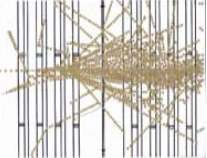


Quality of Mass Produced PWO Crystals

Rihua Mao^{*}, Liyuan Zhang, Ren-yuan Zhu
California Institute of Technology

^{*}Current address: Shanghai Institute of Ceramics, Shanghai, China



Randomly Selected PWO Samples



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

SIC: 20 from production batch for PrimEx

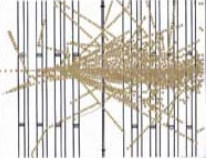


BTCP: $28.5^2 \times 220 \times 30.0^2$ mm

A clear, rectangular PWO crystal sample, likely BTCP, shown horizontally. It has a slightly textured surface and shows some internal reflections.

SIC: $22^2 \times 230 \times 22^2$ mm

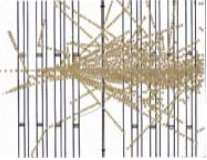
A clear, rectangular PWO crystal sample, likely SIC, shown horizontally. It has a smooth surface and shows some internal reflections.



Experiment

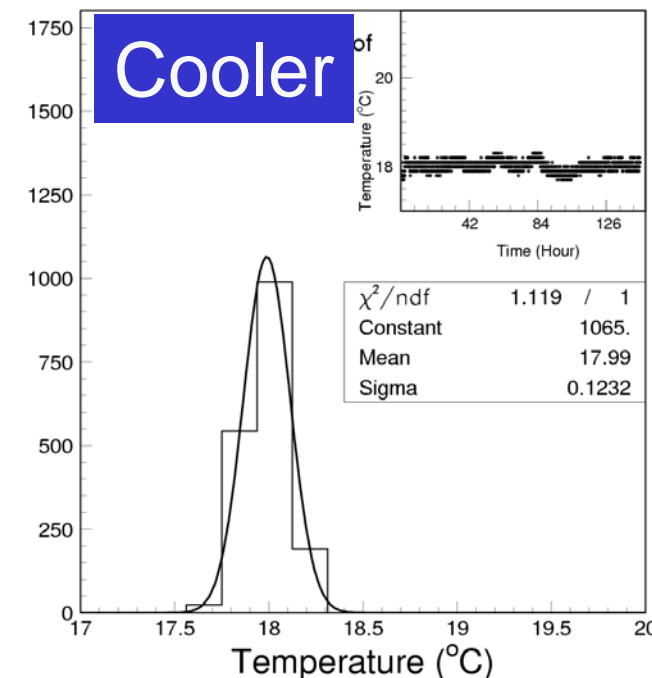
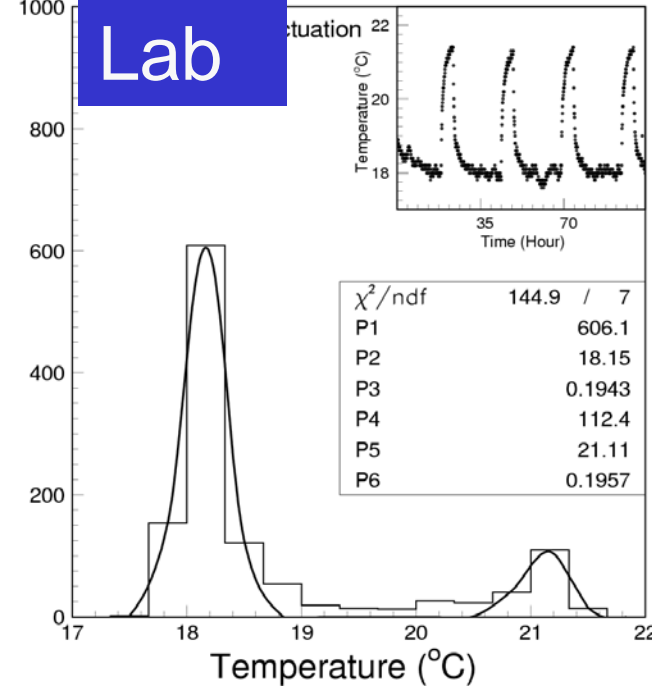


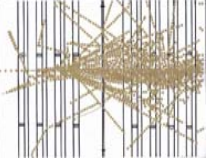
- All crystals went through (1) thermal annealing at 200°C, (2) irradiations by γ -ray at 15, 400 and 9k rad/h until equilibrium and (3) recovery.
- Properties measured: Transmittance, emission and excitation spectrum, light output, decay kinetics and light response uniformity, as well as their degradation, 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.



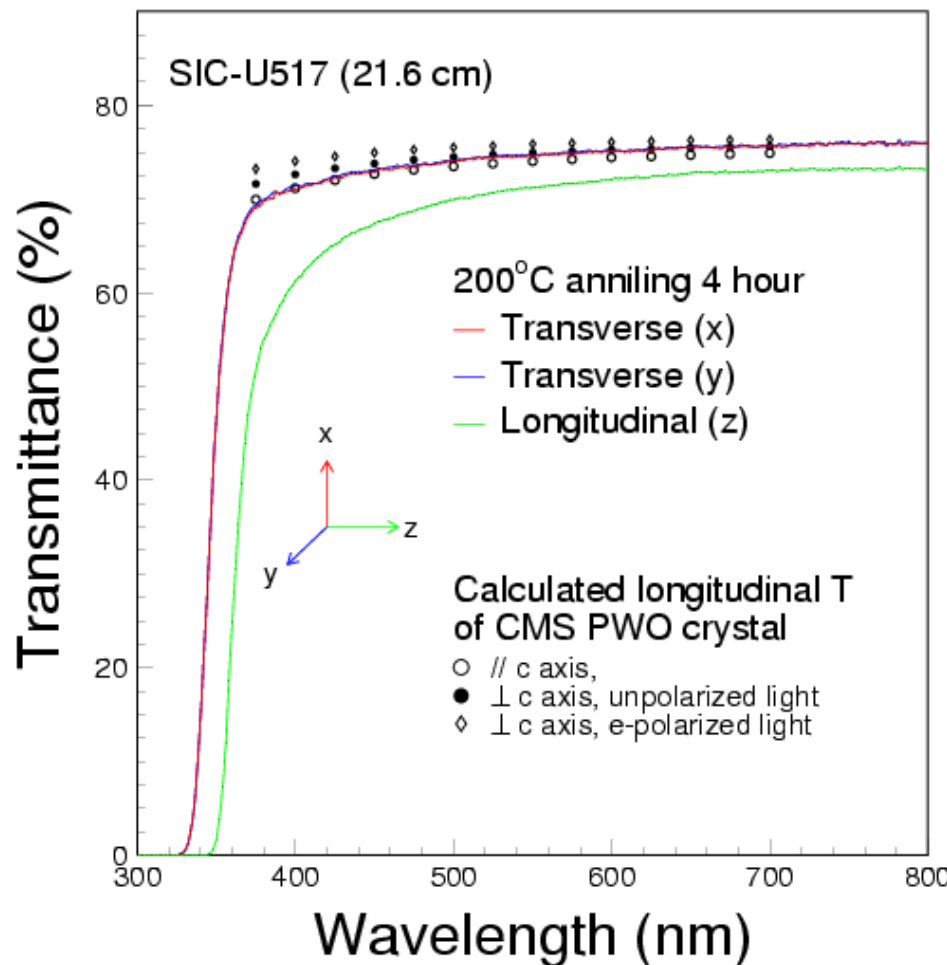
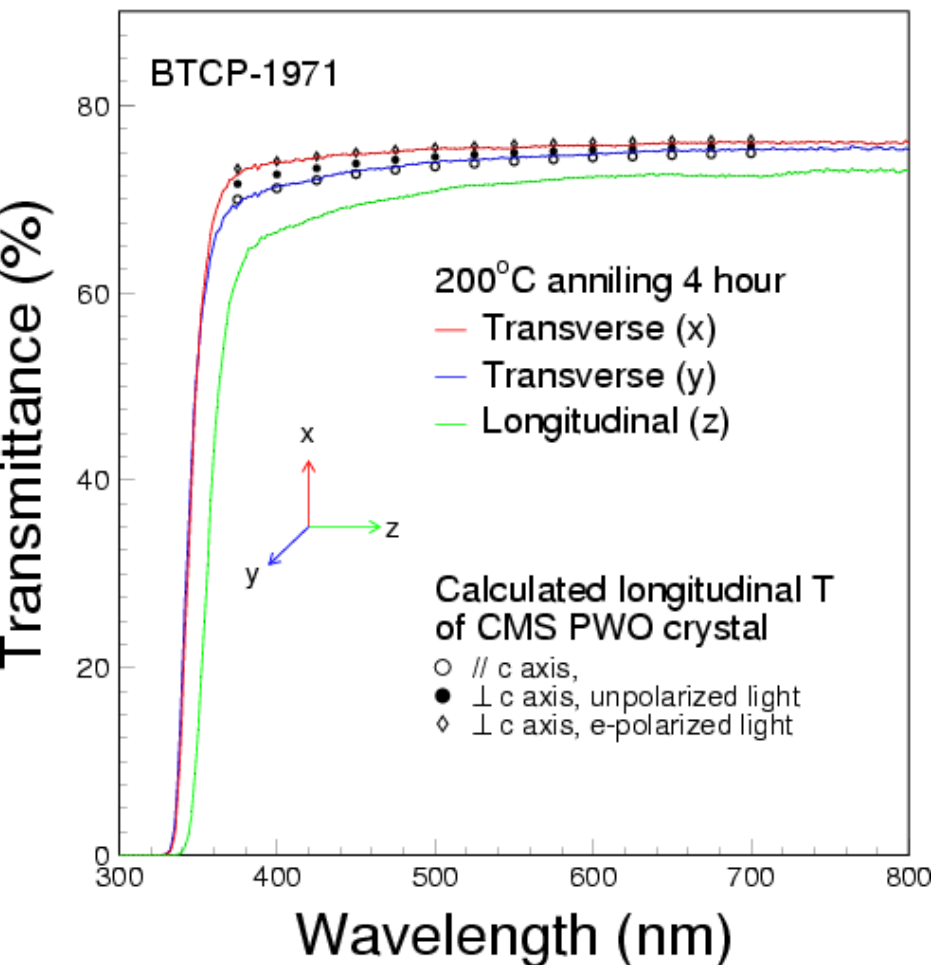


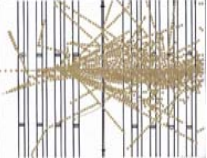
Transmittance and Birefringence

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**

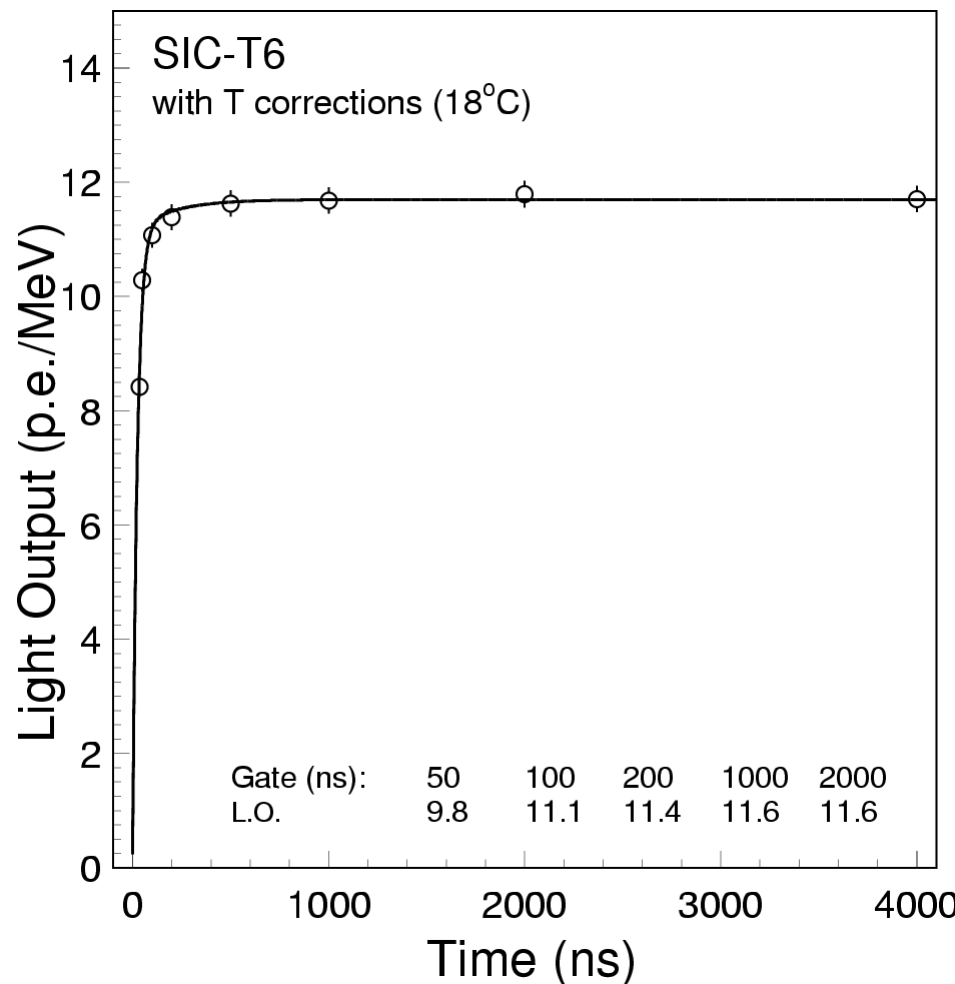
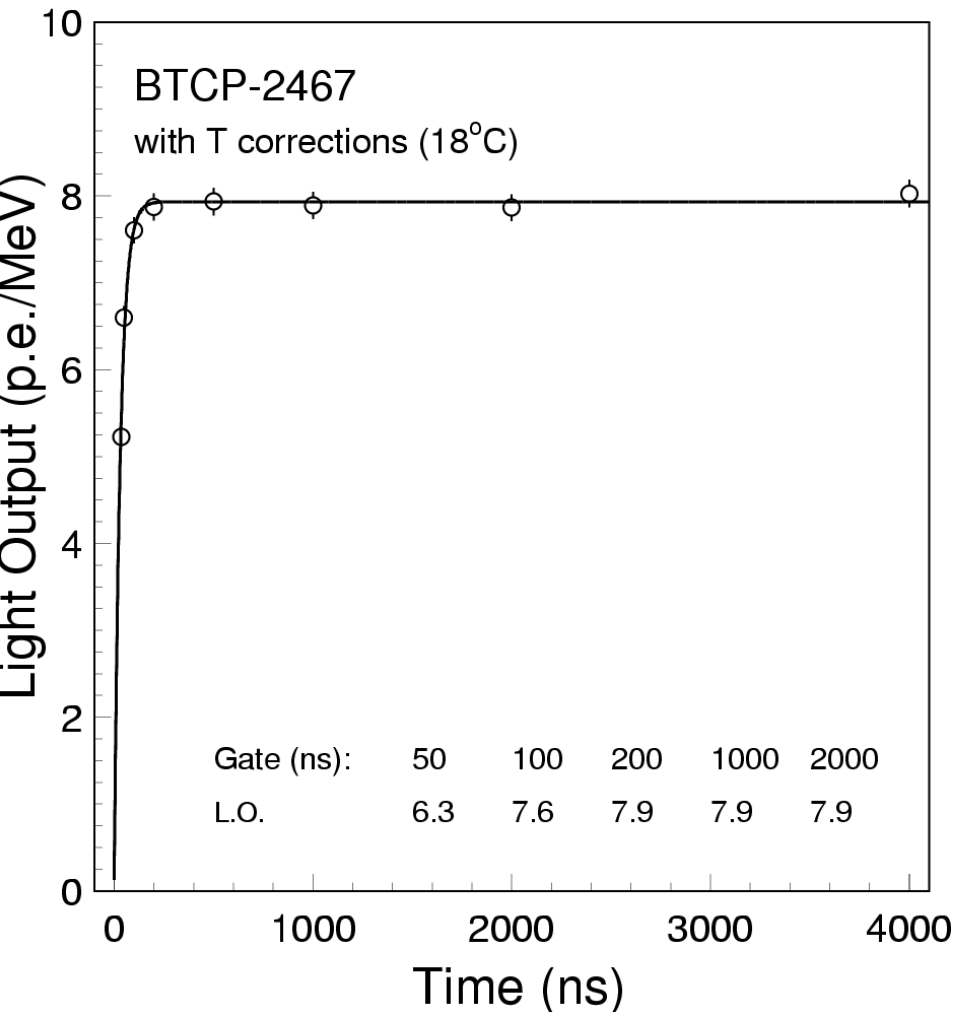




