Rajasthan produces very rich beryl ore
which contains as high as 12.5 to 13.5 per cent of
beryllium.

Beryllium is used industrially in the manufacture of
alloys, specially those of copper, nickel, cobalt and iron.
Heat-treated copper-beryllium alloy (2.35% of beryllium)
has a tensile strength about six times that of copper
without any substantial decrease in its electrical conduc-
tivity. This alloy can suitably replace phosphor-bronze
springs in electrical instruments. Beryllium-copper
alloy is also used in the manufacture of wear resisting
parts, viz., bushings, sleeves, rollers, etc. and also in the
manufacture of tools, nickel-beryllium, iron-nickel-beryl-
lum and iron-chromium-nickel-beryllium alloys find
extensive application in industry. Beryllium-copper
alloy, because of its special physical properties, such
as, high degree of fatigue endurance, non-sparking
quality and resistance to heat and corrosion, is one of
the most valuable alloys and finds wide use in defence
and other industries. Likewise binary and ternary
alloys of beryllium with some non-ferrous metals like
magnesium, aluminium, nickel, cobalt and particularly
those with copper are unique in some of their properties and hence industrially very useful. In fact, it is claimed that “beryllium is to copper what carbon is to iron”.

Beryllium oxide is also an important raw material for manufacture of special types of refractory wares. Because of its extraordinary resistance to thermal shocks and high electrical resistance even at high temperatures, it may rightly be called a “super-refractory” which can be used with great advantage for making crucibles, muffle furnace boats, electric furnace linings and vacuum tubes. It is also an important ingredient in “phosphors” for fluorescent lamps. By far the most important is its use as moderator in nuclear reactors and as an alloying agent for copper and nickel to produce precipitation hardening alloys.

In view of the strategic importance of the metal, investigations were undertaken at the National Metallurgical Laboratory to produce beryllia. A suitable chemical method was developed which was chosen for trial on semi-pilot plant basis. The flow-sheet of the process is shown in Fig. 6. On an average, an extraction of 80-85% of beryllium oxide from beryl was obtained along with a purity of product ranging from 95 to 98%.

An electrolytic process was also developed at the National Metallurgical Laboratory, the flow-sheet of which is shown in Fig. 6. The electrolytic method developed consisted in electrolising a solution of sodium beryllium fluoride obtained by leaching with water a sinter of beryl and sodium silico fluoride. The leach liquor containing the complex salts of beryllium formed the catholyte in the outer vessel and a 10% sodium chloride solution contained in a porous pot placed at the centre of the vessel was the anolyte. On electrolysis of the solution, a slurry containing the beryllium hydroxide was obtained in the cathode compartment which on washing, drying and ignition gave the oxide.

The electrolytic process offers advantages over the conventional chemical process in that the product is purer and the process is simpler and much cheaper with a cost estimate of Re. 0.87 per lb instead of Rs. 6.50 per lb by the chemical process.

Based on the results obtained in course of our experiments on semi-pilot plant basis of the fluoride process, as developed by us, a pilot plant scheme for production of 2,400 lb of beryllium oxide per annum was later drawn up. But it was later considered that further work connected with beryllium would be conducted at the Indian Department of Atomic Energy.

Zirconium

The metal zirconium is another metal like beryllium, which has assumed strategic importance since the last War. The field of application of this metal is already very wide, covering right from the nuclear reactor down to the surgeon’s gadgets and is expanding daily in various directions. The use of zirconium metal and its alloys in the construction of nuclear reactors is of outstanding importance today. Its superior mechanical properties and corrosion resistance even at very high temperature coupled with its very low neutron absorption cross-section makes it almost an ideal constructional material for high temperature thermal reactors.

Zirconium in the form of silicon-zirconium and ferro-silicon-zirconium is used as an alloying addition in the steel industry where it acts as a powerful deoxidiser and scavenger. Zirconium is an intensive grain-refiner for magnesium-base alloys. Finer grain size results in increase in strength and ductility of the alloys. Addition of zirconium to beryllium-copper alloys improves the strength at high temperature. Alloys of nickel containing 2-10% zirconium are suitable for cutlery manufacture and those containing still higher proportions of zirconium from 25 to 30%, for high speed cutting tools. High purity zirconium metal is resistant to attack by hydrochloric acid even at elevated temperatures. Hence it is used particularly in U.S.A. for fabricating various parts of the hydrochloric acid plant. It is an ideal material for spinners for spinning rayon fibres.

