

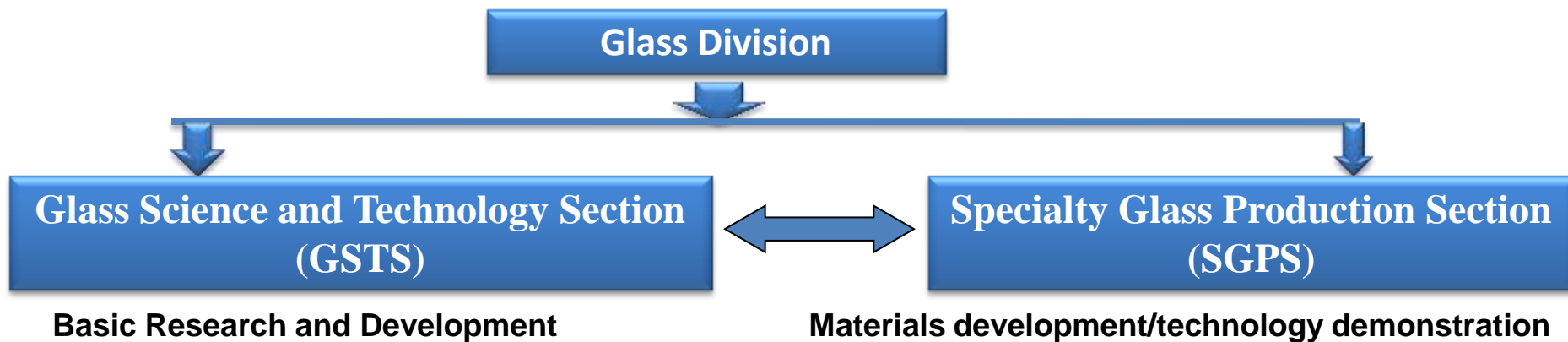


Specialty Glasses and Optical Fibers: CGCRI Perspective

Ranjan Sen

February 24, 2017

Glass Division (CSIR-CGCRI)



Objective/Mandate of the division:

- Development of specialty glass and glass-ceramics for engineering, civil and medical applications
- Basic research to explore new areas for futuristic applications: both product and process development.
- Indigenous technology development depending on user's specific requirement.
- Moderate to pilot scale production to meet country's demand.
- Teaching/ Training of students and researchers.



High Density Glass for Radiation Protection

- An essential item for Glove Boxes/ Cells used for nuclear material processing.
- Only 4 countries in the world are known to have the technology.
- Indigenous technology successfully demonstrated
- 20 MT of high density RSW glass blocks produced for DAE.

Applications

- Nuclear power plants
- Waste treatment facilities
- Isotope production facilities
- Nuclear research centers



Pouring of melt into pre - heated mould



Glass blocks of 400 × 400 × 100 mm sizes



Hot Cell RSW Glass

Management of Radioactive Waste

Development of strategies is the key component for safe, sustainable and broadly acceptable management of all types of radioactive waste

Main emphasis is given to :

- ▶ Waste volume minimization
- ▶ Recovery and recycle of valuables
- ▶ Ultimate goal of near zero release of radioactive wastes to the biosphere
- ▶ Isolation of radioactive waste from human environment for extended period of time

Special Glass Beads For Nuclear Waste Immobilization

The material ensures confinement of the radio isotopes in a stable matrix and safe disposal with no threat to the environment.

Process technology developed by CGCRI and transferred to Industry for manufacturing

Model: Production by Industry partner + Certification of the quality by CSIR-CGCRI

Till date 51.5 MT of glass beads produced and supplied to DAE

The technology is of significant importance for the country's nuclear power program.

NRDC Innovation Award -2013



Borosilicate glass nodules developed at CGCRI, Kolkata



Exchange of Technology Transfer document with M/s H R Johnson

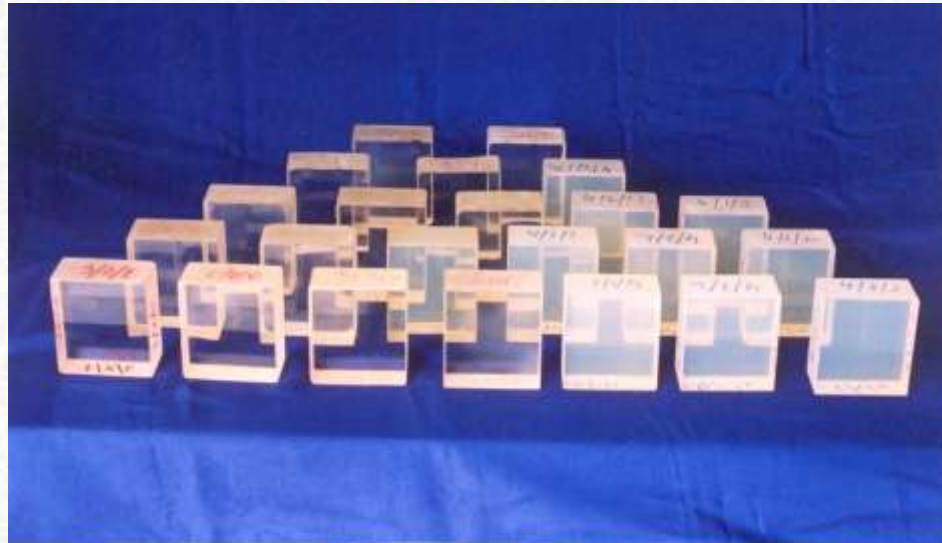


Glass Nodules produced by M/s H R Johnson

Ultra-low thermal Expansion Transparent (ULET) Glass-Ceramic

Material used for making laser gyroscope

**ULET GC has both vitreous and crystalline phases.
Coefficient of thermal expansion is $0.13 \times 10^{-6} \text{ K}^{-1}$ (0-50 °C)**



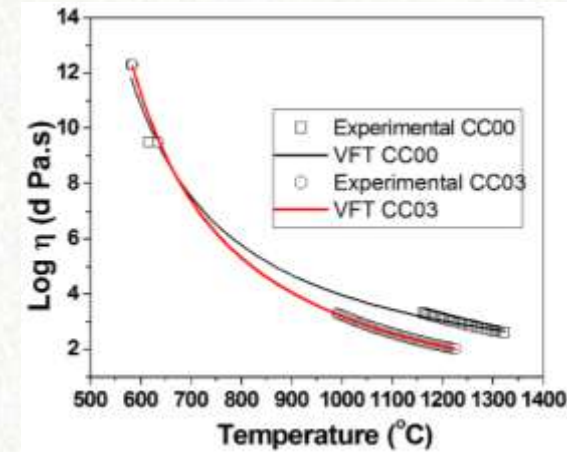
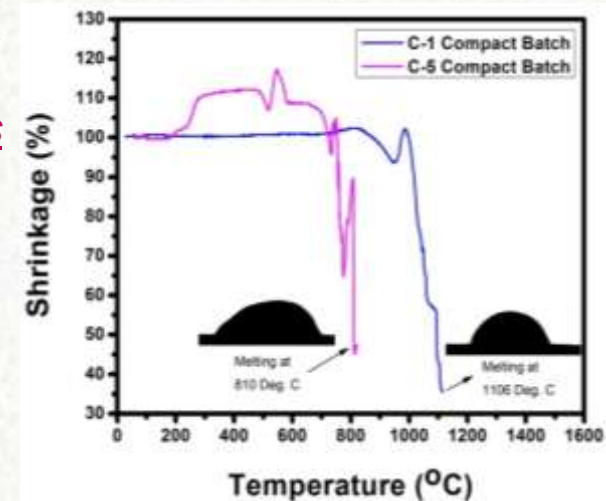
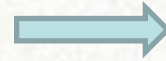
**ULET glass ceramic blocks of dimension $90 \times 90 \times 40 \text{ mm}^3$
produced at CGCRI**