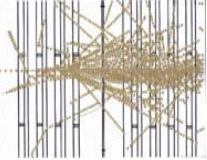
Light Output and Decay Kinetics



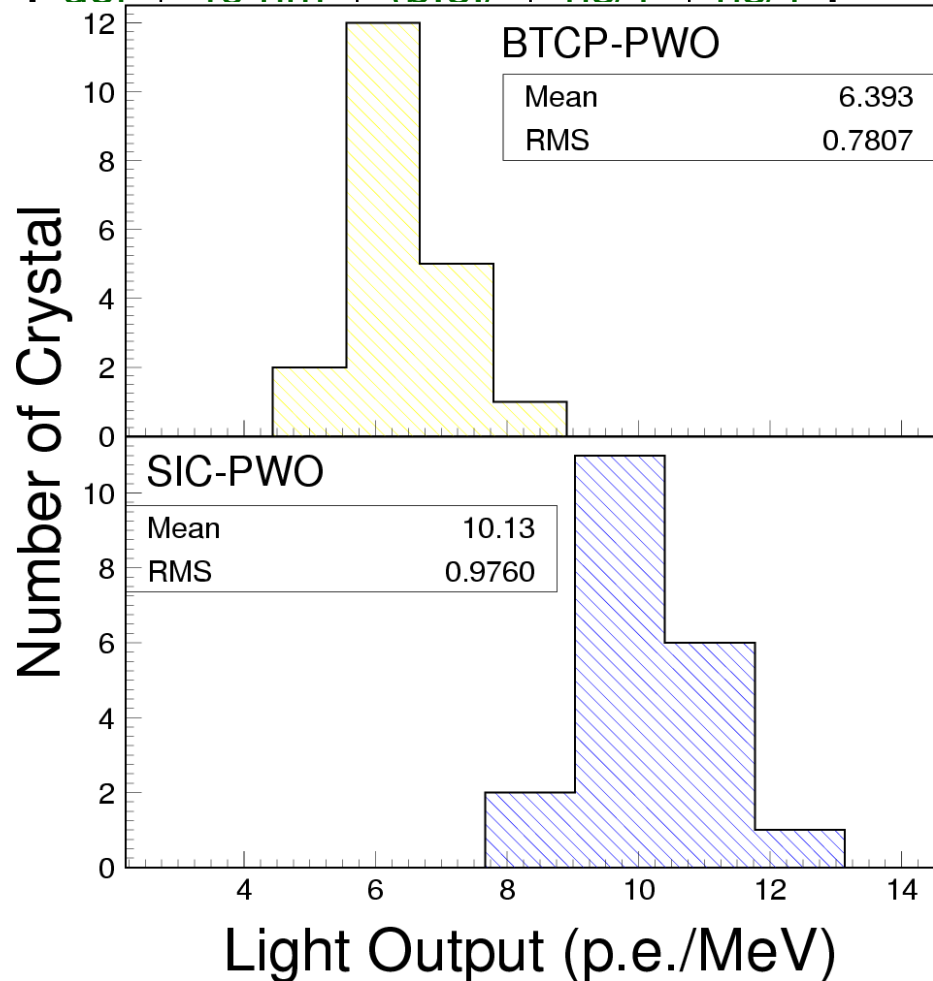
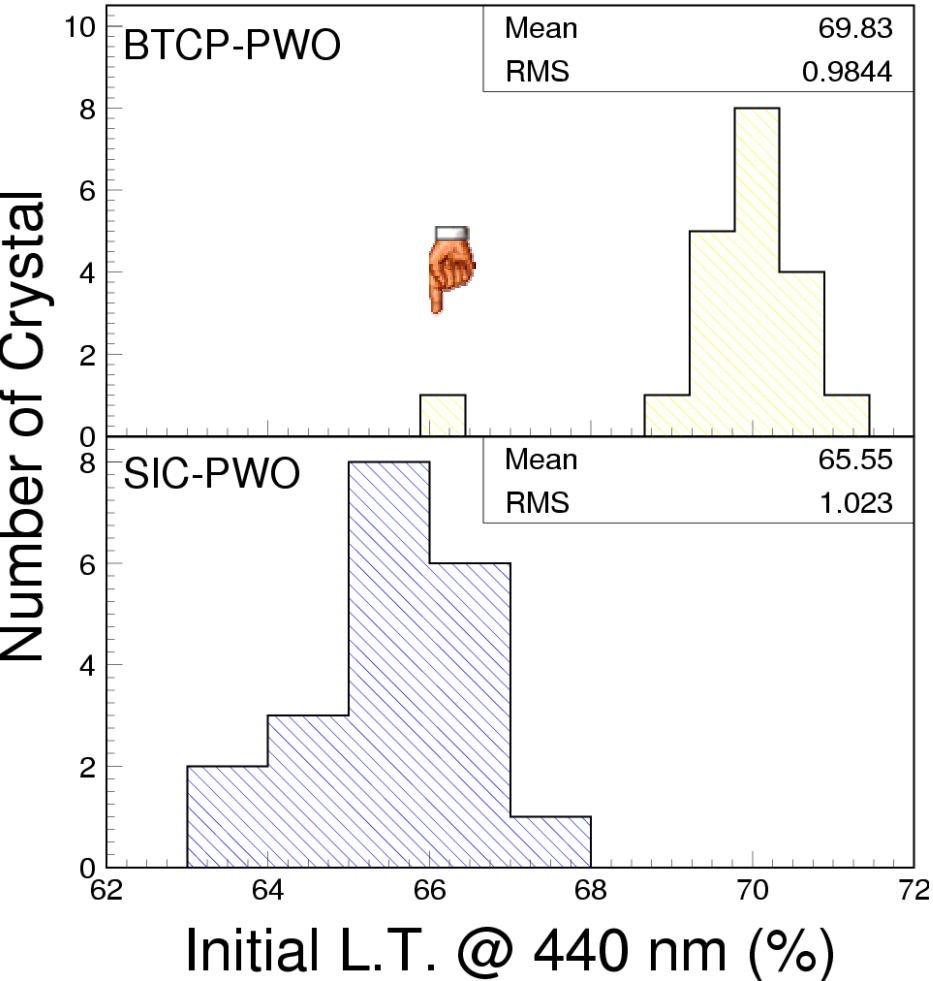
Both are fast, SIC samples have more light

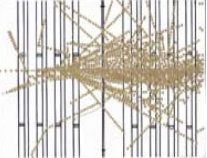


Comparison of L.T. and Light Yield



Vendor	ILT@40 nm	ILO (p.e./ns/1)	50 ns/1	100 ns/1
--------	-----------	-----------------	---------	----------





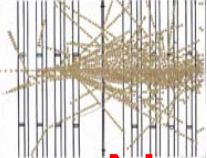
Caltech γ -ray Irradiation Facilities



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

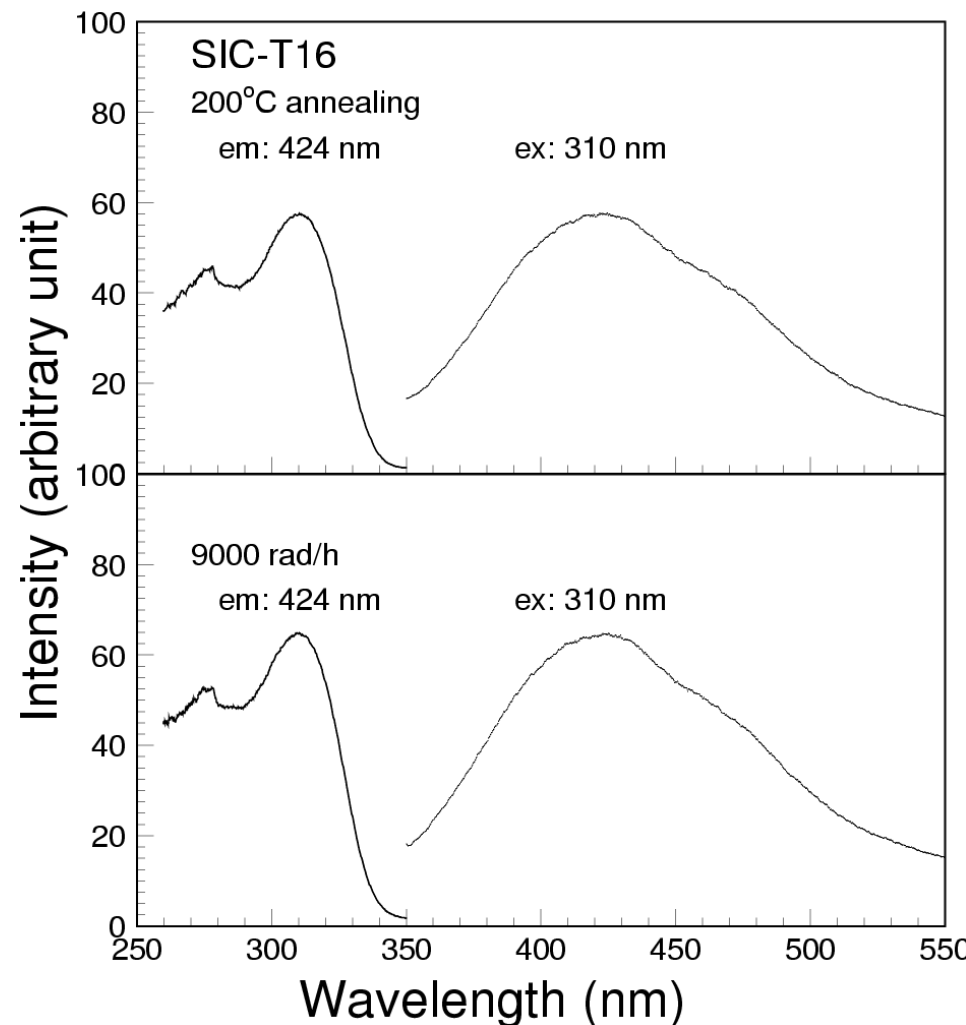
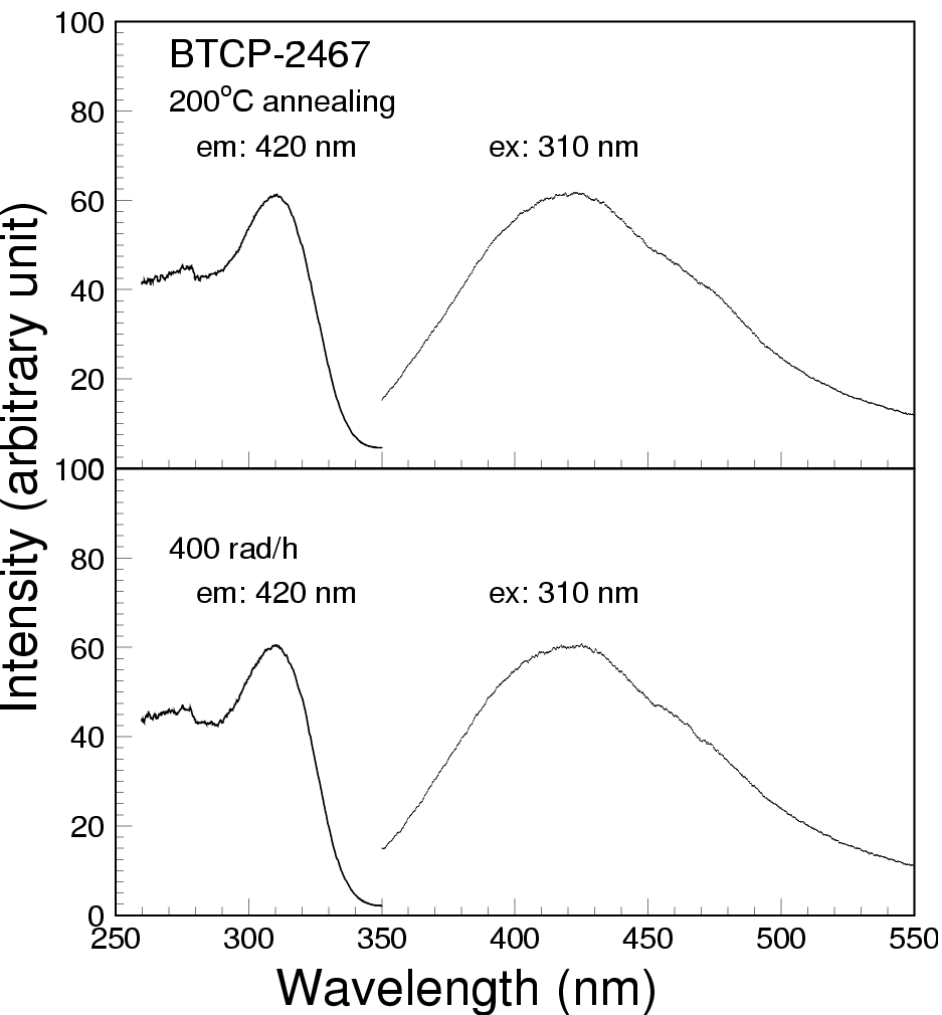




