An Investigation of X-ray Luminosity versus Crystalline Powder Granularity

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***Abstract*—At the High-throughput Discovery of Scintillator Materials Facility at Lawrence Berkeley National Laboratory, scintillators are synthesized by solid-state reaction or melt mixing, forming crystalline powders. These powders are formed in various granularity and the crystal grain size affects the apparent luminosity of the scintillator. To accurately predict a "full-size" scintillator's crystal luminosity, the crystal luminosity as a function of crystal granularity size has to be known. In this study, we examine $Bi_4Ge_3O_{12}$ (BGO), Lu_2SiO_5:Ce (LSO), $YAlO_3$:Ce (YAP:Ce), and $CsBa_2I_5:Eu^{2+}$ (CBI) luminosities as a function of crystalline grain size. The highest luminosities were measured for 600- to 1000-μm crystal grain sizes for BGO and LSO, for 310- to 600-μm crystal grain sizes for CBI, and for crystal grains larger than 165 μm for YAP:Ce. Crystal grains that were larger than 1 mm had a lower packing fraction, and smaller grains were affected by internal scattering. We measured a 34% decrease in luminosity for BGO when decreasing from the 600- to 1000-μm crystal grain size range down to the 20- to 36-μm range. The corresponding luminosity decrease for LSO was 44% for the same grain size decrease. YAP:Ce exhibited a luminosity decrease of 47% when the grain size decreased from the 165- to 310-μm crystal grains to the 20- to 36-μm range, and CBI exhibited a luminosity decrease of 98% when the grain size decreased from the 310- to 600-μm crystal grain range to the 36- to 50-μm range. We were able to very accurately estimate full-size crystal luminosities from crystalline grains that are larger than 90 μm.**

***Keywords*—Bismuth germanate, $Bi_4Ge_3O_{12}$ (BGO), Lutetium oxyorthosilicate, Lu_2SiO_5:Ce (LSO), Yttrium aluminum perovskite, $YAlO_3$:Ce (YAP:Ce), Cesium diabarium iodide ($CsBa_2I_5:Eu^{2+}$)**

1. INTRODUCTION

There is an undergoing surge for discovering new scintillator materials for detection of ionizing radiation. New scintillators are needed in national security, high-energy physics, and medical imaging applications, ranging in uses from detecting nuclear threats at our borders (by improving energy resolution and lowering scintillator costs), to enabling the constructions of time-of-flight PET scanners (by improving timing resolution). The search is currently underway at several national laboratories, including Lawrence Berkeley National Laboratory (LBNL) at the High-Throughput Discovery of Scintillating Materials (HTSD) Facility [1]. At LBNL, scintillating materials are synthesized from high-purity components in computer-controlled furnaces, through solid-state or melt reactions, and the resulting powders are screened in the HTSD Facility for scintillator characteristic properties such as optical excitation luminescence, x-ray luminescence, and scintillation light decay times. The reason powders are synthesized is that it is faster to synthesize a crystalline powder using a solid-state reaction or by melt mixing than to grow a single crystal, thereby allowing us to quickly evaluate a compound for its potential as a scintillator crystal.

For a large scintillation crystal covered with a good reflector, a large portion of the light will reach the photodetector. In this work the scintillator samples are created in the form of crystalline powders, which are held in cuvettes, and the emission light from the crystalline grains is collected with lenses that direct the light into a spectrometer. The solid angle (~45º half-angle) of the first collection lens only collects a fraction of the emission light and there is no reflector attached to the back of the cuvettes, and hence, most of the light will not make it to the photodetector. This light reducing effect can be corrected for since all the samples are measured in the same setup and all the luminosities will have the same geometric light collection efficiency.

The crystalline powders have grain sizes ranging from a few micrometers to a few millimeters and their size has a large effect on the fraction of light detected. For example, for very fine particles light emitted from the deeper layers will be scattered and absorbed, decreasing the apparent luminosity. To accurately estimate the luminosity of a future full-size grown crystal of the same material, the need therefore exists to estimate how the powder's grain size affects the apparent luminosity, and from these results be able to get an accurate estimate of a larger crystal's luminescence. The aim of this work is to examine the crystalline granular size influence on the measured luminosity, and use the results in correcting the measured powder luminosities to estimate larger crystal luminosities.

