PbWO4 Studies in 2014/2015 and Plan for 2015/2016

Summary of activities in 2014-2015

- 15 crystals were delivered from SIC in 2014 (#1-10 in spring & #11-15 in December)
- The measured dimensions of crystals are 20 ± 0.15 mm \times 20 ± 0.15 mm \times 200 ± 0.15 mm.
- Visual inspections show that most of crystals were good quality, but some of them have small scratched on side or front surface.
- The crystals were characterized at JLab and cross-checked at Giessen University Caltech and BNL
- Additional infrastructure for crystal testing has been setup at IPN-Orsay and CUA

Optical properties of crystals

- At *JLab* the longitudinal and transverse transmittances for these 15 crystals have been measured. The reproducibility of the measurements is on the order of a few percent. The main uncertainty is the crystal orientation.
 - For most of crystals transmittance starts around 350 nm and reaches to ~90% at $\lambda > 500$ nm.
 - The longitudinal transmittance (20 cm thickness) varies between 60% and 70% for most crystals at a wavelength 420 nm. One of the crystals (#14) shown a completely different behavior above 480 nm.
 - The transmittance in the transverse direction (2 cm thickness) was measured at several distances ranging between 5 and 55 mm from the face of the crystal. The crystal-to-crystal variation of the transverse transmittance seems to be much less than what was observed for the longitudinal transmission. However, the transmittance of one of the crystals (#9) is significantly lower than that of the other crystals.
- Cross checks of subsets of the 15 crystals at other facilities:
 - *Giessen* studies show that longitudinal transmittance of blocks #2, 3, 6, 8 and #9 on average are lower by 10-15% than data measured at JLab. While, for transverse transmittance there are only small difference between Giessen and JLab data.
 - It is interesting to note that there seems to be a significant difference in the shape of the distributions between the *JLab* and *Giessen* in the region around 400 nm.
 - *BNL* data show that longitudinal transmittance of blocks #7 and #15 are lower than data measured at JLab.
 - *Caltech* studies show that sample #11 has significant scattering centers. Sample #5 shows better LT than sample #11 because of less scattering centers.
 - A comparison *Caltech* and *JLab* data for the crystal (#5) shows that transmittance measured at Caltech with integrating sphere is higher than JLab.

Light yield

- Measurements at Giessen
 - the light yield for a subset of 15 crystals (#2, 3, 6, 8 and #9) at room temperature (+18°C) measured and found on the order of 15-16 pe/MeV though some of crystals show a lower yield of only 12 pe/MeV.
 - data also show that the light yield increases by a factor of about three due to cooling to -25 °C.
- Measurements at *CUA* based on radioactive source ²²Na (511 keV photons) have been used to study temperature dependence of the light yield. Data show that LY increases with temperature by ~2.4% per 1°C which is consistent with CMS and Giessen data.
- *Caltech* studies show that light output of crystals #5 (15.6 p.e./MeV) and #11 (12.4 p.e./MeV) are better than CMS SIC (11.4 p.e./MeV).

Timing

• *Caltech* studies show that two crystals (#5 and #11) are faster than CMS PWO crystals. Fraction of Light Output (LO) in 50/100 ns for sample #5 and #11 is 91/99%, while for CMS crystals is 84/96%.

Radiation hardness

- At *JLab* irradiation using γ -source (single source ¹³⁷Cs) with a dose rate of ~260 rad/h. No sign of radiation damage was observed up to ~300 krad
- At *Idaho* irradiation with 20 MeV electron beam at dose rate 1.3 Mrad/h show high resistance of the SIC crystals.
 - The transmittance of some of crystals (#2, 4 and #8) changed ~15% after accumulated dose 432 krad (at dose rate of 1.3 Mrad/h), while others (#3, 5 and #6) do not seems show any effects of radiation damage.
 - To minimize systematic effects due to recovery of color centers with fast relaxation time all transmittance measurements were carried out 10 minutes after exposures. No effort was made to keep the crystals away from light between end of irradiation and transmittance measurement.
 - Challenge of irradiation tests with beam: temperature control. Difficult to achieve with intense beams, which give high and concentrated dose to crystals and can even result in heating and thermal damage. For example, for irradiation at dose rate 1.3 Mrad/h for 20 min exposure the temperature near the crystal front increased from ~74°F to ~98°F. For similar exposure time at dose rate 2.6 Mrad/h the temperature increases from 76°F to 115°F resulting in crystal damage.
- At *Giessen* the longitudinal transmittance for subset of crystals (#2, 3, 6, 8 and #9) was determined before and after gamma irradiation (5 radioactive sources ⁶⁰Co) with an integral dose of 30 Gy within 15 minutes. The impact of radiation effects were quantified in terms of the change in the absorption coefficient *k*, and found to be from $dk\sim0.4 m^{-1}$ (crystal #03) to $\sim1.3 m^{-1}$ (crystal #02), which is better than CMS quality requirement ($\sim1.6 m^{-1}$).