Zirconium silicate and zirconium dioxide are two useful refractory materials which are used for the lining of electric furnaces and burning chambers, in glass industry, for foundry work and electrical insulators.

In India, zircon occurs in the beach sand of Travancore, mixed with monazite, ilmenite, rutile, etc. It constitutes about 6% of the sand and is recovered as a by-product in the tailings after separation of monazite and ilmenite. In fact, India is one of the important zircon-producing countries. Development work was therefore undertaken at the National Metallurgical Laboratory to explore the possibility of processing this mineral for use in industries and a process was developed for obtaining zirconium dioxide from zircon.

The flow-sheet of the process is shown in Fig. 7. As a first step for the production of zirconium dioxide an electrolytic method was developed consisting in fusing the zircon sand with potassium silico-fluoride. The sintered product was leached and electrolysed. The sediment of the hydroxide was filtered, washed and ignited and a pure zirconium dioxide was obtained. The product could therefore be used for chlorination. High efficiency of extraction of the order of 85-95% has generally been obtained for the process. The purity of the product ranged from 93 to 96%.

Soderberg electrodes

Continuous self-baking electrodes of the Soderberg type are universally used in the electrolytic extraction of aluminium and other light metals. They are also employed in the smelting of pig iron and certain ferro-alloys. In India the main consumers of Soderberg paste are the aluminium producers and manufacturers of ferro-manganese and ferro-silicon. At present the aluminium units are producing their own paste from petroleum coke of Digboi refinery. It was with a view to conserve foreign exchange and explore ways and means of getting good service performance from indigenous pastes, that the National Metallurgical Laboratory undertook a study on the production of suitable Soderberg paste from indigenous materials.
Flow-sheet for the fluoride process for beryllium oxide and alternative process for beryllium oxide.
The work so far done indicates that while calcined petroleum coke is being used for production of this paste, converting it into dense aggregate at a slightly higher cost makes the material more uniform in its density as well as conductivity and the electrodes can consequently be expected to be stronger, more wear resistant and better conductors. Conversion of bituminous coals into dense aggregates will involve addition of low temperature chars. Kinetic studies indicate that it may be possible to conduct low temperature carbonisation so that a pre-determined quantity of residual volatiles remain in the char. Conversion of bituminous coals into dense aggregates is a pre-requisite for their utilisation as raw materials of Soderberg pastes.

Laboratory scale experiments have established that it is possible to obtain hard dense briquettes from petroleum coke or low ash coal which can be used as carbon grist in the Soderberg paste after crushing and grading (Fig. 8).

Results indicate that the paste composition made out of these carbon aggregates are very well comparable with those of imported paste as shown in Tables II and III.

### Table II
Comparison between the green and fired properties of the imported electrode pastes and the author's compositions.

<table>
<thead>
<tr>
<th>Sample</th>
<th>Bending strength of the green paste</th>
<th>Physical characteristics of tamped paste fired to 1,200°C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>kg/cm</td>
<td>Compressive strength</td>
</tr>
<tr>
<td></td>
<td></td>
<td>lb./sq. inch</td>
</tr>
<tr>
<td>French paste</td>
<td>24·20</td>
<td>1,650</td>
</tr>
<tr>
<td>Norwegian paste</td>
<td>22·4</td>
<td>2,000</td>
</tr>
<tr>
<td>Author's compositions</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.</td>
<td>20·0</td>
<td>2,400</td>
</tr>
<tr>
<td>2.</td>
<td>14·0</td>
<td>1,500</td>
</tr>
</tbody>
</table>

### Table III
Electrical resistivities of the author's compositions and French paste calcined at 1,200°C

<table>
<thead>
<tr>
<th>Sample</th>
<th>Binder Composition</th>
<th>Electrical Resistivity ohms/mm²/m</th>
</tr>
</thead>
<tbody>
<tr>
<td>French paste</td>
<td>-</td>
<td>175·0</td>
</tr>
<tr>
<td>Author's compositions</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1.</td>
<td>Electrode pitch and tar</td>
<td>37·0</td>
</tr>
<tr>
<td>2.</td>
<td>Hard pitch and tar</td>
<td>113·0</td>
</tr>
</tbody>
</table>