2. BACKGROUND

The scintillator powders synthesized at the HTSD Facility are placed in cuvettes and the powders are subsequently characterized in the HTSD Facility for, among other characteristics, x-ray luminescence (XRL). In the XRL setup, the powders are illuminated by a 50 keV and 60 mA x-ray source [Nonius FR591, Bruker AXS Inc., Madison, WI] and the resulting luminescence is measured with a SpectraPro 2156 spectrometer [Princeton Instruments, NJ] with a -70ºC cooled PIXIS 100B charge-coupled device (CCD) [Princeton Instruments, NJ] [1]. The light emitted from the sample is directed into the spectrometer with a pair of optical quartz lenses, where the first lens collects the light emitted from the sample and collimates it into a parallel beam, and the second lens focuses the beam into the spectrometer. The scintillation light seen by the CCD is thus limited in intensity by the amount of light that is emitted from the sample in the direction of the first lens, see Fig. 1. This setup is identical for all measured samples, and the apparently luminosity in our setup can be thus be seen as a function of mainly two effects: 1) the penetrations of the x-rays into the sample – exciting the crystalline grains – referred to in this paper as the "x-ray depth", and 2) the penetration of the produced scintillation light out of the sample, referred to in this paper as the "optical depth". The x-ray depth effect means that low-density materials will have a deeper illumination profile within the sample compared to high-density materials, which will have a more of a "half-moon" luminosity profile, as illustrated on the left in Fig. 1, where the sample is more intensely irradiated closer to the x-ray source and less so at the "back" of the sample. The latter effect, the optical depth, means that smaller crystal grains will scatter a higher fraction of the emitted optical light before the light has a chance of escaping the sample and eventually being detected by the CCD, in effect reducing the light making it out of the sample.

3. METHODS

3.1. Crystal selection

We performed our experiments with four known scintillators: 1) Bismuth Germanate ($Bi_4Ge_3O_{12}$, or simply BGO), 2) Lutetium Oxyorthosilicate ($Lu_2SiO_5:Ce^{3+}$, or simply LSO), 3) Yttrium Aluminum Perovskite activated by Cerium ($YAlO_3$:0.2%Ce^{+}, or simply YAP:Ce), and 4) Cesium Diabarium Iodide ($CsBa_2I_5$:4%Eu^{2+}, or simply CBI). The BGO and LSO crystals were selected as we had access to a large amount of scrap pieces that had been left over from crystal boule cutting from previous projects. The downside of using scrap pieces is that we could not measure a gold standard value for the luminosity of the crystals, as the scrap pieces were oddly shaped and small in size. LSO has reported luminescence values between 22,200 and 33,000 photons/MeV in the literature [2-7], while BGO has a well-known luminosity value of ~8,000 photons/MeV that varies very little across samples and in the literature [7-11]. These numbers are however of little importance, as we want to study the luminescence as a function of granularity of the powders, which does not require an "absolute" luminescence value. Since we used the same batch of crystal pieces for all powders, we can safely assume that the only factor between each sample is the crystal grain sizes within that sample.

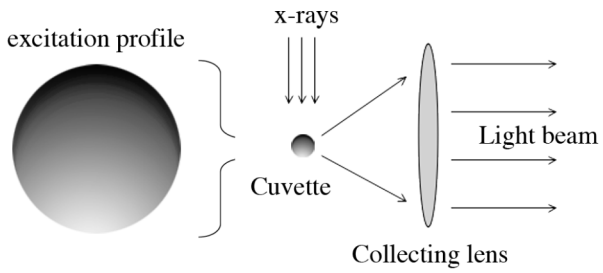

Fig.1. X-ray luminescence setup. The x-rays illuminate the sample from the top in the schematic, and the excitation light that is emitted from the sample (shown from above) towards the right get collected by a quartz lens into a collimated parallel beam. The cuvette is displayed as an hypothetical excitation profile, where darker areas are more luminescent and lighter areas are less luminescent. An enlarged image of the excitation profile of the cuvette is displayed on the left in the figure. The figure is not to scale.

