Result of SIC 2004 Samples and a Damage/Recovery Study for PWO Samples from BTCP and SIC

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Introduction

- 12 endcap size SIC 2004 PWO samples, 570/572 (01/04), 630/641 (05/04), 686/705 (10/04), 713/747 (11/04), 781/782 (12/04) and 841/855 (01/05), went through a standard test procedure at Caltech: a thermal annealing @ 200°C, followed by irradiations in three steps: ~72 h @ 15 rad/h, ~72 h @ 400 rad/h and ~48 h @ 9 krad/h.

- Properties measured: transmittance, emission and excitation spectra, light output, decay kinetics, light response uniformity and their degradation, as well as emission weighted radiation induced absorption coefficient (EWRIAC).

- Results are compared to two previous batches of 20 PWO samples measured at Caltech: BTCP 2001 batch (endcap size) and SIC 2002 batch (CEBAF size).

- 2 SIC samples (570 & 572) and 2 BTCP 2003 samples (2482 & 2531) went through irradiation and recovery cycles: 2 @ 15 rad/h, 3 @ 400 rad/h, and 2 @ 9 krad/h.

- Comments on PWO quality control.
200°C Thermal Annealing

- Carried out in a Lindberg Blue-M tube furnace with automatic control.
- Removed residual absorption and restored the sample to a “defined state”.

Annealing process
- By Lindberg/Blue - M Tube furnace
  - Heating rate: 1°C/min.
  - Annealing @ 200°C for 4 hours
  - Cooling rate: 0.5°C/min.
Caltech γ–ray Irradiation Facilities

Open 50 curie Co-60: 15 and 400 rad/h

Closed 2,000 curie Cs-137: 9k rad/h at center (10% uniformity)
Recovery Temperature Control

After irradiation recovery was measured when samples were kept in a cooler at $18^\circ\text{C}$ with $0.12^\circ\text{C}$ variation.
Photo Luminescence Measurement

HITACHI F4500 Fluorescence Spectrophotometer

Corrections were made for light source (1), gratings efficiencies (2 & 3) & QE of the red-extended PMT (Hamamatsu R928, 4): 185 to 900 nm
Photoluminescence

No variation in excitation and emission before and after 9000 rad/h irradiation

<table>
<thead>
<tr>
<th>Material</th>
<th>Batch</th>
<th>Treatment</th>
<th>Excitation</th>
<th>Emission</th>
</tr>
</thead>
<tbody>
<tr>
<td>SIC-641</td>
<td>May</td>
<td>200°C annealing</td>
<td>em: 424 nm</td>
<td>ex: 310 nm</td>
</tr>
<tr>
<td>SIC-782</td>
<td>December</td>
<td>200°C annealing</td>
<td>em: 424 nm</td>
<td>ex: 310 nm</td>
</tr>
</tbody>
</table>

Intensity (arbitrary unit) vs. Wavelength (nm)
Light Output Measurement

A Hamamatsu R2259 PMT, a LeCroy QVT in Q mode and a $^{137}$Cs source were used for PWO samples. Temperature corrections were made to 18°C.
Light Output and Decay Kinetics

Fast scintillation light observed before and after 15 and 400 rad/h irradiations

![Graphs showing light output and decay kinetics for SIC-641 and SIC-782 after 200°C annealing and irradiations at different rates.](image)
Light Response Uniformity

Fit to a linear function:

\[
\frac{y}{y_{\text{mid}}} = 1 + \delta \left( \frac{x}{x_{\text{mid}}} - 1 \right)
\]

No variation before and after 15 and 400 rad/h irradiations
Comparison of LO & Degradation

2004 batch has more light: **geometry**
Some 2004 samples damages more: **contamination**
SIC samples have 50% more light than BTCP: **doping**
Randomly Selected PWO Samples

BTCP: 20 from 2001 batch for CMS endcaps
SIC: 20 from 2002 production batch for PrimEx

