Quality of Mass Produced PWO Crystals

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Randomly Selected PWO Samples

BTCP: 20 from 1st batch (100) for CMS endcaps
SIC: 20 from production batch for PrimEx

BTCP: 28.5^2 x 220 x 30.0^2 mm
SIC: 22^2 x 230 x 22^2 mm
Experiment

- All crystals went through (1) thermal annealing at 200°C, (2) irradiations by γ-ray at 15, 400 and 9k rad/h until equilibrium and (3) recovery.
- Properties measured: Transmittance, emission and excitation spectrum, light output, decay kinetics and light response uniformity, as well as their degradation, radiation induced color center and emission weighted radiation induced absorption coefficients.
- Light output degradation was only measured at 15 rad/h because of limited light output: less than 8 p.e./MeV for BTCP samples.
Thermal Annealing

- Rigorous temperature control both in amplitude and slope:
  - From RT to 200°C: 200 minutes;
  - Maintain at 200°C: 240 minutes;
  - From 200°C to 25°C: 400 minutes.

- Crystals are kept in dark at RT (18°C) after annealing. The minimum time between annealing and the 1st measurement is 48 hours.
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**

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**BTCP-1971**

200°C anning 4 hour
- Transverse (x)
- Transverse (y)
- Longitudinal (z)

*Calculated longitudinal T of CMS PWO crystal*
- // c axis,
- ⊥ c axis, unpolarized light
- ⊥ c axis, e-polarized light

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**SIC-U517 (21.6 cm)**

200°C anning 4 hour
- Transverse (x)
- Transverse (y)
- Longitudinal (z)

*Calculated longitudinal T of CMS PWO crystal*
- // c axis,
- ⊥ c axis, unpolarized light
- ⊥ c axis, e-polarized light
Light Output and Decay Kinetics

Both are fast, SIC samples have more light

![Graphs showing light output and decay kinetics for BTCP-2467 and SIC-T6 with T corrections (18°C).]
Comparison of L.T. and Light Yield

<table>
<thead>
<tr>
<th>Vendor</th>
<th>ILT@40 nm</th>
<th>ILO (p.e.)</th>
<th>50 ns/1</th>
<th>100 ns/1</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>BTCP-PWO</td>
<td>69.83</td>
<td>6.393</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SIC-PWO</td>
<td>65.55</td>
<td>10.13</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Initial L.T. @ 440 nm (%)

Light Output (p.e./MeV)
Caltech $\gamma$–ray Irradiation Facilities

Open 50 curie Co-60: 15, 100 and 400 rad/h

Closed 2,000 curie Cs-137: 9k rad/h at center, up to 36k rad/h
Photoluminescence

No variation in either excitation or emission spectrum
No damage in scintillation mechanism
No Variation in Light Response Uniformity

The response ($y$) along the axis was fit to a linear function

$$\frac{y}{y_{mid}} = 1 + \delta \left( \frac{x}{x_{mid}} - 1 \right)$$
Light Output Degradation

5-15% and 15-30% light output loss under 15 and 500 rad/h
Damage is dose rate dependent

![Normalized Light Output Graphs](image-url)
Damage in Longitudinal Transmittance

Radiation induced absorption caused by CC formation

**Graphs:**

- **BTCP-2466**
  - Transmittance (%)
  - Wavelength (nm)
  - From top to bottom:
    - 200°C annealing
    - 15 rad/h (65 h)
    - 100 rad/h (63 h)
    - 400 rad/h (62 h)
    - 9000 rad/h (10 h)
    - 35000 rad/h (6.5 h)

- **SIC-T5**
  - Transmittance (%)
  - Wavelength (nm)
  - From top to bottom:
    - 200°C annealing
    - 15 rad/h (108 h)
    - 400 rad/h (72 h)
    - 9000 rad/h (24 h)
Comparison of Radiation Damage

SIC samples seem more radiation hard
Comparison of Transmittance Loss

SIC samples less diverse: Bridgman technology
One BTCP sample shows LT increase under irradiation
Type III Sample: Transmittance Loss

Type III sample: preexisting intrinsic color center at 420 nm after 200 degree annealing, causing difficulty for monitoring with 440 nm light.
Investigation on BTCP Samples (I)

Three samples cut to 5 pieces: 4.3 cm each:
Type I: 2467, Type II: 2436, Type III: 2465

Tail

E

D

C

B

A

Seed

43 (mm)

43 (mm)

43 (mm)

43 (mm)

43 (mm)

28.5² x 220 x 30.0² (mm)
Investigation on BTCP Samples (II)

Anomaly is shown also at the Tail end (E and D)

BTCP-2465

Normalized Light output under certain dose rate in equilibrium for 4.2 cm long small pieces.