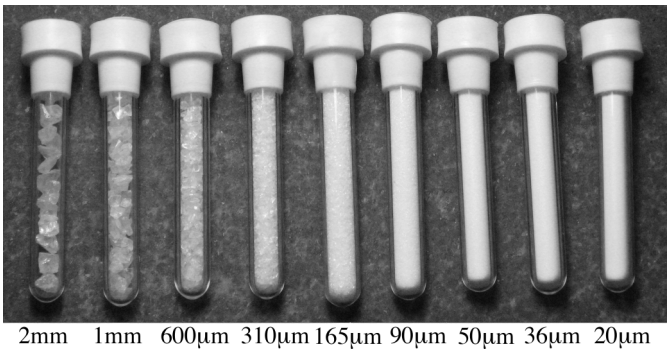

Fig. 2. Cuvettes containing different size grains of LSO. The numbers beneath each cuvette refers to the mesh size openings the grains did NOT pass through; for instance, the "310 μm" cuvette contains grains that are smaller than 600 μm, but larger than 310 μm, as the powder did pass through the next larger mesh size (600 μm), but not the 310 μm mesh.

YAP:Ce was acquired in polished 10 x 10 x 10 mm^3 cubes from Proteus, Inc. [Chagrin Falls, OH]. We used three crystal cubes for our experiments, ensuring enough material to fill our cuvettes. YAP:Ce has a published luminosity of 15,900 to 21,600 photons/MeV [12-16], a density of 5.55 g/cm^3 and is, as BGO and LSO, non-hydroscopic.

CBI is a newly discovered scintillator material with a very high luminescence of 103,000 photons/MeV and a 2.5% energy-resolution at 662 keV [unpublished results], with a decay time of 1.4 μs, and is one of the many new scintillators discovered at the HTDS Facility [17-22]. Furthermore, CBI has a 5.1 eV band gap and a density of 4.8 g/cm^3. The CBI crystal used in this work was grown in our crystal growth facility, before being sacrificed for our granularity tests.

3.2. *Sorting the grains*

The crystal pieces were crushed with a pestle in a mortar. We took great care in making sure not to grind the samples since this would introduce heat into the samples, which

could alter the crystals' intrinsic characteristics (such as the chemical composition). Since we used many scrap pieces of crystal, all pieces were crushed in one batch to make sure any pieces with higher – or lower – luminosities would be evenly distributed in the final powder distributions. The BGO, LSO, and YAP:Ce samples were processed in a Powdersafe™ hood enclosure [AirClean® Systems, Raleigh, NC] to ensure that any airborne particles would not accidentally be inhaled, while the CBI crystal was processed in a water-free environment (<0.1ppm O_2 and <2±1ppm H_2O) inside of an Argon-filled Labmaster SP glove box [MBRAUN, Germany].

The crushed powder was sifted through Sefar Nitex® nylon meshes in a L3P Sonic Sifter from the ATM Corporation [Qual Lab LLC, Bridgewater, NJ] and by hand. We selected to use sieve sizes separated with no more than twice the mesh opening between one mesh size to the next, that is, we used mesh size openings of 2 mm, 1 mm, 600 μm, 310 μm, 165 μm, 90 μm, 50 μm, 36 μm, and 20 μm. The largest mesh opening was selected based on the inner diameter of the cuvettes — which the powder is placed in for the luminosity measurements — and which was measured to be ~3.4 mm in diameter. The smallest mesh size was selected to be 20 μm since any smaller mesh openings had very low mesh opening to surface area ratios (<10%). Our convention in this paper is to label the sieved grain sizes with the mesh size the powder did NOT pass through; for instance, if the powder passed through the 600 μm mesh size, but not the 310 μm mesh size, the granularity of the sifted powder (containing 310- to 600-μm sized grains) is labeled 310 μm.

The sieved material was placed in cuvettes, fully filling each of the cuvettes. The powders in the cuvettes were compacted by gently tapping the cuvettes against a hard surface until the powder had settled and minimized the amount of air in between grains.

3.3. Amount of Grain in Each Cuvette

The cuvettes were weighed before and after they were filled in order to estimate the amount of scintillator material in each container, giving us the packing fraction of each grain size within the cuvettes. The amount of scintillator in each cuvettes (after cuvette and lid weight subtraction) is summarized in Fig. 3. The BGO, LSO, and YAP:Ce cuvettes are filled all the way to the top (unless otherwise noted), while the CBI cuvettes are filled a few millimeters below the top (unless otherwise noted) in order to be able to properly seal them from the surrounding atmosphere.

