

**Improved Neutron Scintillators Based on  
Nanomaterials  
Final Report  
6/30/08**

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## I. Motivation for the Phase I Proposal:

New high intensity neutron sources like the Spallation Neutron Source (SNS) at Oak Ridge National Laboratory and the Japanese Spallation Neutron Source (JSNS) have come on line to more thoroughly study condensed matter physics, materials science and engineering, and the geo- and biosciences. Present day commercially available neutron scintillators are not well suited to the high flux capabilities of these new facilities. To fully exploit the research opportunities provided by them, a new generation of neutron detectors with a higher count rate capability, higher efficiency at short-wavelengths and/or higher spatial resolution are required (Cooper, 2004). As described in a recent white paper, “*the large area coverage combined with small pixel sizes, high neutron detection efficiencies, and 1-  $\mu$ s time resolution eliminates all but the scintillator option*” (Cooper, 2003) [1].

PartTec proposed in this Phase I SBIR to improve the sensitivity and resolution of neutron scintillation detectors by reducing the size of the neutron converting and scintillating particles in these detectors to nanometer dimensions. It was believed, based on preliminary unfunded research, that the use of nano-particles will permit higher concentrations and more uniform distributions of converter materials within the scintillator while reducing the absorption coefficient for the light emitted, thereby making a brighter neutron scintillator. *The physics of smaller particles on neutron converter particle density and detector light transmission seem clear, yet the engineering of these scintillators using  $^6\text{LiF}$ ,  $^7\text{LiF}$  and  $\text{ZnS(Ag)}$  nano-particles have so far failed to produce the anticipated results. Nevertheless, as explained below, the results obtained during this Phase I development dispelled a few misconceptions about the transmission of 420 nm light in neutron scintillators and laid the groundwork for developing mechanisms to produce more efficient and faster neutron scintillators.*

## II. Technical Objectives of Phase I Proposal:

PartTec endeavored to investigate possible advantages of using nano-particles in the fabrication of  $^6\text{LiFZnS(Ag)}$  Neutron Scintillation Detectors. The main technical objectives of the work plan were to develop techniques to;

- Reproduce results previously reported for the nano-particle  $\text{Gd}_2\text{O}_3$  scintillator.
- Fabricate nano-size neutron converter and scintillator particles for  $\text{LiFZnS}$  Neutron Scintillators and measure their properties.
- Determine an accurate and reproducible correlation between the size of the nano-particles used to fabricate a scintillator and their physical properties relevant to the detection of neutrons.
- Compare possible matrix options for neutron scintillation detectors.
- Control the dispersion of nano-particles in the polymeric matrix.
- Determine the benefits and/or weaknesses derived from the use of nano-particles in the fabrication of neutrons detectors.

This proposal was designed to be a chemical engineering development activity with the purpose of proving the feasibility of mass producing  $\text{LiFZnS}$  Neutron Scintillation Detectors

with significantly improved detection efficiency and gamma ray discrimination properties. The work was conducted primarily in the research laboratories of the Chemistry Department at Indiana University under the supervision of Professor Jeff Zaleski and his scientific group. PartTec was given access to the advanced technical equipment and professional services within these laboratories through a subcontract with Professor Zaleski and Indiana University.

### III. Background Information

Typical neutron scintillators consist of neutron conversion and scintillating materials that work together to capture the neutron and convert its energy into detectible light pulses. The  ${}^6\text{LiF}$  has a large neutron capture cross section, producing an energetic Alpha particle that excites the ZnS, which then decays by giving off 420 nm light.  ${}^6\text{Li}$  is chosen for its high neutron capture cross-section and its low gamma radiation. These materials are held together within in a suitable binding material or matrix, such as *Epoxy*, Polystyrene or PMMA. Commercial neutron scintillators mix micron size ZnS and  ${}^6\text{LiF}$  powders in an epoxy binder to form the neutron scintillator. The ratio of ZnS to LiF is 2:1, and the binder is typically 30% of the mixture.

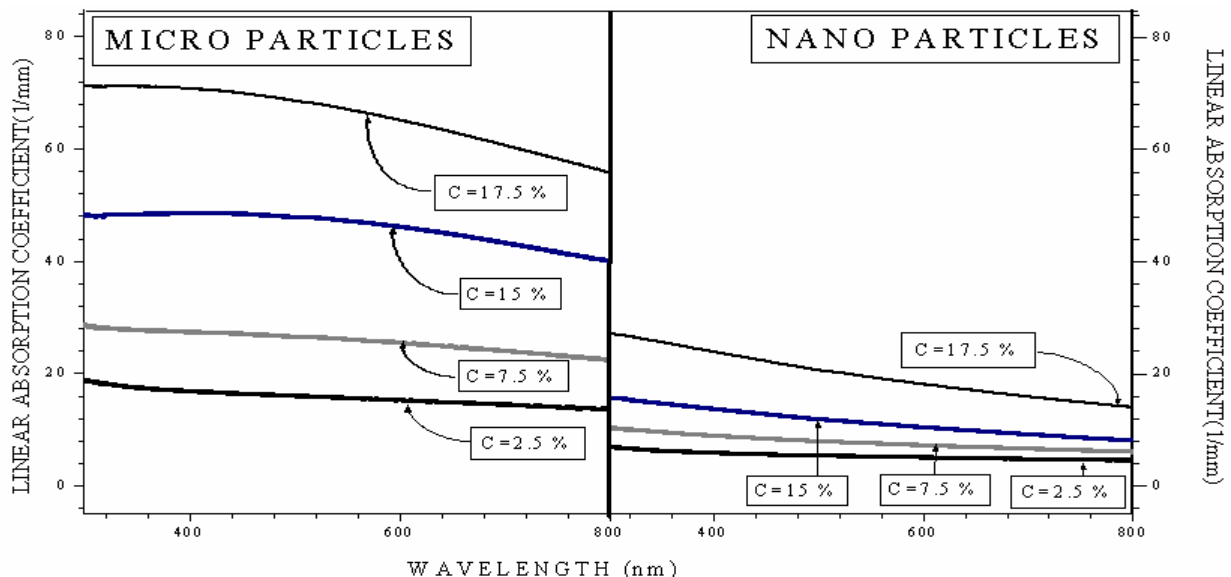
Unfortunately, ZnS has a high index of refraction, making it almost opaque to its own scintillation light, reducing the detection efficiency of the scintillator. To overcome the self absorption of the 420 nm light, available commercial scintillator thicknesses are limited to less than about 0.5 mm. By replacing micron size  ${}^6\text{LiF}$  and ZnS particles with approximately 100 nm particles, it was proposed that the absorption of the 420 nm light would be significantly reduced and that thicker and more efficient scintillators could be produced. PartTec concentrated on developing the  ${}^6\text{LiF}/\text{Zns:Ag}$  neutron converter/scintillator by using nanometer scale conversion and scintillating particles.