**General**

Investigations have also been undertaken on short term, long range and ad hoc basis on processing and metallurgical problems for light metal industries. Technical data and advisory information have also been freely furnished to different firms and plants engaged in the production and processing of light metal alloy products, etc. Long-range service problems have also been likewise undertaken for Government bodies and kindred organisations. A typical example may be the production of aluminium-bronze containing 12% aluminium and less than 10% iron and manganese, the rest being copper, for the Railways. A typical industrial problem may be
Fig. 8.
Soderberg electrode paste qualitative line diagram.
illustrated by investigations undertaken into cause, effect and remedial measures for "dark brown spots" on aluminium sheet met with in a production unit. Elucidation of causes responsible for such "dark brown spots" and remedial measures proposed after ad hoc investigation of the problem from fundamental and applied angles have been furnished. Our investigation in this field revealed that the formation of "dark brown spots" appeared to be due to alternate condensation and evaporation of moisture condensed in the capillary spaces between the stacked sheets. It was suggested that ambient temperature of the storage room should not be allowed to fall below the dew point. Similarly, an active corrosion station has been established at the National Metallurgical Laboratory for corrosion studies under "industrial atmospheric" conditions as are met with in Jamshedpur relating to light alloys under development at the National Metallurgical Laboratory as also of the hot-dip aluminised steel wire and strips, etc. Such corrosion tests have extended over periods of several months to over 5 years' exposure under local atmospheric conditions. Investigations were also carried out in connection with the use of aluminium in distillation stills for essential oils which indicate that in the absence of phenol in the oil, aluminium is an excellent metal for fabrication of the distillation stills.

Other problems have related to the surface treatment of light metals and alloys. Development work on surface treatment of light metals and alloys is wide indeed and has covered several aspects of electro-plating of aluminium, chemical polishing of aluminium, etc. Electro-plating of aluminium permits the advantages of its light weight to combine with specific properties of the plated coating. This also includes chromium plating of aluminium-alloy pistons, etc. for imparting adequate wear resistance. In the particular case of aluminium, the adherence of the electro-plated coating to the base metal has been a serious problem, arising out of the strongly electro-positive character of aluminium and the ease with which it forms tenacious and protective oxide film which has to be removed before electro-plating. Nickel plating of aluminium has been developed by using the modified "iron dip" method. The aluminium sheet is degreased in a mild alkaline cleaner containing trisodium phosphate and a washing soda 3% each, then dipped in 5% NaOH solution and etched in 5% hydrofluoric acid solution followed by a short dip in 70% HNO₃ solution and then an iron dip and nickel plating bath followed by stoving at 250°C for 2 hours and polishing, etc. The adherence of the nickel plating on aluminium was excellent. It was further experimentally established that plating of chromium, gold, etc. can be done on the aluminium surface after the latter has been nickel plated as outlined above.

Having given a general resume of the work underway at the National Metallurgical Laboratory on light metals and their alloys, it will perhaps be not out of place to state some of our projected activities in these fields.

One of the most important light metals of interest from the indigenous angle is aluminium, whose world production today stands at 3 million tons per annum and is likely to be doubled by 1965. Aluminium cannot, however, rival steel since the minimum chemical work involved in extracting a ton of aluminium from its oxide is about 7 times that involved for one ton of iron. Likewise, magnesium required during the last World War as an incendiary is now being produced to the extent of 200,000 tons per year. Titanium claimed as a "wonder metal" has today a world production figure of over 10,000 tons per annum and costs less than £1 per pound to produce.