INVESTIGATION ON THE INCORPORATION OF *BORAX PENTAHYDRATE* AND *COLEMANITE* IN DIFFERENT GLASSES

Feasibility study carried out on using borate minerals such as Borax pentahydrate ($\text{Na}_2\text{B}_4\text{O}_7 \cdot 5\text{H}_2\text{O}$) and Colemanite ($\text{CaB}_3\text{O}_4(\text{OH})_3 \cdot \text{H}_2\text{O}$) as the alternate sources for soda and calcia (lime) in glass melting and establishing process technology for two types of commercial glasses (soda lime silica based Float glass and Container glass) in the lab scale

Salient findings:

- Substantial reduction in melting temperature.
- Improvement in physical, optical, thermal and mechanical properties.
- Discussions are going on to take up industrial scale trial meltings



Patents filed:

- ENERGY EFFICIENT SODA LIME SILICATE GLASS COMPOSITIONS USING BORAX PENTAHYDRATE, PCT/IN2014/000027, WO/2014/128714
- NOVEL SODA LIME SILICATE GLASS COMPOSITION COMPRISING COLEMANITE AND A PROCESS FOR THE PREPARATION THEREOF, PCT/IN2013/000554, WO/2014/195960

Futuristic Work

- ✓ Development of new generation chalcogenide glasses for infrared optics (Night vision camera, Thermal imaging devices for automobiles and defence applications) and photonic devices
- ✓ Energy efficient glass preparation using Microwave radiation: Upscaling up to 500 g glass melting
- ✓ Development of glasses for fluorescence cooling
- ✓ Development of High-Efficiency Si-Photovoltaic Cell with Novel Glass/Glass-Ceramic Layers
- ✓ Development of Low phonon glass and glass-ceramics for MIR emissions

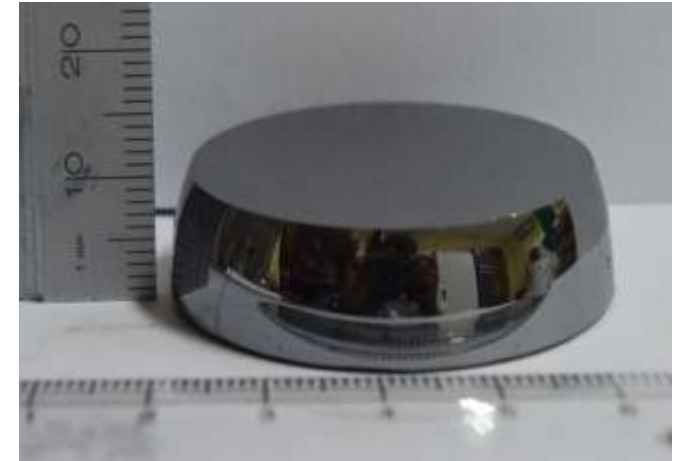
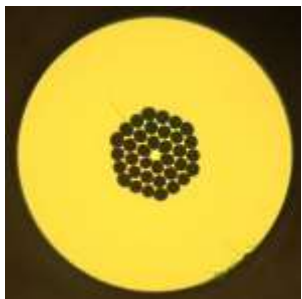
Chalcogenide Glass for IR Optics/ Photonic/ Sensor Applications

Glass developed in different compositions up to 200g. scale

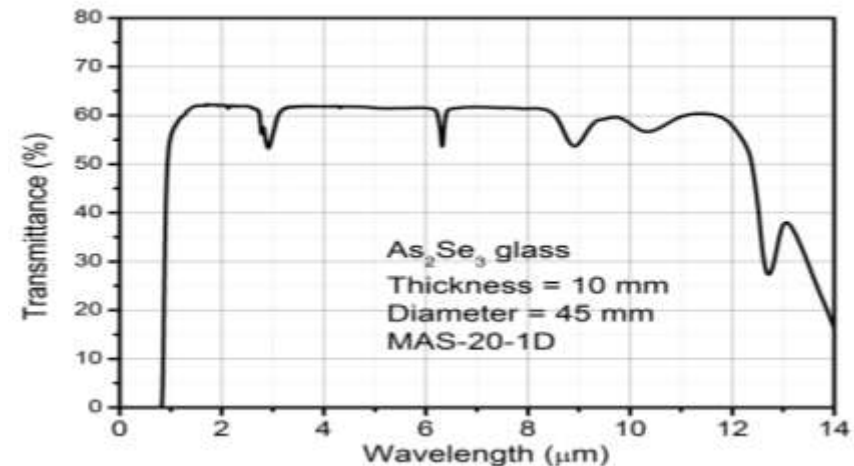
Application and Product development work initiated

Technology development for undoped and RE doped Chalcogenide Glasses

Applications:
Thermal Imaging
Supercontinuum Sources
Lasers/ Amplifier
Gas/ Chemical Sensors

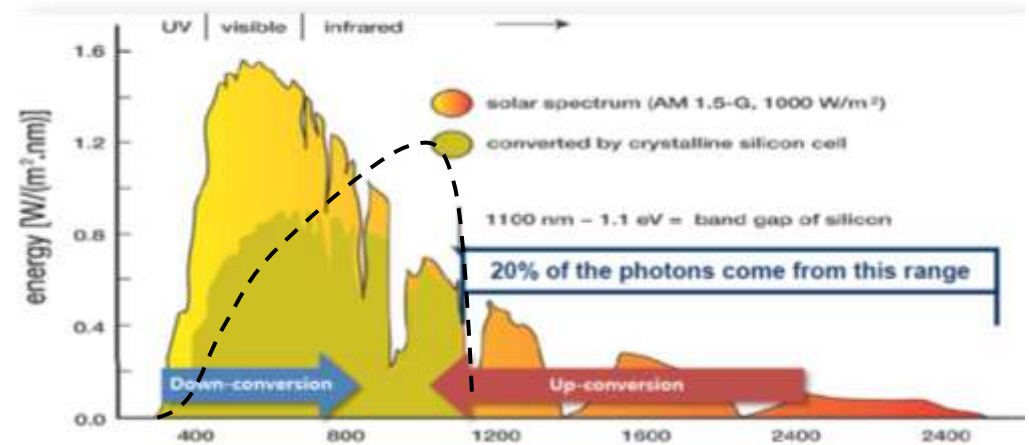
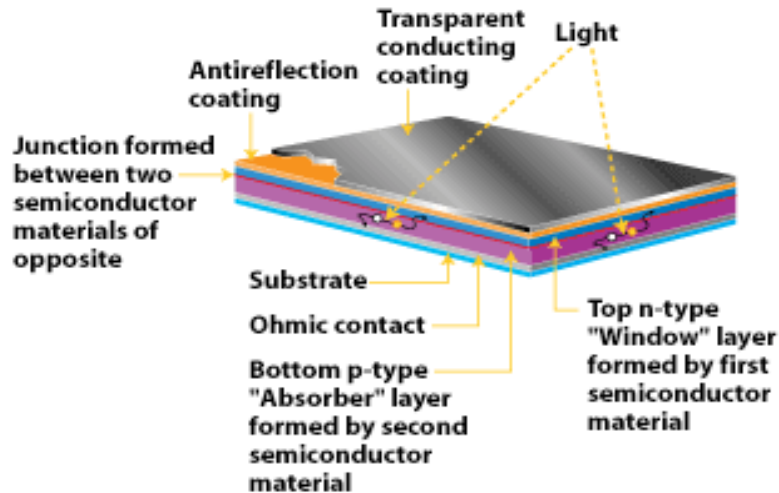