3.4. X-Ray Luminescence (XRL)

Each sample of crystalline powder was exposed to a 50 keV and 60 mA x-ray beam in the HTSD Facility [1], illuminating the sample from the side, as previously illustrated in Fig. 1. A luminosity spectrum from 200 to 1000 nm was recorded for each sample and the luminosity was calculated as the area integral under the spectrum curve. Each experiment was performed at least five times for statistical analysis, where each cuvette was allowed to rotate in between measurements, and thus be exposed from different sides. This is more important for the larger crystal grains, as the grain distribution is less uniform for these samples, and a single crystal (or an air gap) can influence the results greatly. The results were normalized to the highest luminescence value for each compound.

3.5. Additional Experiments

To eliminate that the cuvette-size played a role in the luminescence measurements, the largest and smallest inner diameter cuvettes [Wilmad LabGlass, Vineland, NJ] that we had access to were used for one additional experiment. These cuvettes did not pass our size selection standards and are normally not be used for our powders or experiments. Of these rejected cuvettes, the smallest inner diameter was measured to be 3.08 mm, and the largest was measured to have an inner diameter of 3.86 mm. The cuvettes were filled with 310 μm BGO powder.

4. RESULTS

No detectable amount of powder made it past the smallest mesh size of 20 μm for all compounds, and hence, the smallest powder grains we ended up with were 20 μm in size. For BGO and CBI, we ended up with enough 36-μm grain powder to fill the cuvette for the XRL measurements, however, not enough to completely fill the cuvette, and this weight data point has consequently been omitted from Fig. 3. For CBI, negligible amounts of material passed through the 36 μm sieve, and no results are therefore available for this grain size.

The normalized luminosity for the four compounds as a function of grain size is displayed in Figs. 4 through 7; Fig. 4 shows the luminosity for BGO, Fig. 5 shows the luminosity for LSO, Fig. 6 shows the luminosity for Yap:Ce, and Fig. 7 shows the luminosity for CBI, respectively. The highest luminosity was achieved for 600-μm grains for LSO and BGO, for 310-μm grains for CBI, and for grains larger than 165 μm for YAP:Ce.

For the larger grain sizes (>600 μm), the luminosity decreases as the grain sizes increase. This is especially prominent for grains larger than 1 mm. The lower luminosity for these large grains can be explained by that the packing fraction of the powder is lower and that air in between the grains lowers the amount of scintillator

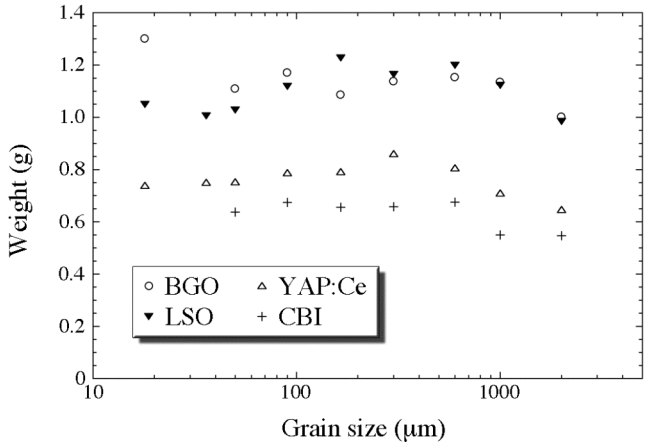

Fig. 3. Weight of BGO (open circles), LSO (filled triangles), and CBI (crosshairs) crystal grains in a full cuvette.

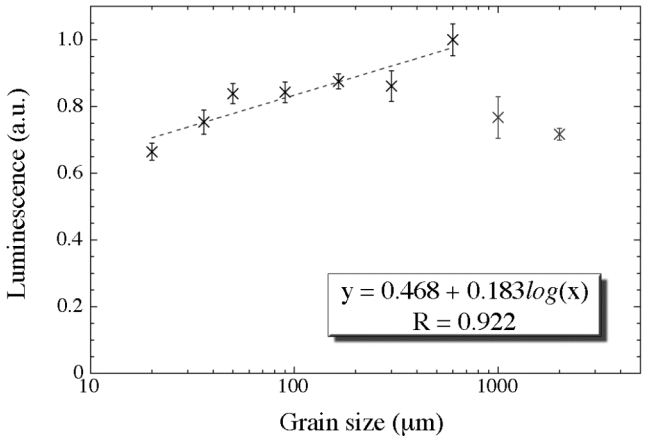

Fig. 4. Apparent luminosity of BGO as a function of crystal grain size. Data has been normalized to the highest luminosity value (for 600-μm crystal grains). Error bars display the standard deviation around the average value. The dotted line is a logarithmic curve fit for all data points smaller than (and including) 600-μm grains.

