Glimpses on the Activities of CSIR-Central Glass & Ceramics Research Institute, Kolkata

Bharat Bhushan Jha
Chief Scientist & Head, BDSD,AMMCD and Glass Division

Business Meet at NAL Bangalore
Date: 17.10.2019

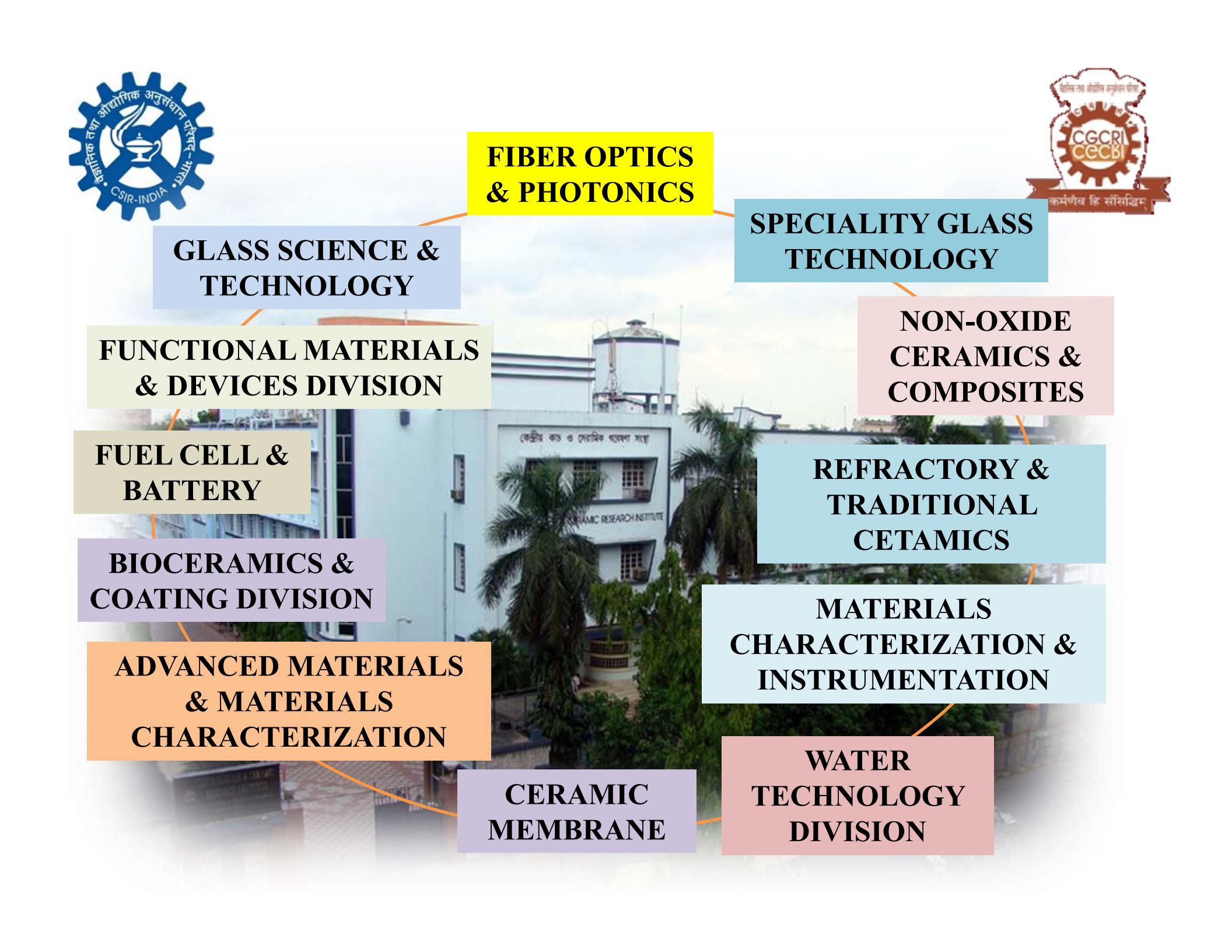
CSIR-Central Glass & Ceramic Research Institute, Kolkata

VISION

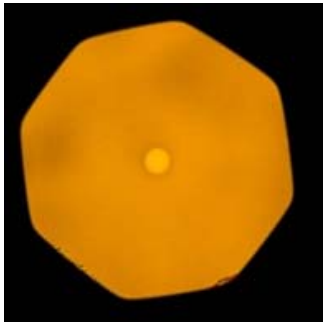
Enduring innovation in science & technology of materials to attain the status of an ultimate centre of excellence in glass and ceramics technology

MISSION

To provide scientific industrial research and development in the area of glass, ceramics and related materials that maximizes the economic, environmental and societal benefit for the people of India


FIBER OPTICS & PHOTONICS
SPECIALITY GLASS TECHNOLOGY
GLASS SCIENCE & TECHNOLOGY
NON-OXIDE CERAMICS & COMPOSITES
FUNCTIONAL MATERIALS & DEVICES DIVISION
REFRACTORY & TRADITIONAL CETAMICS
FUEL CELL & BATTERY
MATERIALS CHARACTERIZATION & INSTRUMENTATION
BIOCERAMICS & COATING DIVISION
WATER TECHNOLOGY DIVISION
ADVANCED MATERIALS & MATERIALS CHARACTERIZATION
CERAMIC MEMBRANE

Important Technologies from CSIR-CGCRI On the Threshold of Commercial Exploitation

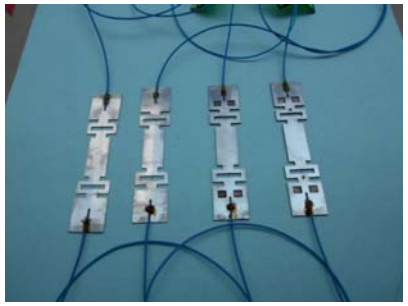


FIBRE FOR INDUSTRIAL, MEDICAL AND STRATEGIC USE

GLASS BEADS FOR HIGH LEVEL NUCLEAR WASTE IMMOBILIZATION

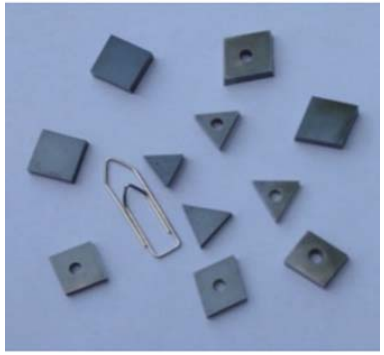
RADIATION SHIELDING WINDOWS

SOFC TECH-NOLOGY



FIBRE BRAGG GRATINGS

LASER GLASS



HIGH SPEED CUTTING TOOLS

CHALCOGENIDE GLASS FOR IRWINDOWS

RBSN MISSILE RADOME

Development of sol-gel based AR coating on large sized RSW glass

- **RSW glass blocks of 400x400×100 mm sizes**

- It is desirable to make antireflective (AR) coating on theses glass surfaces for improving the visible transmittance.
- Knowledge-base on the development of various types of optical coatings including AR coating on glass substrates by sol-gel processing for different applications.
- Challenge has been taken to develop AR coating on large sized (say, 400 x 400 x 100 mm^3) high lead containing RSW glasses for the used in nuclear hot cells of our country.