Methods of crystal recovery

- Stimulated recovery: Two curing strategies were tested: 1) IR curing with 4 LEDs (950 nm) at current 75 mA and distance 1 cm for 12 hrs, 2) Blue light curing with 4 LEDs (360 nm) at current 50 mA for 3 hrs. Curing does not seem to recover the optical quality of crystal #2 irradiated at ~2 Mrad radiation dose.
- *Thermal annealing* for 10 hrs at 200°C (with ramp up-down rate 20°C/h) has been performed for all 15 crystals.

Summary of 2014/2015 studies

• Our studies of a set of fifteen crystals produced by SIC in 2014 seem to indicate that the overall quality mostly conform to CMS/PANDA requirements.

Crystal	LT(%)	LT(%)	LT(%)	LY(p.e.)	dLY/dT	LY(50ns)	dk(m ⁻¹)	dk(m ⁻¹)	dk(m-1)	Test
ID	360nm	420nm	600nm	+18°C	(%/1°C)	LY(100ns)	360nm	420nm	600nm	facility
						(%)				
SIC-1	28.6	65.4	72.7	-	-	-	-	4.69	1.07	JLab
SIC-2	27.7	62.6	70.4	-	-	-	-	7.24	3.79	JLab
	-	-	-	-	-	-	-	1.3	0.72	Giessen
SIC-3	33.5	67.7	74.6	-	-	-	-	0.80	0.18	JLab
	-	-	-	14.3	-	-	-	0.38	0.2	Giessen
SIC-4	32.6	66.7	73.9	-	-	-	-	3.37	2.55	JLab
SIC-5	33.3	66.0	74.1	-	-	-	-	0.14	0.54	JLab
	35.7	62.0	73.7	15.6	-	91/99	-	-	-	CalTech
SIC-6	42.6	65.7	73.4	-	-	-	-	0.56	? (<0)	JLab
	-	-	-	-	-	-	-	0.72	0.4	Giessen
SIC-7	29.1	66.0	73.6	-	-	-	-	1.08	0.77	JLab
SIC-8	36.3	66.1	73.7	-	-	-	-	4.34	4.35	JLab
	34.0	56.0	69.0	-	-	-	-	0.5	0.28	Giessen
SIC-9	32.2	66.1	73.3	-	-	-	-	2.26	1.19	JLab
	-	-	-	-	-	-	-	0.85	0.5	Giessen
SIC-10	23.8	66.8	74.1	-	-	-	-	1.79	0.08	JLab
SIC-11	27.1	62.4	70.2	-	-	-	-	-	-	JLab
	28.7	63.0	70.6	12.4*	-	91/99	-	-	-	CalTech
SIC-12	40.7	70.8	75.3	-	-	-	-	-	-	JLab
SIC-13	31.1	64.1	72.5	-	-	-	-	-	-	JLab
SIC-14	37.0	67.1	55.8	-	-	-	-	-	-	JLab
SIC-15	33.6	67.2	74.0	-	-	-	-	-	-	JLab

Table 1: The absorption coefficient is calculated from

$$dk = \ln \frac{T_0}{T} \frac{1}{d},$$

where T_0 and T are the measured transmittance before and after irradiation respectively and d is the distance over which the measurement was performed. At Giessen dk was determined from measurements of the longitudinal transmittance (d= 20 cm) before and after uniform gamma irradiation (5 radioactive sources ⁶⁰Co) with an integral dose of 3 krad (30 Gy) with a dose rate ~12 krad/h. The JLab data are based on transverse transmittance measurements (d= 2.0 cm thickness at 5 mm distance from the frontal face) before and after frontal irradiation of the crystals with electron beam (at Idaho) with an integral dose of 432 krad with a dose rate ~1.3 Mrad/h.

The results seem to suggest that dk is much higher for the JLab than the Giessen measurements. This could indicate that the rates in the JLab measurements are higher, and in particular in the first few cm of the crystal. It could also suggest that the measurements are not compatible due to the different types of irradiation. It should be noted that the general shapes of the transmittance spectra are consistent.

- The optical transmittance seems consistent with CMS quality and relatively uniform along the crystals. However, there is a global variation from crystal to crystal on the order of 20%.
- The light yield of the crystals also seems to be consistent with CMS quality standards (12-16 p.e./MeV).
- Understanding the effect of systematics on the optical measurements is important for the interpretation of crystal quality.
- Need to understand setup-dependent systematic effects, e.g.,
 - Results from Caltech suggest that the measured values of the transmittance are higher than those for JLab measurement.
 - Results from measurements at Giessen and BNL show similar features in that the measured values of the transmittance are lower than those for the JLab measurement.
- A quantitative analysis of the homogeneity of the crystals must be done to fully characterize the crystal quality achievable.

