Cryogenic Research and Technology at IISc

Prof. Subhash Jacob

Centre for Cryogenic Technology
Indian Institute of Science, Bangalore, India.
Facility at Centre for Cryogenic Technology (CCT)

- Liquid nitrogen plants
  - StirLIN4: 50 l/hr
  - PLN 430S with PSA system: 55 l/hr
Liquid helium plant

- KOCH 1410 liquefier: 17 l/hr (with 2 helium compressors)
- Production rate: 25 l/hr (with 3 helium compressors)

CCT produces around 4,00,000 liters of liquid nitrogen and 30,000 liters of liquid helium per annum for the users in the Institute and is the largest in producing cryogens among the academic sector institutions in the country.

CCT also supplies cryogens to various R&D and medical doctors and institutions in and around Bangalore.
CCT has machine shop, TIG and micro plasma welding machines, high vacuum systems and helium mass spectrometer leak detectors for most of the in-house fabrication and testing.
CCT has successfully completed many R&D projects

220 Liters LOX dispenser, HAL

Rotating Helium transfer coupling for Superconducting generator, BHEL
Liquid Cryogen Level Sensor, ISRO

CCD Cryostat, IIA
Current Research projects

R & D Projects

- Enhancement of vortex tube LOX separator technology for HTV
- Design and development of novel optical coolers
- Pulse tube cryocoolers
- Cryogenic heat pipes
- Liquid helium FRP cryostat for operation of SQUIDs for NDT studies
- Cryogenic treatment of metals
- SQUID detection of low frequency NMR/NQR

Consultancy Projects

- Calibration of temperature sensors for cryogenic upper stage project, ISRO
- Advice on cryomilling system, M/s KAPS Engineers, Baroda
Enhancement of vortex tube LOX separator for HTV

- The project aims at enhancement of vortex tube LOX (liquid oxygen) separation technology to achieve high purity (≈ 98%) with high separation efficiency (≈ 63.5%), required parameters for Hypersonic Test Vehicle.
CFD modeling of vortex tube

- The first step in CFD modeling - generate sufficient confidence level in modeling of the vortex tube for single phase flow and achieving maximum temperature separation.
- The numerical modeling of the vortex tube carried out with the code system of Star-CD.
- As the Reynolds number is very high, ‘Renormalization Group’ (RNG) version of k-ε model has been used.
- Analysis carried out to evaluate:
  - Optimum number of nozzles
  - Nozzle profile
  - Cold end diameter (d_c)
  - Length to diameter ratio (L/D)
  - Cold and hot gas fraction

3-d model and Temperature distribution of vortex tube
Design and fabrication of vortex tubes based on CFD studies.

- Based on CFD analysis and available compressor throughput, 12 mm diameter straight and conical vortex tubes (divergence angle of 2.5° towards the hot end) have been fabricated.
- The assembly has the provision of varying cold end orifice diameter, L/D ratio and hot end outlet area for various investigations.

Exploded View of Straight Vortex Tube Assembly

Exploded and sectional View of Conical Vortex Tube Assembly.
Photographic view of SS straight vortex tubes

Photographic view of SS and Al 2219 conical vortex tubes
Experimental setup

- Four numbers of turbine flow meters (Flow Technology Inc., USA) incorporated to measure the amount of fluid flowing through main inlet, cryostat, hot and cold end of the vortex tube.
- Data acquisition for the system using Lab VIEW and Field point.
- Teledyne purity monitor for LOX purity.
There is very small increase in oxygen purity until the inlet temperature reaches about 105K.

From 105K to about 102 K, there is a sharp increase in oxygen purity.

As the inlet temperature decreases further to ~ 98 K, the oxygen purity reaches a value close to 96%.

For achieving high LOX purity, Pressure at the inlet should be in the range of 6-7 bar (90-105 psi)
First stage experiments

Experimental results:

<table>
<thead>
<tr>
<th>LOX purity</th>
<th>Separation efficiency</th>
</tr>
</thead>
<tbody>
<tr>
<td>Target</td>
<td>46.87%</td>
</tr>
<tr>
<td>Achieved</td>
<td>48%</td>
</tr>
</tbody>
</table>

(For Conical vortex tube with L/D = 25, dc=7mm, inlet pr=87psig, inlet temp=102.3K)
Second stage experiments

- L/D=20, dc=7
- L/D=20, dc=7, 3 nozzles
- L/D=10, dc=7
- 6mm st. vortex tube
- Target

Graph showing separation efficiency (%) vs. oxygen purity (%) for different conditions.
Optical cryocoolers

- Solid state cryocoolers based on anti Stokes fluorescence is known as optical cryocooler

- Anti Stokes fluorescence is a phenomenon by which a substance excited by radiation at one wavelength will have fluorescent emission at a lower wavelength.