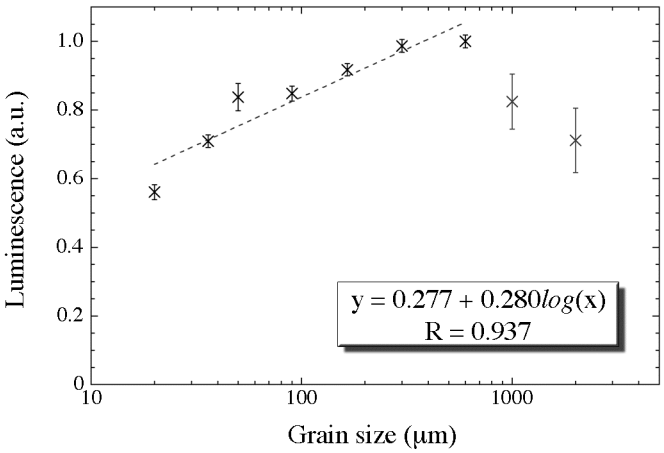

Fig. 5. Apparent luminosity of LSO as a function of crystal grain size. Data has been normalized to the highest luminosity value (for 600-μm crystal grains). Error bars display the standard deviation around the average value. The dotted line is a logarithmic curve fit for all data points smaller than (and including) 600-μm grains.

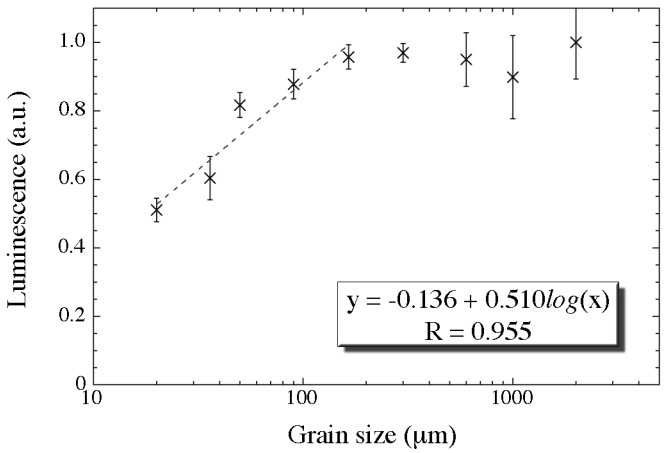

Fig. 6. Apparent luminosity of YAP:Ce as a function of crystal grain size. Data has been normalized to the highest luminosity value (for 165-μm crystal grains). Error bars display the standard deviation around the average value. The dotted line is a logarithmic curve fit for all data points smaller than (and including) 165-μm grains.

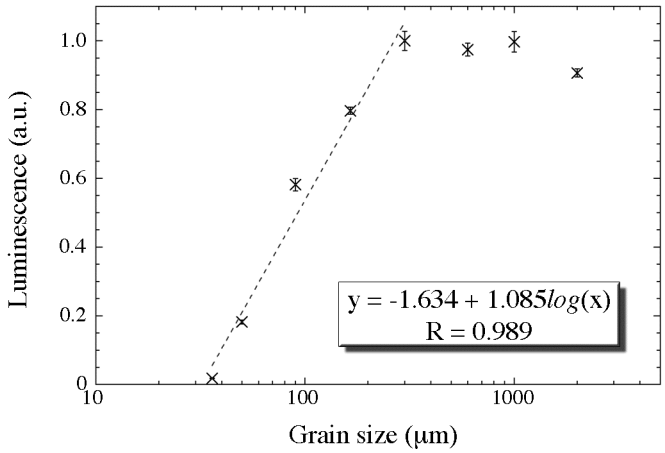

Fig. 7. Apparent luminosity of CBI as a function of crystal grain size. Data has been normalized to the highest luminosity value (for 310 μm crystal grains). Error bars display the standard deviation around the average value. The dotted line is a logarithmic curve fit for all data points smaller than (and including) 310-μm grains.