We are rapidly moving in the world of supersonic jet flights, missiles, more and more powerful and faster and complex machines. In this unabated race, resources of metals in general are getting depleted at an enormous rate; for example, proved world reserves of copper will barely last 30 years and if its production continues to multiply fourfold over 2-3 decades, we shall have to look around for metals of the "rare-earth group" of metals which today have relatively much smaller industrial scale uses but will be needed far more abundantly in years to come. It has now been clearly shown that metals of the rare-earth group are valuable alloying elements both in micro and macro-additions in the metallurgy of iron and steel, in highly alloyed and stainless steels, in light alloys of aluminium and magnesium, etc. Researches and development work are actively being pursued all over the world in these metals of the complex rare-earth group of which India has enormous resources. Naturally, therefore, one of the most active themes under pursuit at the National Metallurgical Laboratory is to study the effects of relatively minor additions of rare-earth group of metals to different ferrous and non-ferrous metals including light metal and their family of alloys as also employing the rare-earth group of metals as integral constituent alloying elements in potential series of...
alloys. The extraction of rare-earth minerals, despite their abundant resources, is highly complex and costly. The extraction techniques, however, are being steadily improved upon and the cost of production thereof is expected to drop in course of time. Cerium, of very high purity now being produced, has three times the electrical conductivity of the commercial grade. It has unique properties of readily absorbing impurity contents in other metals when molten. It will not, perhaps, be out of place to outline such applications on rare-earth group of metals in light alloys, representing the fields on which projected research and development work at the National Metallurgical Laboratory is being actively planned and intensifies.

The use of rare-earth metals in light alloys has aroused considerable interest during the last few years. Such metallurgical applications of rare-earth may be classified in two broad headings:

1. Applications in which rare-earths form an alloy with some other metals e.g. with aluminium and magnesium.
2. Applications where rare-earths play a weak role as alloying agents but function fundamentally as purifying elements.

In each case, rare-earths are added in the form of a mixture and not in their pure state. Two types of mixtures are widely used, one of which is Lan-Ceramp (Lanthanum, cerium) whose average analysis is as follows:

<table>
<thead>
<tr>
<th>Metal</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lanthanum</td>
<td>30%</td>
</tr>
<tr>
<td>Cerium</td>
<td>45 to 50%</td>
</tr>
<tr>
<td>Mixture of neodymium</td>
<td>20 to 24%</td>
</tr>
<tr>
<td>Praseodymium and Samarium and Iron</td>
<td>10% max</td>
</tr>
</tbody>
</table>

The other mixture is "misch metal" which is derived from monazite and contains 22 to 25% lanthanum, 50 to 55% cerium, 15 to 17% neodymium, 8 to 10% other rare-earth metals (varying according to origin of monazite), and small amounts of iron. Cerium is said to exercise beneficial influence in aluminium production and to increase the toughness of pure aluminium by reducing the silicon content. It is claimed that an addition of 0.2% cerium will reduce the silicon content of 0.1% to 0.02%. For this purpose, cerium is added to the aluminium in the fused state, preferably in the form of a high cerium containing aluminium alloy, or the form of cerium fluoride to the electrolytic bath in which aluminium is prepared.

Rare-earths are chiefly used in light alloys of aluminium and magnesium. 10 to 12% of the Lan-Ceramp or misch metal added to aluminium will considerably improve its mechanical resistance to heat. However, relative additions of rare-earths required for magnesium are not so large. A few per cent of rare-earths added to magnesium improves its mechanical resistance to heat, refines the grain structure and produces a more easily workable material.

Leontis has evaluated the specific effects of various rare-earth metals at room and elevated temperatures on properties of magnesium. Alloys containing didymium exhibited the highest tensile and compressive strengths at room and elevated temperatures. All rare-earth metals increased the creep resistance of extruded magnesium between 400 to 600°F, but the increase depended on the temperature and concentration of the added metal.

Magnesium rare-earth metal alloys were investigated in Germany many years ago and the findings were published in Becks Technology of Magnesium in 1939. Cast alloys of magnesium with rare-earth have now become commercially available. They all use zirconium as a second essential alloying element because of its grain-refining effect, thus preventing cracking during freezing.

For aircraft applications, engineers and metallurgists have sought alloys with high strength-weight ratio and ability to withstand increasing stress at elevated temperatures. Patton has described how by adding 2 to 4% rare-earths to Mg-Zr alloys, Dow Chemicals have obtained new alloys with outstanding high temperature properties.