Specialty Refractory Crucible Development


10 L Specialty Refractory Pot & Stirrer


60 L Specialty Refractory Pot and Stirrer

Pot sustained six melting with repeated heating and cooling and survived 231 hrs.

GLASS BEAD MANUFACTURING TECHNOLOGY: A MATERIAL OF CHOICE FOR ENHANCED NUCLEAR SAFETY

2006-10: Lab Scale Development

2010-11: Scaled up 150 Kg Batch Scale

2011 onwards: Production of Glass Beads in Tonnage Scale

Latest supply of Glass Bead with varying compositions to DAE: 73 MT

Total Consignment Valuation over the supply of 73 MT Glass Bead: Rs. 499 lakhs

2011, 2016 : License Agreements with M/s H & R Johnson for production of both Frit and Nodule of Five Component system; 3 Compositions

- Technology License Agreement with H & R Johnson (India)
  1. For Regular Composition Glass Beadin May 2012
  2. For Low Sodium Glass Bead in February 2014
  3. For High Sodium Glass Bead in November 2016

2014 onwards: R & D on New Seven Component Glass Bead

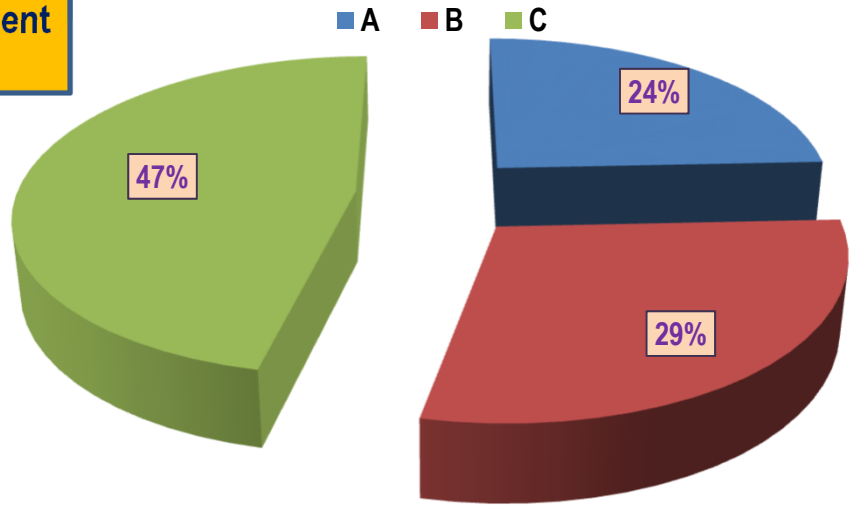
- Development of new five compositions in seven component system
- Supply of 1 MT bead of Centriod Composition to DAE's Kalpakkam facility

DAE's Projected Annual Requirement: 50T

Estimated commercial Value: Rs. 4 Cr / annum

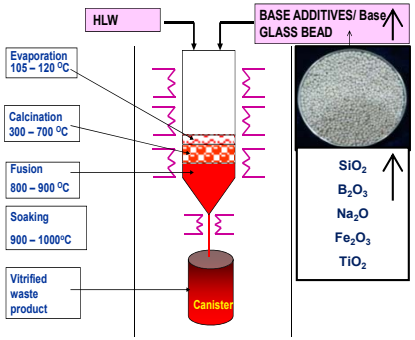
Total Revenue generated through Licensing & Transfer of Technology: Rs.148 lakhs

A. Technology License Fees 36 lacs out of 148 lacs i.e 24 %
B. Royalty Charges 43 lacs out of 148 lacs i.e 29 %
C. Technology Support Fees: 69 lacs out of 148 lacs



Future: Technology to more partners:
New up-coming requirement:15 MT of Seven Component Borosilicate Glass Bead from DAE

Immobilization of Nuclear Waste



73 MT of Glass Bead of varying compositions
(Cost: - 5.0 Cr) supplied so far to
Department of Atomic Energy (DAE)

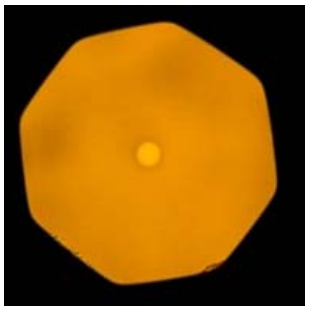
- Tarapur Atomic Power Station
- IGCAR, Kalpakkam

MOU between NRG, BARC & CSIR-CGCRI on RSW glass technology and specialty refractory crucible on 22/10/2018

CSIR-CGCRI and BARC representatives are signing the documents on behalf of their organizations.

Packaged Fibre Laser Modules for Strategic, Industrial & Medical Applications

- **Prototype laser modules: 100 W CW and 20W pulsed at 1 µm developed for industrial applications.**
- **Prototype 30W CW laser at 2µm developed for medical applications.**



CSIR-CGCRI fabricated laser fiber & Prototype Module

CSIR-CGCRI Technology for Fiber Lasers developed under FTT Project

Ytterbium Pulsed Laser for Marking & Scientific Applications

- Wavelength: 1.06 µm
- Maximum Average Power: 20 W
- Maximum pulse energy: 1.0 mJ
- Beam Quality : $M_2 < 1.8$
- Pulse width: 80-200 ns