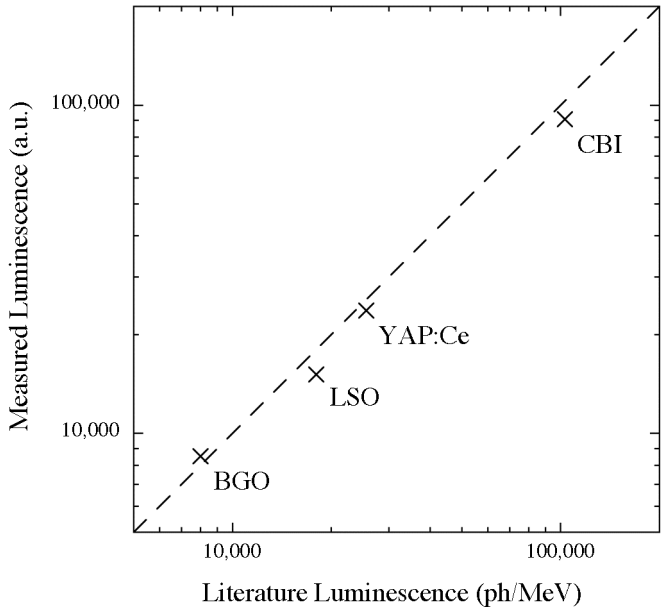

Fig. 8. Measured luminosity as a function of published luminosity for the crystal size that produced the highest luminosity. The dashed line illustrates a one-to-one correspondence between the measure values and the literature values. See article text for details.

material that can contribute to the measured luminosity. This hypothesis is supported from the results displayed in Fig. 3, where the amount of material in each cuvette is fairly independent of crystal size except for the largest crystal grains. The large standard deviation (i.e., the error bars) on the 1- and 2-mm data points in Figs. 4, 5, and 7 (i.e., BGO, LSO, and YAP:Ce) also indicate that the orientation of the cuvette in the setup plays an important role, where the position of one single large crystal grain (or air gap) can create a large (or small) optical signal. The luminosity of these larger grains should however not produce a higher luminosity than the ”mid-size” grains since the packing fraction is lower and less scintillator material contributes to the luminescence signal. This conclusion does not fully hold for YAP:Ce, where the 2 mm crystal grains produced slightly higher luminescence

(103%) over the mid-size grains. The reason for this might be that we started with polished surfaces and a very hard material, and ended up with 2-mm crystals with very transparent surfaces (compared to the smaller grain sizes).

For BGO and LSO, the luminosity for crystal grains smaller than the 600-μm grains decreases with decreasing grain size. This effect is also true for CBI and YAP:Ce, but for crystal grain sizes smaller than 310 μm and 165 μm, respectively. The reason for this effect is that scatter becomes an important factor for smaller grain sizes, where the excitation light from the scintillator grains will be attenuated while trying to penetrate out of the sample. In Figs. 4 through 7, a logarithmic curve fit has been added to the data points below (and including) the grain size that creates the largest luminescence signal (165 μm grains for YAP:Ce). The luminosity as a function of grain size is not a simple logarithmic function however, and the curve fit has only been added to illustrate the effect. A more realistic function should involve both the crystal grain size and the x-ray depth, however, surface defects are most likely also influencing the sharp decreases in apparent luminescence for YAP:Ce and CBI (more on this in the Discussion section).

The luminosity for the larger and smaller inner diameter cuvettes with 310-μm BGO powder was measured to be 28,300±700 photons/MeV versus 27,500±700 photons/MeV, respectively. This is equivalent to a 2.8% increase in luminosity for the larger cuvette, with a 2.3% error. The larger cuvette has a 25% larger inner diameter, or 57% larger volume, and hence, we can from this experiment draw the conclusion that the cuvette size plays very little – if any – role in the luminescence measurements. The explanation for this is that only the light that is emitted from around the centerline (in relation to the collimator lens) of each cuvette will make into the parallel beam by the first lens, and any light from the periphery of the cuvette will not be focused correctly into the spectrometer.