At 300°F and above, cast magnesium-zirconium alloys with 2 to 4% additions of rare-earth metals, have shown as much as five times the creep strength compared to older magnesium-aluminium-zirconium type alloys. For aircraft engines which require outstanding elevated temperature properties and high strength-weight ratios, the properties exhibited by these alloys with rare-earth additions are particularly useful. Several alloys have been developed e.g. the addition of 0.3% rare-earths to aluminium-copper-silicon piston alloys, the use of 0.05 to 0.3% rare-earth metal in aluminium alloys containing manganese and the recent use of rare-earth zirconium alloys for jet engine parts. The latter alloys contain 0.1 to 0.9% Zr and up to 4% rare-earths and sometimes 1 to 6% Th and 0.5 to 5% Zr. The didymium metals, particularly neodymium, are especially interesting in this application but their uses may be.

Three-high reversing rolling mill installed at the National Metallurgical Laboratory.
restrict due to limited availability of neodymium. Misch metal contents up to 11% in an aluminium alloy containing silicon, copper, nickel, manganese and small amounts of chromium and titanium have been recommended for high temperature aircraft engine service.

Two magnesium base alloys have recently been announced by Magnesium Elektron\(^1\) to which thorium and rare-earth metals are added: one being a wrought alloy with 0.7 Th, 0.5 Zr, 0.6 Zr-magnesium available in sheet, plate, extrusions and forgings possessing good properties at elevated temperatures and the second being a magnesium base casting 2.5 Ag-2 rare-earth-0.6 Zr-alloy with a room-temperature proof stress comparable with high-strength aluminium alloys and which has very attractive creep properties at elevated temperatures, especially at 200°C.

For uses in cryogenics, the 4 Mg-Mn-Ti-aluminium alloy B54SM developed by Northern Aluminium Company is a serious rival to new nickel steels developed by International Nickel Company for pressure vessels required for holding liquid nitrogen at temperatures as low as -200°C.

Another recent announcement\(^6\) a few months back from France has indicated the development of as-cast and heat-treatable aluminium-magnesium alloy containing 11% magnesium.

In this connection, the application of lithium in metallurgy of light alloys forms an interesting field of application to be potentially developed. Resources of lithium have been located in India such as, for example, in considerable quantities in northern Hazaribagh, in Bastar in Madhya Pradesh and other places. Lithium is an important de-gassing agent and de-oxidiser in metal refining, such as, of copper bronzes, grey iron, low carbon steel, etc. Considerable research has been conducted in the Armour Research Institute on silver-lithium brazing alloys. The Aluminium Company of America has developed new aluminium alloys in which 1% lithium is an important alloying element besides copper up to 4%. Likewise, magnesium-lithium alloys are also being developed containing 10-15% lithium. It is proposed to study the scope of the applications of lithium under Indian conditions for the development of aluminium-magnesium alloys.

There are several other applications\(^6\) of substitute aluminium alloys, such as the development of aluminium bronze for replacing tin-bronzes, used in worm gears of trolley bus reducers. Considerable work in this connection has been done in Russia on the production of aluminium-bronze for the above purpose wherein coarse granularity is overcome by the addition of 3-6% Fe and 2.5% Mn.

The Austrian Casting Institute\(^7\) has announced the development of aluminium-silicon alloys containing 7-9% Si, 5-11% Zn, 0.2-0.5% Mg. This is an unusual composition because alloys with Zn contents are not suitable for casting. However, it is claimed to possess good tensile, bending and hardness characteristics. This alloy known as "Durofondal" allows scrap zinc alloys to be used in its production.

An aluminium-base anti-friction alloy\(^8\) containing Sb 3.5-4.5%, Mg 0.3-0.7%, has been developed in the U.S.S.R. This anti-friction alloy is successfully applied as a thin layer on a steel base and rolled at 350-450°C followed by annealing at 460°C.

Yeshwant P. Telang\(^9\), of the Ford Scientific Laboratory's applied science department, has recently described aluminium—21% silicon alloys with ultimate tensile strengths approaching 50,000 pounds per square inch at a symposium sponsored by the American Society for Metals during the 42nd National Metal Congress. The new alloys are claimed to possess unprecedented mechanical properties particularly for high silicon alloys, in effect higher than those of commercial automotive casting or heavy-duty piston alloys currently in use. In addition to their high strength, the alloys are shown to have excellent hardness, impact and anti-friction properties, low density and lowered thermal expansion coefficients. Alloys of similar composition have been used for pistons in Europe for some time but their poor foundry characteristics, excessive variation in properties and poor machineability have caused them to be generally neglected in U.S.A. To achieve the superior properties demonstrated by the Ford metallurgists, the alloys have been modified to produce a material with the finest possible microstructure. From the foundryman's point of view, the alloys possess the advantage of little or no loss in refinement during remelting, superheating or holding at elevated temperatures. It is proposed to further investigate development of similar alloys under Indian conditions considering that 50% of the light alloy castings in U.S.A. fall in the aluminium-silicon family of alloys which claim 80% of the world's light alloys.