#### III.1 Results from Preliminary Research

PartTec performed preliminary research on the use of  $\text{Gd}_2\text{O}_3$  nano-particles prior to the submission of the Phase I Scintillator proposal. Both micron and nanometer size  $\text{Gd}_2\text{O}_3$  neutron converting particles were sonicated and bound in a polystyrene matrix to form 0.04 mm thick scintillator foils via spin coating. These preliminary results, reported by a previous PartTec research team in 2004, show a significant improvement in light transmission for nanometer size  $\text{Gd}_2\text{O}_3$  particles bound in polystyrene compared with similarly bound micro sized particles, demonstrating the feasibility of the improvement in neutron conversion and scintillation efficiency by using nano-particles. The linear absorption coefficient of polymer composites obtained by thoroughly dispersing micron and nanometer sized particles of  $\text{Gd}_2\text{O}_3$ , shown in Fig. 1, demonstrated a measurable difference. It was observed that, at 400 nm wavelength, the same linear absorption coefficient is obtained for a 2.5 % loading with micron size particles as for a 15 % loading of the same polymeric matrix with nano-particles.

Based on the description of the fabrication method and these preliminary results, PartTec submitted an invention disclosure to the US Patent office and sought external funding for the

development of this process into a commercially viable Neutron Scintillator product, eventually resulting in the Phase I SBIR proposal reported here.



**Figure 1.** Comparison of light absorption for  $Gd_2O_3$  micro and nano-particles bound in a polystyrene matrix at various concentrations from preliminary research performed at Parttec.

#### IV. Chemical/Equipment Acquisition & Work Plan (SEPT 07)

The first order of business for the execution of this development effort was the acquisition of all necessary equipment and micro/nano particles to both verify the previous  $Gd_2O_3$  film results and demonstrate that the process works as well for nanoparticle  $LiFZnS$  films. The equipment list used for this development effort is provided in Appendix A.

Many of the nanomaterials used in this work were commercially available from chemical supply houses. However, the  $LiF$  and Silver doped  $ZnS$ , while available commercially as micro-particles, were not commercially available as nano-particles. The nano-particle  $^{6,7}LiF$  were obtained by milling 5  $\mu m$   $LiF$ , while the silver doped  $ZnS$  was fabricated in the laboratory using a well published preparation to produced particles as small as 20 nm. A description of the nano-particle  $ZnS:Ag$  preparation and the TEM measurements of the resulting particles are provided in Appendix B.  $^7LiF$  was significantly less expensive than the  $^6LiF$ , and was therefore used for much of the preliminary Phase I development work. The sizes of all particles used here were verified using the Indiana University TEM machine.

#### V. Work Plan

The first development task was to reproduce the results of the previous investigators using  $Gd_2O_3$  micro- and nano-particles in a Polystyrene binder. The procedure described in the Patent Application called for sonicating 2.5 grams of nano-particle  $Gd_2O_3$  in 500 mL of 99.9% pure Toluene until the mixture became transparent, as determined by a Perkin Elmer UV-VIS spectrometer. This process was reported to take between 150 and 300 hours of sonication at 500 watts. The mixture would then be added to Polystyrene dissolved in Toluene before being spin coated into thin foils for transparency tests using the UV-VIS

spectrometer.

The techniques developed for the Gd<sub>2</sub>O<sub>3</sub> films would then be used to produce <sup>7</sup>LiFZnS and finally <sup>6</sup>LiFZnS micro- and nano-particle films for comparison of their light absorption and neutron scintillation light transmission as a function of film thickness. One additional technique developed for this phase of the work was the production of useable amounts of surface doped ZnS:Ag in the I.U. Chemistry lab as discussed in Appendix B.