Photoluminescence



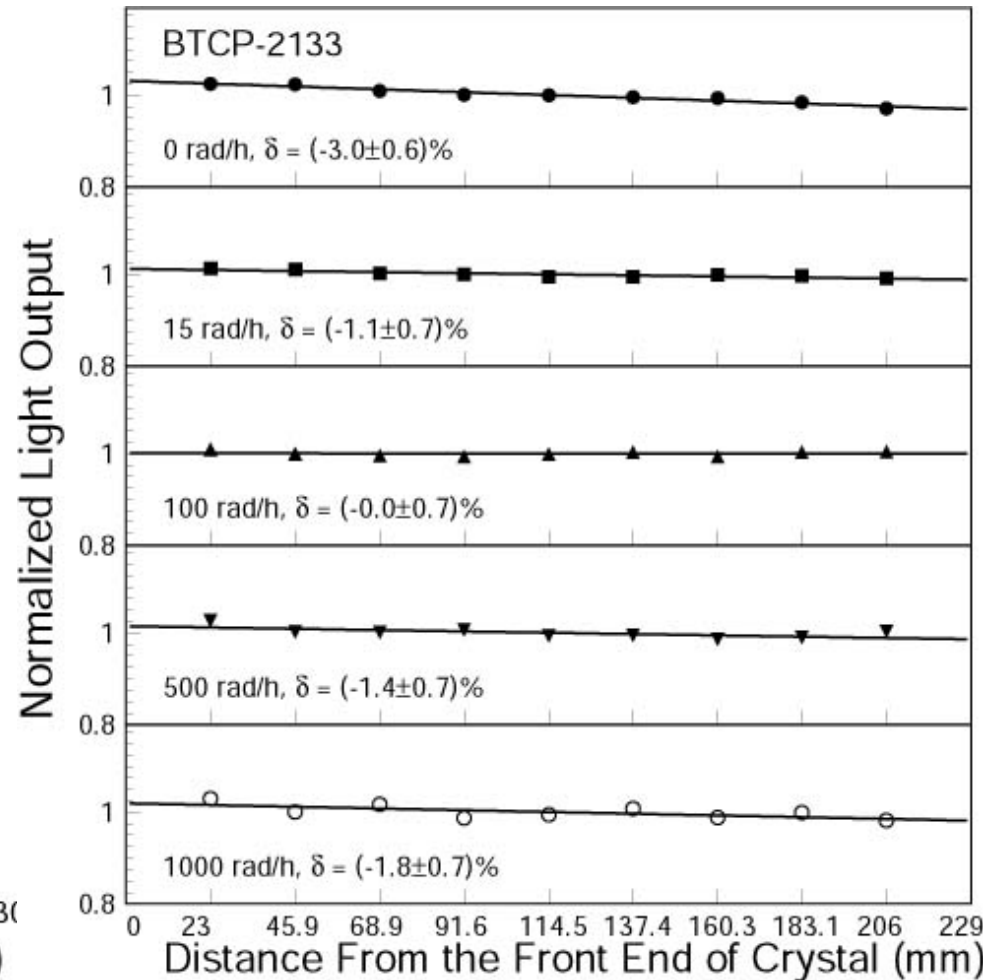
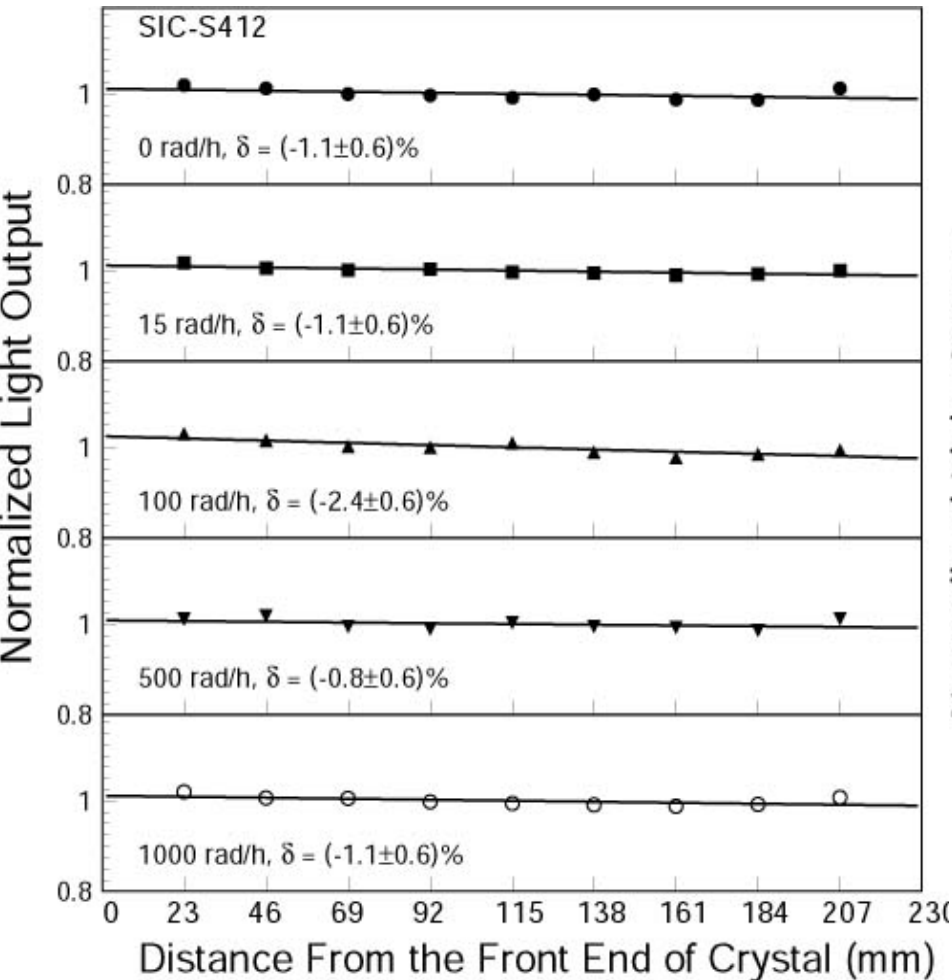
No variation in either excitation or emission spectrum
No damage in scintillation mechanism

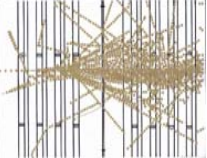


No Variation in Light Response Uniformity

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

$$\frac{y}{y_{mid}} = 1 + \delta(x / x_{mid} - 1)$$

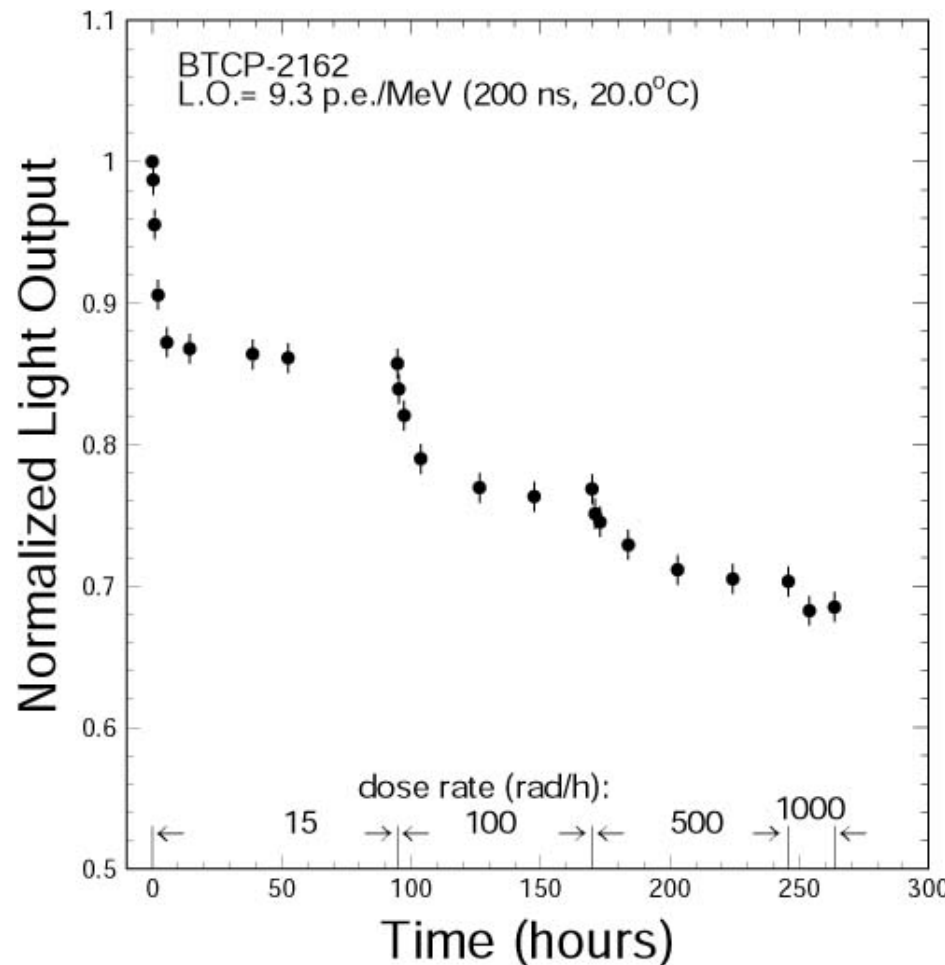
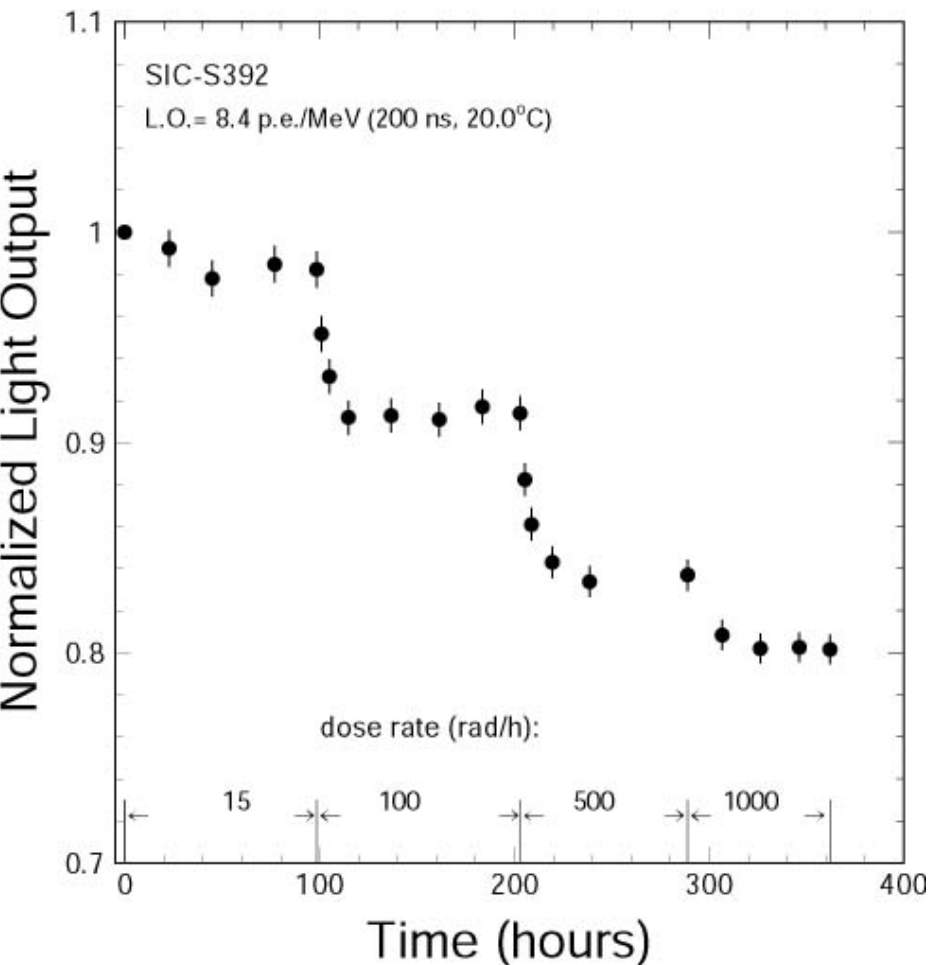


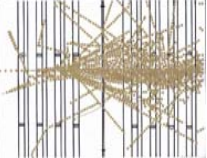


Light Output Degradation



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

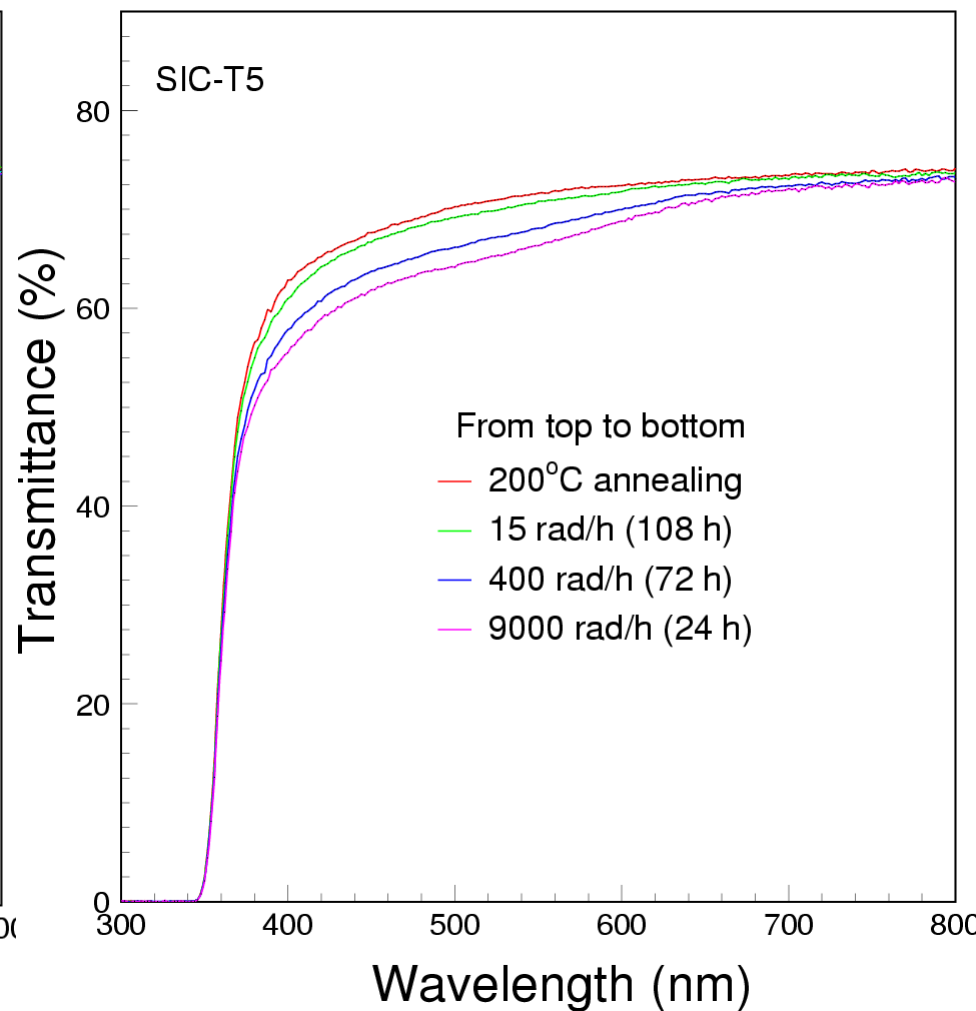
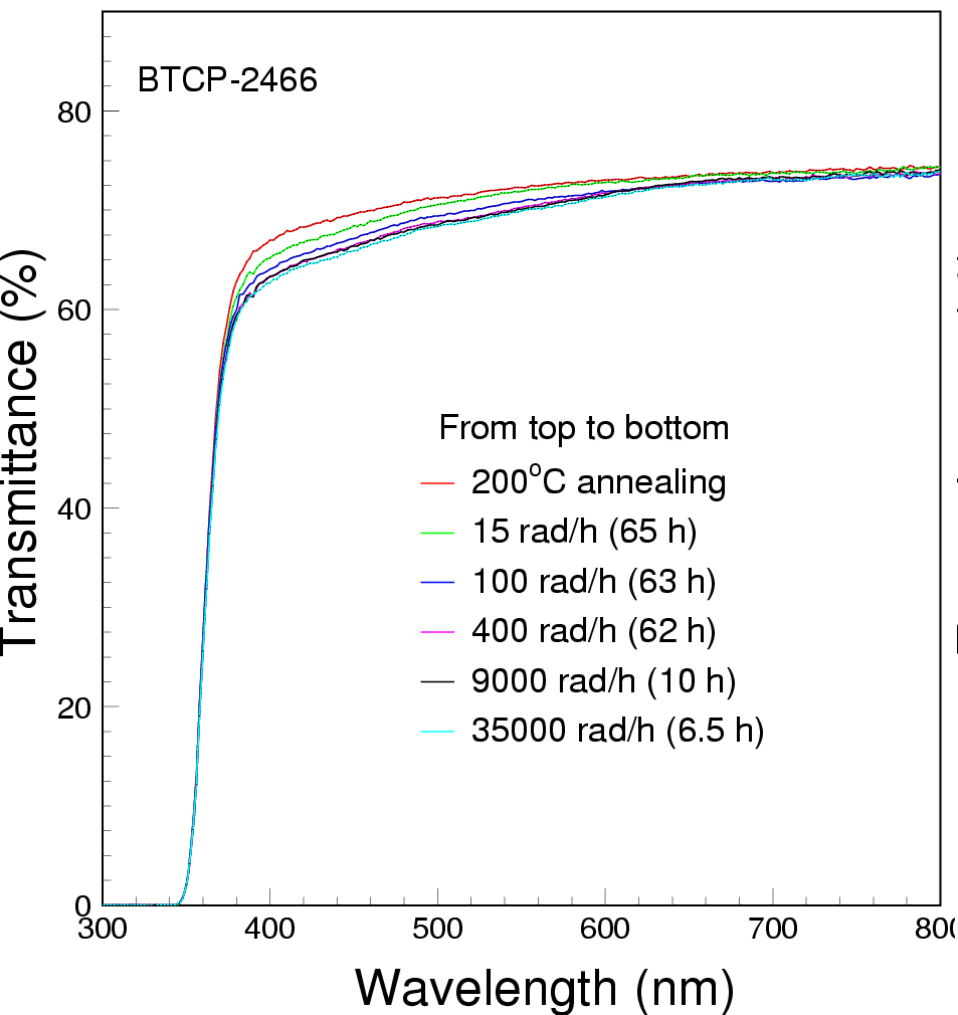


