

TECHNOLOGY FOCUS



Bulletin of Defence Research & Development Organisation

Technology Focus is published bi-monthly by the Defence Scientific Information & Documentation Centre (DESIDOC) on behalf of the Defence Research & Development Organisation (DRDO), Ministry of Defence, Government of India. It focuses on the technological developments in the Organisation, covering the products, processes and technologies.

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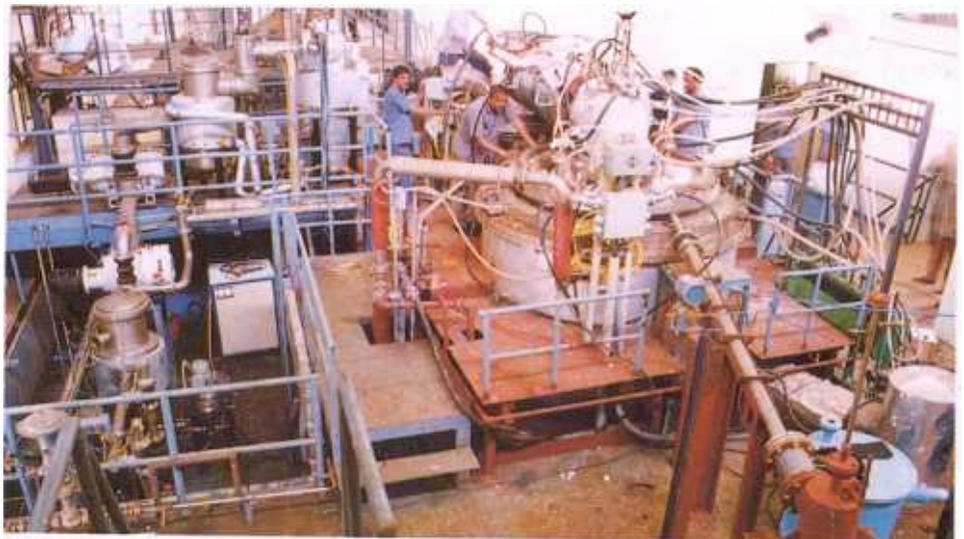
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TECHNOLOGY FOR COMMERCIAL PRODUCTION OF TITANIUM SPONGE

DRDO has recently demonstrated state-of-the-art Combined Process Technology for the production of titanium—a material of strategic importance. With this, the country is now poised for commercial production of titanium the technology for which is currently available with a select group of developed countries.



Combined process equipment for *Ti* sponge production in 4000 kg batches



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MESSAGE

The Defence Research and Development Organisation (DRDO) has successfully developed several products, processes and technologies, most of which have already been inducted into the Services.

The products developed by DRDO are of international quality and many of which are proving to be cheaper as compared to those of developed countries. I am happy that *Technology Focus*, DRDO's bi-monthly publication, has been projecting the achievements of the Organisation at both national and international level in proper perspective.

I wish the DRDO and the publication *Technology Focus* success in their current and future endeavours.

P.R. Sivasubramanian

(P.R. SIVASUBRAMANIAN)

Titanium—A Strategic Material

Titanium known for its excellent corrosion resistance, high strength and low density, is widely used for aero-engine and airframe structures in both civilian and military aircraft. Titanium-alloy components also find applications in satellite launch vehicles, rockets and missiles. Titanium is thus regarded as a metal of strategic importance. Excellent corrosion resistance of titanium to a wide range of chemicals, acids and alkalis has led to its use in thermal and nuclear power plants, desalination plants, fertilizer and petrochemical plants, and electroplating industries. Bio-medical implants, sports goods, automobiles, and architecture and consumer durables are some of the other emerging areas of titanium applications.