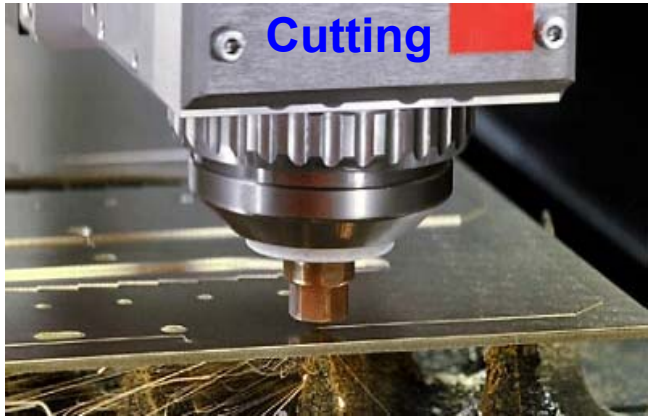
- Field-trial of Pulsed Yb-fiber laser is successfully completed at M/s Spinks-Global (Gurgaon)

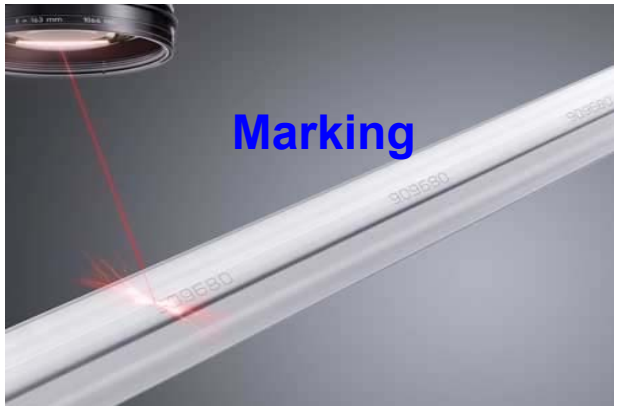
Thulium CW Laser for Medical & Scientific Applications

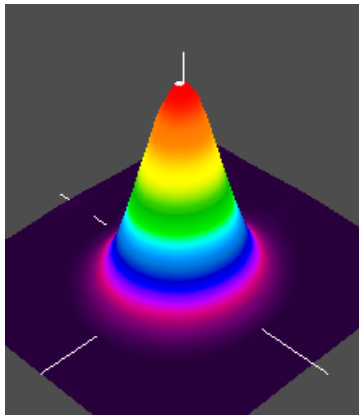
- Operating Wavelength: 1.94 µm
- Maximum Output Power (CW) : 30 W
- Modulated: 10 Hz-1 KHz

- Pre-Clinical trial of Tm-fiber laser is done

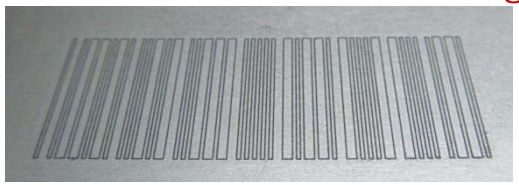
Marking Results Compared to Commercial Laser