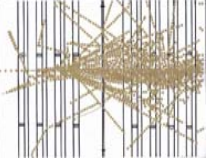


Damage in Longitudinal Transmittance



Radiation induced absorption caused by CC formation



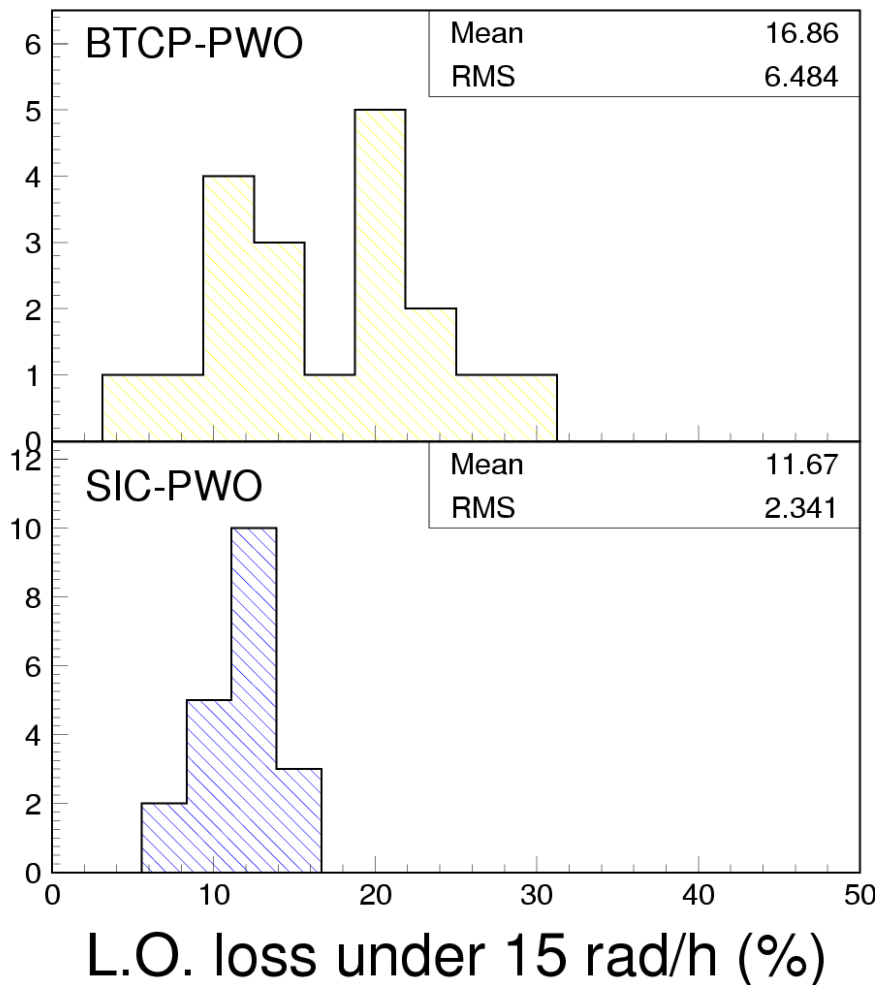


Comparison of Radiation Damage

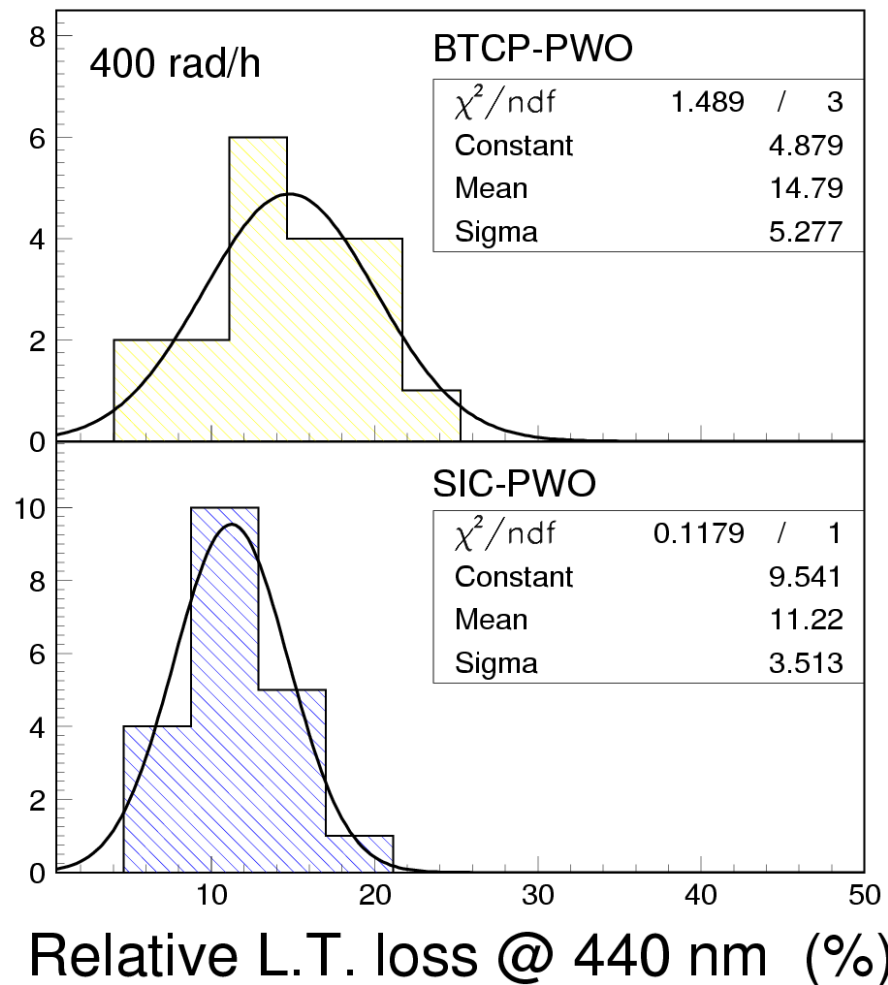


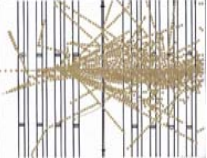
SIC samples seem more radiation hard

Number of Crystal



Number of Crystal

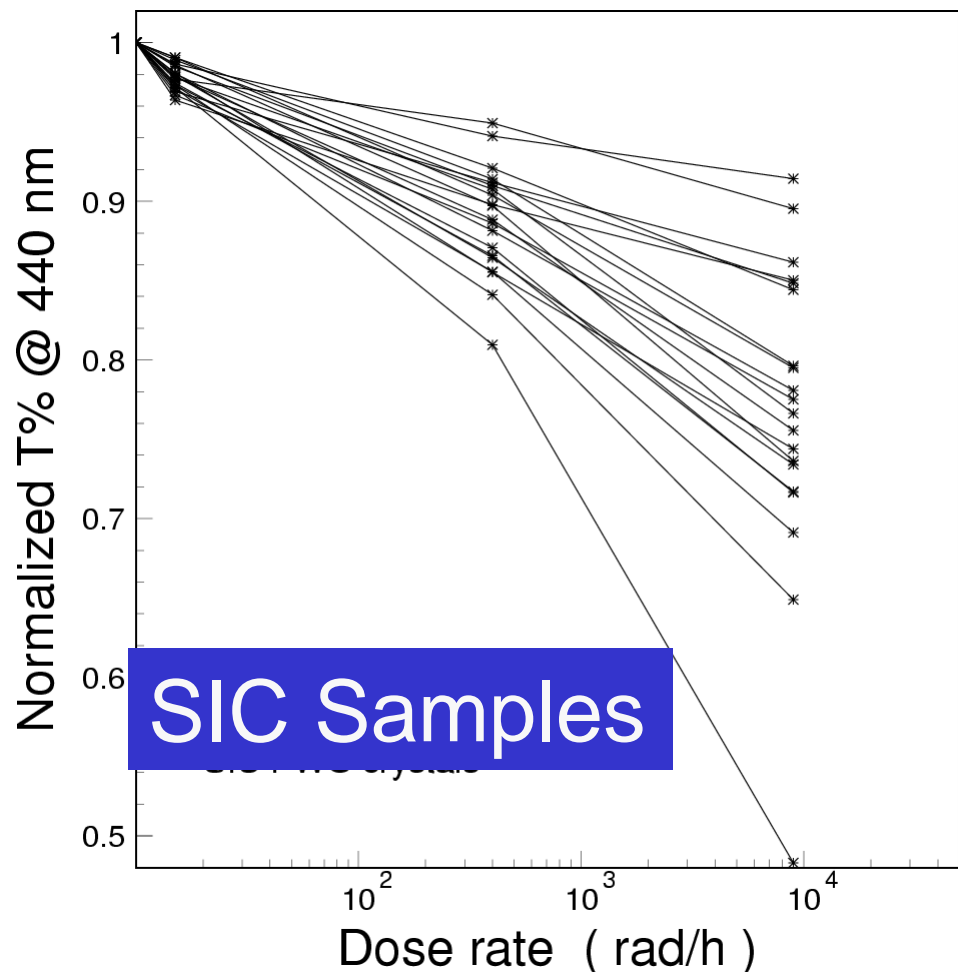
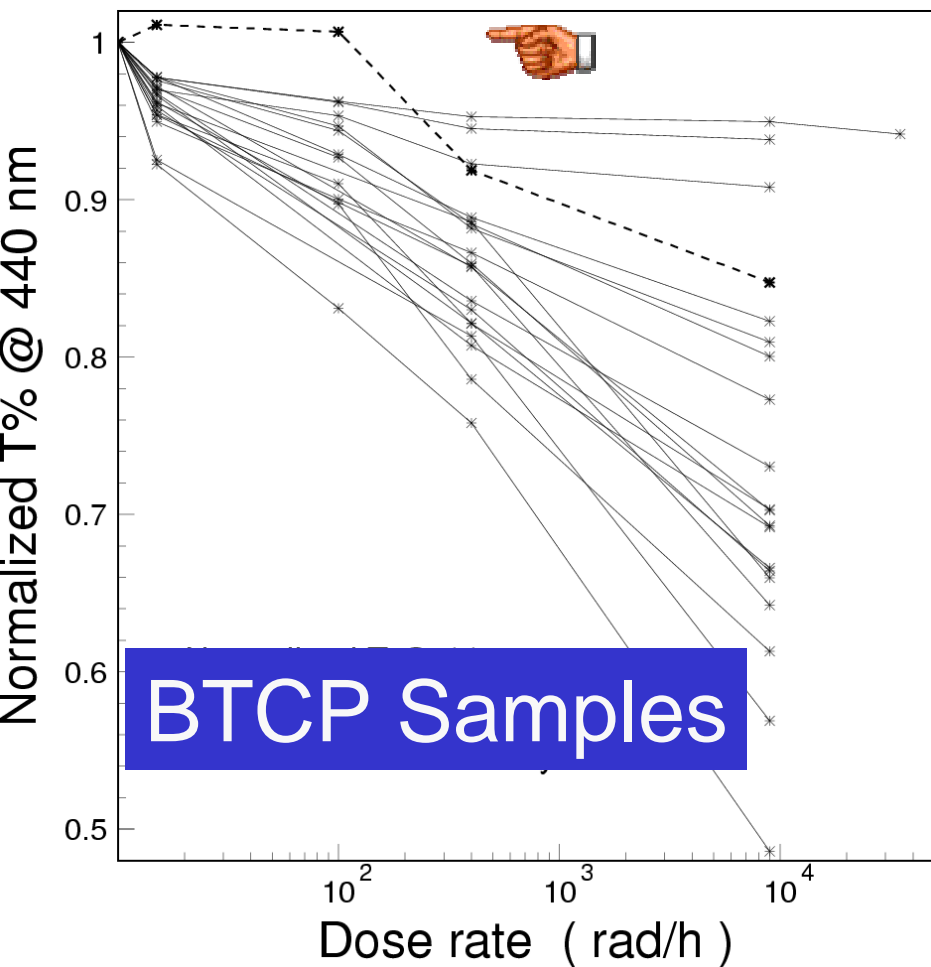