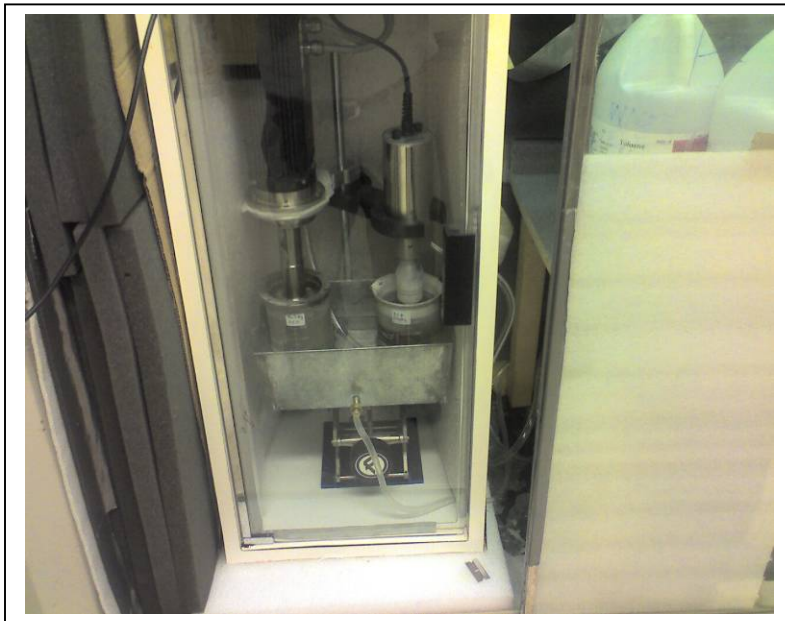
## **VI. Reproduction of Previously Reported Gd<sub>2</sub>O<sub>3</sub> Results.**

### **VI.1 Toluene Sonication Issues & Results**

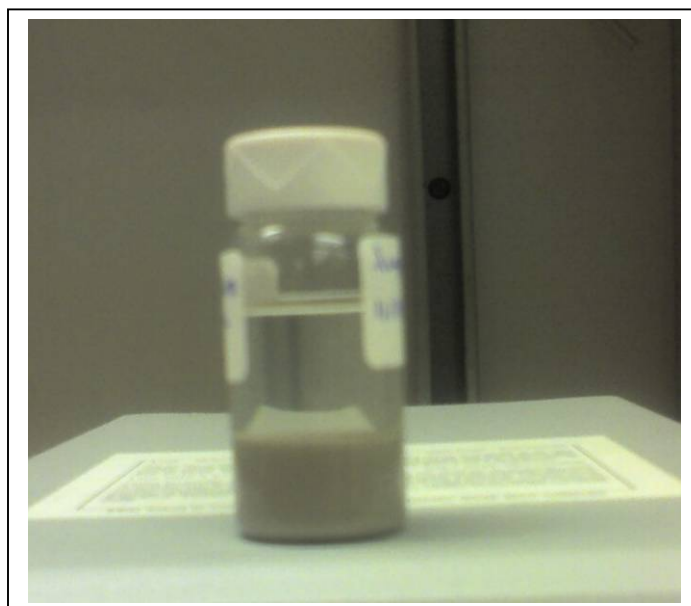
Several attempts to reproduce the Gd<sub>2</sub>O<sub>3</sub> film results using the method prescribed in the patent application met with various difficulties at all stages of development that were eventually understood, but not all were overcome. The un-cooled Toluene-Gd<sub>2</sub>O<sub>3</sub> mixture temperature quickly rose to above 85°C under continuous sonication at 500 Watts. At this temperature, the Toluene boiled off into the hood at the rate of 250 mL per day and was replenished as necessary. In addition, rather than becoming more transparent with time, the Toluene-Gd<sub>2</sub>O<sub>3</sub> solution, which started out as a milky-white solution, became less transparent with time, exhibiting a rather dense brownish cloudy mixture. The Gd<sub>2</sub>O<sub>3</sub> particles appeared to grow in size (i.e., clump together) and settled to the bottom of the beaker within minutes when the sonicator was switched off. Attempts to resolve this were made by placing the beaker of solution in an external continuous water cooling bath that kept the mixture at 38°C during sonication, but to no avail. Sonicating at lower power produced the same results, but after proportionally longer periods of time.

Continuous sonication at 500 watts also presented an unacceptable noise problem within the laboratory, which was resolved by the purchase of a commercial sound proof sonication box that was placed in a large hood. The sound box accommodated both the Heilshire and Cole-Palmer sonicators so that multiple batches could be processed simultaneously. Photos of the final water cooled sonication system and the Toluene-Gd<sub>2</sub>O<sub>3</sub> mixture after 24 hours of continuous sonication are shown in Figs. 2 and 3. The sonicated Gd<sub>2</sub>O<sub>3</sub> has turned brown and settled to the bottom of the container in Fig. 3. The volume of the sediment in the container in fig 3 is significantly larger than the initial volume of Gd<sub>2</sub>O<sub>3</sub> put into the Toluene (2.5 grams), indicating that the particles have clumped together to make larger size aggregates of material. These results were confirmed by a TEM measurement of the Sonicate material during 10 days of continuous sonication. TEM measurements of the 100 nm Gd<sub>2</sub>O<sub>3</sub> placed in the Toluene after 4 and 10 days of continuous sonication are compared in figs. 4a and b respectively.

Finally, UV-VIS measurements were taken at various times during a 302 hour sonication (from 10/16/07 through 10/30/07) of 100 nm Gd<sub>2</sub>O<sub>3</sub> in Toluene. The results, presented in Fig. 5, show that the absorption of light increased with sonication time, i.e., exactly the opposite of what was expected. The UV-VIS of the sonicated Toluene-Gd<sub>2</sub>O<sub>3</sub> is compared with a sample of 99.9% pure toluene in this figure. Multiple measurements of the same samples were made, yielding very consistent UV-VIS plots.