Production of Titanium Sponge

Titanium is extracted in the form of titanium sponge from its oxide minerals through magnesium reduction of its chloride intermediate. However, the sponge production process is complex and cumbersome. It involves a number of complicated unit operations at high temperature and high levels of vacuum, involving a variety of corrosive media. The technical know-how for the production of titanium sponge has so far been available with a select group of developed countries comprising Japan, USA, Commonwealth of Industrial States, UK and China. As of now, India's total titanium requirement is being met by imports even though it possesses a large reserve base of titanium minerals. Ilmenite ($FeO \cdot TiO_2$), an important titanium mineral, is widely distributed along the Indian coastal lines. With proven reserves exceeding 270 million tonnes, about 37 per cent of the total world Ilmenite reserves is located in



7 KA, 2 module multipolar cell

the beach sands of the southern peninsular India.

Combined Process Technology

DRDO has been working on the development of an integrated flow sheet and technology for large scale production of titanium sponge starting from titanium tetrachloride which is



500 tonne capacity *Ti* sponge ejection press

produced by high temperature chlorination of oxide concentrates. Production of titanium sponge by Kroll process involving magnesium reduction of $TiCl_4$ and vacuum separation of reduced mass for preparing sponge, free from entrapped $Mg/MgCl_2$, has been one of the thrust areas of development at DRDO. Initially, DRDO demonstrated the technology for development of titanium sponge in 2000 kg batch size using the conventional two-step Kroll process involving separate equipment for 'reduction' and 'vacuum distillation'. DRDO has recently demonstrated state-of-the-art Combined Process Technology, wherein reduction and vacuum separation processes are carried out in a single equipment thereby gaining benefits of energy and labour savings, improved productivity and also superior product quality. Further, this new technology has been demonstrated on a larger 4000 kg batch size to enable the establishment of a commercial plant without involving any further scale up.

Magnesium chloride is obtained as a by-product during the reduction of titanium tetrachloride by magnesium. It is customary in large industrial plants to recover magnesium and chlorine by fused-salt electrolysis and

recycle them in the plant for achieving a reduced cost of production of titanium and thus an improved commercial viability. Fused-salt electrolysis is a complex technology especially for the production of magnesium. Realising the importance of the by-product recovery step for economic production of titanium, DRDO has fully developed the technology for the production of magnesium by fused-salt electrolysis in a state-of-the-art prototype multipolar cell capable of recovering 220 kg of Mg per day.

Salient Features

- Use of a set of two identical stainless steel reactors. One is located inside a furnace for carrying out sequentially titanium tetrachloride reduction with magnesium and pyro-vacuum treatment of the reduced mass, and the other inside a water-cooled chamber to receive the distillates of excess magnesium and residual by-product magnesium chloride during the pyro-vacuum treatment step.
- Operation of reduction and vacuum separation steps using a single, double purpose, pit-type electric resistance furnace, specially designed for air-cooling during the



4000 kg titanium sponge cake

reduction stage and for vacuum operation during the pyro-vacuum treatment stage.

- Use of a stainless steel interconnecting pipe, provided with specially designed electric heaters, to combine the two stainless steel reactors positioned side by side.
- Use of a valveless top transfer system for tapping the by-product liquid magnesium chloride from the

reactor during the reduction stage into a custom designed ladle by the application of pressure/vacuum.

- Use of a microprocessor-based Digital Distributed Control System for automatic control of the various process parameters, viz., $TiCl_4$ purification, reduction and vacuum separation.
- Use of a specially designed equipment comprising hydraulic pressure jaw crushers, roll crushers, vibrating screens, sponge blenders for grading and processing of the sponge cake into 2 to 25 mm size fraction.
- Recycling of magnesium chloride, the reaction by-product in the 'reduction' step, by fused-salt electrolysis in multipolar cell.

With the successful demonstration of titanium sponge production technology by the advanced 'Combined Process Route' and magnesium metal by 'Fused-Salt Electrolysis' in multipolar cell, the country is poised for commercial production of titanium.

CHROME MASK BLANKS

Chrome mask blanks find extensive use in the fabrication of masks for various lithographic processes used in solid-state device fabrication. The masks fabricated on these blanks have various advantages like fine resolution, edge acuity and longer shelf-life in repeated process of lithography. Further, where sub-micron geometries are encountered, the use of a mask fabricated on these

chrome blanks by E-beam writing is the only solution. Chrome blanks are therefore the basic material for directly writing masks by E-beam process.