BTCP: 28.5$^2$ x 220 x 30.0$^2$ mm

SIC: 22$^2$ x 230 x 22$^2$ mm
Transmittance Measurement

HITACHI U-3210 UV/VIS Spectrophotometer with a Large Sample Compartment
Longitudinal Transmittance

Radiation induced absorption, caused by CC formation, observed after 15, 400 and 4k rad/h
EWRIAC Measured after Irradiations

Note: emission weighted and multiple bounces

\[ R_{iae} = 1/LAL_{equilibrium} - 1/LAL_{before} \]

\[ LAL = \frac{\ell}{\ln\left\{ \frac{T(1-T_s^2)}{\sqrt{4T_s^4 + T^2(1-T_s^2)^2 - 2T_s^2}} \right\}} \]

\[ T_s = (1-R)^2 + R^2(1-R)^2 + \ldots = (1-R)/(1+R) \]

\[ R = \frac{(n_{crystal} - n_{air})^2}{(n_{crystal} + n_{air})^2} \]

\[
\begin{array}{ll}
\text{Wavelength (nm)} & \text{Wavelength (nm)} \\
800 & 800 \\
700 & 700 \\
600 & 600 \\
500 & 500 \\
400 & 400 \\
\end{array}
\]

\[
\begin{array}{ll}
\text{Absorption coefficient (m}^{-1}) & \text{Absorption coefficient (m}^{-1}) \\
0 & 0 \\
0.2 & 0.2 \\
0.4 & 0.4 \\
0.6 & 0.6 \\
1 & 1 \\
2 & 2 \\
3 & 3 \\
4 & 4 \\
\end{array}
\]

\[
\begin{array}{ll}
\text{Photon energy (eV)} & \text{Photon energy (eV)} \\
1.5 & 1.5 \\
2 & 2 \\
2.5 & 2.5 \\
3 & 3 \\
3.5 & 3.5 \\
4 & 4 \\
\end{array}
\]

- SIC-641
  - 15 rad/h
  - \( A_1 = 0.01 \) \( E_{\alpha_1} = 2.32 \) \( \sigma_1 = 0.2 \)
  - \( A_2 = 0.19 \) \( E_{\alpha_2} = 3.15 \) \( \sigma_2 = 0.76 \)
  - \( \chi^2/DOF = 1.0 \)
  - E.W.RIAC=0.18

- 400 rad/h
  - \( A_1 = 0.06 \) \( E_{\alpha_1} = 2.32 \) \( \sigma_1 = 0.2 \)
  - \( A_2 = 0.62 \) \( E_{\alpha_2} = 3.15 \) \( \sigma_2 = 0.76 \)
  - \( \chi^2/DOF = 0.3 \)
  - E.W.RIAC=0.56

- 9000 rad/h
  - \( A_1 = 0.63 \) \( E_{\alpha_1} = 2.32 \) \( \sigma_1 = 0.2 \)
  - \( A_2 = 2.34 \) \( E_{\alpha_2} = 3.15 \) \( \sigma_2 = 0.76 \)
  - \( \chi^2/DOF = 2.8 \)
  - E.W.RIAC=2.15

- SIC-782
  - 15 rad/h
  - \( A_1 = 0.01 \) \( E_{\alpha_1} = 2.32 \) \( \sigma_1 = 0.2 \)
  - \( A_2 = 0.29 \) \( E_{\alpha_2} = 3.15 \) \( \sigma_2 = 0.76 \)
  - \( \chi^2/DOF = 0.1 \)
  - E.W.RIAC=0.25

- 400 rad/h
  - \( A_1 = 0.08 \) \( E_{\alpha_1} = 2.32 \) \( \sigma_1 = 0.2 \)
  - \( A_2 = 0.56 \) \( E_{\alpha_2} = 3.15 \) \( \sigma_2 = 0.76 \)
  - \( \chi^2/DOF = 0.3 \)
  - E.W.RIAC=0.50

- 9000 rad/h
  - \( A_1 = 0.02 \) \( E_{\alpha_1} = 2.32 \) \( \sigma_1 = 0.2 \)
  - \( A_2 = 0.87 \) \( E_{\alpha_2} = 3.15 \) \( \sigma_2 = 0.76 \)
  - \( \chi^2/DOF = 1.3 \)
  - E.W.RIAC=0.77
Comparison of LT and Damage

2004 batch has higher initial LT: **1 cm shorter**
BTCP samples have 3% higher LT: **Orientation**
Transmittance of a PWO Cube

Transmittances measured along the c and a axis are **NOT** directly comparable.