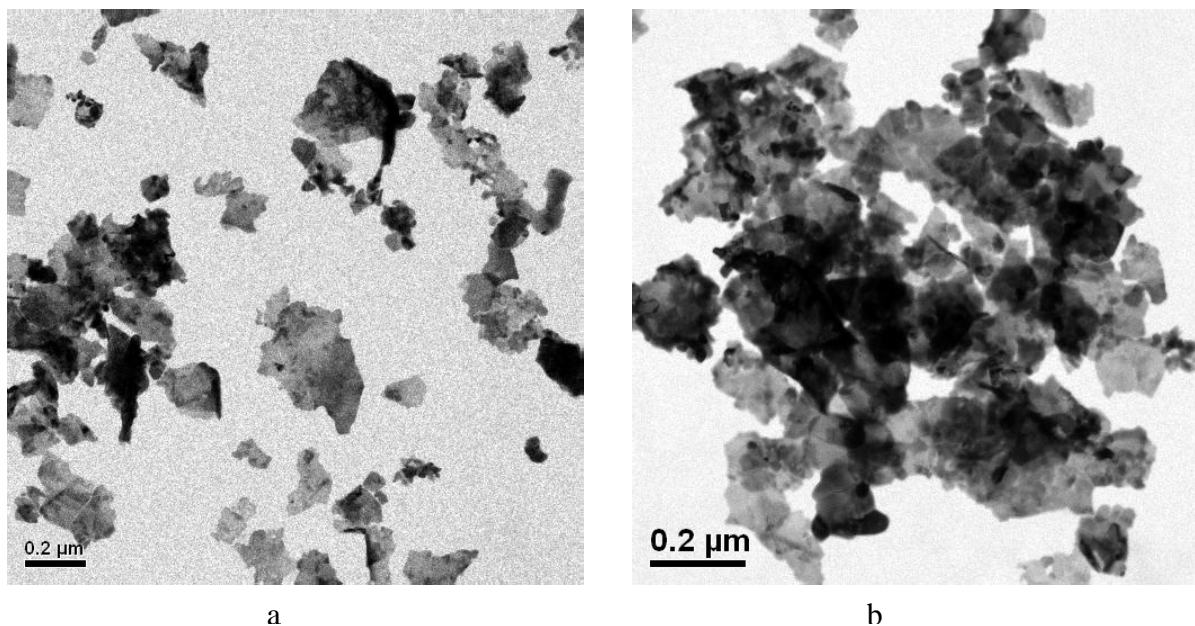
**Figure 2:** Dual Sonicator setup in sound proof box showing water cooling bath used during



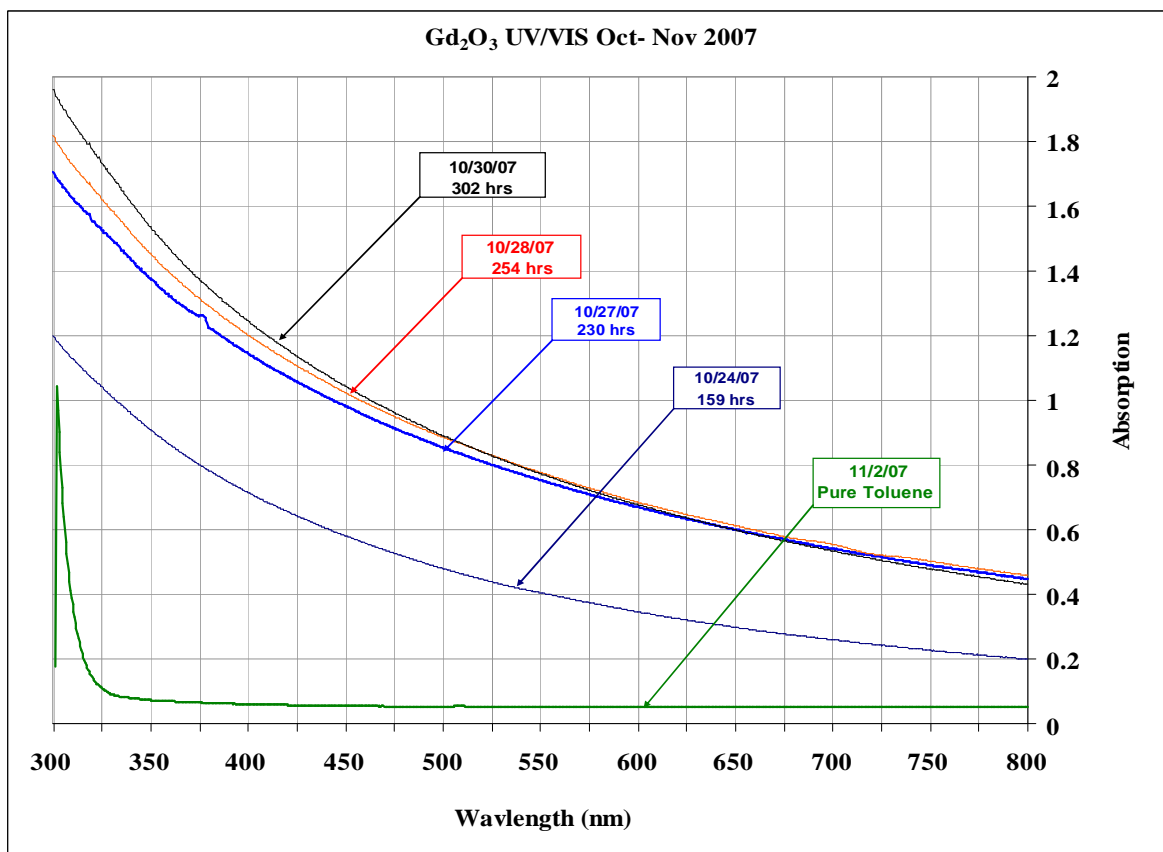
**Figure 3.** Toluene-  $Gd_2O_3$  mixture after 24 hours of Continuous sonication at 500 Watts.