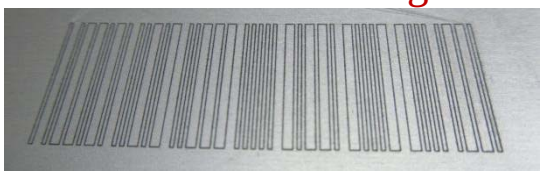




Commercial Laser Marking



CGCRI Marking



CGCRI Marking

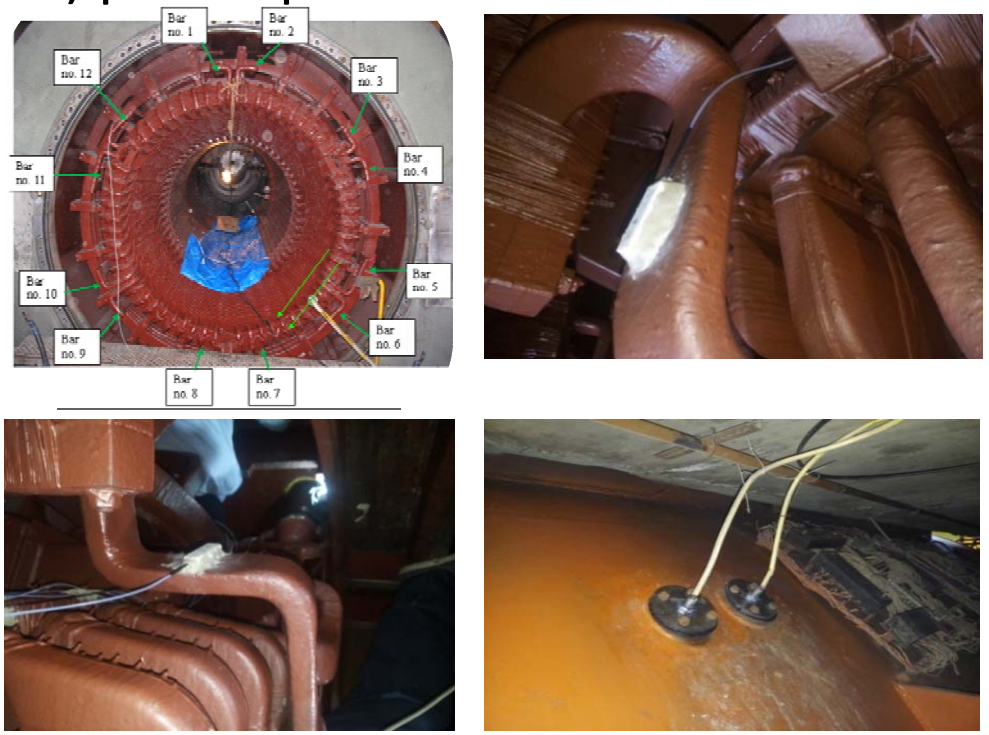
Marking on Aluminum

Commercial Laser Marking

FBG Sensor for power plants

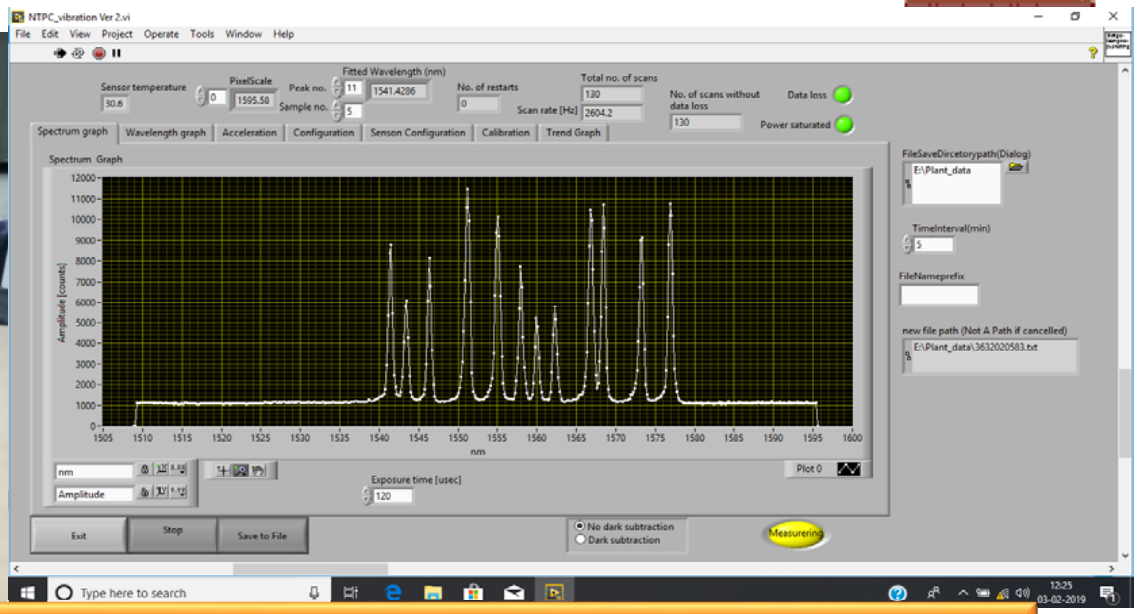
Deployed FBG based vibration sensors and FBG interrogation system for stator end winding vibration monitoring at DADRI (210 MW) power plant of NTPC

Packaged vibration sensors



Sensors Installed in the generator

Signal processing system and the PC in the control room



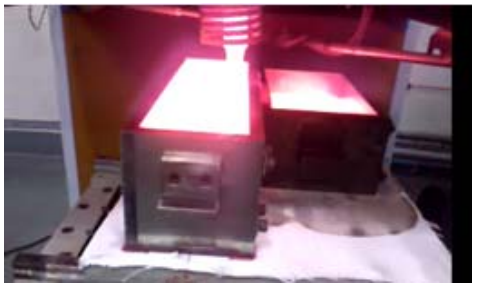
- **Present status:**
  - **Patent application: In process**
  - **MOU to be signed for the supply of 5 units**

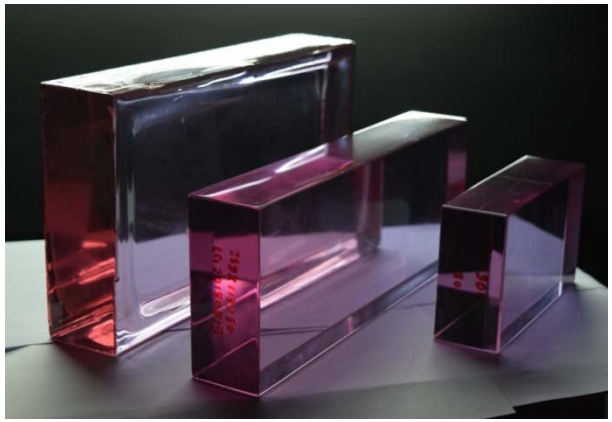
  (One unit consists of 16 vibration sensor and one Signal processing unit system)
    - Cost of one unit is ~ 35 lakhs

Process Technology Demonstration in 5 L scale

- Adoption of bottom pouring flow casting technique based on Induction heating

Bottom pouring and flow casting of glass





Standardized the process parameters for casting of glass Discs of 250x125x40 mm^3 supplied to RRCAT for testing bulk homogeneity

Setting up of infrastructure and up scaling the technology to 15 L scale

- Optimization of process parameters to produce 300 x 150 x 40 mm glass Discs
- 30 no of glass blocks to be delivered

To meet the requirement for the development high power laser of 1 kJ at RRCAT

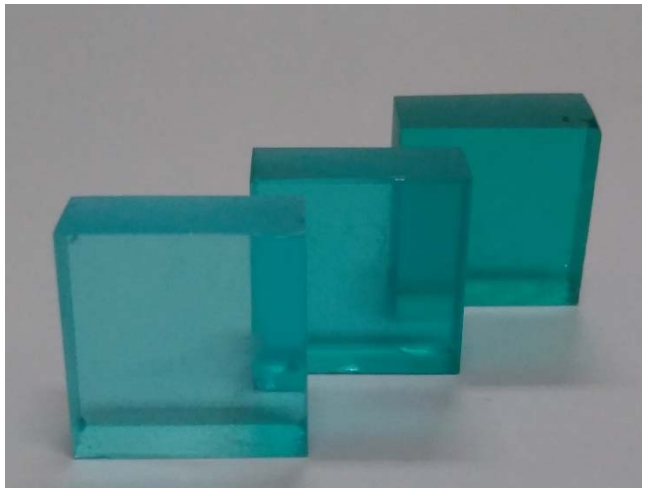
Edge Cladding glass for Nd- glass discs

- Nd: glass discs have large sizes. parasitic oscillations and Amplified Spontaneous Emission (ASE losses cause “Loss of inversion” before actual arrival of laser pulse. Hence, the discs are edge cladded with high absorption glass / glass ceramic.

CGCRI Developed CuO doped **Phosphate** Glass for edge cladding of Nd-phosphate laser glass

Absorption coefficient ~7.5 cm^{-1}

- Refractive Index, Coefficient of Linear Thermal Expansion and Hardness matching with the Nd- laser glass



Developed Edge cladding glasses

Facility establishment and Development of Space grade Optical glasses

Signing of MoU With VSSC, ISRO is under progress

Objectives

Phase I

Formulation and optimization of composition for specified optical Flint and Crown glasses

(VSSC has placed order to supply 5 no. each)

Sl. No	General Classification	Equivalent to Schott
1	Crown Glass	N-SK5
2	Crown Glass	N-SK14
3	Crown Glass	N-LAK10
4	Flint Glass	N-SF14
5	Flint Glass	N-LAF21

Phase II

Establishment of pilot scale (5 L capacity) glass melting facility for the realization of flint and crown glasses