From top to bottom
- 2465E
- 2465D
- 2465C
- 2465B
- 2465A

Normalized L.O. versus irradiation time under 15 rad/h for 4.2 cm long small pieces.

From top to bottom
- 2465E
- 2465D
- 2465C
- 2465B
- 2465A

March 30, 2004
Calor2004, Rihua Mao, SIC
Investigation on SIC Samples (I)

Two anomalous samples were cut to pieces

Crystal ID: NO.4-1-20
Dopant: Y/150 at ppm

<table>
<thead>
<tr>
<th>Seed</th>
<th>1</th>
<th>2</th>
<th>A</th>
<th>B</th>
<th>C</th>
<th>D</th>
<th>E</th>
<th>F</th>
<th>G</th>
<th>H</th>
<th>I</th>
<th>J</th>
<th>3</th>
<th>4</th>
</tr>
</thead>
</table>

The length of seed is 20.0 mm, thickness of 1, 2, 3, 4 is 5.0 mm.
Dimension of AB, CD, EF, GH and IJ is: 25.0 x 25.0 x 44.3 mm$^3$.

Crystal ID: B13
Dopant: Y/150 at ppm

Seed Side

<table>
<thead>
<tr>
<th></th>
<th>B13a</th>
<th></th>
</tr>
</thead>
</table>

B13a

Dimension of B13a: 22.0 x 22.0 x 177.0 x 25.0 x 25.0 mm$^3$.
Dimension of B13b: 22.0 x 22.0 x 50.0 x 23.0 x 23.0 mm$^3$.
Investigation on SIC Samples (II)

Anomaly was found at the tail end: impurity related?

- AB  L.Y. As-received = 17.9 p.e./MeV  1.8 rad/h
- CD  L.Y. As-received = 17.9 p.e./MeV  1.8 rad/h
- EF  L.Y. As-received = 16.7 p.e./MeV  1.8 rad/h
- GH  L.Y. As-received = 15.7 p.e./MeV  1.8 rad/h
- IJ  L.Y. As-received = 12.9 p.e./MeV  1.8 rad/h

B13b
After 50°C, 4h annealing
Dose rate: 4.0 rad/h

B13a
After 50°C, 4h annealing
Dose rate: 4.0 rad/h
Trace Analysis on SIC Samples

GDMS on SIC PWO(Y) Samples (ppmw)
by Shiva Technology West (November, 1999)

<table>
<thead>
<tr>
<th>Element</th>
<th>Seed/Tail 1</th>
<th>Seed/Tail 2</th>
<th>Seed/Tail 3</th>
<th>Seed/Middle/Tail 4</th>
<th>Tail 5</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na</td>
<td>0.2/0.8</td>
<td>0.2/2.3</td>
<td>0.4/0.8</td>
<td>0.2/0.8/1.9</td>
<td>0.8</td>
</tr>
<tr>
<td>Si</td>
<td>0.5/0.2</td>
<td>0.7/1.3</td>
<td>0.5/1.2</td>
<td>0.5/0.4/0.1</td>
<td>0.05</td>
</tr>
<tr>
<td>K</td>
<td>0.3/1.8</td>
<td>0.4/2.9</td>
<td>0.7/1.2</td>
<td>0.5/0.9/2.0</td>
<td>1.3</td>
</tr>
<tr>
<td>Ca</td>
<td>0.9/&lt;0.05</td>
<td>0.6/0.08</td>
<td>0.12/0.15</td>
<td>0.8/0.6/0.2</td>
<td>0.15</td>
</tr>
<tr>
<td>Cu</td>
<td>0.04/0.2</td>
<td>0.04/0.4</td>
<td>0.3/0.35</td>
<td>0.08/0.1/0.54</td>
<td>0.23</td>
</tr>
<tr>
<td>As</td>
<td>0.15/0.35</td>
<td>0.1/0.6</td>
<td>0.5/0.5</td>
<td>0.14/0.16/0.6</td>
<td>0.54</td>
</tr>
<tr>
<td>Y</td>
<td>40/45</td>
<td>40/50</td>
<td>30/35</td>
<td>40/40/60</td>
<td>50</td>
</tr>
<tr>
<td>Nb</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
</tr>
<tr>
<td>Mo</td>
<td>0.3/0.55</td>
<td>0.3/0.9</td>
<td>0.6/0.8</td>
<td>0.2/0.5/0.8</td>
<td>1.0</td>
</tr>
<tr>
<td>Sb</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
</tr>
<tr>
<td>Ba</td>
<td>0.1/0.1</td>
<td>0.1/0.1</td>
<td>&lt;0.05/0.06</td>
<td>0.3/0.15/0.07</td>
<td>0.1</td>
</tr>
<tr>
<td>La</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
</tr>
<tr>
<td>Eu</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
</tr>
<tr>
<td>TC†</td>
<td>3.8/2.1</td>
<td>4.9/4.6</td>
<td>4.4/3.4</td>
<td>5.3/4.0/2.5</td>
<td>4.3</td>
</tr>
</tbody>
</table>