To understand the cause for the unexpected  $Gd_2O_3$  clumping and the yellowing of the pure toluene under sonication, two beakers of 99.9% pure Toluene were sonicated simultaneously, one with the Heilshire at 500 Watts and the other with the Cole-Palmer at 200 watts. Both beakers of Toluene exhibited significant yellowing after only 24 hours of continuous sonication, as shown in Fig. 6. The amount of discoloration of the toluene was observed to be higher for the beaker sonicated with the Cole-Palmer sonicator. UV-VIS measurements of the Toluene before and after sonication for 24 hrs are provided in Fig.7. It was also

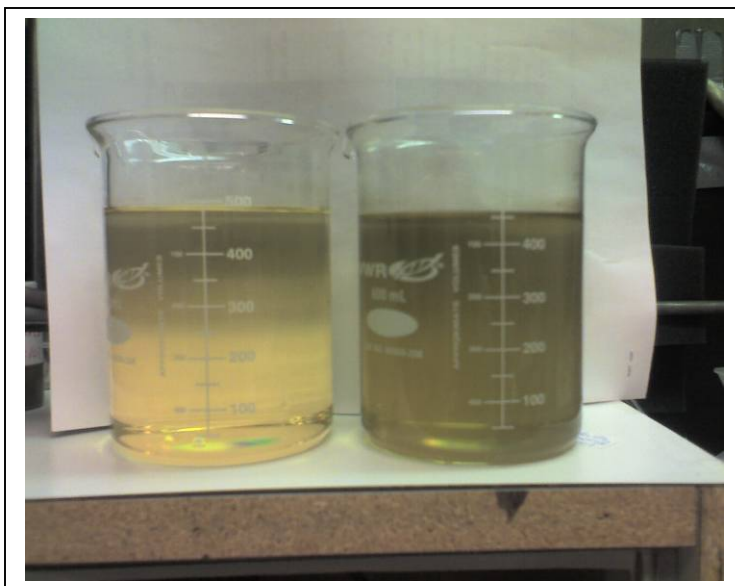




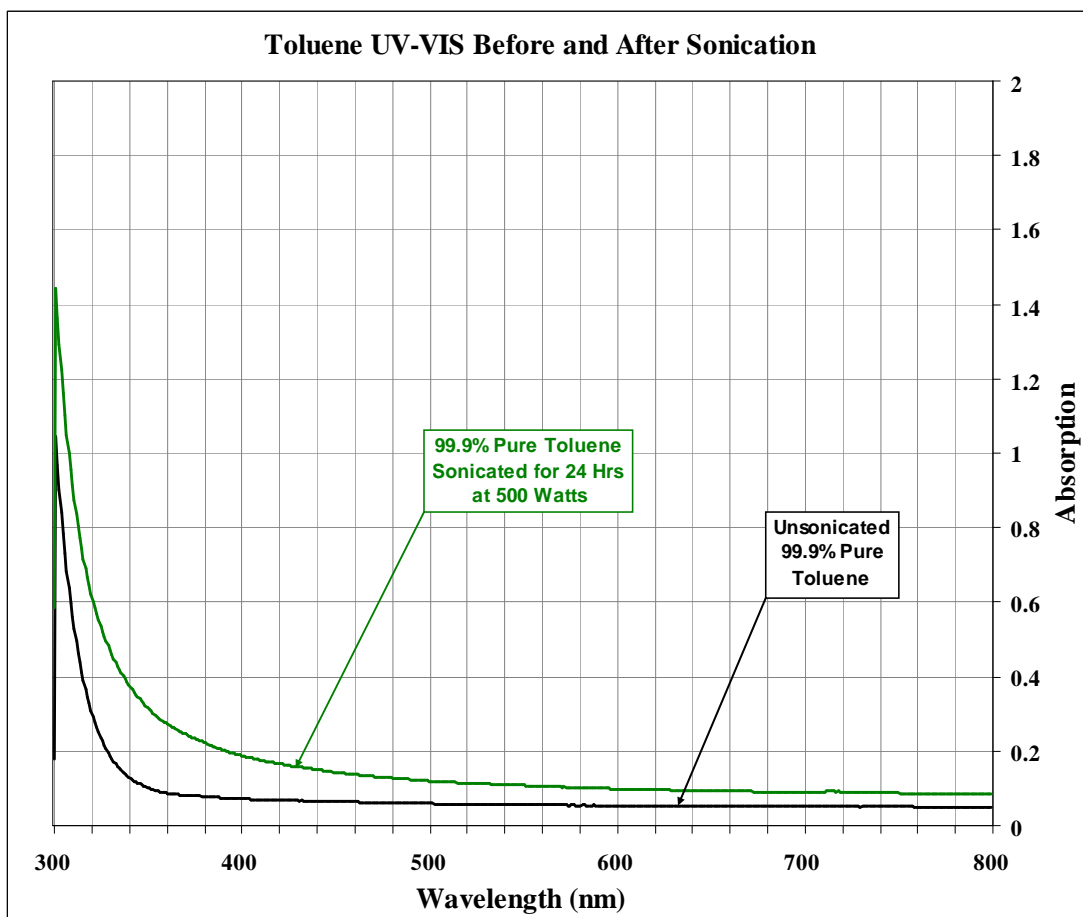
**Figure 4.** a. 100 nm Gd<sub>2</sub>O<sub>3</sub> after 4 days of continuous sonication at 500 Watts.  
b. again after 10 days of continuous sonication at 500 Watts. Significant clumping of the Gd<sub>2</sub>O<sub>3</sub> is visible. The resolution of both measurements is 200 nm.