Tremendous interest has been aroused recently by the announcement of a basically new aluminium making process to be used by Aluminium Limited at 4 million dollars to be built at Arvida, Quebec. Designed as a pilot plant unit, it will have a production capacity of 6000-8000 tons of aluminium a year. The process is covered.
by patent right and some details are available. Though power requirements remain almost the same, the new process of Aluminium Limited cuts the cost of production by fifty per cent reduction in capital cost. Impure alumina is partially reduced electrically, converting about 50% of aluminium content to metal. Partly reduced mass then contacts AlCl₃-vapour at over 1000°C and at about 1 atm. Under these conditions, the aluminium in the converter reacts with vapour to form AlCl₃. This monohalide then flows to a condenser leaving behind impurities, such as, titanium—aluminium vapour cools by coming into contact with a violently agitated pool of molten aluminium when temperature drops and the initial reaction reverses, forming AlCl₃ and Al. Product Al can be tapped continuously and the AlCl₃ is recycled to the converter. Further details of this new process are available in several announcements on the subject.

From an appraisal of the developments in the field of light metals and their family of alloys referred to in this paper, it is obvious that tremendous scope for research and development in these fields exists under Indian raw-materials and processing conditions. The National Metallurgical Laboratory will be actively engaged in studying such developments, both in the fields of metallurgy of light metals and in the development of light alloys based on indigenous alloying elements including the rare-earth group of metals. Considerable ground in these fields has been covered and good beginnings made. In concluding, it may be stated that progress in research and development work in diverse fields of the metallurgy of light metals and their family of alloys at the National Metallurgical Laboratory has been steady and rewarding, placing a high premium on scientific ingenuity and rationalised planning and yet far more remains to be accomplished during successive Five Year Plans of our country to which end, this young Laboratory is fully geared today.

References

Mr. U. P. Mullick, Institute of Consulting Engineers, Calcutta: Dr. Nijhawan in his paper has referred to various tests on aluminium coating on steel wires and the good anti-corrosive properties of this process. I would like to know (i) whether the problem has been considered taking into account the rates of expansion of steel and aluminium which are different, (ii) the extent of anti-corrosive properties of aluminium of thick mild-steel rods so that they can be used in structural works like reinforced concrete works, (iii) whether the aluminised steel wire or rods will have sufficient adhesive strength and tensile strength, etc. and (iv) whether abrasive resistance of aluminised steel coatings will be comparable with galvanised coating, say, for wire, rod and tubes of ½" diameter and larger sections. Dr. B. R. Nijhawan (Author): Personally, I would not advocate the use of aluminised steel rods for the purpose of reinforced concrete at all. It is embedded in the concrete and the corrosion resistance of steel rods vis-a-vis reinforced concrete is being separately studied at other laboratories of the Council of Scientific and Industrial Research. I would not consider at all aluminising necessary for reinforced concrete structures. The coating of steel with the hot-dip aluminium should possess certain minimum adhesion strength as there are tests specified to determine the adhesive properties of this bond and all attempts are directed towards reducing the thickness of the intermetallic iron-aluminium compound. If the thickness of the intermetallic layer is within control and within specified limits, the coating will possess the requisite rigidity and adhesion strength. If the thickness of the intermetallic compound exceeds these limits, spalling of the coating may result. But for certain conductivity uses, where we want to substitute copper conductor by this aluminised steel wire, we have suitably increased the thickness of the aluminium coating in comparison with what is normally used for Posts and Telegraphs purposes, where we currently employ galvanised wire. It is quite a complex subject and I would invite Mr. Mullick to come and discuss with us the detailed investigation reports that we have prepared on the subject.