Titanium sponge developments in India

Defence Metallurgical Research Laboratory
Hyderabad, India

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THE INDIAN TITANIUM SCENE

Agencies engaged in the ‘Titanium Ore to Product Cycle’

TITANIUM SPONGE
KMML, 500 tpy

TITANIUM MINERAL WEALTH
ILMENITE: 520 million tons
RUTILE: 30 million tons

TITANIUM DIOXIDE
PIGMENT
KMML: 60000 tpy
TTPL: 25000 tpy
Kolmac: 5000 tpy
Kilburn: 5000 tpy

TITANIUM ALLOY R&D
DMRL, VSSC, BARC, BHU, NCML, IISC, NAL, IIT (M)

INGOT MELTING & MILL PRODUCTS
MIDHANI: 200-250 tpy

TITANIUM FABRICATION
TEAM, TITAN, TITANOR, EXOFAB, ZIRCOTAN, L&T, ALFA-LAVAL, BHPV

ILMENITE
IREL: 600000 tpy
KMML: 200000 tpy †
TOTAL: 800000 tpy

RUTILE
IREL: 19,000 tpy
KMML: 22,000 tpy
TOTAL: 41,000 tpy

SYNTHETIC RUTILE
IREL: 100000 tpy
DCW: 50000 tpy
KMML: 100000 tpy †
CMML: 50000 tpy

Agencies engaged in the ‘Titanium Ore to Product Cycle’
Titanium consumption in India

- Annual consumption expected to be doubled in the coming 5 years
- Higher costs, infrastructural limitations, lack of awareness on long term benefits of Ti in down the line limiting the consumption
DMRL technology (Kroll process) for titanium sponge production – from TiCl₄ purification to preparation of finished sponge lots of 1.75 MT

Technology demonstrated and proven to produce premium grade sponge in industrial scale batches
Technology transfer to KMML

A small commercial facility for titanium sponge set up at KMML, Kerala, India with DMRL technology and funding support from Dep. of Space

KMML sponge plant:
Capacity: 500 MT per year
(expandable to 1000 MT per year)
- Commissioned in June 2012
- TiCl4 source: KMML oxide plant
- Magnesium: imported
- 5 Reduction stations
- 5 Vacuum distillation stations
- Sponge cake ejection
- Sponge crushing, cutting & blending to prepare homogeneous finished lots in a separate housing
- Regular production taking place in 3-3.5 MT batches
Some engineering improvements in the technology at KMML sponge plant

<table>
<thead>
<tr>
<th>S.No.</th>
<th>Technical /Engineering improvement</th>
<th>Overall result/benefit</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Process control through Thyristor based temperature controllers/re-designed air duct work/varying speed air blowers/zone wise energy recorders</td>
<td>Significant energy savings/smooth process operations</td>
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<tr>
<td>2.</td>
<td>Fine tuned operating conditions of TiCl₄ purification, Reactor pressure control, MgCl₂ tapping, vacuum distillation operations</td>
<td>Consistent batch operations, enhanced performance, reduction in overall cycle time, improved physical characteristics of sponge, improved yield</td>
</tr>
<tr>
<td>3.</td>
<td>MgCl₂ ladle for regular collection of hot chloride from a battery of reactors and teeming of salt at a selected working place</td>
<td>Ease of process operations, improved working environment, ease of solid waste disposal, scope for supply of hot salt to the proposed MgCl₂ recycling cell</td>
</tr>
<tr>
<td>4.</td>
<td>New infrastructural facilities for sponge handling (de-humidified space, belt conveyor system etc)</td>
<td>Improved sponge yield and implementation of quality assurance practice</td>
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</tbody>
</table>
Implementation of sponge quality assurance practice

Sampling procedure for evaluation of quality of a finished Sponge Lot

1750 kg Lot
Blending
Five way Chute
Rotary Sampler

20 kg

10 kg
10 kg

5 kg
5 kg

2.5 kg
2.5 kg
2.5 kg
2.5 kg

1.25 kg
1.25 kg

Coring & Quartering
Button Melting for analysis & Hardness Testing

Coring & Quartering
Compact & drilling for Mg & Cb analysis

Primary Sample
40 increments 500 gm/increment

Secondary Sample
Typical analysis of a finished lot sample

<table>
<thead>
<tr>
<th>Element</th>
<th>Content (wt%)</th>
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</thead>
<tbody>
<tr>
<td>Iron (Fe)</td>
<td>0.023</td>
</tr>
<tr>
<td>Oxygen (O)</td>
<td>0.0375</td>
</tr>
<tr>
<td>Silicon (Si)</td>
<td>not detected</td>
</tr>
<tr>
<td>Nickel (Ni)</td>
<td>0.0063</td>
</tr>
<tr>
<td>Carbon (C)</td>
<td>0.0033</td>
</tr>
<tr>
<td>Chloride (Cl)</td>
<td>0.0243</td>
</tr>
<tr>
<td>Nitrogen (N)</td>
<td>&lt; 0.0020</td>
</tr>
<tr>
<td>Magnesium (Mg)</td>
<td>0.0122</td>
</tr>
<tr>
<td>Chromium (Cr)</td>
<td>0.0012</td>
</tr>
<tr>
<td>Hydrogen (H)</td>
<td>0.0019</td>
</tr>
<tr>
<td>Copper (Cu)</td>
<td>0.0071</td>
</tr>
<tr>
<td>Tin (Sn)</td>
<td>not detected</td>
</tr>
<tr>
<td>Manganese (Mn)</td>
<td>0.0009</td>
</tr>
<tr>
<td>Ti (by difference)</td>
<td>99.901</td>
</tr>
<tr>
<td>Hardness (BHN)</td>
<td>78</td>
</tr>
<tr>
<td>Year</td>
<td>Activity</td>
</tr>
<tr>
<td>----------</td>
<td>--------------------------------------------------------------------------</td>
</tr>
<tr>
<td>1982-93</td>
<td>Establishment of Magnesium pilot plant facilities – Development of Monopolar cell technology in 30 kA cells (150 kg/day)</td>
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<tr>
<td>1994-2000</td>
<td>Setting up of multipolar cells and experimental operations in 7 kA with solid feed &amp; 4.8 kA cells with molten salt feed</td>
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<tr>
<td>2003-2006</td>
<td>Setting up &amp; operation of 8 kA multipolar cells with molten MgCl₂ feed – Study of cell performance</td>
</tr>
<tr>
<td>2012</td>
<td>Sanction of funds for setting up of another Magnesium Pilot Plant for resolving technical issues before its implementation on industrial scale</td>
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</tbody>
</table>
Flow sheet for MgCl₂ recycling developed at DMRL

Technical issues to be resolved:
- Mechanization of magnesium metal ladling and improvement systems for chlorine recovery
- Al pick up by metal from refractory
- Improvement in current efficiency and metal yield
- DMRL to set up another pilot plant to work on above

Magnesium metal produced in the DMRL pilot plant
DMRL titanium sponge technology for large capacity plants

M/s
Steel Authority of India Limited (SAIL)
Tata Steel
Indian Rare Earths Limited
Vedanta Industries

expressed interest in DMRL technology for setting up of large capacity titanium sponge plants

-SAIL also explored tie-up with TIMET for such a venture

-DMRL technology is under evaluation for its suitability for a titanium sponge plant of 5000 / 10000 MT per year
Prospects for titanium sponge in India

- Excellent mineral reserves
- Established mineral processing industry
- Scope for increased consumption of titanium due to availability of sponge locally (without importing)
- Projections indicate increased demand in a large number of applications
Acknowledgements

Thanks to International Titanium Association (ITA) for giving this opportunity