**Figure 5.** UV-VIS measurements of the Toluene-Gd<sub>2</sub>O<sub>3</sub> mixture as a function of sonication time at 500 Watts compared with pure Toluene.



**Figure 6.** 99.9% pure Toluene sonicated at 500 (left) and 200 Watts (right) for 24 hours.



**Figure 7.** Comparison of Pure Toluene UV-VIS before and after sonication for 24 hrs. These data are plotted on the same scale as Fig. 5



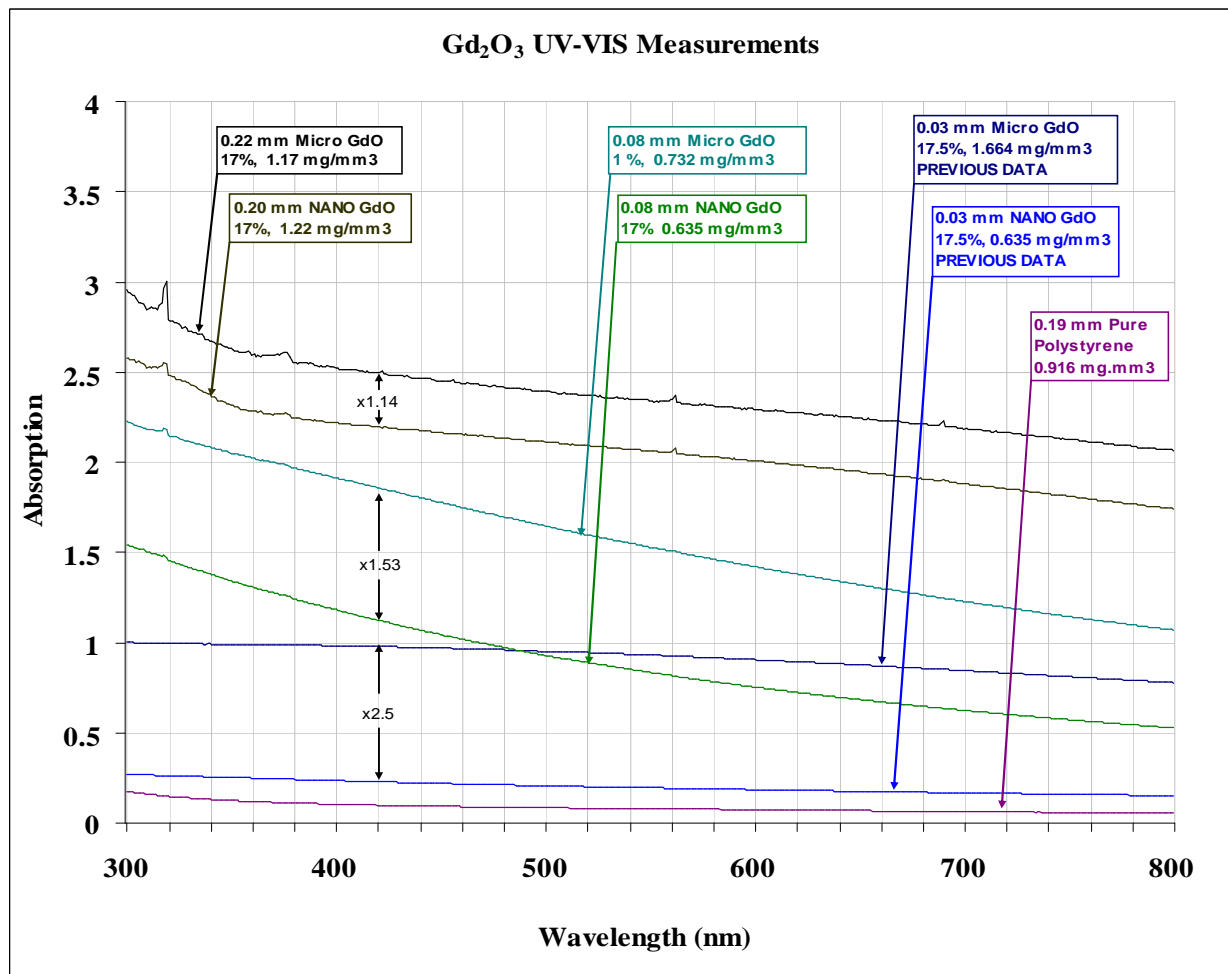
determined that the pure Toluene turned yellowish in color at lower power settings of the sonicators (<25%), but again after commensurately longer sonication times.

The cause for the yellowing was determined to be polymerization of the Toluene under high power sonication. This phenomenon, although initially unknown to us, is a well documented phenomenon [2]. Gas Chromatography Mass Spectral analysis (GCMS) of the sonicated Toluene confirmed the polymerization. This also showed that there was little to no oxidation and less than 1% contaminating materials in the Toluene. Based on the body of evidence presented here, PartTec determined that the advertised process for producing transparent Gd<sub>2</sub>O<sub>3</sub> could not possibly work, and alternative means of reproducing the preliminary results were sought.