- This results in more energy being radiated than being absorbed for each photon, which results in cooling of the material.
Significance of Solid state Optical coolers

- The very nature of Solid State Optical coolers namely,
  - No vibrations
  - No moving parts
  - Compact and low mass
  - No/low EMI signatures
  - Capability to cool to 77 K
  - Maintenance free, long life operation,
  - Moderately good efficiency \( \approx 1\% \) (cooling power / DC power)
  - Requirement of only Diode Laser power to cool fluoride glass/crystals

- Makes it the most outstanding candidate for Military, Space & Research applications and represents a new approach to cryogenic refrigeration.
CCT is working for development of an optical cryocooler for cooling power of about 400mW at 80K in collaboration with Dept. of Physics and Material Research Centre at IISc (*Patent filed)

Fig 1: Optical cooler with thermal shield cooled by thermo-electric cooler
Optical Cooler Laboratory, Dept, of Physics, IISc.
(Mbraun LAB Master Glove Box)
Photo-thermal Deflection Spectroscopy

Photograph of the Photo-thermal Deflection Spectroscopy on Optical Top.
Thermal Lens Method

The Research Consultant, Prof. R. Srinivasan has proposed thermal lens method, which will give sensitive estimation of phase angle changes when a sample is getting heated or cooled by the pump beam.
Thermal Modelling of Optical Cryocooler
Temperature Distribution in Combined Model Counter 2 with No Load Condition

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<th>Tc</th>
<th>T1</th>
<th>T2</th>
<th>T3</th>
<th>T4</th>
<th>T5</th>
<th>T6</th>
<th>T7</th>
<th>T8</th>
<th>T9</th>
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<td>308.07</td>
<td>325.21</td>
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<td>300*</td>
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<td>301.89</td>
<td>301.55</td>
<td>301.54</td>
<td>301.97</td>
<td>300.94</td>
<td>301.14</td>
<td>310.14</td>
<td>310.14</td>
<td>308.87</td>
<td>327.11</td>
<td>300.94</td>
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</table>
CCT has developed two types of PTR

- **Single stage PTR - GM type, Cam operated valve**
  - Novel cam operated pressure wave generator have been developed
  - Producing a refrigeration power of about 7W at 77K and a no load temperature of ≈ 40K
  - Uses an air-cooled compressor of 1.6 kW
Warm end heat exchanger cooled with thermo-electric cooler.

Figure 2. TEC arrangement at warm end heat exchanger
Single stage PTR – GM type, Rotary valve

- Indigenous rotary valve have been developed
- Producing a refrigeration power of 7.3W at 77K and a no load temperature of $\approx 37.5K$
- Uses a water-cooled compressor of 3 kW
Two stage Pulse Tube Cryocooler

Photo of the system

Schematic
Typical Cool Down

COOLDOWN BEHAVIOUR OF
TWO STAGE PULSE TUBE REFRIGERATOR

TIME (min)

TEMPERATURE (K)

1st STAGE
2nd STAGE

64.2K
3.5K

P16_12_5
Typical Heat Load Characteristics
Numerical simulation of pulse tube refrigerator with inertance tube

- The governing equations (continuity, momentum, energy and the equation of state) are set up for the individual components.
- One-dimensional form of the time-dependent equations are employed.
- Equations are discretised using second-order upwind differencing for the convective terms.
- Regenerator matrix energy equation is to be additionally considered for regenerator.
- The formulation consists of other algebraic equations for quantities such as the properties of the gas and matrix, friction factor and the Nusselt number.
Variations of the matrix and the helium gas temperatures over one cycle
Loop Heat Pipe (LHP)

- Two phase heat transport system
- Capillary pressure in the evaporator wick is used to circulate the working fluid
- Built-in diode action
- Adverse tilt has no effect on the performance
- Wick only in the evaporator
- Easy start-up and self priming
Cryogenic Loop Heat Pipe (CLHP)
Liquid helium cryostat for operation of SQUIDs for NDT studies

- SQUID magnetometer/Gradiometer probes are very sensitive magnetic flux detectors and are useful for measuring extremely small magnetic fields.