![Graph showing transmittance vs. wavelength for PWO Cube](image)

\[ \Delta T(\%) = 100 \times \left( \frac{T_a - T_c}{T_c} \right) \]

- \( \Delta T(\%) @ 360 \text{ nm} = 8.2 \)
- \( \Delta T(\%) @ 420 \text{ nm} = 2.6 \)
Transmittance and Birefringence

*a axis*: better L.T., but non-isotropic transverse T.
Both approaching theoretical limit

BTCP: grown along the *a axis*

SIC: grown along the *c axis*
Initial Transmittance

SIC samples are consistent: \textit{c axis} orientation

\begin{figure}
\centering
\includegraphics[width=\textwidth]{transmittance_graphs}
\caption{Transmittance graphs for SIC-641 and SIC-782.}
\end{figure}
Emission Weighted RIAC

Radiation hardness is more divergent at high dose rate
01/04 and 12/04 batches are better: **need more statistics**!
All samples: EWRIAC < 1 m\(^{-1}\) up to 400 rad/h
EWRIAC & Comparison

Two 2003 BTCP samples are better than the 2001 batch
Some SIC 2004 samples are softer caused by Contamination

BTCP-PWO

2001 batch
Comparison of EWRIAC

Radiation hardness is more divergent at high dose rate
01/04 and 12/04 batches are good and two BTCP 2003 samples are better than the 2001 batch: need more statistics!
Summary I: 2004 SIC Samples

- SIC samples produce 50% more light than BTCP samples because of doping.
- The overall performance of SIC 2004 samples is consistent with the 2001 batch but has a batch to batch fluctuation (soft batches) caused by contamination.
- The EWRIAC data shows that all samples tested so far are qualified for CMS barrel, where dose rate is less than a few hundred rad/h even at SLHC. Stringent QC, however, is required for crystals to be used in endcaps where up to 10,000 rad/h is expected at SLHC.
Damage and Recovery Test

With RIAC under control we have to deal with LO variations by using knowledge of LT: monitoring.

Four PWO samples delivered around the end of 2003 (2482 & 2531 from BTCP and 2570 & 2572 from SIC) went through identical damage-recovery cycles.
EWRIAC of 2 BTCP Samples

Average EWRIAC = 0.05, 0.28 & 0.71 m⁻¹ @ 15, 400 and 9,000 rad/h
EWRIAC of 2 SIC Samples

Average EWRIAC = 0.30, 0.59 & 1.19 m⁻¹ @ 15, 400 and 9,000 rad/h

A factor of 6, 2 & 1.7 of BTCP
History of BTCP L.T.

Average variation @ 400 rad/h: 2.4%

\[ \Delta_{400} = \frac{(T_1 - T_2) + (T_1 + T_2)}{} = 2.4\% \]

dose rate (rad/h):

BTCP-2482
- Normalized after 2nd 15rad/h irradiation and recovery

BTCP-2531
- Normalized after 2nd 15rad/h irradiation and recovery

Time (hours)
History of SIC L. T.