We also examined the absolute value of the measured luminosity versus the luminescence value in the literature. The data in Figs. 4 through 7 were all normalized to the highest luminescence signal in order to focus the attention to the apparent luminosity as a function of crystalline grain size. In Fig. 8, the measured (absolute) luminescence for the grain size that produced the highest luminescence is plotted against the literature value for the crystal compound. The measured values used in this plot are for the 600-μm grains for BGO and LSO, for the 2-mm grains for YAP:Ce, and for the 310-μm grains for CBI. For LSO, we used the average of the literature values (27,130 ph/MeV) [2-7], for YAP:Ce we used the company supplied value (18,000 ph/MeV), and for CBI, we used 103,000 ph/MeV. As can be seen from the graph, the measured values are excellent at predicting the crystal luminescence.

5. Discussion

In our daily operations of the HTSD Facility, we estimate the representative grain size for each sample to be able to get a better estimate of possible luminosity. The compounds with the highest relative x-ray luminosity are then grown in our crystal growth facility. From the grown crystals, the luminosity is determined by measuring the crystal luminosity (and energy resolution) on a photomultiplier tube (PMT) and normalizing this value to a photopeak from known scintillator, such as NaI(Tl). The measured luminosity of the crystalline powder is hence an intermediate result to estimate the possible crystal luminosity, and although we use the crystalline grain x-ray luminosity value to estimate possible crystal luminosity, the results above show that there is no simple formula that can be used to guess the crystal luminosity knowing the crystal grain size – unless the grains are large. The results from this study do however validate 1) synthesizing scintillating crystals by solid-state or melt reactions (i.e., the method of production), and 2) estimating the crystal grain luminosities from these crystalline grains (i.e., the quality testing of the crystals). The measured crystalline grain x-ray luminosity for grains larger than 90 μm provided luminosity values within 40% of a "full-size" crystal for the crystals examined in this study, and larger crystal sizes were even better at predicting "full-size" crystal luminosities.

The reason YAP:Ce and CBI showed such a fast decrease in luminosity as the crystal size decreased is most likely due to defects at the surface level in the crystal grains. The smaller the crystal, the larger the effect the surface has on the luminosity — as the size of the crystal decreases, the volume decrease by the power of three, while the surface area decreases by the power of two. Therefore, as the crystal decreases in size, the surface area will limit the luminosity by a factor proportional to the crystal size. This effect is not enough to explain the sharp decline in the luminosity for CBI, and another factor can be CBI's hydroscopic nature. We were not able to produce any amounts of <36-μm crystals, and one possible explanation for this can be that the 2ppm of water in the glove box binds to these smaller particles, making them stick together as well as deactivating their luminescent properties. We did examine the crystals for all samples under a microscope, but no obvious differences were visible between the different (non-hydroscopic) materials for small (50-μm) crystal grains.

6. Conclusions

We have measured the optical luminosity for x-ray excited crystal grains of BGO, LSO, YAP:Ce, and CBI. All the examined crystals exhibited a decreased luminescence for decreasing crystal grain size when the packing fraction did not limit the setup. The amount of luminescence loss for smaller crystal grains was in this study measured to be 34% for BGO when the crystal size decreased from 600 μm to 20 μm, while LSO had a decrease in luminosity of 44% for the same grain size decrease. YAP:Ce exhibited a 47% decrease in luminosity when decreasing the crystal grain size from 165 μm to 20 μm, while CBI was measured to have a 98% luminescence loss when the crystal size decreased from 310 μm to 36 μm. A decrease in luminescence was also measured for larger crystals (>1 mm), where the decrease of scintillating material in the

x-ray excited volume (i.e., the packing fraction) limited the amount of light collected by our setup. The decrease in luminosity with decreasing crystal grain size is explained by an increased scatter fraction, where the amount of light that makes it out of the sample is limited by the increased number of surfaces the light has to penetrate through, and the associated increased absorption. Surface defects also decrease the light collection for smaller grains. No simple equation for correcting for these effects was established since all four materials exhibited different decrease rates.

The results obtained in this study validate the crystal synthesizing method as a means of evaluating compounds for their scintillating properties, as well as using x-ray luminescence on large (>90 μm) crystalline grains to predict "full-size" crystal luminosity.

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