- SQUIDS are especially unique for detecting surface flaws in Non Destructive Test (NDT) in which signals of the order of 1 nT in unscreened laboratories has to be detected. It is difficult to achieve satisfactory spatial resolution at such level without using SQUID.

- CCT has developed an indigenous FRP (non metallic and non magnetic) LHe cryostat of about 1 mm (standard is in the range of 2-5mm) spatial resolution at the pick-up coil. The holding time of this cryostat is about a day.
The salient features of the cryostat are:

- Made of FRP except for the top part of the cryostat.
- Two radiation shields made of carbon fabric blanket.
- Distance between the pick up coil and the DUT is 6 mm.
- We have used PTFE coating at the outer surface of the inner vessel and inner surface of the outer vessel to reduce the helium diffusion and outgassing.
Cryotreatment of metals

Salient features of the Cryotreatment system

- No direct contact of liquid Nitrogen with specimens.
- Efficient transfer of cold to specimens.
- Capacity to Cryotreat large no of samples.
- Option of various cooling / soak / heating rates.
- Easy movement of the unit.
Objectives of cryotreatment studies

- Wear studies of HSS tools (M42)
- Study of dimensional stability and residual stresses of diaphragm materials (APX4) of Integral Diaphragm Pressure Transducers developed LPSC, ISRO.
Cryotreatment unit connected with LN2 dewar
Cryotreatment effects on wear properties of tool

<table>
<thead>
<tr>
<th></th>
<th>Untreated Tool</th>
<th>Cryotreated Tool</th>
<th>Percentage Improvement</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Wear (microns)</strong></td>
<td>88</td>
<td>59</td>
<td>33 %</td>
</tr>
<tr>
<td><strong>Frictional Force (Newton)</strong></td>
<td>10.5</td>
<td>7.4</td>
<td>30 %</td>
</tr>
</tbody>
</table>

Tool: Molybdenum based HSS Tool (M42)

Wear tests: Standard pin on disc wear testing unit

Test procedure: as per ASTM standard designation G99-95
# Effect of cryotreatment on APX4

<table>
<thead>
<tr>
<th>Sl No</th>
<th>Test</th>
<th>Raw Material</th>
<th>Cryotreated</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Yield Strength</td>
<td>523 MPa</td>
<td>525 MPa</td>
</tr>
<tr>
<td>2</td>
<td>Ultimate Tensile strength</td>
<td>1003 MPa</td>
<td>1011 MPa</td>
</tr>
<tr>
<td>3</td>
<td>% Elongation</td>
<td>19.64 %</td>
<td>24.39 %</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Sl No</th>
<th>Test</th>
<th>Raw Material</th>
<th>Cryotreated</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Hardness (BHN)</td>
<td>292</td>
<td>300</td>
</tr>
<tr>
<td>2</td>
<td>Residual stresses</td>
<td>-535 MPa</td>
<td>-372 MPa</td>
</tr>
<tr>
<td>3</td>
<td>Retained Austenite</td>
<td>3 - 4.1 %</td>
<td>1.9 – 2.2 %</td>
</tr>
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</table>
SQUID detection of low frequency NMR / NQR

- SQUIDs can measure extremely small magnetic field (in femtoTesla) and is being used in many fields such as geophysics, biomagnetism, low frequency NMR, NQR etc.

- It is important to study the magnetization as a function of applied magnetic field and temperature for SQUID magnetometers

- CCT is developing a low frequency NMR / NQR spectrometer with DC SQUIDs (developed by IGCAR, Kalpakam) as detectors for detection of explosives and non-destructive testing and evaluation, etc.