Average variation @ 400 rad/h: 3%
a factor of 1.3 of BTCP
Long Term Recovery (BTCP 2001 Batch)

Fast: 40% and 42% with time constant of 31 and 31 h
Slow: 22% and 24% with time constant of 1363 and 1232 h

35% unrecoverable damage
Long Term Recovery (SIC 2004 Batch)

Fast: 25% and 25% with time constant of 65 and 43 h
Slow: 30% and 21% with time constant of 2644 and 1756 h

50% unrecoverable damage
History of BTCP Light Output

Average variations: 2.7% @ 15 rad/h, 12% @ 400 rad/h

BTCP-2482
L.O. = 8.2 p.e./MeV (200 ns, 18°C)
\[ \Delta = \frac{p_{e_1} - p_{e_2}}{p_{e_1} + p_{e_2}} \]
\[ \Delta_{15} = 2.9\% \]
\[ \Delta_{400} = 11.5\% \]

Dose rate (rad/h):

15 Rec 15 Rec 400 Rec 400 Rec 400 Rec

Time (hours)

BTCP-2531
L.O. = 8.7 p.e./MeV (200 ns, 18°C)
\[ \Delta = \frac{p_{e_1} - p_{e_2}}{p_{e_1} + p_{e_2}} \]
\[ \Delta_{15} = 2.5\% \]
\[ \Delta_{400} = 12.0\% \]

Dose rate (rad/h):

15 Rec 15 Rec 400 Rec 400 Rec 400 Rec

Time (hours)
History of SIC Light Output

Average variations: 2.1% @ 15 rad/h, 8.3% @ 400 rad/h

A factor of 0.78 and 0.69 of BTCP

SIC-2570
L.O.: 13.9 p.e./MeV (200 ns, 18°C)

\[ \Delta = \frac{p_{e_1} - p_{e_2}}{p_{e_1} + p_{e_2}} \]

\[ \Delta_{15} = 2.0\% \]

\[ \Delta_{400} = 8.2\% \]

SIC-2572
L.O.: 11.7 p.e./MeV (200 ns, 18°C)

\[ \Delta = \frac{p_{e_1} - p_{e_2}}{p_{e_1} + p_{e_2}} \]

\[ \Delta_{15} = 2.1\% \]

\[ \Delta_{400} = 8.3\% \]
History of BTCP LT & LO @ 400 rad/h

Normalization point:
after 2 cycles of damage & recovery @ 15 rad/h
History of SIC LT & LO @ 400 rad/h

Normalized point:
after 2 cycles of damage & recovery @ 15 rad/h

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**SIC-2570**  
Dose rate: 400 rad/h  
Normalized after 2nd 15 rad/h irradiation and recovery

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**SIC-2572**  
Dose rate: 400 rad/h  
Normalized after 2nd 15 rad/h irradiation and recovery

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- **T**, irradiation  
- **T**, recovery  
- **L.O.**, irradiation  
- **L.O.**, recovery
BTCP Monitoring @ 400 rad/h

Average Slope$_{BTCP} = 3.82$

\[
\Delta L_0/L_0 = -0.03 + 3.66 \times (\Delta L_T/L_T)
\]

\[
\Delta L_0/L_0 = -0.04 + 3.95 \times (\Delta L_T/L_T)
\]
SIC Monitoring @ 400 rad/h

Average Slope$_{SIC}$ = 2.53 = Slope$_{BTCP}$/1.5
Consistent with beam test observation

\[ \Delta L_{O}/L_{O_0} = -0.02 + 2.52 \times (\Delta L_{T}/L_{T_0}) \]
Summary II: Damage/Recovery test

- All four samples have EWRIAC < 1.5 m⁻¹ @ 9,000 rad/h.
- BTCP samples have a factor of 1.3 smaller variation in longitudinal transmittance @ 400 rad/h, but have a factor of 1.3 and 1.4 larger variation in light output @ 15 and 400 rad/h respectively, as compared to SIC samples, indicating more difficult in monitoring. This is caused by faster recovery and smaller unrecoverable damage fraction in BTCP samples.
- The variations of LO can be monitored (and corrected) by the variations of LT @ 400 rad/h. The slope between these two variations are 3.82 and 2.53 for BTCP and SIC samples respectively. The observed ratio (1.5) consists with the 2004 beam test result.
- To be studied: the nature of this difference observed.
Comparison between Caltech & SIC