†: Total contamination, excluding Y.

Impurity segregation:

Na, K, Cu, As, Mo: <1;  
Ca, Ba: >1;  
Y: slightly less, but close to 1.

SIC samples are doped with Y only.
Trace Analysis on BTCP Samples

GDMS on BTCP PWO(Y/Nb/La) Samples (ppmw)
by Shiva Technology (November, 2003)

<table>
<thead>
<tr>
<th>Element</th>
<th>2467 Seed/Tail</th>
<th>2436 Seed/Tail</th>
<th>2465 Seed/Middle/Tail</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na</td>
<td>0.95/0.98</td>
<td>2.5/5.2</td>
<td>3.8/3.4/5.2</td>
</tr>
<tr>
<td>Si</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
</tr>
<tr>
<td>K</td>
<td>0.36/0.58</td>
<td>0.45/0.90</td>
<td>0.71/0.56/1.6</td>
</tr>
<tr>
<td>Ca</td>
<td>2.4/1.8</td>
<td>1.3/0.9</td>
<td>1.7/1.3/1.2</td>
</tr>
<tr>
<td>Cu</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
</tr>
<tr>
<td>As</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
</tr>
<tr>
<td>Y</td>
<td>71/74</td>
<td>94/120</td>
<td>98/83/100</td>
</tr>
<tr>
<td>Nb</td>
<td>0.06/0.11</td>
<td>0.07/&lt;0.05</td>
<td>&lt;0.05/0.27/0.26</td>
</tr>
<tr>
<td>Mo</td>
<td>0.2/0.23</td>
<td>0.33/0.38</td>
<td>0.37/0.37/0.41</td>
</tr>
<tr>
<td>Sb</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
<td>&lt;0.05</td>
</tr>
<tr>
<td>Ba</td>
<td>1.7/1.5</td>
<td>1.5/1.2</td>
<td>5.3/1.7/2.5</td>
</tr>
<tr>
<td>La</td>
<td>250/140</td>
<td>200/130</td>
<td>280/160/150</td>
</tr>
<tr>
<td>Eu</td>
<td>0.6/0.5</td>
<td>0.8/1.4</td>
<td>1.1/0.53/0.3</td>
</tr>
<tr>
<td>TC†</td>
<td>6.4/5.7</td>
<td>7.0/10</td>
<td>13/7.9/11</td>
</tr>
</tbody>
</table>

†: Total contamination, excluding Y, Nb and La.

Impurity segregation:

Na, K, Nb, Mo: <1;

Ca, Ba, La: >1;

Y: slightly less, but close to 1.

BTCP PWO is triple doped with Y/Nb/La!!!
Light Output & La Concentration

- The anti-correlation between the light output of PWO and its La concentration, may explain the low light yield of BTCP PWO.