Development and demonstration of process technology in 5 L melt scale

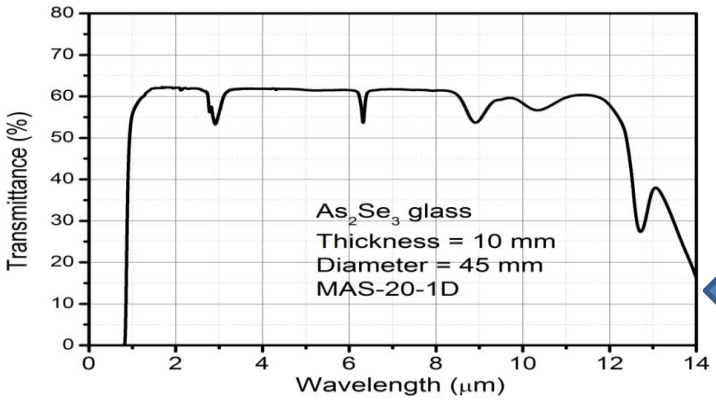
Supply of optical glasses of specified dimensions to ISRO

Phase III

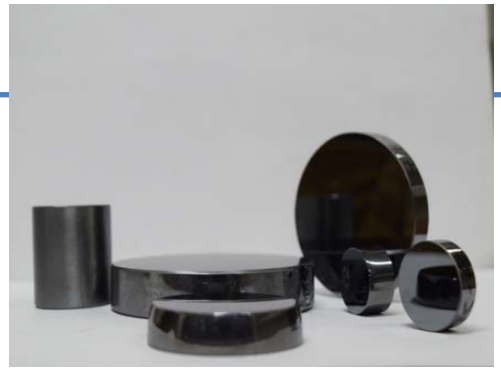
Upscaling of technology with industrial collaboration

Chalcogenide Glass (ChG) for IR Optics Applications

Salient Achievements



UV-vis-IR Transmission Spectrum



Processed bigger As_2Se_3 glass (varied dimensions)

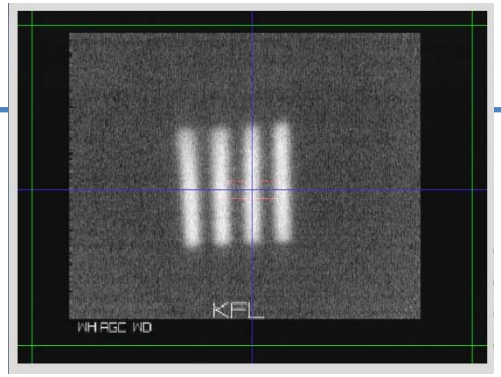


Image formation

- **Arsenic-Selenium (As-Se) based chalcogenide glass**

Property	IRG-26 glass of Schott, Germany	Equivalent glass developed at CGCRI
Density, g/cm^3	4.63	4.61
RI@ 1.5 µm	2.84	2.83

- Supplied few glass pieces of desired dimensions to BEL
- BEL showed interest in As-free chalcogenide glasses as well
- ToT to BEL (their projected requirement is in tune of few hundreds to thousand pcs per year) for As-free glass

Radome Facilities

Radome Furnace Bay

Nitridation Furnace

CSZ De-Humidifier

Fired Astra Radome

Project Details and Deliverables.

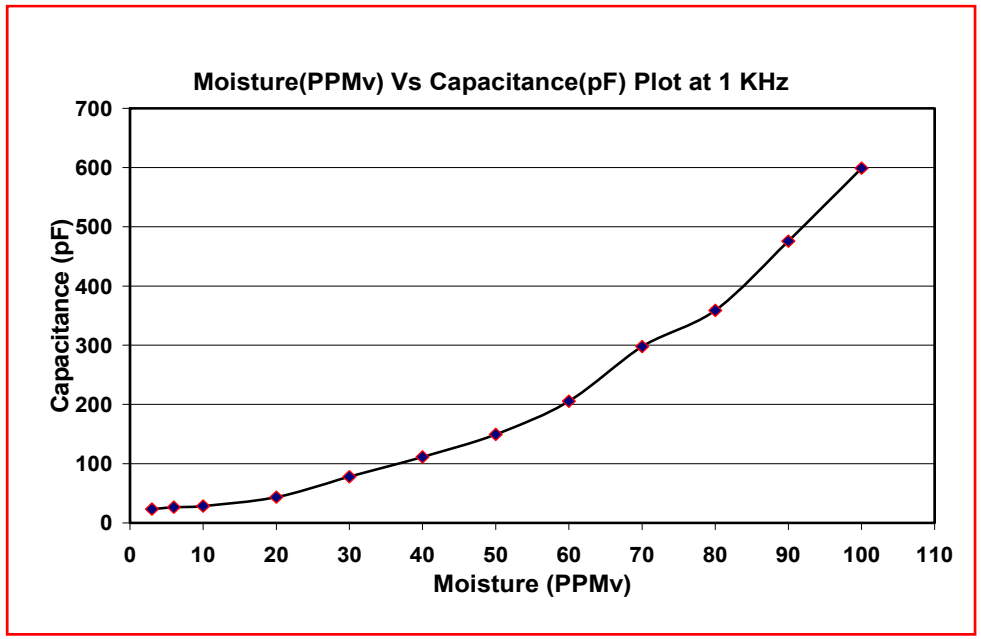
- Project initiation date : **14th December 2015**
- Project completion date : **31st March 2020**
- Initial Cost of Project : **Rs 1103 lakhs**
- An extra Amount allocated : **Rs 105 Lakhs**
- **PMRC meeting was held on 14th June 2109 at CSIR-CGCRI in presence of Director RCI and Director CSIR-CGCRI**
- **Two Radome** has been supplied to RCI for conducting different testing (Machining, Thermo-mechanical, EM, High temperature Shear)
- **Two more qualified Radomes (Size:- 180 mm Dia & 472 mm ht.)** are
- ready for dispatching.
- **Six more Radomes** will be ready within August 2019.
- Within September 2019, **5 qualified Radomes** are required to be supplied to RCI and we are well within our schedule.
- In PMRC meeting RCI Director expressed his satisfaction on the progress of the project which is well within the schedule time.

Low ppm Moisture Meter developed at CGCRI under NTPC Project

sensor

Description	Trace moisture sensor γ- Alumina
Measuring Range	5 -100 ppm
Accuracy(at 30^{0}C)	± 1%
Temperature Limits	10^0 C – 120^{0}C
Response Time (90%)	10 s
Powder Supply/Consumption	9 Volt DC, <10 mA
Weight	200g
Options	Digital/LCD OR LED Display

Sensor and meter tested at Nisha Engg. Co. Baruipur



- Delivery Mode Project Sanctioned by NTPC Rs.47.00 Lakhs.
- Indian Navy requirement for installation of low ppm moisture meter for high pressure air stream in the war ship.