Caltech LO is 3% higher: QE of PMT
Caltech LT is 3% lower: need identical QC instruments
Investigation on RIAC at SIC

- To understand the SIC irradiation facility, Dr. Liyuan Zhang visited SIC on January 3-7, 2005.
- Two CEBAF size SIC 2002 PWO samples were irradiated for 24h at the $^{60}$Co facility used by SIC. Their transmittance before and after irradiation was measured. The calculated RIAC @ 420 nm result consists with what measured at Caltech under 90 Gy/h irradiation, indicating no fundamental problem in the measurement.
- The dose rate in previous irradiations, however, has a large uncertainty.

<table>
<thead>
<tr>
<th>Sample</th>
<th>@XX Gy/h</th>
<th>RIAC (1/m)</th>
<th>@90 Gy/h</th>
<th>RIAC (1/m)</th>
</tr>
</thead>
<tbody>
<tr>
<td>T12</td>
<td>24 h</td>
<td>1.7</td>
<td>24 h</td>
<td>1.4</td>
</tr>
<tr>
<td>T13</td>
<td>24 h</td>
<td>2.0</td>
<td>32 h</td>
<td>1.9</td>
</tr>
</tbody>
</table>
EWRIAC Measured at Caltech

Note: emission weighted and multiple bounces

EWRIAC has number to be converted to RIAC @ 420 nm
220k Curie $^{60}$Co Source used by SIC

Photos by courtesy of Dr. Hui Yuan
The dose rate at previous sample location was affected by commercial goods being irradiated at the same time.

Better stability of the dose rate is expected when samples are placed on a rack at the designated location.

Attention must be paid to avoid commercial goods, including moving goods, between samples and the source during irradiations.

Dose rate: ~80 Gy/h.
Summary III: PWO Quality Control

- Comparison between LT and LO data between Caltech and SIC indicates the importance of using identical QC equipment.

- There was a large uncertainty in dose rate in SIC’s RIAC data, which is the origin of the discrepancy discussed in the December DPG meeting. Current sample location has a dose rate of about 80 Gy/h with uncertainty reduced.

- A well controlled irradiation facility is crucial for PWO QC, which should be used to check every endcap crystals delivered, and sort them out according to their radiation hardness.
Light Attenuation Length Affects LRU


Ray-Tracing simulation for CMS PWO crystals shows no change in LRU if LAL is longer than 3.5 crystal length

Light collection efficiency, fit to a linear function of distance to the small end of the crystal, was determined with two parameters: the light collection efficiency at the middle of the crystal and the uniformity.

<table>
<thead>
<tr>
<th>LAL (cm)</th>
<th>20</th>
<th>40</th>
<th>60</th>
<th>80</th>
<th>200</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Large Area Photo Detector</strong>, covering 100% back face</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\eta_m$ (%)</td>
<td>9.5±.2</td>
<td>15.7±.4</td>
<td>19.2±.5</td>
<td>21.6±.6</td>
<td>26.9±.7</td>
</tr>
<tr>
<td>$\delta$ (%)</td>
<td>23±1</td>
<td>-4.6±.8</td>
<td>-11±1</td>
<td>-15±1</td>
<td>-15±1</td>
</tr>
<tr>
<td><strong>ϕ5 mm Photo Detector</strong>, covering 3.7% back face</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$\eta_m$ (%)</td>
<td>.38±.04</td>
<td>.74±.08</td>
<td>1.1±.1</td>
<td>1.4±.2</td>
<td>3.0±.3</td>
</tr>
<tr>
<td>$\delta$ (%)</td>
<td>23±4</td>
<td>-3.5±4</td>
<td>-12±4</td>
<td>-16±4</td>
<td>-17±3</td>
</tr>
<tr>
<td>$\frac{\eta_m(ϕ5mm)}{\eta_m(Full)}$ (%)</td>
<td>4.0</td>
<td>4.7</td>
<td>5.7</td>
<td>6.5</td>
<td>11</td>
</tr>
</tbody>
</table>