Processed bigger As_2Se_3 glass (10 mm thick & 45 mm dia)



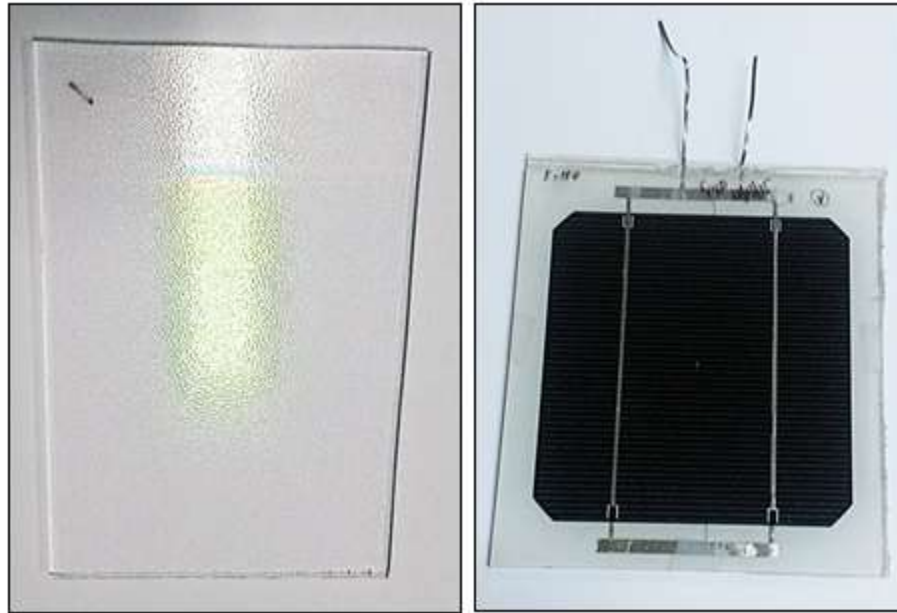
Transmission Spectrum

Development of High-Efficiency Si-Photovoltaic Cell with Novel Glass/Glass-Ceramic Layers



- Band gap energy of Si is constrained to 1.12 eV
- A large part of the incident solar spectrum remains unutilized as photons with energy equal to or greater than the band gap can only produce photocurrent
- However, excess energy leads to cell heating and causes thermalization losses
- Low energy photons (sub-band gap) gives rise to transmission losses
- Solar spectrum conversion by upconversion (UC), down-conversion (DC) and down shifting (DS) at appropriate wavelengths can lead to about 60-70% efficiency enhancement
- Rare earth doped glasses and nanometal glass nanocomposites are advantageous for such applications

AR coating on Solar Cell Cover Glasses with hydrophobic surfaces



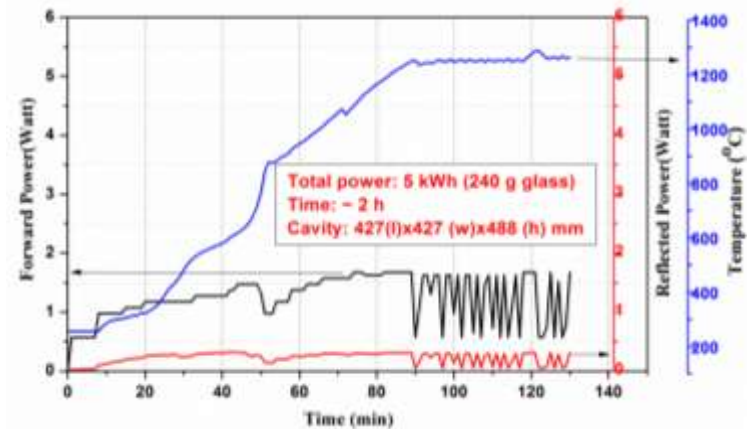
Salient features:

- **Single layer AR coating with mesoporous structure**
- **Pencil Hardness >5-6H (ASTM D 3363)**
- **Passed Adhesion test (ASTM D 3359)**
- **Reflection decreases 4-5% in the wavelength range 380-1000 nm in comparison to the uncoated solar cover glass**
- **Photo-current (I_{sc}) increased 3-4% in compare to the uncoated solar cover**
- **After application of thin hydrophobic coating water contact angle can be achieved up to 125° with contact angle hysteresis $10 \pm 2^\circ$**

Alternate Glass melting using Microwave heating

Advantage of Microwave heating:

- ❖ Shorter processing times ; Time saving (60-70 %)
- ❖ Energy saving (50-60 %) ; Eco-friendly
- ❖ Improved properties.
- ❖ Less evaporation, less contamination from crucible wall, improved chemical durability
- ❖ Development of new properties; Higher Fe-redox ratio/ retention of more ferrous ion in glass.



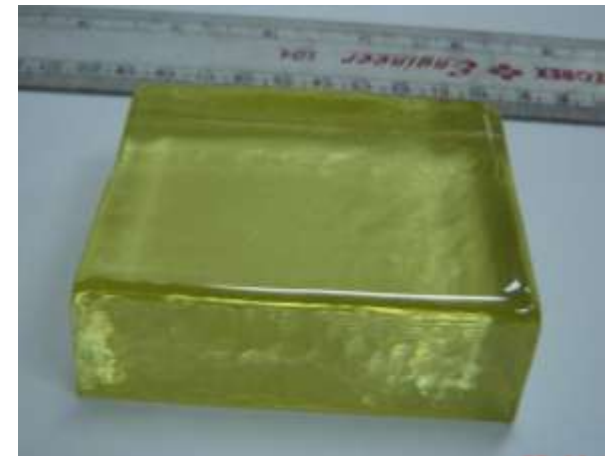
240 g RSW Glass:
Power: 5-6 kWh; Time: 2 h