Chrome blanks are basically glass plates with specific optical transmission (85 per cent at $\lambda = 0.35 \mu\text{m}$), mechanical flatness ($2 \mu\text{m}$), and parallelism on which a controlled film of chromium (800-1000 Å) is

deposited maintaining excellent adhesion and uniformity of thickness (± 5 per cent) over its extended surface.

Further, since these blanks are to be used for fabrication of masks by E-beam process, film resistivity (20 ohm/sq) is an essential requirement. In addition, deposited chromium film

should have pinhole density of ≤ 0.05 Pcs/cm².

DRDO has been regularly importing these chrome glass blanks for writing the masks of micron and sub-micron geometries. Appreciating the strategic technological importance of chrome glass blanks, DRDO planned a crash programme to indigenously develop these chrome blanks using existing facilities and expertise in collaboration with the Department of Electronics.

DRDO has now successfully developed and demonstrated the technology for fabrication of chrome mask blanks for 2.5 and 3.5 in. size. The blanks meet all the required specifications. The performance evaluation of fabricated blanks has also been carried out by Bharat Electronics Ltd, Bangalore and SAC, Ahmedabad, in addition to DRDO.

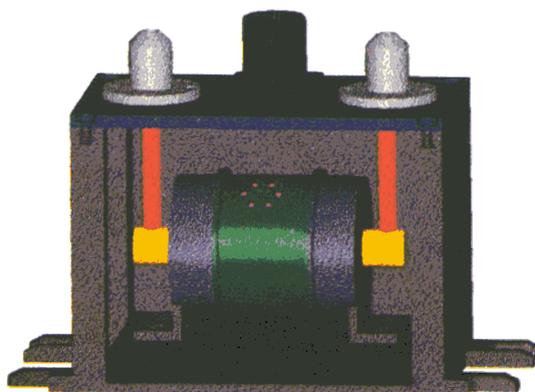
The following specifications have been achieved:

Film thickness	$900 \pm 70 \text{ \AA}$
Film thickness uniformity	± 5 per cent
Sheet resistance	20 ohm/sq
Optical density at 4000 \AA	2.8
Pinhole density (1-4 μm)	≤ 0.05 Pcs/cm ²
Pattern roughness	$\sim 0.2 \mu\text{m}$

SOLID-STATE ELECTROLYTE OXYGEN SENSOR

Solid-state electrolyte oxygen sensor is gaining popularity in medical and aeromedical applications due to its fast response to satisfy physiological protection requirements, long operational and storage life, least maintenance and calibrating problems and easy interface to electronic systems. Solid-state electrolyte oxygen sensors are made from a number of ceramic metal oxides; but the most commonly used material is ZrO_2 .

ZrO_2 is normally an electrical insulator but when doped with traces of di- or trivalent metal oxides (Y_2O_3) and heated above 600 °C, it becomes conductive due to the presence of oxygen vacancies in the crystal lattice. Since conductivity is determined by this specific mechanism, the electrical properties of this material are directly influenced by surrounding oxygen concentration and the material provides direct analogue electrical



output. This sensor does not cross-couple with argon or other gases.

DRDO has successfully developed the above sensor for oxygen concentration measurements. The sensor consists of zirconia disc coated with porous platinum electrodes on both sides and electrical wires are taken out from the cell for

measurements. One side of the disc is exposed to reference gas while the other to the gas in which oxygen concentration needs to be measured. The disc is placed in an alumina tube and is properly secured using high temperature ceramic seal. Alumina tube is held between a pair of NILO-475 diaphragms to provide stability against mechanical and thermal induced stresses. The nichrome wire wound on alumina tube forms the heater. Chrome-alumel thermocouple measures the temperature of operation. The electronic control unit based on 80196 microcontroller provides the required circuits for temperature control, temperature sensing and signal measurements.

The above sensor has been developed for the first time in India specifically for aeromedical applications. It will also find useful application in biomedical engineering.