Feld trial at Nisha Engg. Co

CGCRI -Line Ministry-Industry-Connections

Technologies

- Specialty Glass (RSW, Laser Glass)
- Ceramic Radomes and Cutting Tools
- Specialty Fibers and laser amplifiers
- Fibre Bragg Grating Sensors and Ceramic based Sensors
- Fibre Lasers
- Ceramic Membrane Based Water purification
- Sol-gel coatings

Major Clients

DAE
DRDO
ISRO
NTPC
DST
BEL
Prism Johnson
BHEL

Dr Harsh Vardhan, Hon'ble Union Minister, Science & Technology, Earth Sciences, Forest & Climate Change, visits CSIR-CGCRI on 14th September 2018

Visit of Honourable Governor, West Bengal during TOLIC meeting at CSIR-CGCRI, Kolkata On 28.01.2019

Delegation from the Bangladesh Council of Scientific and Industrial Research from 14.01.2019 to 05.04.2019

Visit of DG-CSIR to CSIR-CGCRI on 22.01.2019

XXVII ICG Congress to be held in Kolkata, India during 2025
&
ICG-2019 Council Meeting at Boston, USA (June 11, 2019)

ICG
International Commission on Glass

ICG INTERNATIONAL COMMISSION ON GLASS
A SOCIETY OF SCIENTIFIC AND TECHNICAL ORGANISATIONS

Alicia Durán
President of the ICG
c/Kelsen 5, Campus de Cantoblanco
28049 Madrid, SPAIN

Dr K Muraleedhara
Director of CGCRI
Copy to Dr. Rajan Sen

Dear Dr Muralledhara and Dr. Rajan Sen,

It is my great pleasure to communicate you, as President of the International Commission on Glass, that Kolkata, India, has been selected as the next venue of the ICG congress in 2025.

The Council of ICG has considered two proposals from Japan and India, both very strong and well justified that have been voted by secret ballot. India obtained 22 votes and Japan 21. So, India will be the next venue of the ICG congress in 2025.

I congratulate you and the committees that have hardly worked for preparing the proposal.

Now, it is time to begin to work for arriving to a successful congress in Kolkata.

Best regards

Alicia Durán
Research Professor of CSIC
President of the ICG

ICG – International Commission on Glass
www.icglass.org

ICG-2019 Council Meeting at Boston, USA (June 11, 2019)

Visit of RC Chairman to CSIR CSIR-CGCRI on 22.02.2019

Post Graduate Seminar at CSIR CSIR-CGCRI

DELEGATION FROM CHINA 03.04.2019

World Intellectual Property Organization Seminar

On 26.04.2019

Other Major Events

One Day Technical Seminar by Asian Forum of Materials Testing on 22.02.2019

National Conclave on 'Water Resources Management' During 17-18 Jan 2019

Theme Meeting on Precision Electron Diffraction Seminar on 19.02.2019

THANK YOU

Facility Created at NOCCD at CSIR-CGCRI

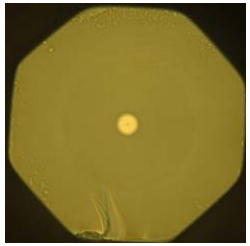
High Temperature Controlled Atmosphere Nitridation Furnace, M/S Nabertherm, Germany Make

RADOME

Packaged High Power Fiber Optical Amplifier Module

CSIR-CGCRI

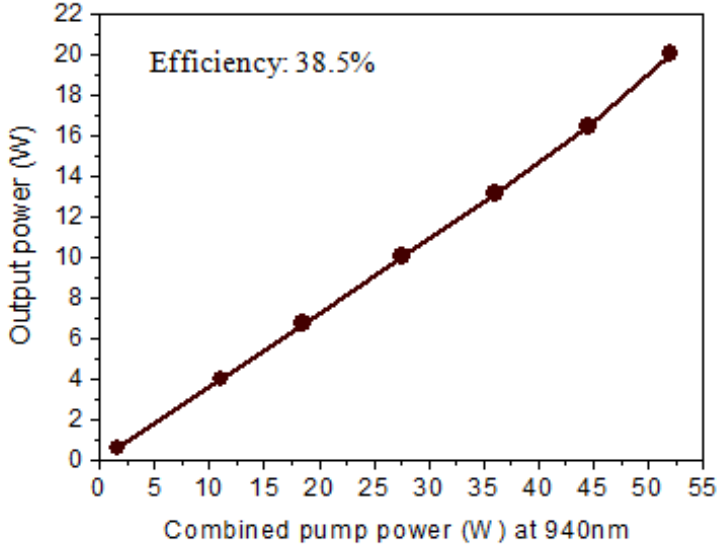
Octagonal shaped Er/Yb codoped fiber



High power Optical amplifier

High power EDFA module launched at CSIR-HQ on 04.04.2018

VINVISH



Output signal power at 1560nm against absorbed pump power at 940nm

Upscaling of Process technology (15L scale) for Nd:Glass blocks

Setting up the infrastructural facility for upscaling the technology to 15 L scale is in progress

Deliverables

Targeted sizes

Sl. No.	Nd_2O_3 wt %	Size of Nd:glass blocks	
1	0.5	90X90 x320 mm^3	Rods
2	2.2	260 x 135 x 40 mm^3 with edge cladding glass	Discs
3	2.2	310 x 160 x 40 mm^3 with edge cladding glass	Discs