## **VI.2 Reproduction of Gd<sub>2</sub>O<sub>3</sub> Results without Long Term Sonication.**

Micro- and nano-foils of Gd<sub>2</sub>O<sub>3</sub> in a polystyrene binder were eventually fabricated by simply mixing the Gd<sub>2</sub>O<sub>3</sub> in Toluene using an IKAMAC combination hot plate/magnetic stirrer. One gram each of 40 nm and 325 mesh (< few um) Gd<sub>2</sub>O<sub>3</sub> were mixed in 100 ml beakers of pure Toluene, sonicated for less than 1 hr to evenly disperse the particles, and stirred continuously thereafter. Simultaneously, two samples of polystyrene, each weighing 4.88 grams, were dissolved in 50 ml of Toluene. When the Polystyrene was uniformly dissolved, the micro- and nano-particle Gd<sub>2</sub>O<sub>3</sub> solutions were poured, one into each beaker of the polystyrene solutions, producing a 17% concentration of Gd<sub>2</sub>O<sub>3</sub> in Polystyrene, very close to the 17.5% concentration used in the preliminary study. Both beakers were then magnetically stirred at 70°C until the solutions evaporated to a honey like mixture. 0.08 and 0.20 mm thick foils of nano- and micrometer Gd<sub>2</sub>O<sub>3</sub> particles in polystyrene were produced for comparison with the foils previously produced and reported. The foils were cast by evenly dispersing 0.5 mL and 1.0 mL amounts of the Gd<sub>2</sub>O<sub>3</sub>/Polystyrene solution on 23 x 30 mm glass slides. The Toluene evaporated within 24 hours, after which the foils could be easily lifted from the slides. A sample foil of pure polystyrene was also fabricated using this method for comparison with the Gd<sub>2</sub>O<sub>3</sub> foils. These cast foils are self supporting and have a thickness variation of about 10% across their area. The foils produced in the preliminary study were fabricated by spin coating on glass. The density of all foils, including the foils produced in the preliminary study, was determined to verify the makeup of the foils for comparison purposes. UV-VIS comparisons of the newly produced Gd<sub>2</sub>O<sub>3</sub> foils with those produced in the preliminary study and with the pure polystyrene foil are shown in Fig. 8. Note that the absorption scale on this graph is twice that of figs. 5 and 7.

The primary result is that the work of the previous PartTec investigators was verified, although by a significantly less time consuming process that did not depend on long sonication times. The newly fabricated 17% nano-particle Gd<sub>2</sub>O<sub>3</sub> foils were significantly more transparent to 420 nm light than the micro-particle foils. It was also found that the ratio of 420 nm light absorption for nano-particle vs. micro-particle foils decreases with foil thickness, from 2.5 for the thinnest to 1.14 for the thickest foils produced. These data suggest that the transparency ratio between nano- and micro-particle foils to 420 nm light asymptotically approaches one as the thickness of the foils increases above 0.2 mm. A summary of the foil properties and their light absorption at 420 nm is shown in Table I.



**Figure 8.** UV-VIS measurements of new 0.08 and 0.2 mm thick 17% Gd<sub>2</sub>O<sub>3</sub> foils compared with 0.03 mm thick 17.5% foils produced in preliminary study and with a 0.19 mm thick pure polystyrene foil.

**Table I**  
**Comparison of Gd<sub>2</sub>O<sub>3</sub> foil properties as a function of particle size and foil thickness**

Foil ID	Particle size	% Gd <sub>2</sub> O <sub>3</sub>	Thickness (mm)	Density (mg/mm <sup>3</sup> )	Absorption @ 420 nm	Comments
Pure Polystyrene	N/A	0	0.19	0.916	0.0987	Very transparent to the eye, uniform thickness, cast
GdOum17	< 10 um	17.5	0.03	1.66	0.974	Preliminary Study; spun
GdOnm17	40 nm	17.5	0.03	0.635	0.228	Preliminary Study; spun
GdOum20	< 10 um	20.0	0.04	0.96	0.948	Preliminary Study; spun
GdOnm20	40 nm	20.0	0.04	1.22	0.459	Preliminary Study; spun
020208	<10 um	17.0	0.08	0.732	1.859	Cast on glass
020108	40 nm	17.0	0.08	0.635	1.124	Cast on glass
020408	<10 um	17.0	0.22	1.17	2.497	Cast on glass
020308	40 nm	17.0	0.20	1.22	2.195	Cast on glass

## VII. LiFZnS(Ag) Fabrication in Polystyrene

The results of the Gd<sub>2</sub>O<sub>3</sub> work precipitated the abandonment of long term sonication of the LiF and/or ZnS in toluene as originally proposed. An attempt to sonicate ZnS in Toluene for several days produced the same results as observed with the Gd<sub>2</sub>O<sub>3</sub>. All LiFZnS:Ag foils discussed below were fabricated using the same mixing and casting process used successfully for the Gd<sub>2</sub>O<sub>3</sub> foils. Nano- and micro-particle <sup>7</sup>LiFZnS:Ag, <sup>6</sup>LiFZnS:Ag, ZnS:Ag foils (17% particles, 83% Polystyrene), and pure polystyrene foils were fabricated and tested. Two different binding agents were used: Polystyrene dissolved in Toluene and Epoxy dissolved in Acetone. A summary of the properties of the many foils produced are provided in a Table in Appendix C. The general result of these fabrications and data is that, unlike the results for Gd<sub>2</sub>O<sub>3</sub>, no statistical difference was observed in the absorption of 420 nm light for LiFZnS:Ag foils made from nano-particles and micro-particles. It was again observed that the percent change in transparency to 420 nm light decreased as the thickness of the foils increased for both nano- and micro particle foils.