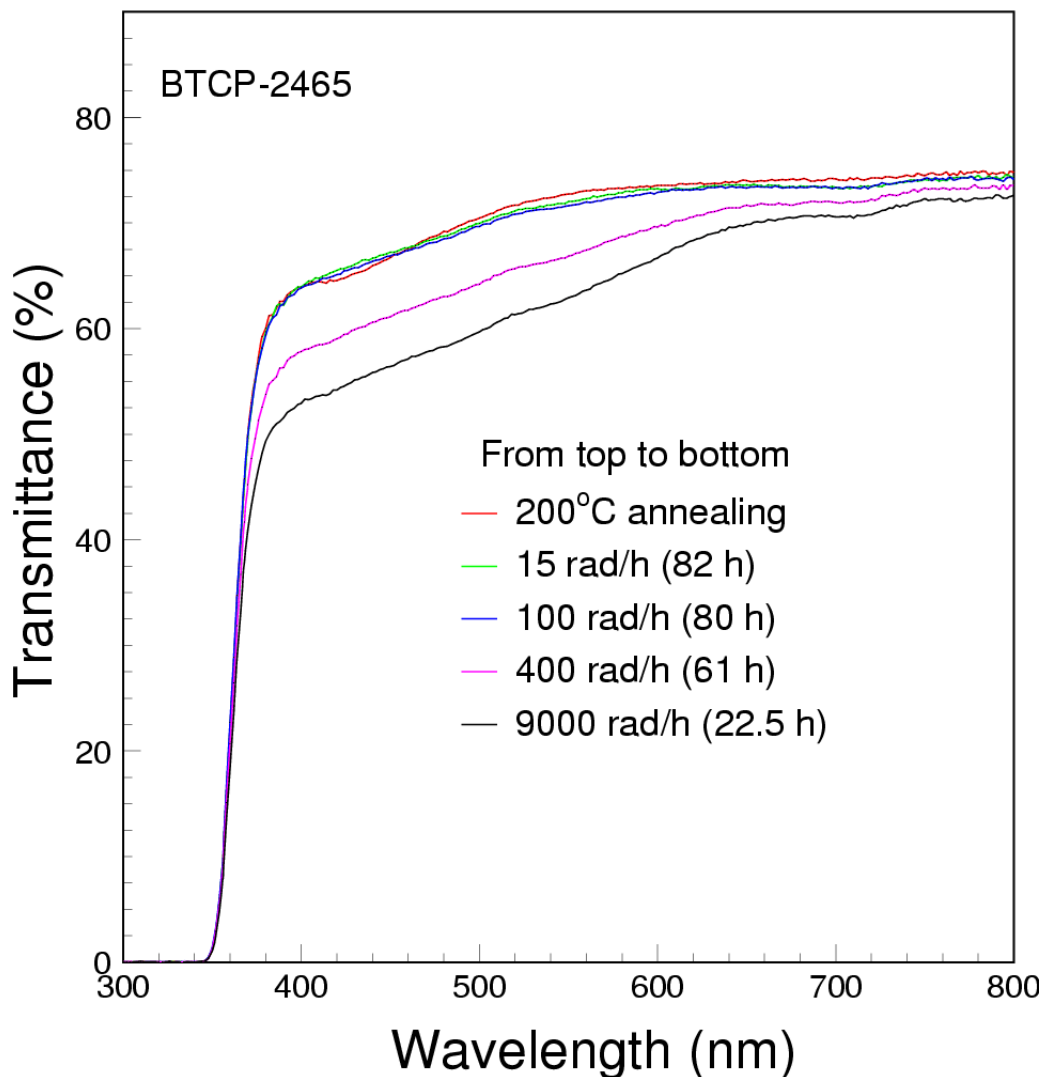
Comparison of Transmittance Loss



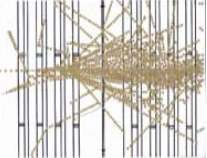
SIC samples less diverse: Bridgman technology
One BTCP sample shows LT increase under irradiation



Type III Sample: Transmittance Loss



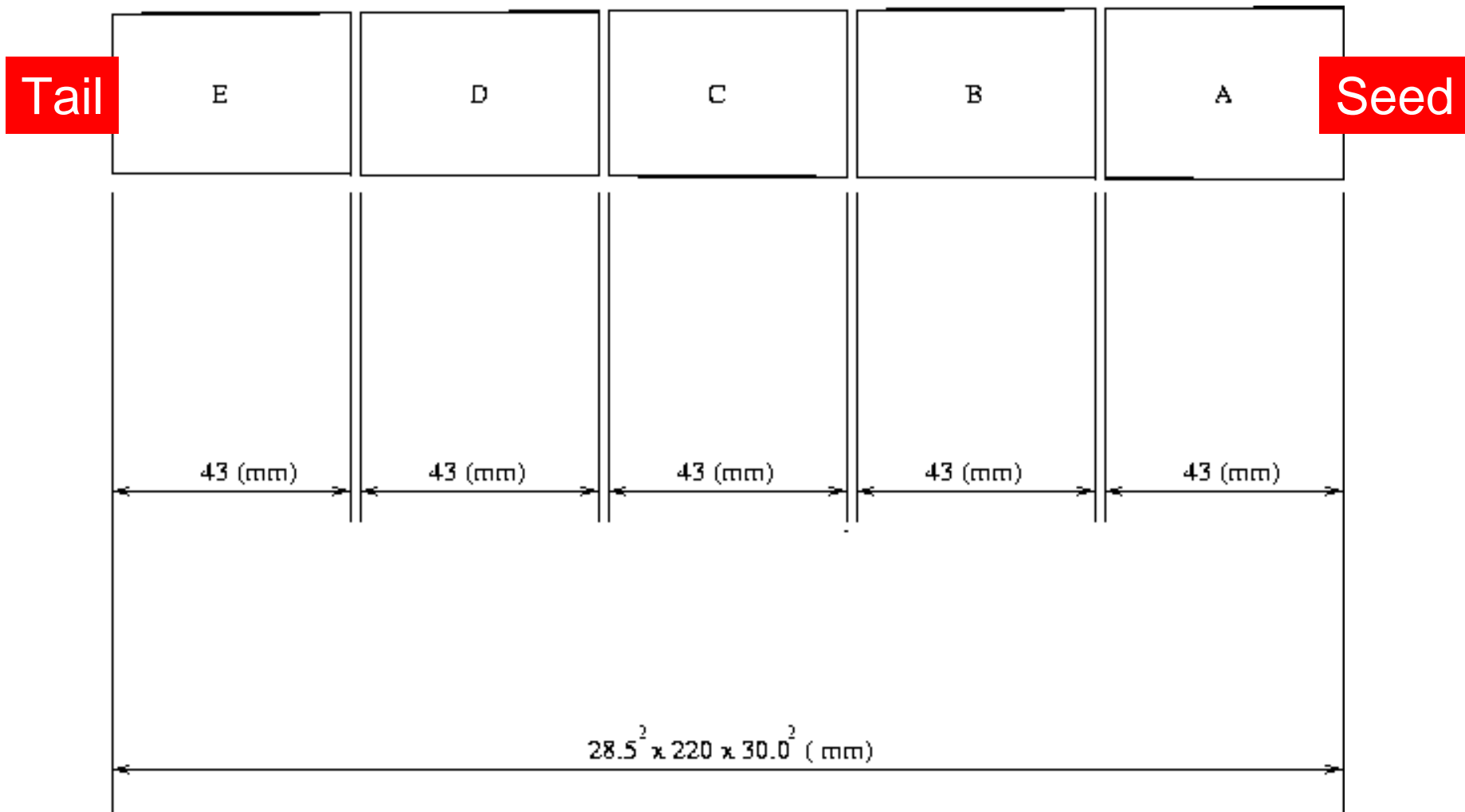
Type III sample:
preexisting
intrinsic color
center at 420 nm
after 200 degree
annealing,
causing difficulty
for monitoring
with 440 nm light

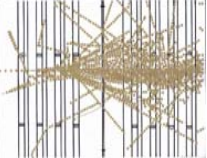


Investigation on BTCP Samples (I)



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

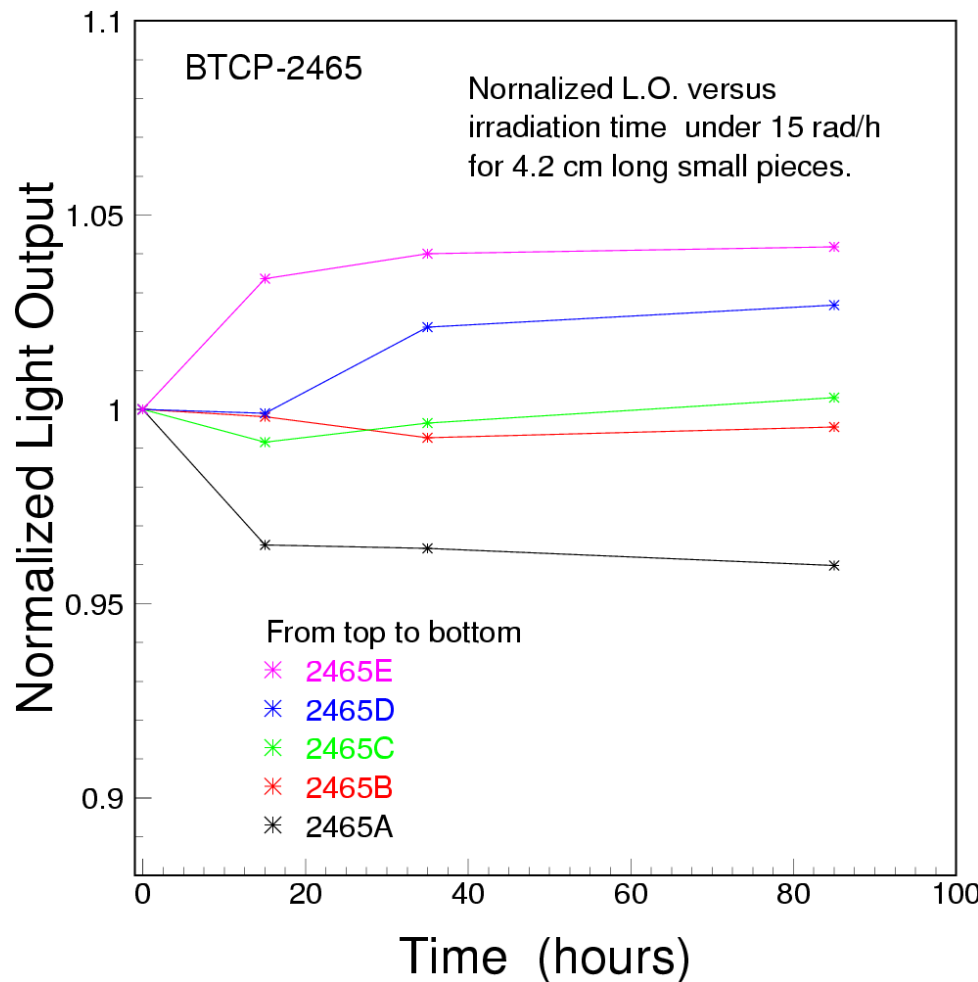
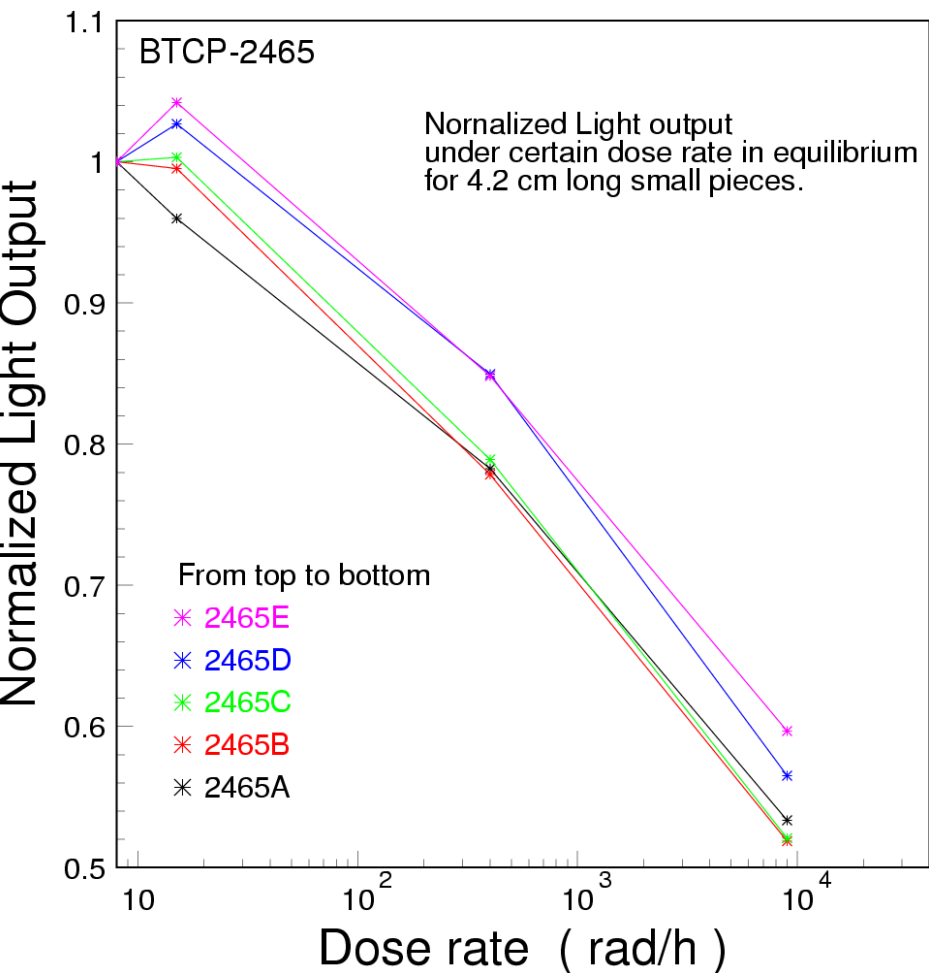


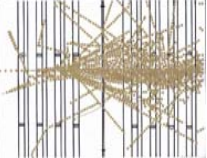


Investigation on BTCP Samples (II)



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





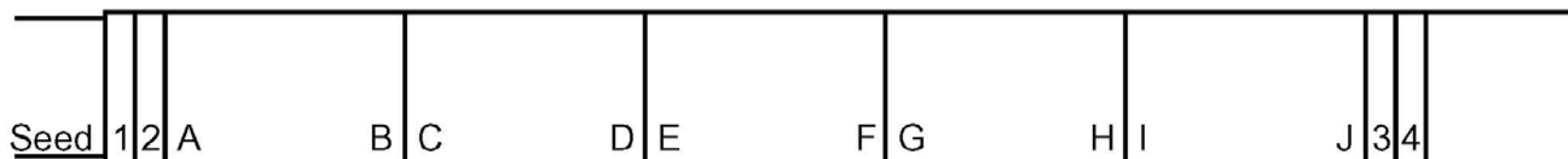
Investigation on SIC Samples (I)



Two anomalous samples were cut to pieces

Crystal ID: NO.4-1-20

Dopant: Y/150 at ppm

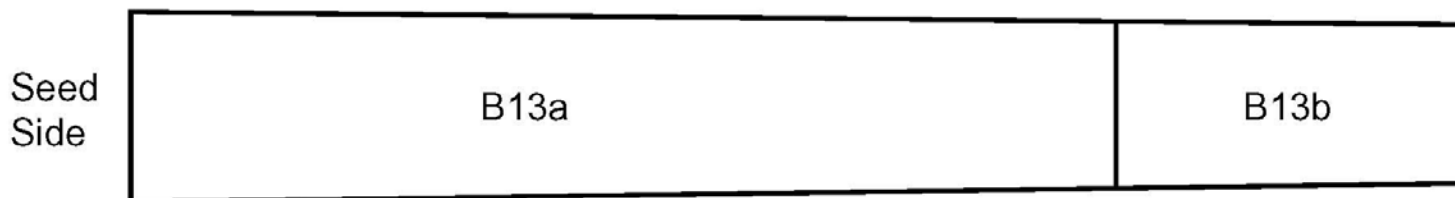


The length of seed is 20.0 mm, thickness of 1, 2, 3, 4 is 5.0 mm.