Raising hearth furnace

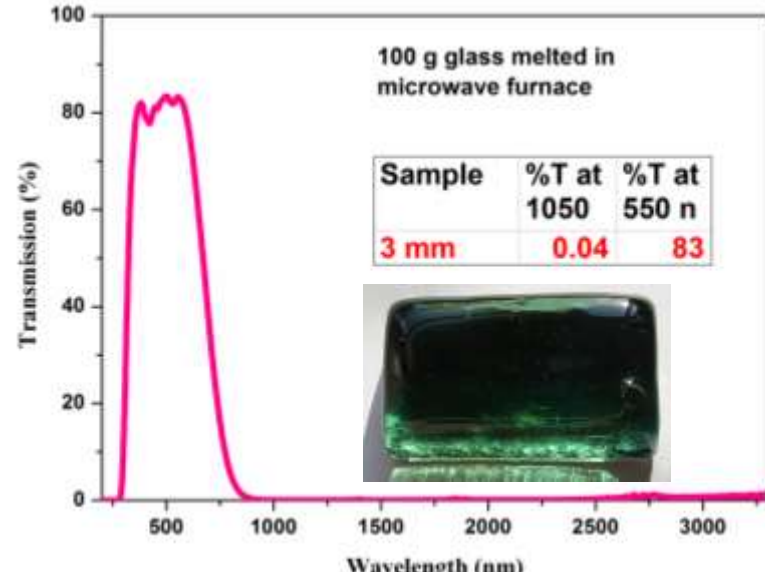
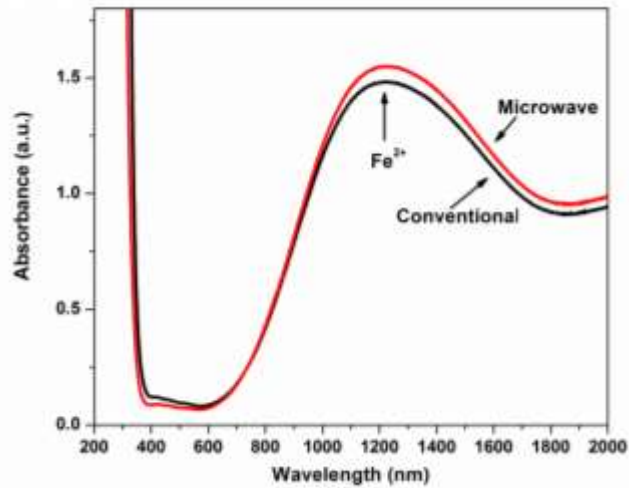


Microwave heating



500 g RSW Glass

Microwave heating yield Higher Fe[II] in glass



Patent Filed:

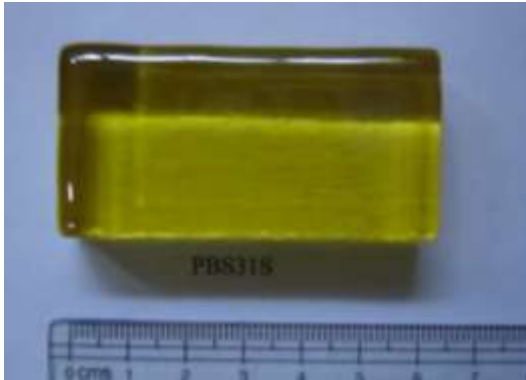
Indian Patent :0022NF2016 ; Process for preparation of iron-doped aluminophosphate glass using microwave energy in air atmosphere for heat absorbing application. Date of filing : 16-03-2016



Manufactured in the U

IR Protective goggles Can be made by this glass

Preparation of Bulk Glasses



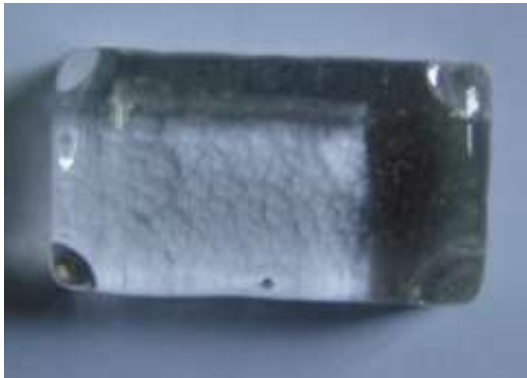
RSW Glass @1250°C



**Zinc Borate glass
@ 1200 ° C**



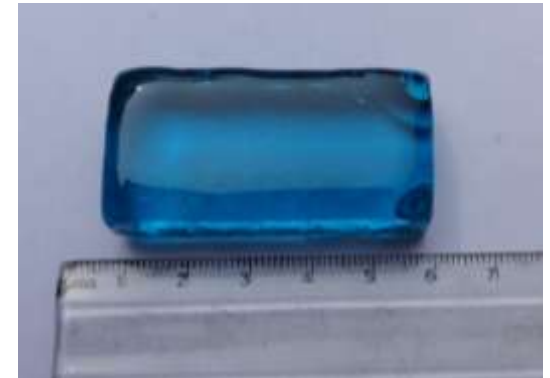
**Cu-doped phosphate glass
@ 1200° C**



**Barium-boro-silicate glass
@1200° C**



**IR absorbing glass
@ 1450° C**



**Phosphate Glass
@ 1250°C**

Melting Facility



Melting Furnaces (Raising Hearth) : 5 Nos. (Maximum 1400 °C)
2 No. (Maximum 1500 °C)
2 No (Maximum 1700 °C)

Induction furnace: 2 No. (1400-1450 °C) (5 lit and 40 lit. capacity)

High Temperature Observation furnace: 1 No. (Maximum 1600 °C)

Annealing/ Ceramization Furnaces : 8 Nos.

Microwave Melting Furnace: 3 No.

Specialized Furnaces



5 lit Induction furnace



6 kW Microwave Furnace



High temperature observation furnace



Rocking furnace

Characterization

➤ **Thermal Properties:**

Simultaneous Thermal Analysis (DSC, DTA, TGA) (STA 449, Jupiter)
Dilatometer (Netzsch DIL402PC)

➤ **Spectroscopic Characterization:**

Fluorescence spectrophotometer (Quantum Master-enhanced NIR
from Photon Technologies International)

FTIR spectrometer (Perkin Elmer, IRL 1280119, Frontier)

Raman Spectrometer (HORIBA JobinYvon, France; Model: Lab Ram
HR 800 EV)

➤ **Optical Properties:**

Prism Coupler (Metricon Model-2010),

UV–Vis-NIR spectrophotometer (LAMDA 950, Perkin Elmer),

Polarimeter (M/s Strainoptics Inc., USA, Model :DIAS-1600)

Interferometer (Zygo corporation, USA, Model: GPI system-XP/D)

➤ **Mechanical Properties:** Microhardness Tester (Mastuzawa, MMT-X7B)

Characterization Facilities



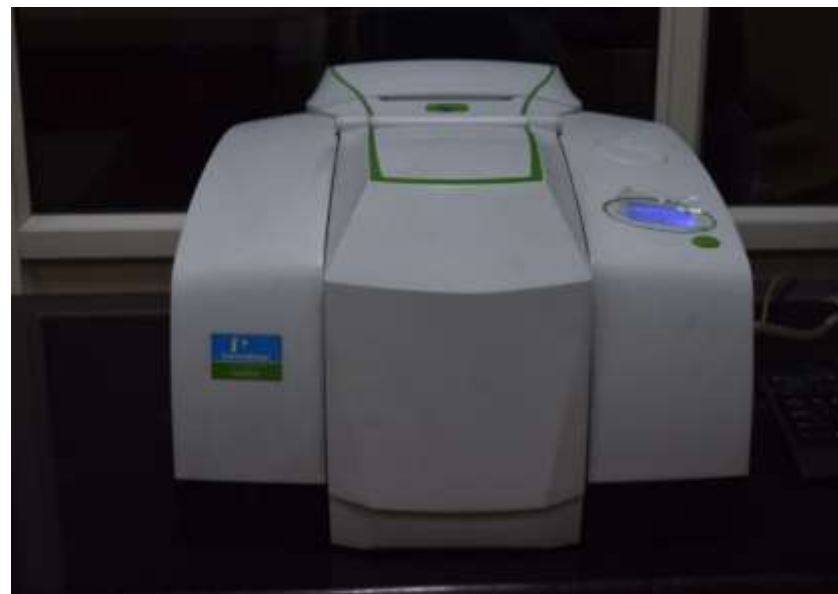
Raman Spectrometer (HORIBA, Lab Ram HR 800 EV)