LARGE CALIBRE FLEXIBLE LINEAR-SHAPED CHARGES

Conventional cutting techniques are not suitable for cutting hard metallic and non-metallic targets in milliseconds, especially in hostile conditions. To perform the cutting of targets in adverse environment, directed explosive energy systems preferably in the form of Flexible Liner Shaped Charges (FLSCs) can be deployed.



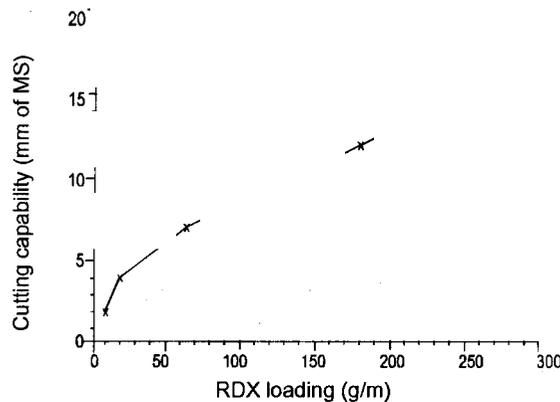
250 g/m flexible linear shaped charge

The FLSC essentially consists of an explosive core enclosed in a linear ductile metal sheath. On detonation, the shock-wave collapses and transforms the inverted 'V' section (called liner) into high velocity metallic jet, which performs the task of progressive linear cut in the target plate. DRDO has already developed a number of low calibre FLSCs with explosive loading ranging from 0.8 to 25 g/m length of charge.

DRDO has now developed the design of high calibre FLSC with an explosive core of hexagonal shape with inverted 'V' in contrast to half-moon shape explosive core of low calibre FLSCs. The machinery required for the extrusion of large calibre FLSCs

has been designed and fabricated indigenously. Large calibre FLSCs

with RDX loading from 65 to 250 g/m, were extruded and evaluated for their cutting capability in mild steel plates. These systems have been successfully tested for cutting of different weight class of Air Force bombs. The lengths up to 1 m, which meet all operational requirements, can be extruded using this facility.



Cutting capability of FLSC

This development will considerably improve the capability of rapid action forces entrusted with the task of quick cutting of strategic targets encountered in offensive operations.

MM WAVE COMMUNICATION SYSTEM

The bulk of communication systems, including line-of-sight and terrestrial systems are currently operating in microwave band. However, the overcrowding of microwave spectrum has led to the development and use of communication systems in millimeter (MM) wave region also. The MM wave

communication systems provide secured, short haul, reliable communication with large channel capacity. These systems are particularly suitable where a clear line-of-sight is available and are considered ideal for providing secured point-to-point communication due to their high directivity, low probability of

intercept, large bandwidth, and compact size.

DRDO has developed MM Wave Communication System in 35 GHz frequency band. The system consists of MM wave transceiver and base band. The transmitter consists of a 35 GHz Gunn oscillator, PIN diode

modulator and the receiver consists of BPF, mixer, IF and AGC amplifiers and detector. The antenna is common to both transmitter and receiver and is connected through an orthomode transducer. Being a homodyne system, a part of the transmitter power is coupled to mixer through a directional coupler. The PIN diode modulator provides on-off keying to MM wave signal in accordance with digital data stream. The base band circuit consists of voice codec, data amplifier, PCM-MUX and provides 120 voice channel transmission.

Various components of MM wave transmitter/receiver are realised in planar waveguide configuration and hybrid MIC technology. A built-in test facility is incorporated in the MM wave system to monitor the health of the terminals. A separate voice channel is used as supervisory channel and also as hot line in case PCM-MUX fails to



MM wave communication system

operate/function. The system has undergone extensive field trials and is now under trial for its commercial application. Presently, the system has been set up at some of the DRDO

labs/estts providing hotline communication link between these establishments.

DRDO PATENTS

ELECTRONIC MATERIALS

Optical Window Grade Zinc Sulphide

Zinc sulphide is an important material for the fabrication of infrared transmitting windows/domes used in areas like remote sensing. This is because of its excellent thermo-mechanical properties coupled with its very good transmission in 2-12 micron range of infrared.