Dimension of AB, CD, EF, GH and IJ is: $25.0 \times 25.0 \times 44.3 \text{ mm}^3$

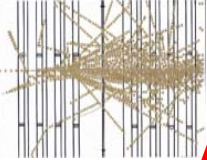
Crystal ID: B13

Dopant: Y/150 at ppm



Dimension of B13a: $22.0 \times 22.0 \times 177.0 \times 25.0 \times 25.0 \text{ mm}^3$

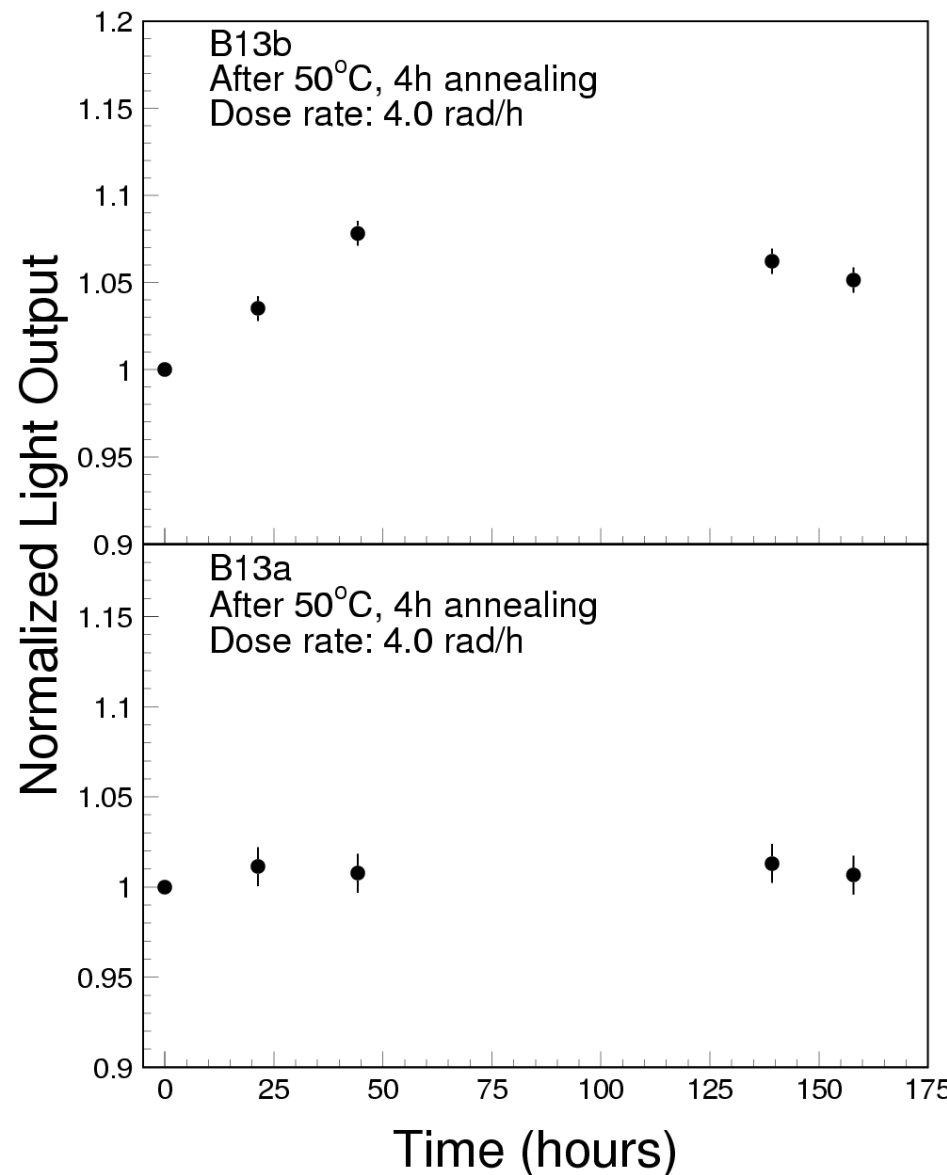
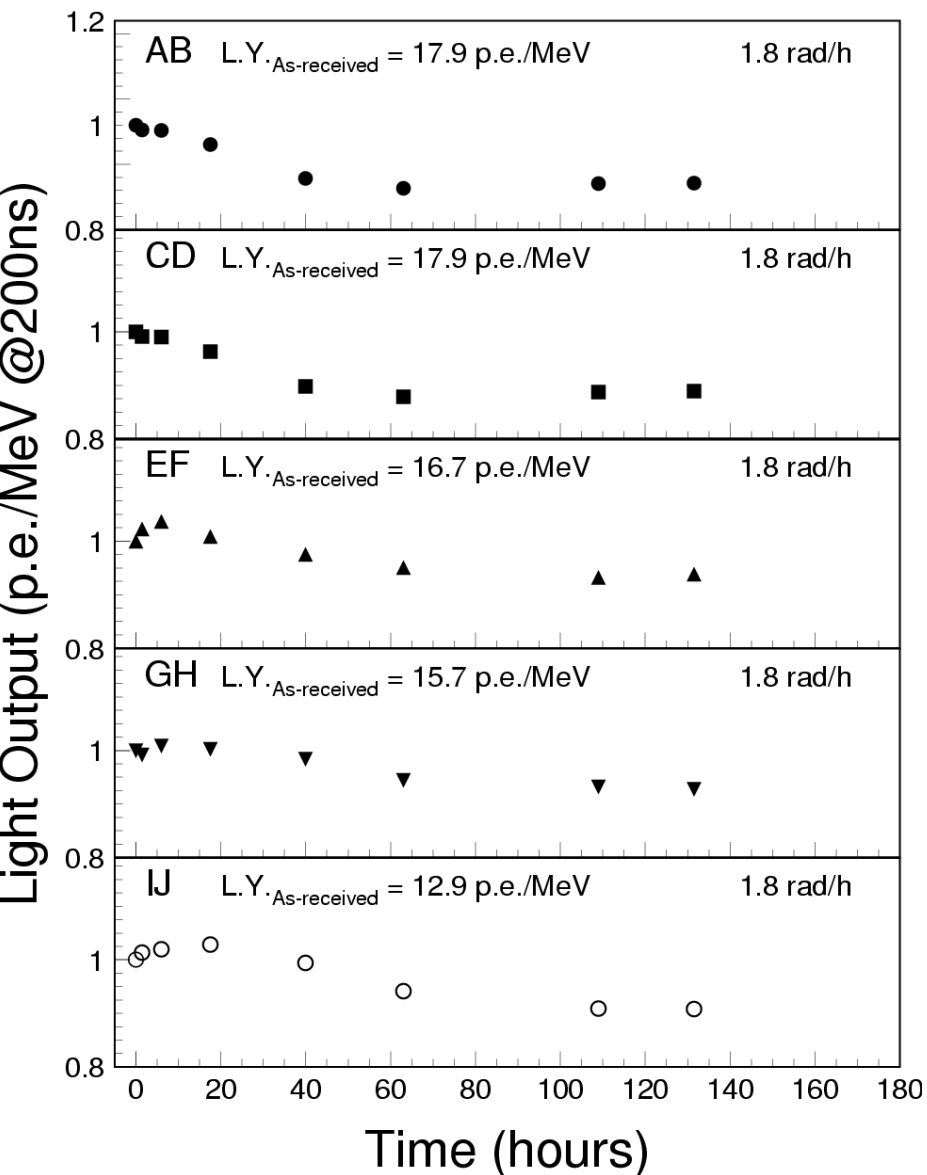
Dimension of B13b: $22.0 \times 22.0 \times 50.0 \times 23.0 \times 23.0 \text{ mm}^3$



Investigation on SIC Samples (II)



Anomaly was found at the tail end: impurity related?



Trace Analysis on SIC Samples



GDMS on SIC PWO(Y) Samples (ppmw)

by Shiva Technology West (November, 1999)

4-1-20-2/3

4-1-20-AB/EF/IJ

Impurity segregation:

Na, K, Cu,
As, Mo: <1;

Ca, Ba: >1;

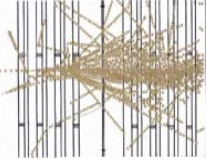
Y: slightly less, but close to 1.

SIC samples are doped with Y only.

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



[†]: Total contamination, excluding Y.



Trace Analysis on BTCP Samples



GDMS on BTCP PWO(Y/Nb/La) Samples (ppmw)

by Shiva Technology (November, 2003)

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



Impurity segregation:

Na, K, Nb, Mo: <1;

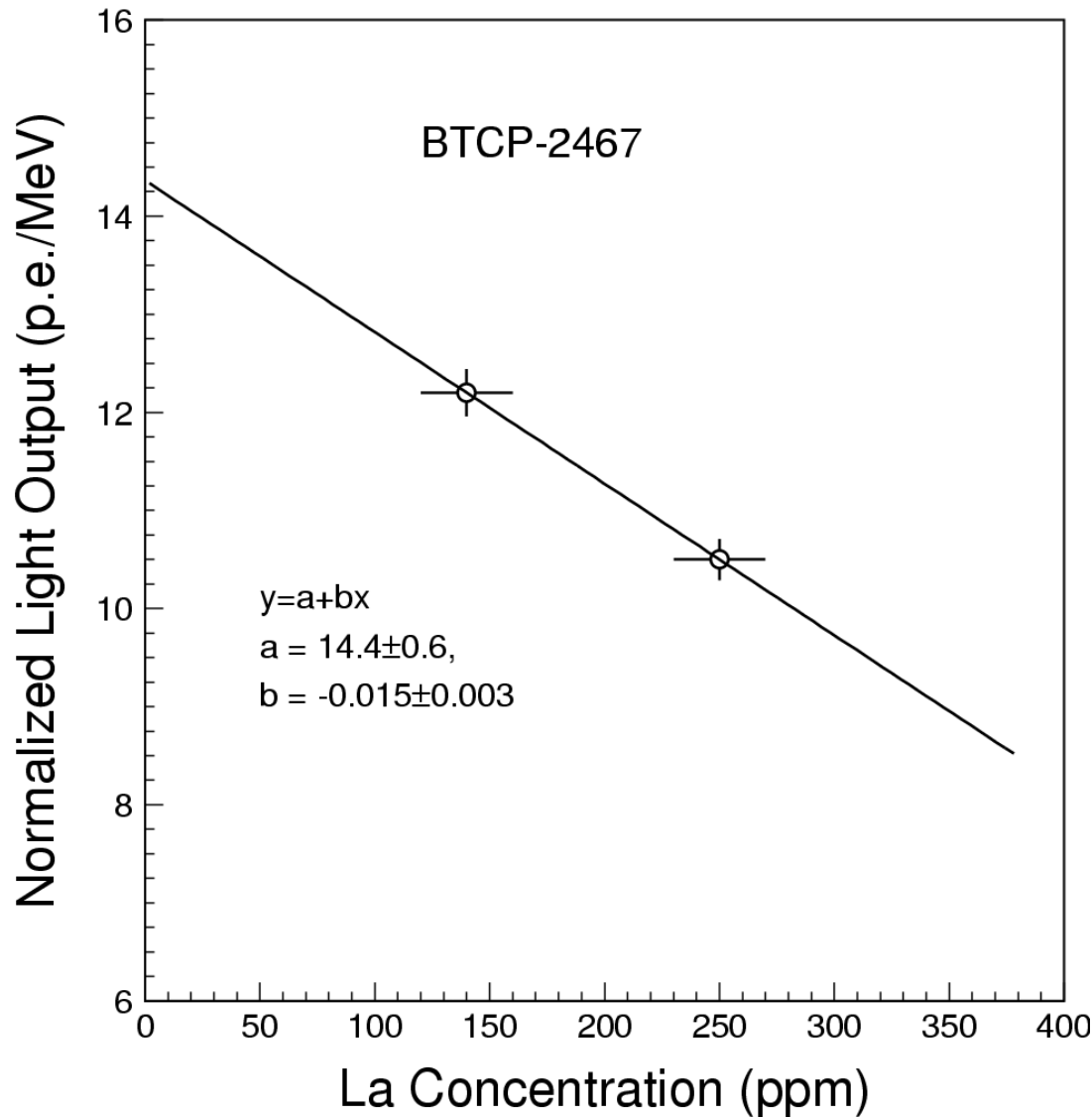
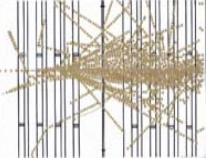
Ca, Ba, La: >1;

Y: slightly less, but close to 1.

BTCP PWO is triple doped with Y/Nb/La!!!

†: Total contamination, excluding Y, Nb and 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.

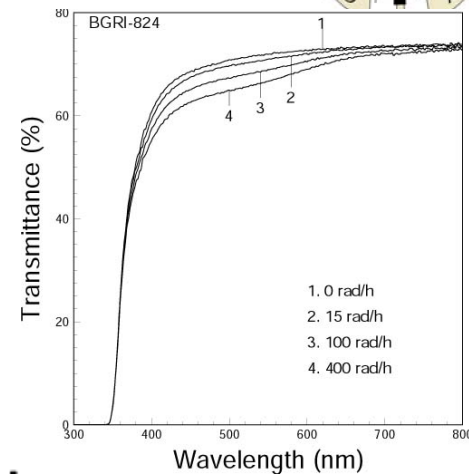
- Further study is under way to clarify this issue.