STA (DSC, DTA, TGA) (NETZSCH, Jupiter)



UV-Vis-NIR Fluorescence Spectrophotometer
(M/s Photon Technology International)



FTIR Spectrometer (Perkin Elmer,
Frontier FT-IR/FIR Spectrometer)

Services towards Testings of Glass/Glass products

➤ Optical properties:

Refractive Index and Abbe number
Dispersion curve
Visible Light transmission (VLT)
Reflectance
Absorption
Yellow Index etc

➤ Mechanical Properties

Micro Hardness
Residual stress/toughened glass test

➤ Total chemical analysis

➤ Thermal Properties:

Coefficient of thermal expansion (CTE)
sagging temperature
DSC/DTA
Thermal shock resistance

➤ Optical Homogeneity

➤ Identification of types of glasses

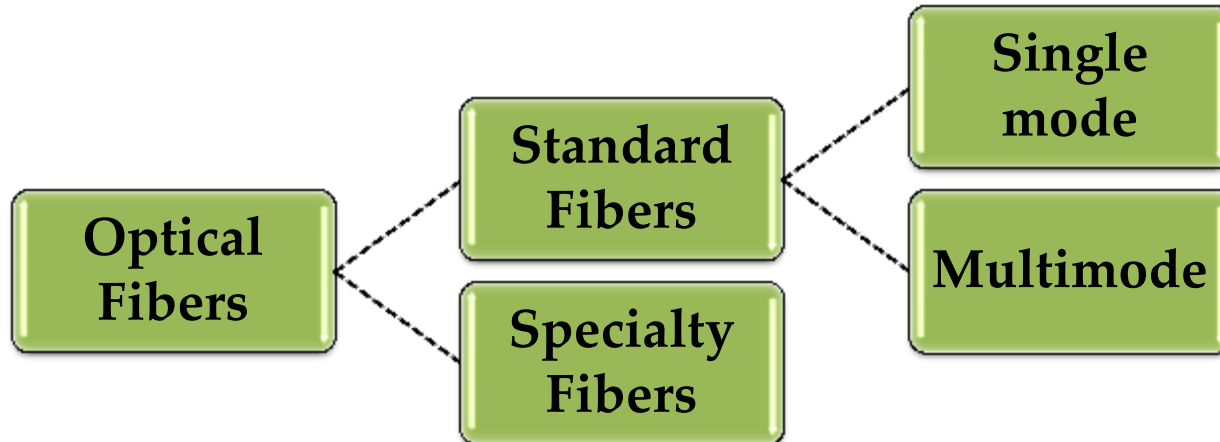
Laboratory scale trail melting; specific problem solving tests

Initiative taken for establishing facility for :

- Energy Performance testing of Architectural Glass and DGU.
- Safety and Structural Performance testing of Architectural Glass

SPECIALTY OPTICAL FIBRE

Types of Optical Fibers

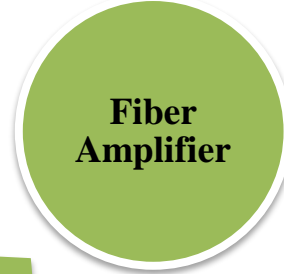
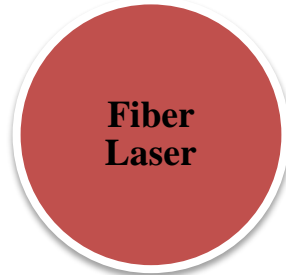
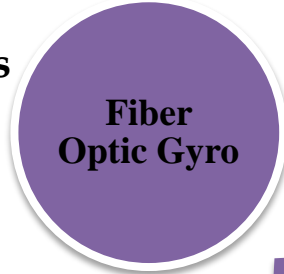


- **Standard Fibers are used in Telecommunication**
 - Produced in millions of Km

- **Specialty Fibers**
 - Vary in design, composition & functionality from standard fibers
 - Required in hundreds of Km
 - Price 100 to 1000 times greater than SM fiber

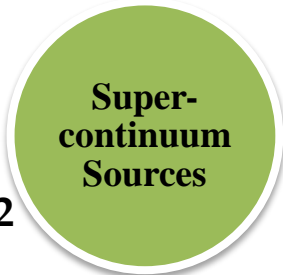
Yb or Tm doped fiber
Core: (Low Al) Yb/Tm+Al/P+SiO₂; Clad: SiO₂

Polarization preserving fiber
Core: GeO₂+SiO₂;
Clad: B₂O₃+SiO₂ stress



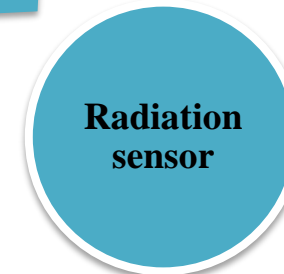
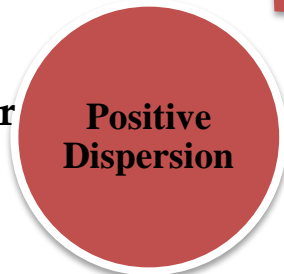
Er doped fiber
Core: (High Al)
Er+Al/Ge+SiO₂;
Clad: SiO₂

Microstructured optical fiber
Core: SiO₂;
Clad: Air & SiO₂



Photosensitive fiber
Core: (High Ge)
GeO₂+SiO₂;
Clad: SiO₂

Dispersion compensating fiber
Core: (High Ge)
GeO₂+SiO₂;
Clad: SiO₂

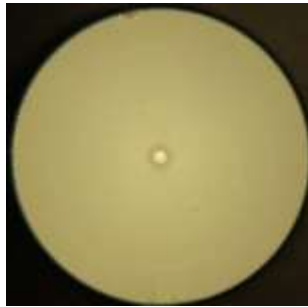


Radiation sensitive fiber
Core: (High P)
P+Ge+SiO₂;
Clad: SiO₂

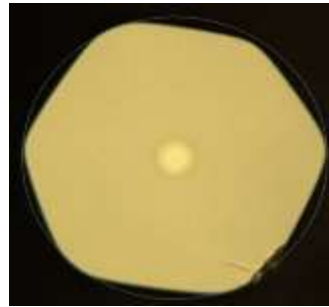
Radiation resistant fiber
Core: SiO₂; Clad: F+SiO₂



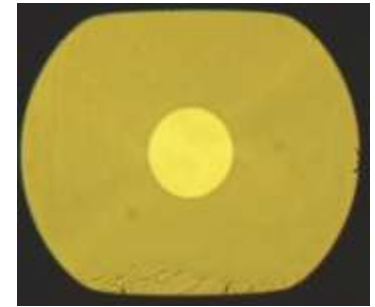
Cross Sectional View of Different Specialty Fiber