From 5L scale

Cast Glass blocks of size 285 x 165 x 80 mm^3

To fabricate 250x125x40mm^3 disc

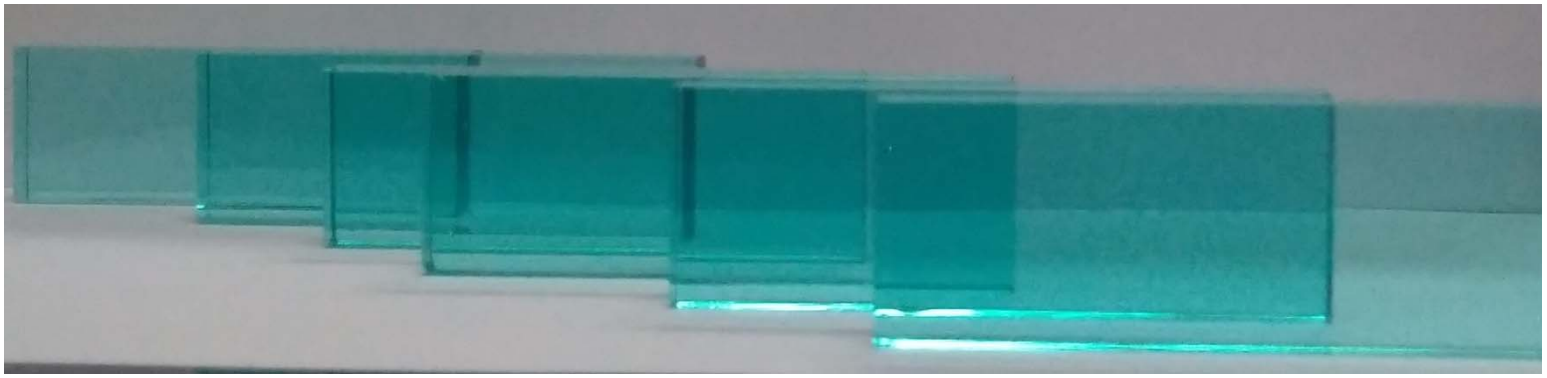
Edge Cladding glass for Nd- glass discs

- **For large sized Nd: glass discs, to limit parasitic oscillations and ASE losses the discs are edge cladded with high absorption glass / glass ceramic**

CGCRI has optimised the cladding glass having

- Absorption coefficient ~7.5 cm^{-1} &
- Real part of RI,
- Coefficient of Linear Thermal Expansion
- Hardness

matching with the Nd-laser glass



Cladding glass samples of dimension 80 x 25 x 10 mm^3

Facility establishment and Development of Space grade Optical glasses

Signing of MoU With VSSC, ISRO is under progress

Budget outlay: Rs. 20.56 Cr

Objectives

i) Formulation and optimization of composition for specified optical Flint and Crown glasses

ii) Establishment of pilot scale (5 L capacity) glass melting facility for the realization of flint and crown glasses

iii) Development and demonstration of process technology in 5 L melt scale

iv) Supply of optical glasses of specified dimensions to ISRO

Sl. No	General Classification	Equivalent to Schott, Germany
1	Crown Glass	N-SK5
2	Crown Glass	N-SK14
3	Crown Glass	N-LAK10
4	Flint Glass	N-SF14
5	Flint Glass	N-LAF21

Stipulated technical specifications of optical Glasses

Sl. No	Parameter	Specification
1	Refractive Index tolerance w.r.t base value	$\pm$ 1 $\times 10^{-4}$
2	Homogeneity of refractive index (n_d)	$\pm$ 1 $\times 10^{-6}$
3	Tolerance for Abbe value (V_d)	$\pm$0.2% to $\pm$0.3%
4	Thermo optical Coefficient (dn/dT)	To be decided
5	Stress Birefringence	$\leq$ 6nm/cm across 80% diameter
6	Maximum allowable cross-section area of bubbles in mm^2 per 100 cm^3 of glass volume	0.03 VB-Grade
7	Maximum number of allowed bubbles per 100 cm^3 of glass	4 Nos
8	Striae	None in sanctioned direction

Development of Process Technology for Hydrophobic Anti Reflective (AR) Coating on Radiation Shielding Window Glass

Lead-silicate glass with high lead oxide content (~71%) is known as Radiation Shielding Window (RSW) glass used in nuclear hot cells as viewing glass.

Problem:

- **High refractive index of RSW glass reduces visible transmission (VT)**
- **RSW glass absorbs moisture. So, in nuclear hot cells, VT of the glass further reduces due to the formation of a translucent layer**

Solution:

To enhance VT and to protect the glass surface, hydrophobic antireflective (AR) coating on RSW glass is highly desired

CSIR-CGCRI has vast expertise and knowledge-base on the development of coatings like hydrophobic, AR coatings on glasses by sol-gel processing

- **Hydrophobic AR coating deposited on RSW glasses.**
- **Coated samples are under examination by NRG, BARC.**

- **Sol-gel based AR coating (~1.2% reflection per surface) on RSW glass blocks (size: 200 x 200 x 20 mm^3) has been deposited by sol-gel dip coating technique.**
- **Result of required tests done by NRG, BARC, Mumbai is positive.**

Creation of a facility including a drain coater for deposition of the coating on RSW glass blocks of 400 x 400 x 100 mm^3 is in progress.

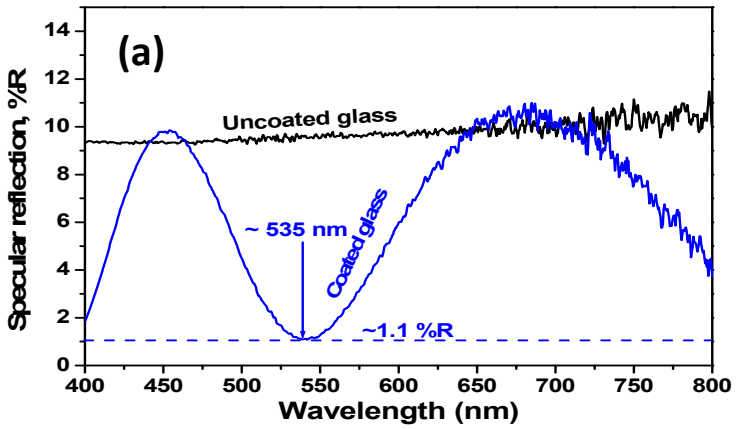
Final goal: Development of a process technology for deposit of the sol-gel **hydrophobic, AR coating** *on 1000 x 1000 x 200 mm^3 sized RSW glass blocks*

Characteristics of hydrophobic AR coated RSW glass blocks

- **Material:** Inorganic-organic hybrid
- **Single surface specular reflection (R):** ~1.2 % (RSW Glass, R >9% at 550 nm)
- **Pencil hardness:** 5H (ASTM D3363)
- **Adhesion test:** 5B Classification (Scotch tape test, ASTM D3359)
- **Static water contact angle:** 90-95^{0} (Hydrophobic)

AR coated Un- Stabilized RSW Glass

AR Stabilized RSW Glass



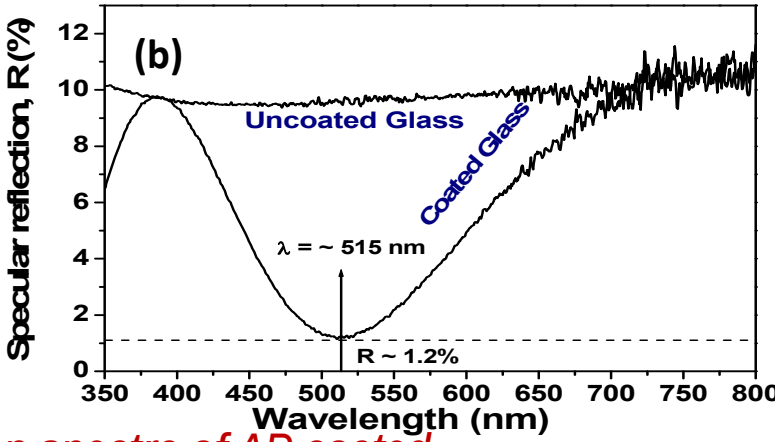


Fig. Single surface specular reflection spectra of AR coated (a) Un-stabilized and (b) Ce-stabilized RSW glasses.