Radiation Induced Color Center Density



Nucl. Instr. And Meth. A332 (1993) 442

RIAC or radiation induced color center density can be calculated precisely by using longitudinal transmittance (0.2%)



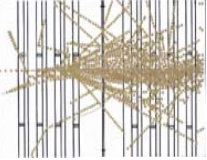
$$RIAC \text{ or } D_{Color-Center} = 1/LAL;$$

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

where T is transmittance measured along crystal length ℓ and T_s is the theoretical transmittance without internal absorption:

$$T_s = (1 - R)^2 + R^2(1 - R)^2 + \dots = (1 - R)/(1 + R), \text{ with}$$

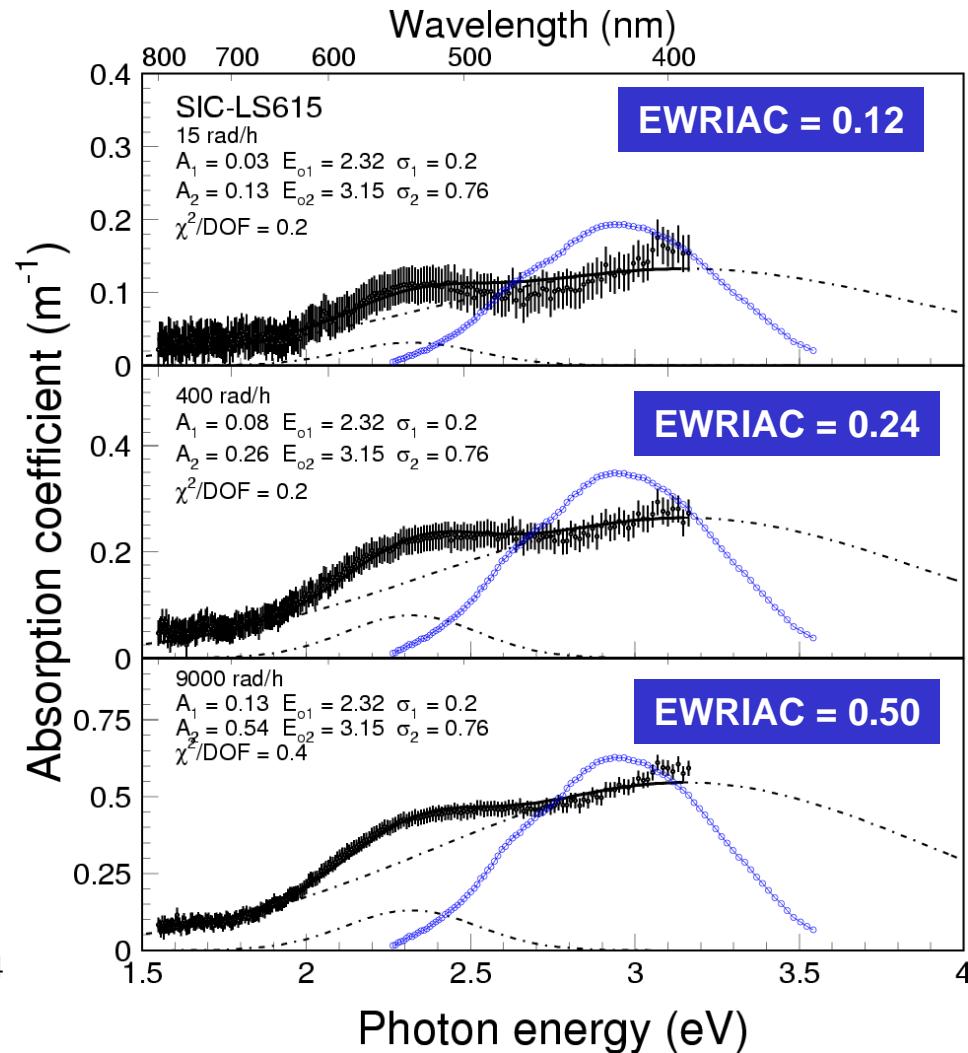
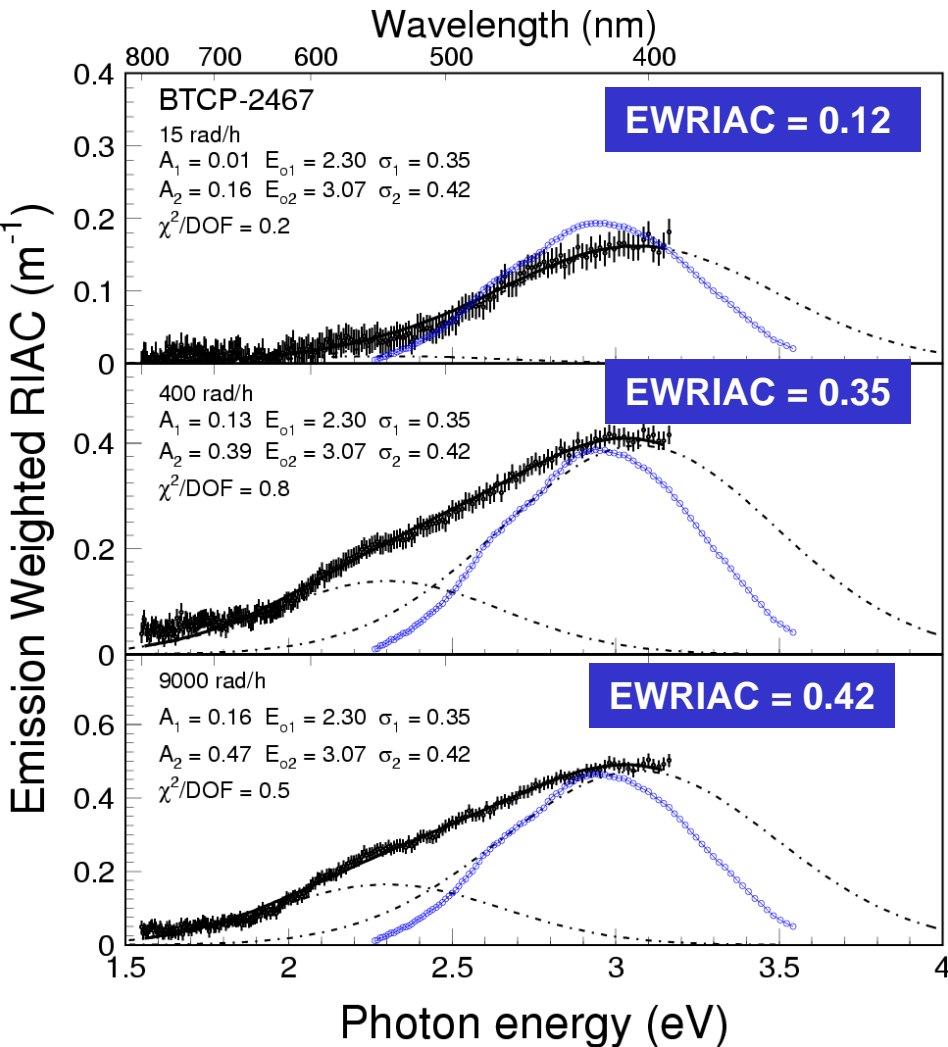
$$R = \frac{(n_{crystal} - n_{air})^2}{(n_{crystal} + n_{air})^2}.$$

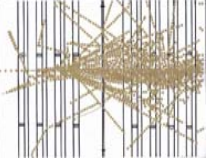


Emission Weighted RIAC



$$EWRIAC = \frac{\int Ri ac(\lambda) Em(\lambda) d\lambda}{\int Em(\lambda) d\lambda} \quad \text{a good measure of rad. damage}$$

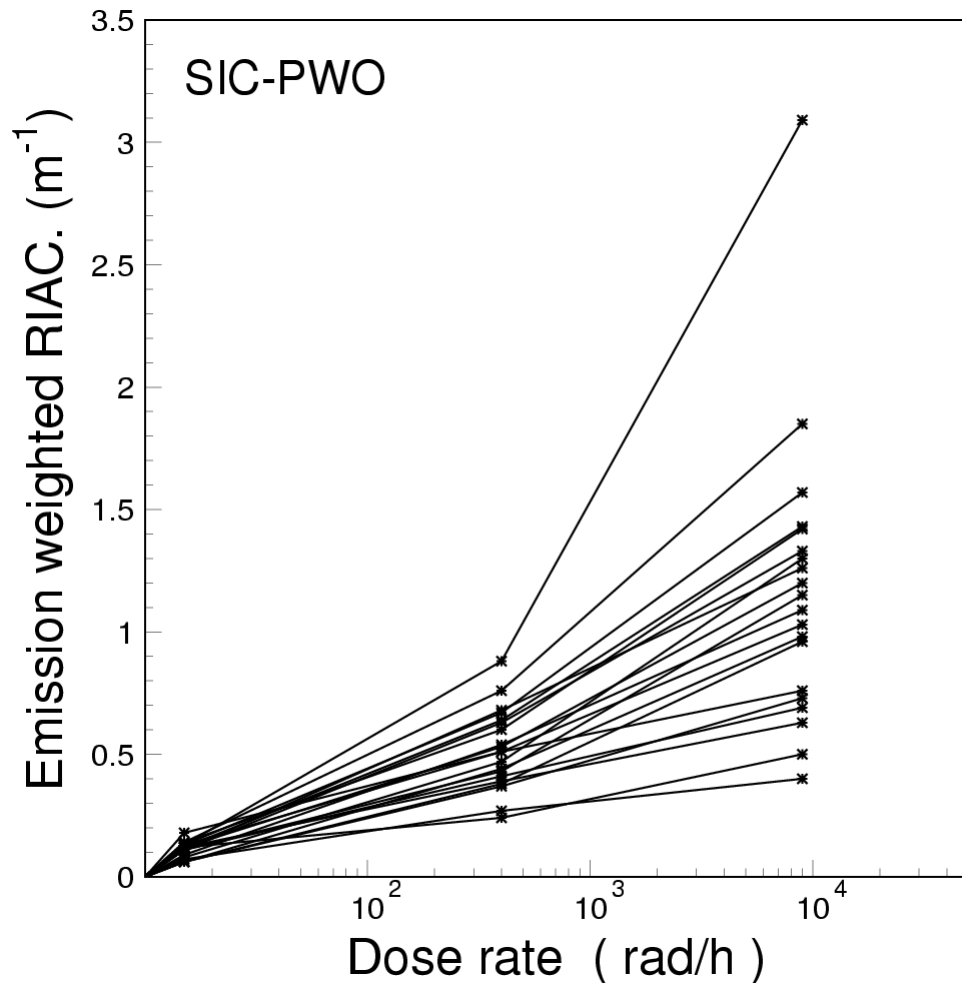
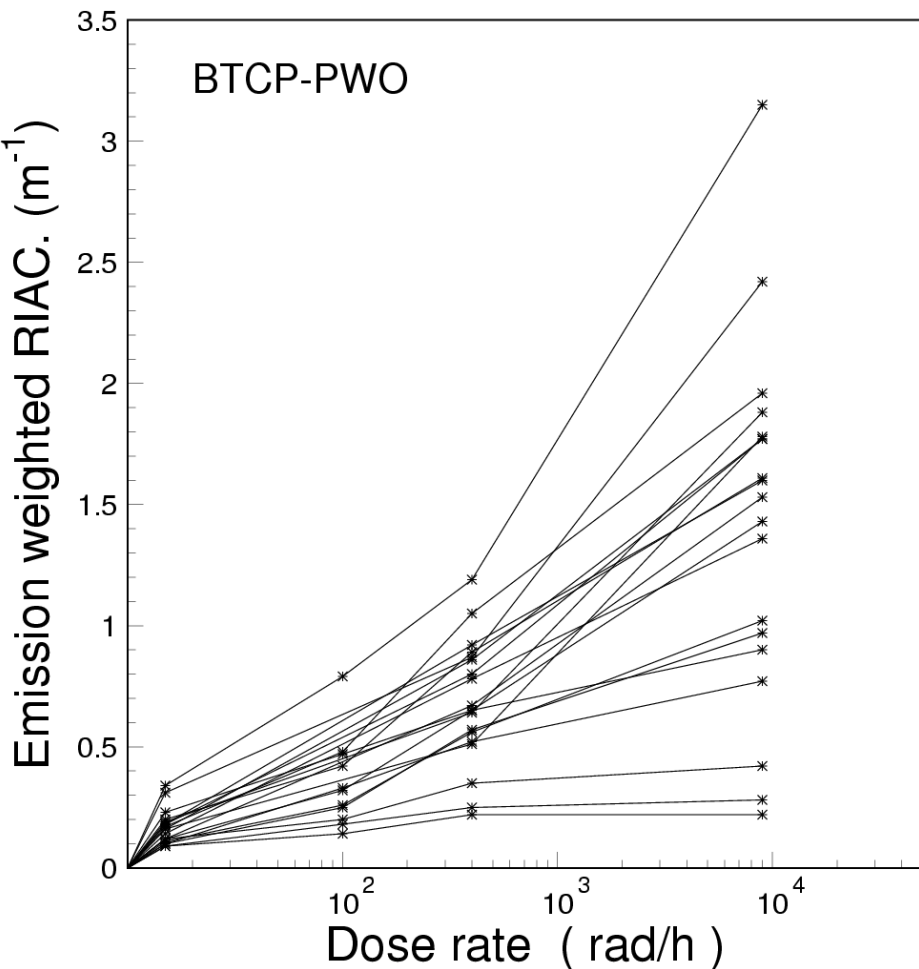


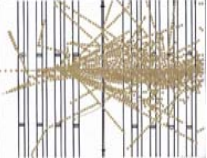


EWRIAC (1/m) and Normalized r.m.s



Vendor	15 rad/h	400 rad/h	9.000 rad/h
BTCP	0.16 (45%)	0.69 (37%)	1.43 (50%)
SIC	0.10 (33%)	0.51 (32%)	1.16 (48%)

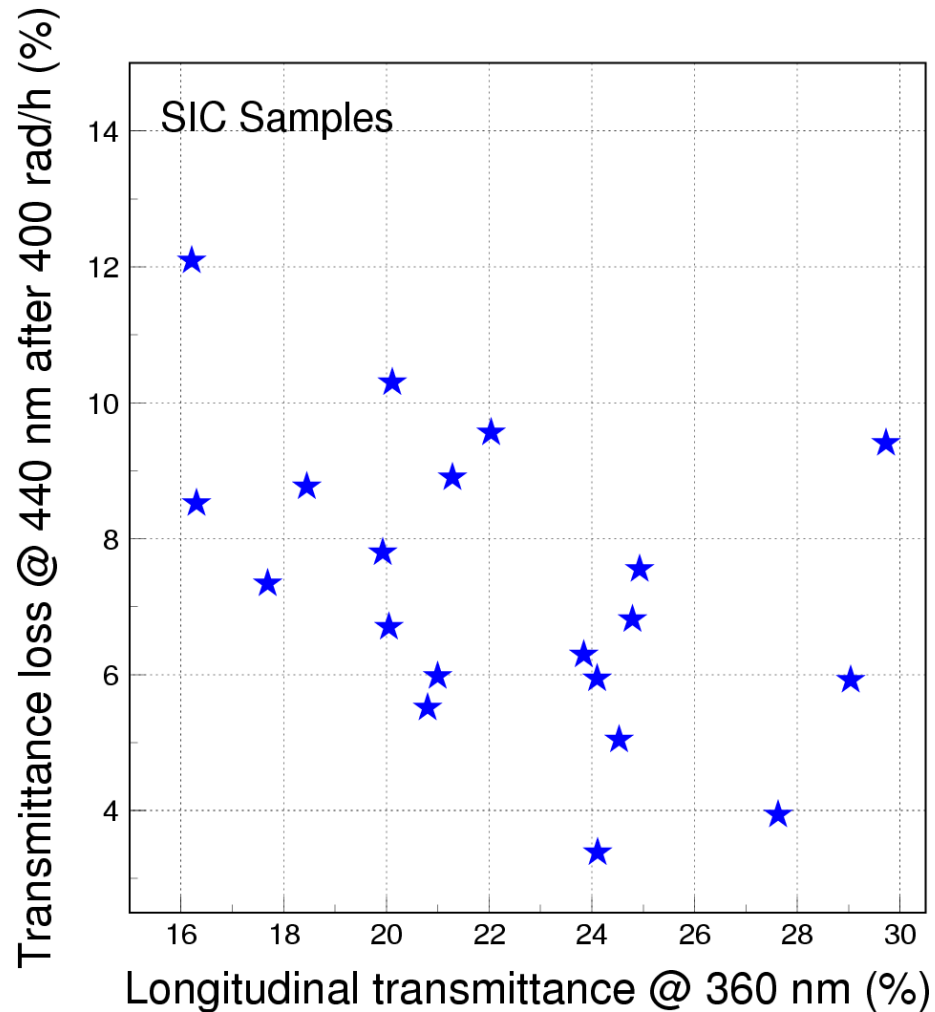
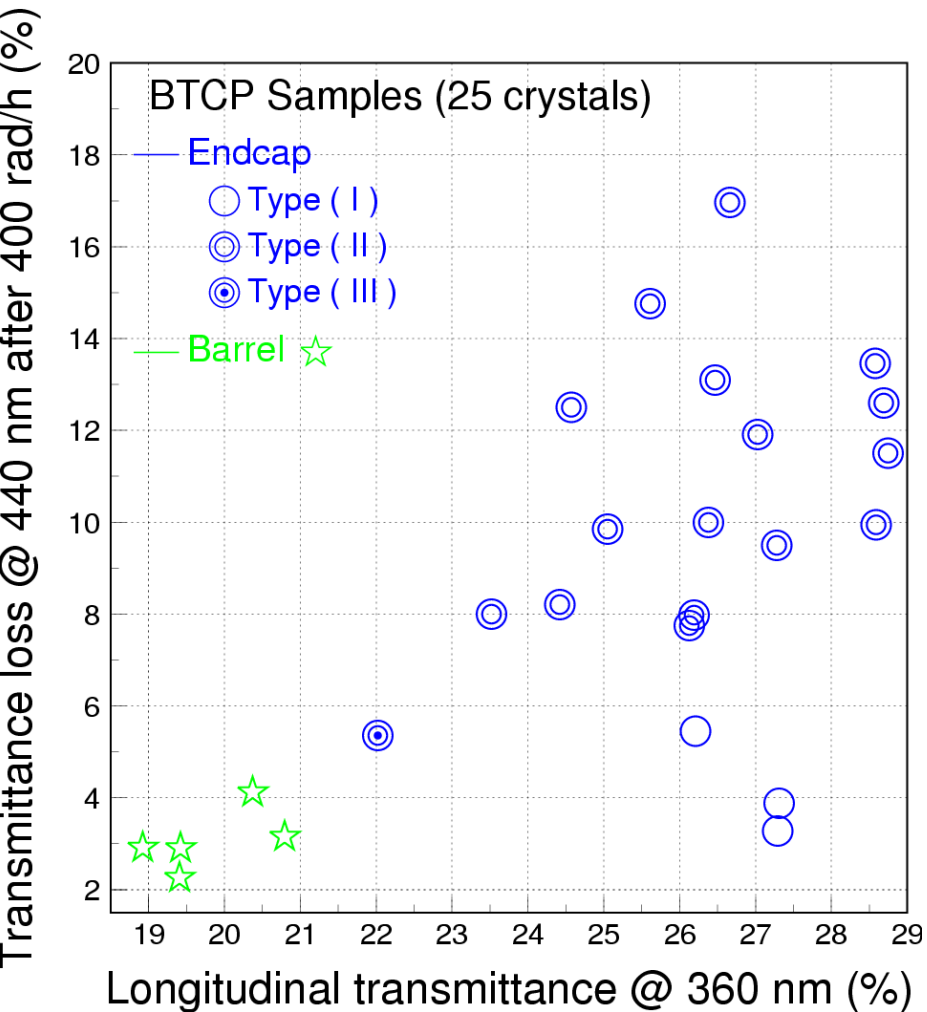