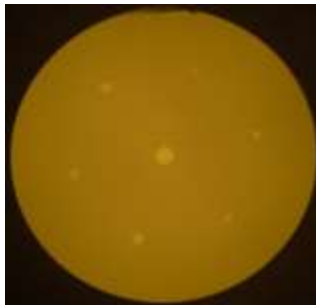
Er/Yb/Tm (RE) fiber



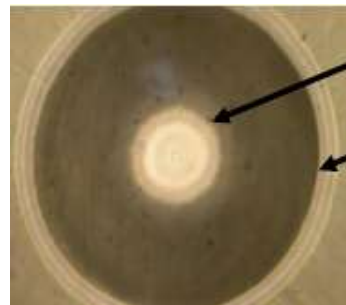
Hexagonal DC RE- fiber



D-shaped DC RE-Fiber



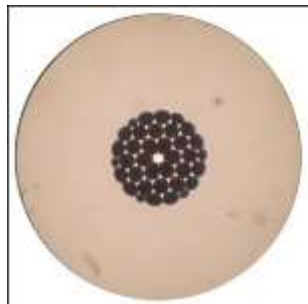
Multi-core Fiber



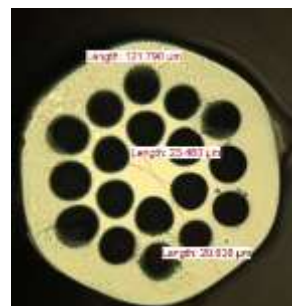
Ring core Er-fiber



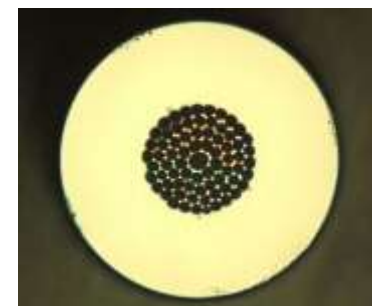
Air-clad RE-fiber



Non-linear PCF

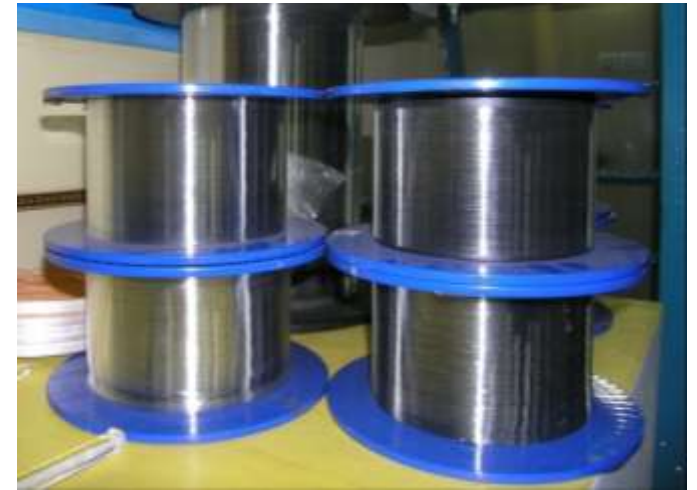
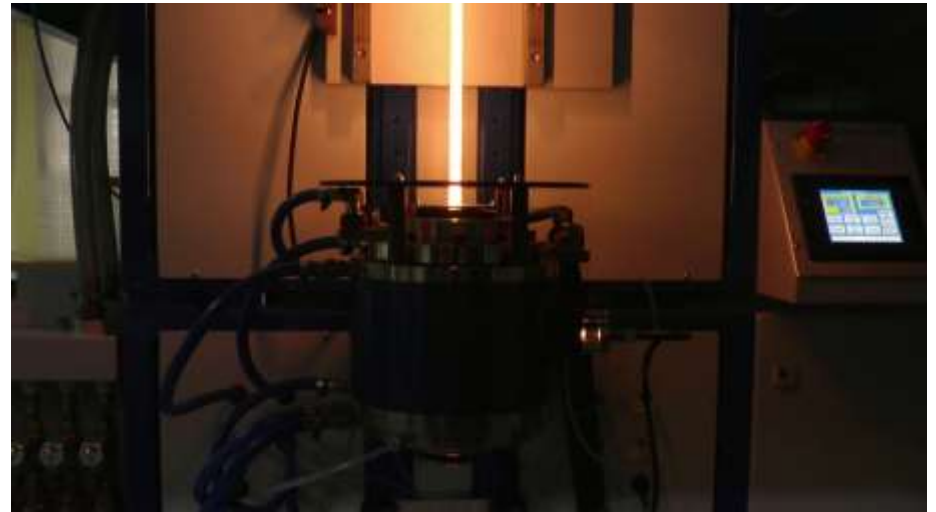
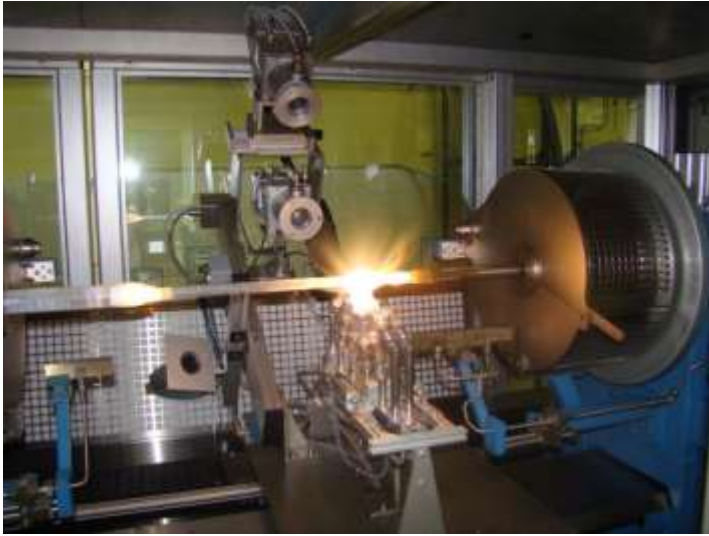


Leakage Channel Fiber



Hollow core PCF

Preform Fabrication to Fiber Drawing



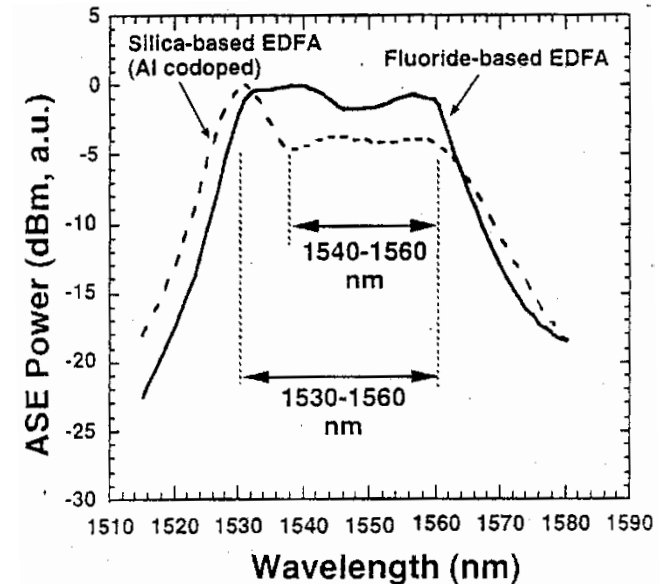
Erbium Doped Fiber Amplifier (EDFA)

➤ Key component behind high speed internet service. EDFAs can provide high gain over bandwidth as wide as 80 nm and output power as high as +37 dBm.