- The project involves the development of the cryogenic probe with the NMR / NQR transmitter and pick up coils, SQUIDs coupling and flux locked loop electronics, etc. along with the available cryostat and accessories

- The second part involve the development of a full-fledged stand-alone SQUID based low frequency NMR / NQR spectrometer
Liquid helium cryostat design of magnetic shield

Cryostat & top flange

Mu Metal Shield

Lead Shield

CryoPerm & Lead Shield
Cryoprobe design for SQUID NMR/NQR
NQR Signals

- Strongest peak at 39-40kHz
- 128 averages with a rate of 1Hz
- Broader signal, may be due to impurity in the sample (reported T2* ~ 1 ms)

FFT of FID following a 51.77 µS pulse

FID and its power spectrum following a RF pulse of width 51.77 µs
Calibration of temperature sensors for cryogenic upper stage project

- CCT has a temperature sensors calibration facility, being utilized for calibration of temperature sensors, required for various organizations. The most important among them is the calibration of temperature sensors for cryogenic upper stage project of ISRO, Mahendragiri

- Calibration temperature range: 4.2K – 300K
Advice on cryomilling systems

- The latest modern technology in milling is cryomilling/cryogrinding, which is becoming quite popular and produces finer and superior ground product in comparison with conventional ground products.
- Cryogrinding is a process in which the material to be ground is cooled to a desired temperature by means of a cryogenic fluid such as liquid nitrogen and ground in a low temperature compatible mill.
- Some of the significant advantages of cryogrinding over conventional grinding are:
  - no chemical degradation of the product
  - finer particle size
  - higher throughput
  - lower energy consumption
  - no clogging and gumming of the mill
  - possibility to grind difficult materials
- CCT has developed a pilot plant for cryogrinding of spices and a pilot plant for cryogrinding of plastics/PVC scrap. (*Patent filed)
The pilot plant for PVC scraps has been developed for a Private company in Bangalore with a throughput of ~ 500 kg per 8 hrs and about 85-90% of the ground product is less than 212 microns.

Throughput and fineness

- Throughput ≈ 60 kg/hr for PVC scrap (at -90°C).
- Throughput ≈ 35 kg/hr for polypropylene (at -130°C).

<table>
<thead>
<tr>
<th>Particle size (µm)</th>
<th>Residual weight (%) (PVC powder)</th>
<th>Residual weight (%) (Polypropylene powder)</th>
</tr>
</thead>
<tbody>
<tr>
<td>&gt;1000</td>
<td>0.0</td>
<td>0.5</td>
</tr>
<tr>
<td>850 –1000</td>
<td>0.4</td>
<td>7.3</td>
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<tr>
<td>500 – 850</td>
<td>3.3</td>
<td>31.5</td>
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<tr>
<td>300 – 500</td>
<td>2.8</td>
<td>23.7</td>
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<tr>
<td>212 – 300</td>
<td>5.9</td>
<td>15.1</td>
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<tr>
<td>150 – 212</td>
<td>10.5</td>
<td>7.0</td>
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<tr>
<td>106 – 150</td>
<td>34.8</td>
<td>10.1</td>
</tr>
<tr>
<td>&lt;106</td>
<td>42.3</td>
<td>4.8</td>
</tr>
</tbody>
</table>

Technology has been transferred to KAPS Engineers, Boroda.
New projects

- Development of Space Pulse tube cryocooler
  (project sanctioned on 28/03/2006)
  - Moving magnet linear motor compressor
  - Cooling power = 0.5 W @65K
  - Input power = < 30 W
  - Weight (w/o electronics) = <2.5 kg

- Zero-helium loss magnet cryostat using hybrid cryocoolers
  (Project submitted to DST)
  - Multi-stage high frequency pulse tube cooler
  - Oil-free, linear motor compressor based J-T expander
  - Counter flow heat exchangers
  - Re-condensation cryostat
  - Transfer tube
  - Helium re-condensation rate ≈ 3 litres of liquid helium in 24 hours
Pulse Tube Based Liquid Helium Re-condenser System
Looking for new faculty

POSITIONS OPEN FOR

ASSISTANT PROFESSORS

in the CENTRE FOR CRYOGENIC TECHNOLOGY, INDIAN INSTITUTE OF SCIENCE, BANGALORE 560 012, INDIA

For Indian nationals having Ph.D degree and with 2 to 3 years of Postdoctoral research experience in the areas of Heat Transfer / Cryocoolers / Cryogenic Instrumentation / Cryogenic System Design etc.

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&
http://www.iisc.ernet.in
Thank you