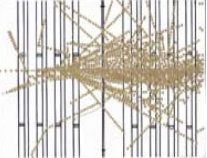


L. T. Loss versus Initial L.T. @ 360 nm



No correlation

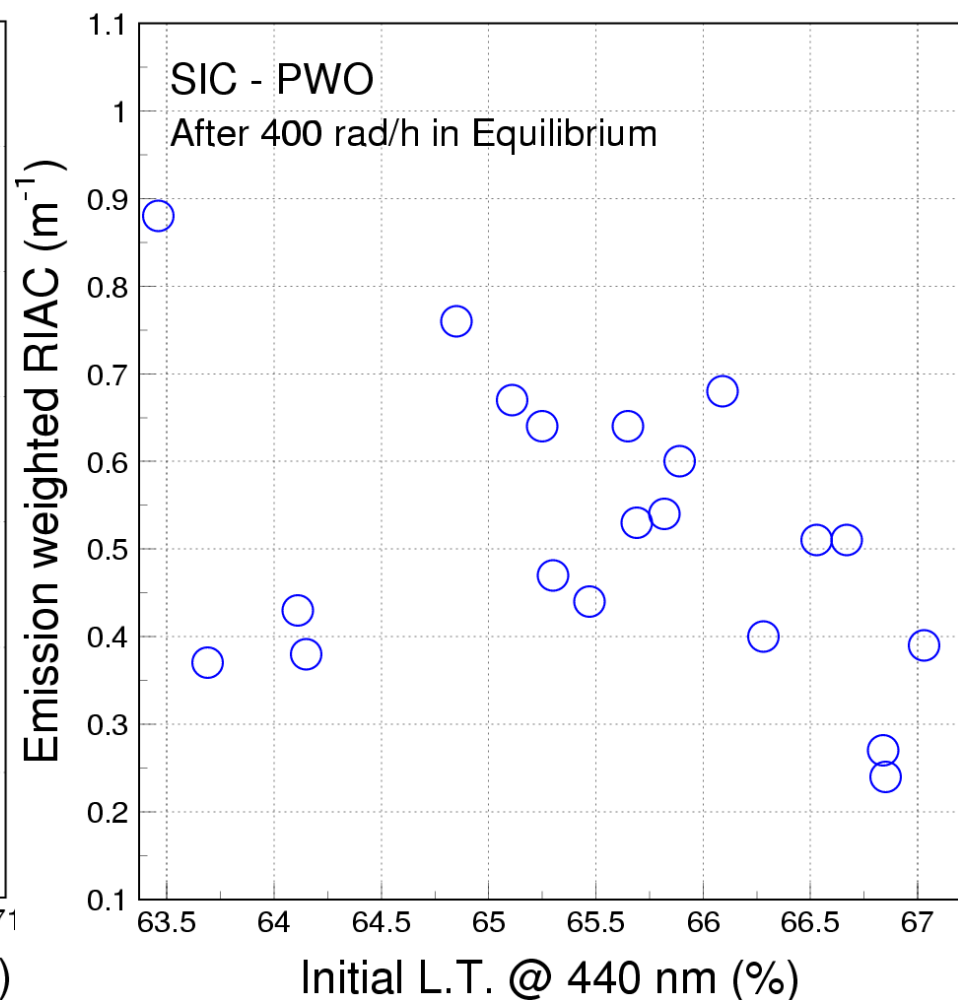
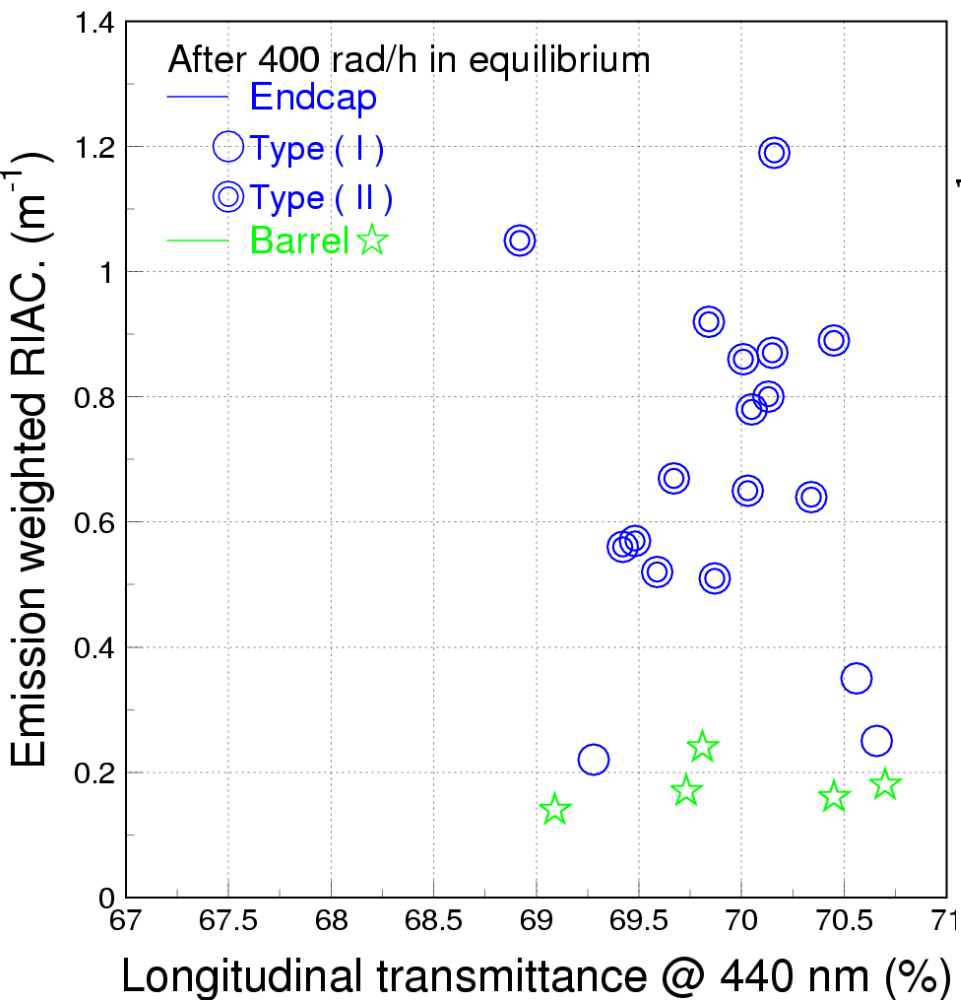


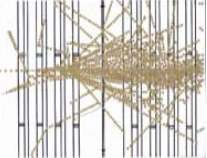


EWRIAC versus Initial L.T. @ 440 nm



No correlation

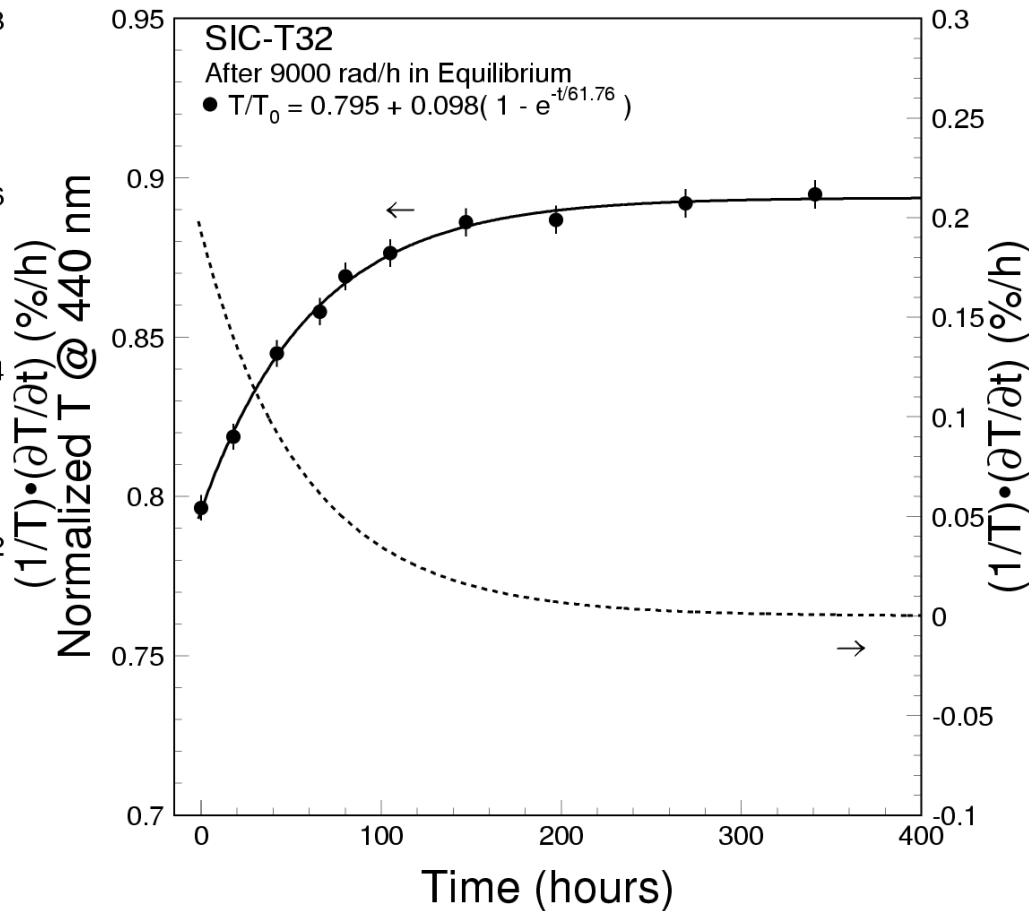
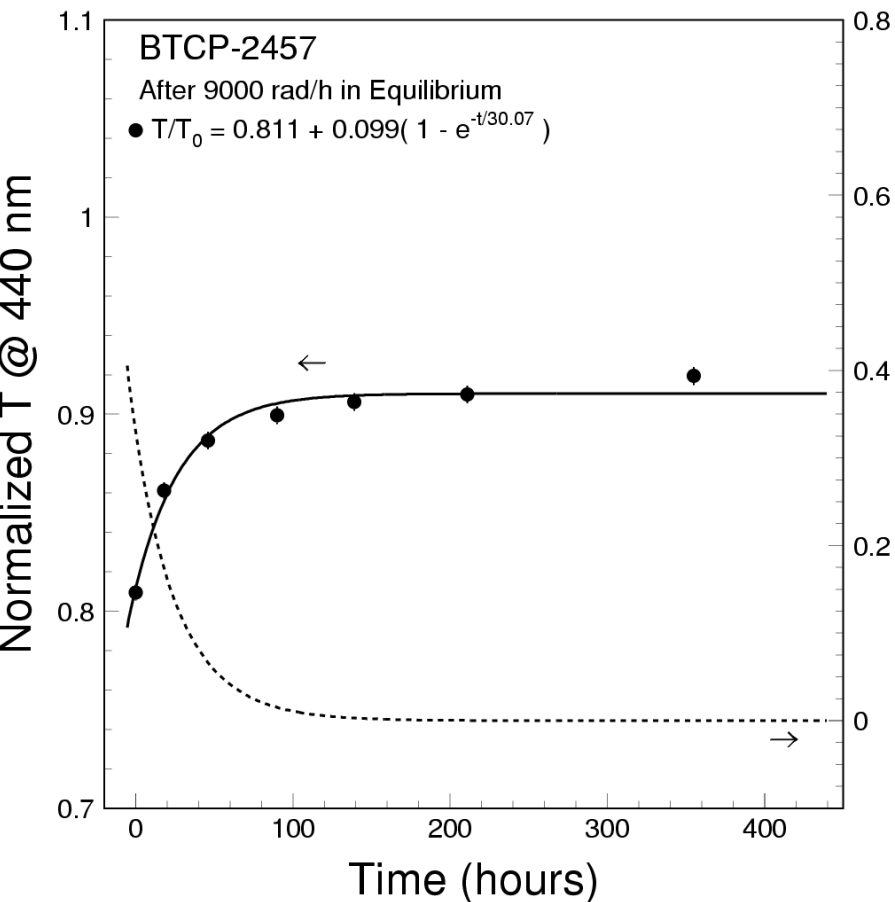


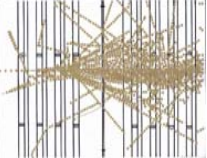


Recovery Speed and Time Constant



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





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.