TOT Values

Title	Agency	MOU Date	Value (Rs Cr)
Transfer of technology for packaged fibre laser	BEL	06.06.2018	0.675
Sponsored project : "A process for the synthesis of graphene oxide and its application in paints"	Berger Paints	30.10.2018	0.05
Development of Technology for manufacturing 700 X 700 X 150 mm^3 RSW Glass Slabs using 120 L Refractory Crucible	BARC	22.10.2018	8.03
Multi-use Refractory Crucibles and RSW Cullet	Prism Johnson	11.05.2018	1.40

TECHNOLOGY OF MANUFACTURING SPECIAL GLASS BEADS FOR NUCLEAR WASTE IMMOBILIZATION

Glass Frit

Glass Bead

CSIR Technology Award 2018

SUPPORT FOR NUCLEAR SAFETY MEASURES

RADIATION SHIELDING WINDOW GLASS TECHNOLOGY

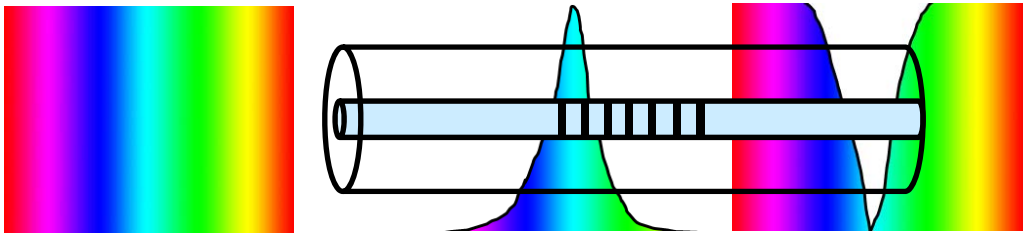
- Platinum pot technology ready for ToT
- Refractory pot technology being developed
- Incubation of Technology (6-18 months)
  - Kolkata (10L refractory pot, new furnace)
  - Naroda (Demonstration @ 140L Melting)

Collaborative efforts with DAE

Fiber Bragg Grating

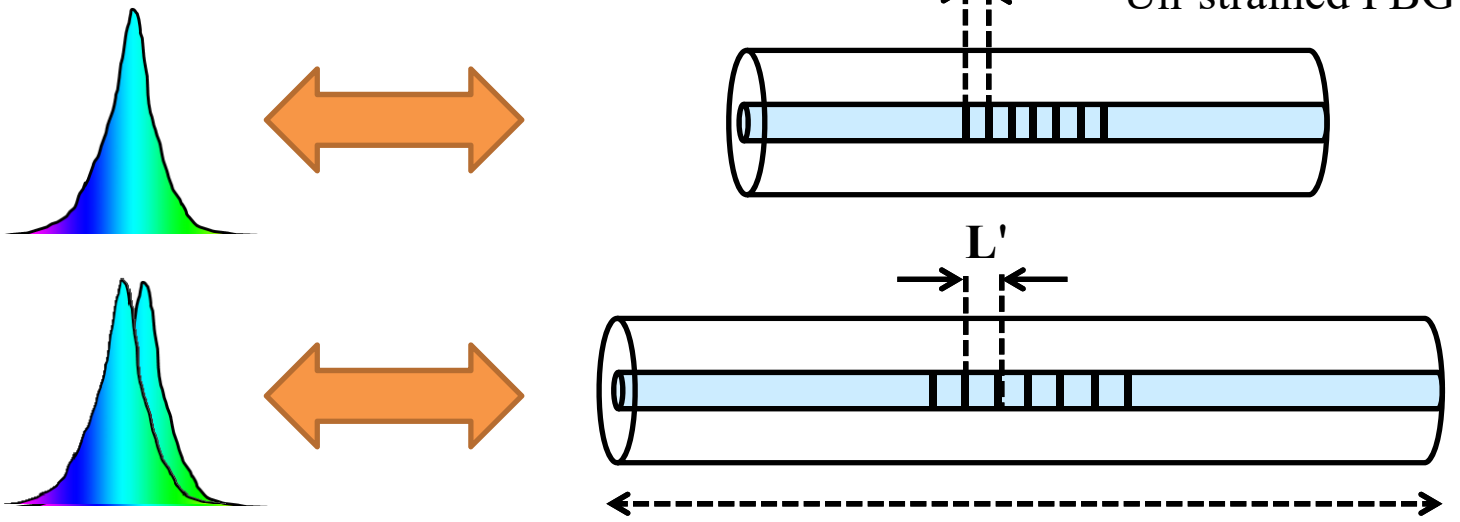
Principle of Fiber Bragg Grating: $\lambda = 2n_{eff}\Lambda$

Λ – grating period
n_{eff} – effective refractive index
λ – Bragg wavelength



Incident Spectrum Reflected Spectrum Output Spectrum

Strain Sensitivity (pm/με): 1.2
Temperature Sensitivity (pm/°C): 13



Development of RBSN Radome

- Internationally, a few manufacturers of radome (e.g., Raytheon and Ceradyne USA) use silicon nitride radomes for high speed missiles.
- RCI Hyderabad and CSIR have funded two large projects to CSIR-CGCRI for developing a technology for fabricating reaction bonded silicon nitride radomes.
- CSIR-CGCRI has successfully fabricated PRALAY radomes.

RADOMES FOR MISSILES

RCI, HYDERABAD (Rs. 11.3Cr.)

PoP mould for ASTRA size radome

Slip casting process

Dried radome

For ASTRA Air-to-Air Missile

Size:178 mm dia x 471 mm height

Application : Ku band transmission

- **Understanding of Silicon nitride ceramic for radome applications**
- **Extensive characterisation under progress**