- Further study is under way to clarify this issue.
RIAC or radiation induced color center density can be calculated precisely by using longitudinal transmittance (0.2%)

\[
RIAC \text{ or } D_{\text{Color-Center}} = \frac{1}{LAL};
\]

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

where \( T \) is transmittance measured along crystal length \( \ell \) and \( T_s \) is the theoretical transmittance without internal absorption:

\[
T_s = (1 - R)^2 + R^2(1 - R)^2 + ... = \frac{(1 - R)}{(1 + R)}, \text{ with}
\]

\[
R = \frac{(n_{\text{crystal}} - n_{\text{air}})^2}{(n_{\text{crystal}} + n_{\text{air}})^2}.
\]
Emission Weighted RIAC

\[ EWRIAC = \frac{\int Riac(\lambda)Em(\lambda)d\lambda}{\int Em(\lambda)d\lambda} \]

a good measure of rad. damage

- **BTCP-2467**
  - EWRIAC = 0.12
  - $A_1 = 0.01\ E_{e1} = 2.30\ \sigma_1 = 0.35$
  - $A_2 = 0.16\ E_{e2} = 3.07\ \sigma_2 = 0.42$
  - $\chi^2$/DOF = 0.2

- **SIC-LS615**
  - EWRIAC = 0.12
  - $A_1 = 0.03\ E_{e1} = 2.32\ \sigma_1 = 0.2$
  - $A_2 = 0.13\ E_{e2} = 3.15\ \sigma_2 = 0.76$
  - $\chi^2$/DOF = 0.2

- **400 rad/h**
  - EWRIAC = 0.35
  - $A_1 = 0.13\ E_{e1} = 2.30\ \sigma_1 = 0.35$
  - $A_2 = 0.39\ E_{e2} = 3.07\ \sigma_2 = 0.42$
  - $\chi^2$/DOF = 0.8

- **SIC-LS615**
  - EWRIAC = 0.24
  - $A_1 = 0.08\ E_{e1} = 2.32\ \sigma_1 = 0.2$
  - $A_2 = 0.26\ E_{e2} = 3.15\ \sigma_2 = 0.76$
  - $\chi^2$/DOF = 0.2

- **9000 rad/h**
  - EWRIAC = 0.42
  - $A_1 = 0.16\ E_{e1} = 2.30\ \sigma_1 = 0.35$
  - $A_2 = 0.47\ E_{e2} = 3.07\ \sigma_2 = 0.42$
  - $\chi^2$/DOF = 0.5

- **SIC-LS615**
  - EWRIAC = 0.50
  - $A_1 = 0.13\ E_{e1} = 2.32\ \sigma_1 = 0.2$
  - $A_2 = 0.54\ E_{e2} = 3.15\ \sigma_2 = 0.76$
  - $\chi^2$/DOF = 0.4
EWRIAC (1/m) and Normalized r.m.s.

<table>
<thead>
<tr>
<th>Vendor</th>
<th>15 rad/h</th>
<th>400 rad/h</th>
<th>9,000 rad/h</th>
</tr>
</thead>
<tbody>
<tr>
<td>BTCP</td>
<td>0.16 (45%)</td>
<td>0.69 (37%)</td>
<td>1.43 (50%)</td>
</tr>
<tr>
<td>SIC</td>
<td>0.10 (33%)</td>
<td>0.51 (32%)</td>
<td>1.16 (48%)</td>
</tr>
</tbody>
</table>
L. T. Loss versus Initial L.T. @ 360 nm

No correlation
EWRIAC versus Initial L.T. @ 440 nm

No correlation

After 400 rad/h in equilibrium
- Endcap
- Type (I)
- Type (II)
- Barrel

SIC - PWO
After 400 rad/h in Equilibrium

Emission weighted RIAC (m⁻¹)

Longitudinal transmittance @ 440 nm (%)

Initial L.T. @ 440 nm (%)
Recovery Speed and Time Constant

Recovery at 18°C in 160 days: two time constants
Short recovery: BTCP: 36.0 h (27%), SIC: 43.6 h (33%)

BTCP-2457
After 9000 rad/h in Equilibrium
\[ \frac{T}{T_0} = 0.811 + 0.099(1 - e^{-t/36.07}) \]

SIC-T32
After 9000 rad/h in Equilibrium
\[ \frac{T}{T_0} = 0.795 + 0.098(1 - e^{-t/61.76}) \]
Summary

- PWO samples from both BTCP and SIC have very good transmittance and fast light output. SIC samples produce 58% more light, which may be explained by 130-280 ppmw La doping in BTCP samples.
- Preexisting CC, causing light output increase under irradiation, is caused by contamination of mono-valent impurities.
- No correlations between radiation hardness and initial longitudinal transmittance was observed.
- Requiring degraded LAL > 1 m, current mass-produced PWO crystals are radiation hard enough for environment of up to a few hundreds rad/h --- a great achievement for HEP and MS.