The known processes for the preparation of zinc sulphide have the drawback that either the yield of optical window grade zinc sulphide is low or the process leads to irregular-sized particles with irregular physical properties or has impurities like zinc oxide.

DRDO has developed a process for the preparation of zinc sulphide which leads to high yield of the order of 70 to 80 per cent of optical window grade zinc sulphide. The grain size of the optical window grade zinc sulphide is relatively larger which reduces IR transmission losses and therefore accounts for its improved IR transmission capability.

Organo-Nickel Coordinated Compounds

The organo-nickel coordinated compounds having applications in some laser and laser-based systems are prepared by multistep processes wherein before each step, the intermediate has to be transferred to a different reaction vessel. These

processes result in lower yield and low purity of the product.

DRDO has developed an improved process for the synthesis of organo-nickel coordinate compounds. The process involves only two steps and is convenient as it does not require transfer of intermediates to a different reaction vessel at each step. The process leads to higher yield and higher purity of product and is cost-effective.

Inorganic High Temperature Resistant Sealant

The high temperature resistant sealants are required in laser and electronic fields wherein the temperature encountered may vary from 800 to 1400 °C. The known

organic sealants lose their property of adhesion or sealing at temperatures over 300 °C.

DRDO has developed a process for preparation of high temperature sealant which can withstand temperatures up to 1600 °C and high pressure of 4 to 6 atm. The process leads to higher yield and uses non-toxic oxides and water as solvent making it free from fire hazards. The sealant is capable of joining metal to ceramic as well as ceramic to ceramic.

NAVAL MATERIALS

Epoxy Non-Skid Paint

The non-skid paint is used to provide rough texture with requisite coefficient of friction to prevent slippage of personnel and VTOL aircraft while taking off and landing on the ship. The paint is also required to possess adequate thermal resistance to withstand stresses generated by hot fuel gases from the aircraft. The known non-skid paints have relatively short life and inadequate thermal resistance due to which the decks of ships necessitate frequent repainting.

DRDO has developed a process for the preparation of a non-skid paint which is abrasion and impact resistant to a fairly high degree. The paint has thermal resistance to withstand hot fuel gas emanating from the aircraft operating from the ship deck. The paint is resistant to sea water and kerosene and also has weathering resistance under ultraviolet radiations.

Rubber-based Passive Acoustic Materials

Acoustic baffles are used in large high performance transducer arrays for isolating ship noise as well as for improving directivity and sensitivity of transducer elements. The materials used for construction of baffles are required to possess specific acoustic and dynamic mechanical properties and have to be compatible with hostile marine conditions, such as salinity, sub-ambient temperature, hydrostatic pressure etc.

DRDO has developed a process for preparation of rubber-based passive acoustic material for construction of acoustic baffles. The material has a flat acoustic velocity in the operational frequency of 1-10 Hz, which has been possible because of the specific compounding materials used in the process for preparation of the material. The material obtained has the requisite mechanical properties for operations in deep sea conditions and has sea water resistance for conditions deployment around five years.

FOOD TECHNOLOGY

Rapid Estimation of Deterioration in Foods

Food materials deteriorate during storage as a result of chemical and bio-chemical reactions forming chemicals, such as acids, peroxides, aldehydes, ketones, hydrocarbons etc. These chemicals are responsible

for off-tastes, off-flavour and deterioration in quality. The known kits for estimation of the deterioration are based on estimation of acidity, peroxide concentration, aldehyde level etc in the spoiled food material. The tests based on the known formulations used for testing the deterioration in foods are lengthy and time consuming. These tests require laboratory facilities for measuring deterioration in foods and therefore cannot be used for on-the-spot assessment of deterioration in foods.

DRDO has developed a formulation which is capable of testing the storage deterioration in food materials within 3 to 4 min. The testing with this formulation does not require any laboratory facilities. The formulation is stable with shelf-life of around eight months at ambient conditions.

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