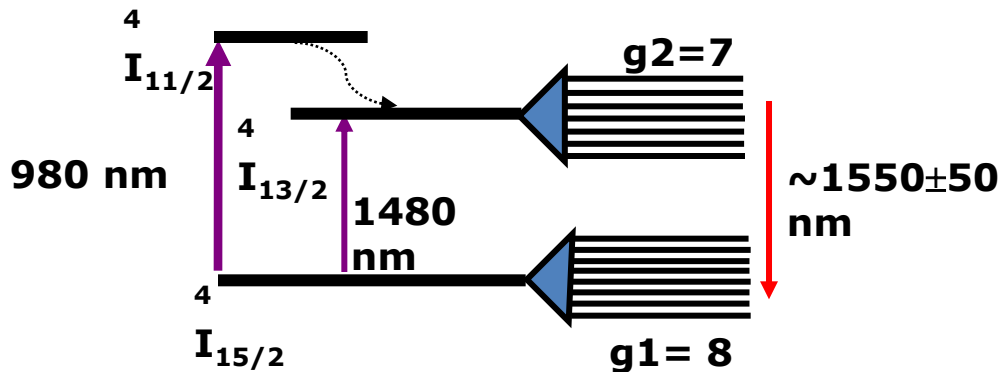
➤ The global optical amplifier market in 2012 was at \$900 million which is forecasted to reach \$2.8 billion by 2019.

Advantage of EDFA:

- Commercially available in C-band & L-band
- High gain and low noise: 4.5 dB – 6 dB
- Flat gain can be achieved by Al co-doping
- Independent of bit rate
- Simultaneous amplification of WDM signals

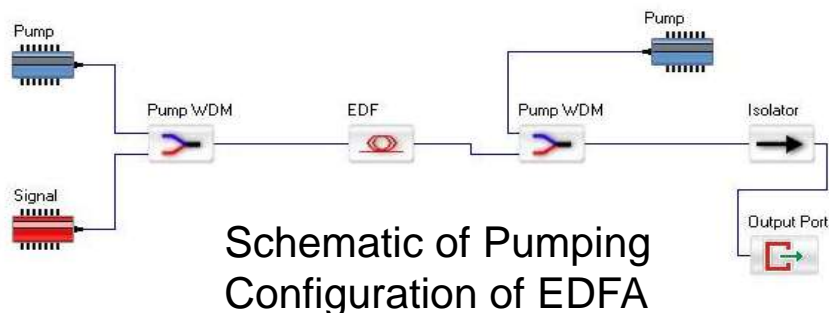


Energy levels of Erbium in Silica

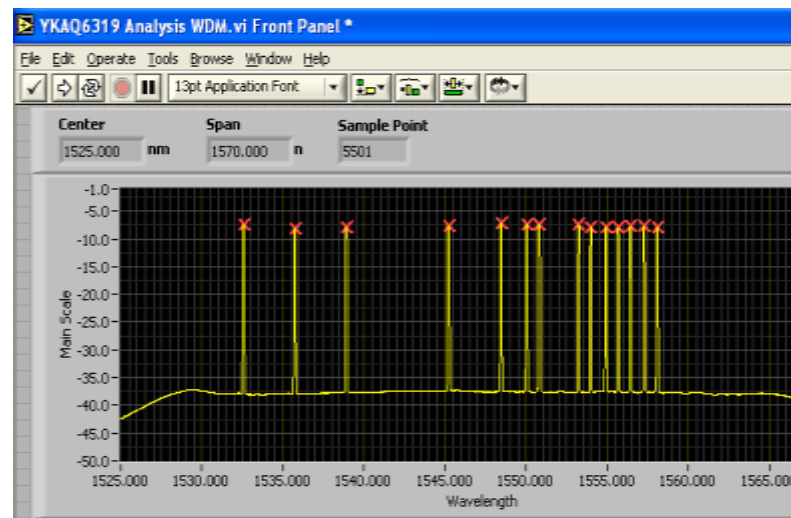


- Single channel and multi channel Amplifier for optical communication
- Fiber laser at 1550 nm

EDFA Modules for CATV & Optical Communication (Industry Partner: NeST Photonics)



Parameters	EDFA for CATV	EDFA for Telecom
Operating Wavelength (nm)	1540-1560	1530 – 1565
No. of Channels	1	16, 32, 40
Input Signal	-3 to +3 dBm	-20 to -3 dBm
Output Signal	16-22 dBm	18-20 dBm
Gain Flatness	-----	< 1 dB
Noise Figure	< 6 dB	< 5.5 dB

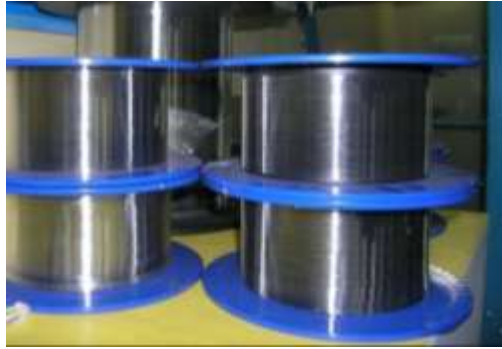


Gain-Flattened EDFA

Technology Award for most significant CSIR Technology of the Five Year Plan Period – 2012

Hundreds of EDFA modules have been sold in the CATV market in India & Abroad

MAKE IN INDIA: Erbium doped optical fiber amplifier



Successfully commercialized



- High Power Optical Amplifier (4-6 Watt) For Smart Cities
- Industry Partners: SFO Technologies, Kochi and Vinvish, Thiruvananthapuram



Optical Amplifier in production at NeST-Cochin (SFO Technologies)

Advantages of a Fiber Laser

- High stability
- Excellent beam quality
- Better efficiency
- Minimum thermal impairment
- Wide selectivity of operating wavelengths
- High power density
- Easy to use

Key Parameters of a Fiber Laser

- Operating Wavelength
- Operation Mode (CW or Pulse)
- Pumping Scheme
- Output average power
- Pulse width and peak power
- Pulse energy
- Beam quality (M^2)



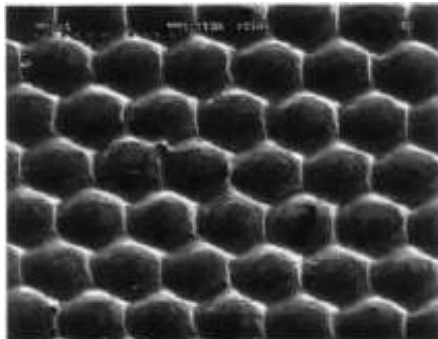
Stent Cutting



Cutting



Marking



Printing/gravure



Pacemakers



Welding

Indigenous technology of CW, QCW and Pulsed Fiber Laser at 1 μm and 2 μm

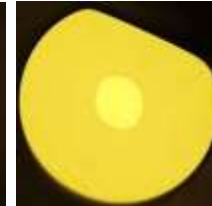
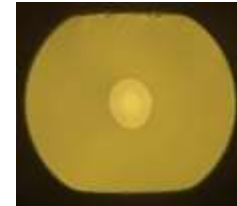
Development of prototype as well as commercialized laser modules with industrial partners



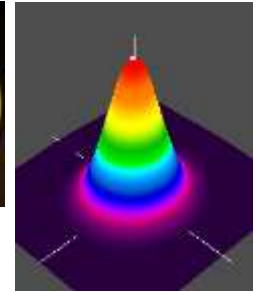
Laser Cut Angioplasty Stent with 100 W 1 μm Laser



Kidney Stone breaking with developed 2 μm laser



CSIR-CGCRI fabricated active fibers at both 1 μm and 2 μm



Laser Beam Profile

Application Areas: 1 μm fiber laser:

- ❑ Medical stent cutting, diamond processing with CW (continuous wave) laser with 20 – 100 W average power
- ❑ Marking, engraving on orthopaedic parts and solar cell scribing with pulsed fiber laser

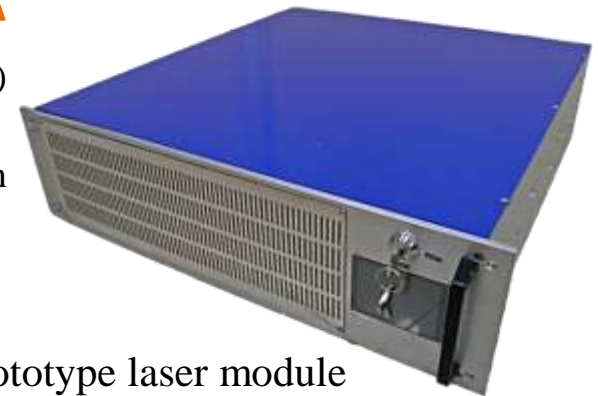
Application Areas: 2 μm fiber laser:

- ❑ Soft/Hard tissue surgery - limited adjacent tissue damage, rapid healing, minimal scarring and remote site accessibility.
- ❑ Lithotripsy irrespective of size and composition of the stone

Future Endeavors:

- ❑ Commercial laser modules
- ❑ Field trials
- ❑ High power fiber lasers beyond 1 kW for strategic sectors

MAKE IN INDIA



Prototype laser module

Achievements:

- 100 W CW Fiber laser at 1 μm
- Prototype demonstration of Pulsed fiber laser at 1 μm with 20W average power and 1 mJ pulse energy
- 10 W CW and 104 mJ QCW fiber laser at 2 μm
- Successful fragmentation of COM stone of size > 2 cm

Fiber Bragg Grating Sensors for Structural Health Monitoring

(Strain and Temperature sensors)

FBG sensors have application in many fields: Energy, Civil engineering sectors, Aerospace, Oil & Gas Industries and others

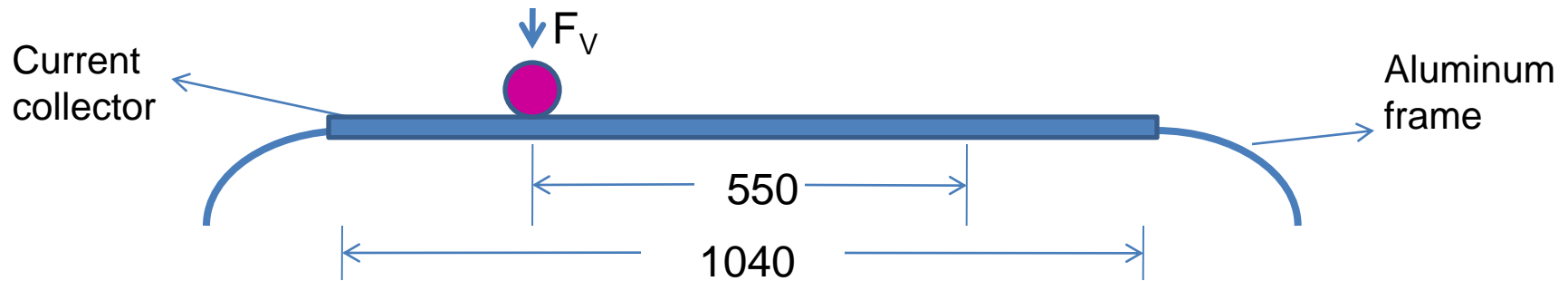
EMI/RFI immunity, High multiplexing capability, No electrical signal/ explosive safe, Tiny sensors embeddable in structures

CGCRI has developed the complete technology starting from fiber fabrication to sensor development, packaging and instrumentation



Some sensors and instruments developed

Smart /Instrumented Pantograph



**Real time condition monitoring of Catenary and Pantograph.
Measurement of:**

- **contact force,**
- **contact position,**
- **impact oscillations between the pantograph and the overhead conductor**

Applications: In Railways



Instrumented Pantograph under test at the facility of Industrial Partner at Kolkata

Step Forward

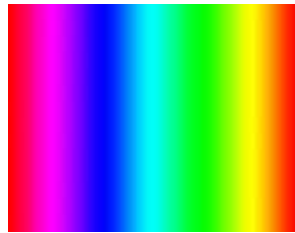
CGCRI looks forward to work jointly with interested industrial partners for indigenous technology development and manufacturing specialty products towards country's capability building in the areas of:

- **Specialty Glasses**
- **Specialty Optical Fibers**
- **Novel and futuristic technologies**
- **Innovative products**

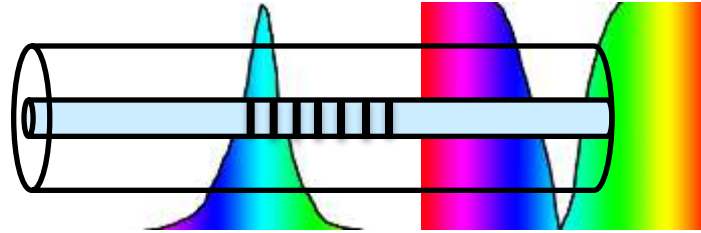
Thank You

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

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



Incident Spectrum

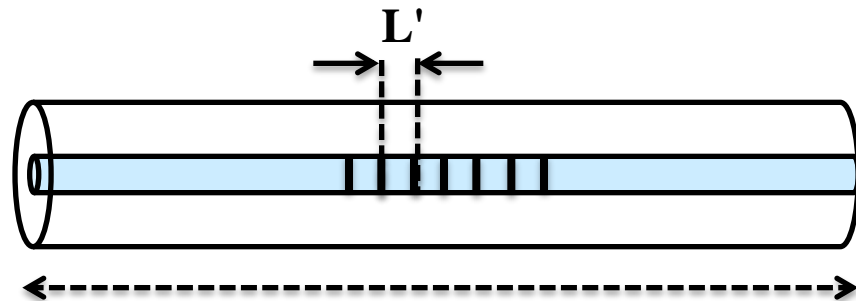
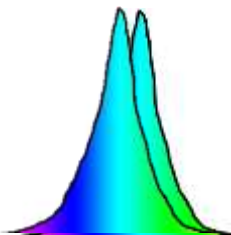
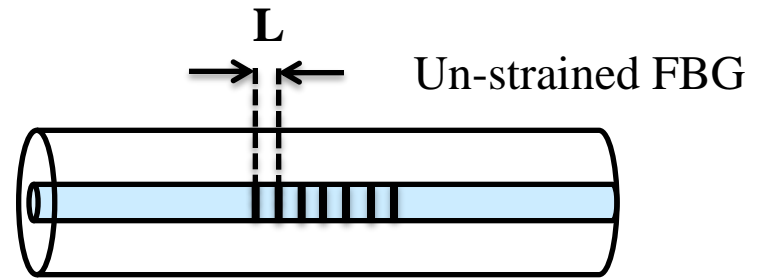
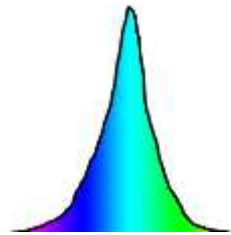


Reflected Spectrum

Output Spectrum

Strain Sensitivity (pm/ $\mu\epsilon$): 1.2

Temperature Sensitivity (pm/ $^{\circ}\text{C}$): 13



Strained FBG