Plan for 2015/2016

30 more crystals were ordered from SIC in spring 2015 Planning to procure crystals from CRYTUR as part of EIC R&D

- Need full information about Composition of the crystals
- Chemical composition of raw materials. (Need information from SIC).
- Details of growing process. Check: Are all crystals made from the same raw material? (Need information from SIC).
- Check Geometrical accuracy and surface quality of the crystals
- Visual inspection, cracks. (Check at JLab upon delivery)
- Tolerance in dimensions. (Define at JLab upon delivery)
- Surface quality/roughness. (Need information from SIC)
- Check crystals intrinsic radioactivity
- Activity of the crystals and decay products (Not a first priority but interesting!) The intrinsic radioactivity of the crystals can be tested by either using them as scintillator at sea level or using ultra-low background HPGe γ-spectrometry.

Comment [h1]: For all following bullets: where will these tests be carried out? And by which group(s)?

Intrinsic radioactivity of the crystals is not a critical problem for high energy calorimetry though it is the main limiting factor for low-background experiments (such as double β -decay, dark matter search). Nevertheless, the use a low-radioactive raw material or purified materials for growing crystals is preferable in all applications. Radioactive background levels of PbWO crystals are due to the presence of isotope ¹¹⁰Pb and contaminations (isotopes Mo, Nb, ...). Note, to address this issue BTCP carried out special work on purification of archeological lead (discovered in Ukraine) for growth of PbWO crystals with low radioactivity background.

- Light yield measurements
- Photon per 1 MeV
- Temperature dependence
- Timing quality
- o LY(100 ns)/LY(1 μs)
- Optical Transmission
- o Study accuracy of the measurements
- Longitudinal transmission
- Transverse transmission (at 5-6 positions along the crystal)
- Radiation hardness
- \circ Irradiation with γ -source
- Irradiation with electron beam

In general, irradiation with electron beam and with γ -source must give similar results if the crystals are uniformly irradiated (not only from the face) and similar procedures are followed during irradiation and transmittance measurements. Special infrastructure could allow more uniform irradiation with electron beam. If similar conditions are ensured then only one of these irradiation tests is necessary.

o Transmission measurements before and after irradiation

Spontaneous recovery of radiation damage and curing

- Spontaneous recovery of the radiation induced damage (dk)
- o Blue and Infra-red curing (Stimulated recovery)

Since our studies of SIC 10 crystals show that even at high dose rates and accumulated doses the effect of radiation damage is very small (below 15%), tests of stimulated curing must be done only after measuring method will be improved!

- Photo-sensors for NPS (selection of option)
- o Phototube: geometric efficiency, QE, Gain, dark current, rate/timing, price/channel
- o LAAPD: geometric efficiency, QE, Gain, dark current, rate/timing, price/channel
- NPS prototype tests

Comment [h2]: Are both tests with beam and with source needed?

- \circ $\;$ Improve/Modify existing 3×3 prototype or build new 5×5 prototype
- Study energy resolution
- Calibration and monitoring
- Temperature-controlled frame

PbWO4 crystal qua	lity spec	ifications f	or mass p	production	order

Parameter	Unit	NPS	NPS	CMS	PANDA	Inform.
		Required	Acceptable	Limit	Limit	Source
Light Yield (LY) at RT	pe/MeV	≥15	≥10	≥8	≥16	Test with
(for all sides polished crystals)						y-source
LY uniformity between blocks	%	10%	20%			Test
LY(100ns)/LY(1µs)	%	>95	>90	>90	>90	Test
Longitudinal Transmission						Optic.
at λ=360 nm	%			≥25	≥35	Measure.
at λ=420 nm	%			≥55	≥60	
at λ=620 nm	%			≥65	≥70	
Transverse Transmission and LY	%	10	15			Optic.
uniformity along crystal						Measure.
Inhomogeneity of Transverse	nm	≤5	≤10	≤3	≤3	Optic.
Transmission $\Delta\lambda$ at T=50%						Measure.
Induced radiation absorption						
coefficient Δk at λ =420 nm and	m ⁻¹	<1.0	<1.5	≤1.6	≤1.1	Test
RT, for integral dose >100 Gy						
Mean value of dk	m -1	≤0.75	≤1.0	≤1.5	≤0.75	Test
Tolerance in Length	μm	≤±100 -	≤±150	+0., -100	±50	Measure.
Tolerance in sides	μm	≤±50	≤±100	≤ ±50	±50	
Surface polished, roughness Ra	μm	≤0.02	≤0.05	≤0.02		Company
Tolerance in Rectangularity (90°)	degree	≤0.1	≤0.2	≤0.12	<0.01	Measure.
Purity specific. (raw material)				5N-6N		Company
Mo contamination	ppm	<10		<10	<1	Company
La, Y, Nb, Lu contamination	ppm	?		≤100	≤40	Company