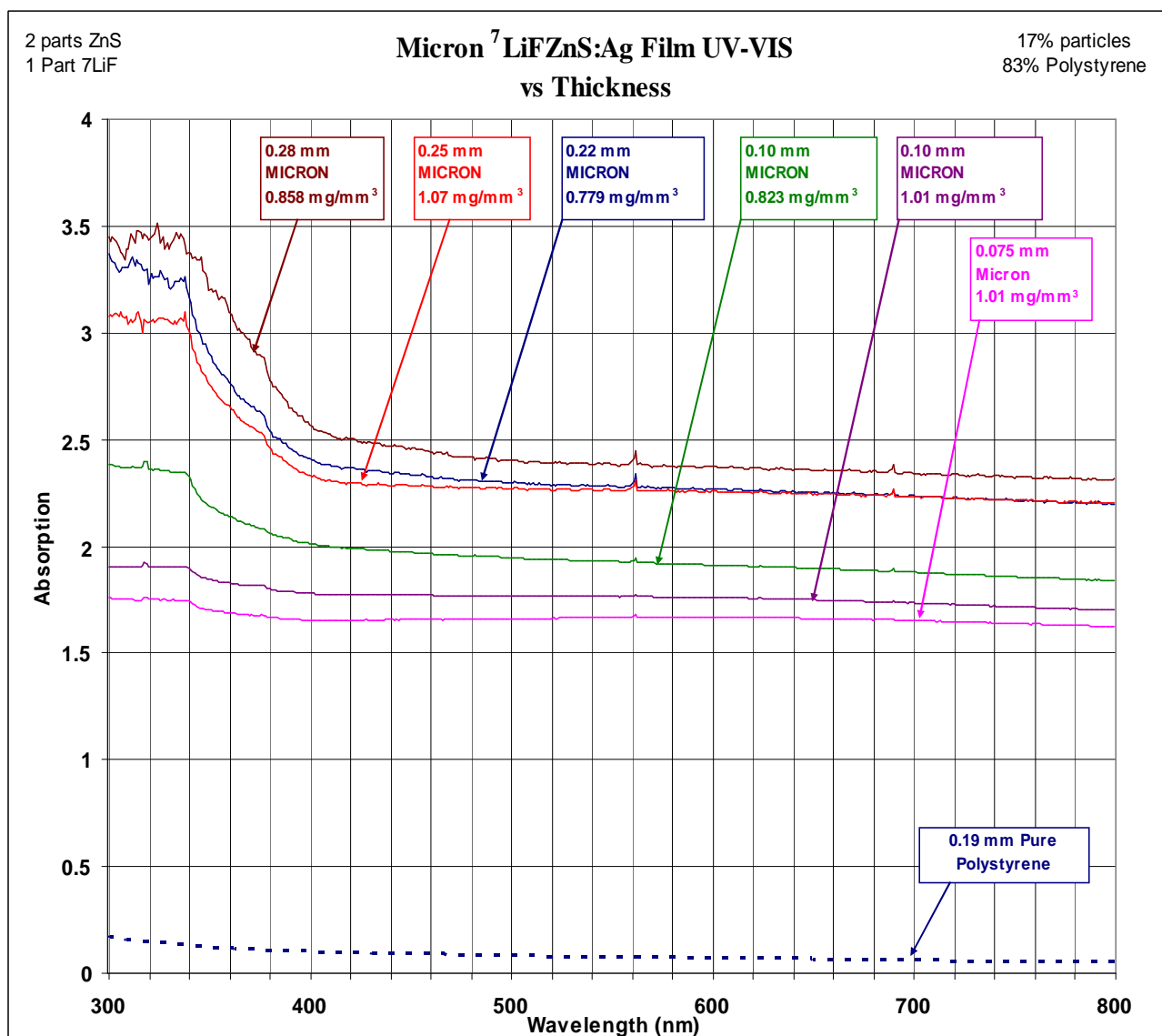
The first attempts to make LiFZnS foils, listed in Appendix C as Batch 0, experimented with casting and spinning foils from Toluene, LiF and/or ZnS, and Polystyrene mixtures of various viscosities. Nine batches (0 through 7b) of various mixtures of neutron scintillator materials and binders were prepared for the fabrication of the foils listed in Appendix C. The fabrication details and materials used for each batch are provided in Appendix D. Batch 6, for example, details the preparation of the Gd<sub>2</sub>O<sub>3</sub> foils discussed in section VI above. A 17% scintillator material and 83% binder mixing ratio (obtained by simple chemical material weight measurement) was established as a standard throughout the program as this was deemed thick enough to accurately represent the UV-VIS performance of a real scintillator, yet contain enough scintillator material to detect neutrons with a weak source, such as the Am<sup>241</sup> source at Indiana University. The LiFZnS scintillator foils were all prepared using 1 part LiF and 2 parts ZnS:Ag to make up 17% of the foil mass by weight, similar to the ratio in commercial scintillators used at the Oak Ridge SNS. The remaining 83% was either polystyrene or epoxy. The Silver doping content was 0.025% and is a surface, rather than bulk, doping. The actual mixing ratio of the various batches, as listed in Appendix D, varies by about a percent from 17%, which seemed to have a negligible effect on the results presented here.

### VII.1 <sup>7</sup>LiFZnS:Ag Foil Fabrication and Measurements.

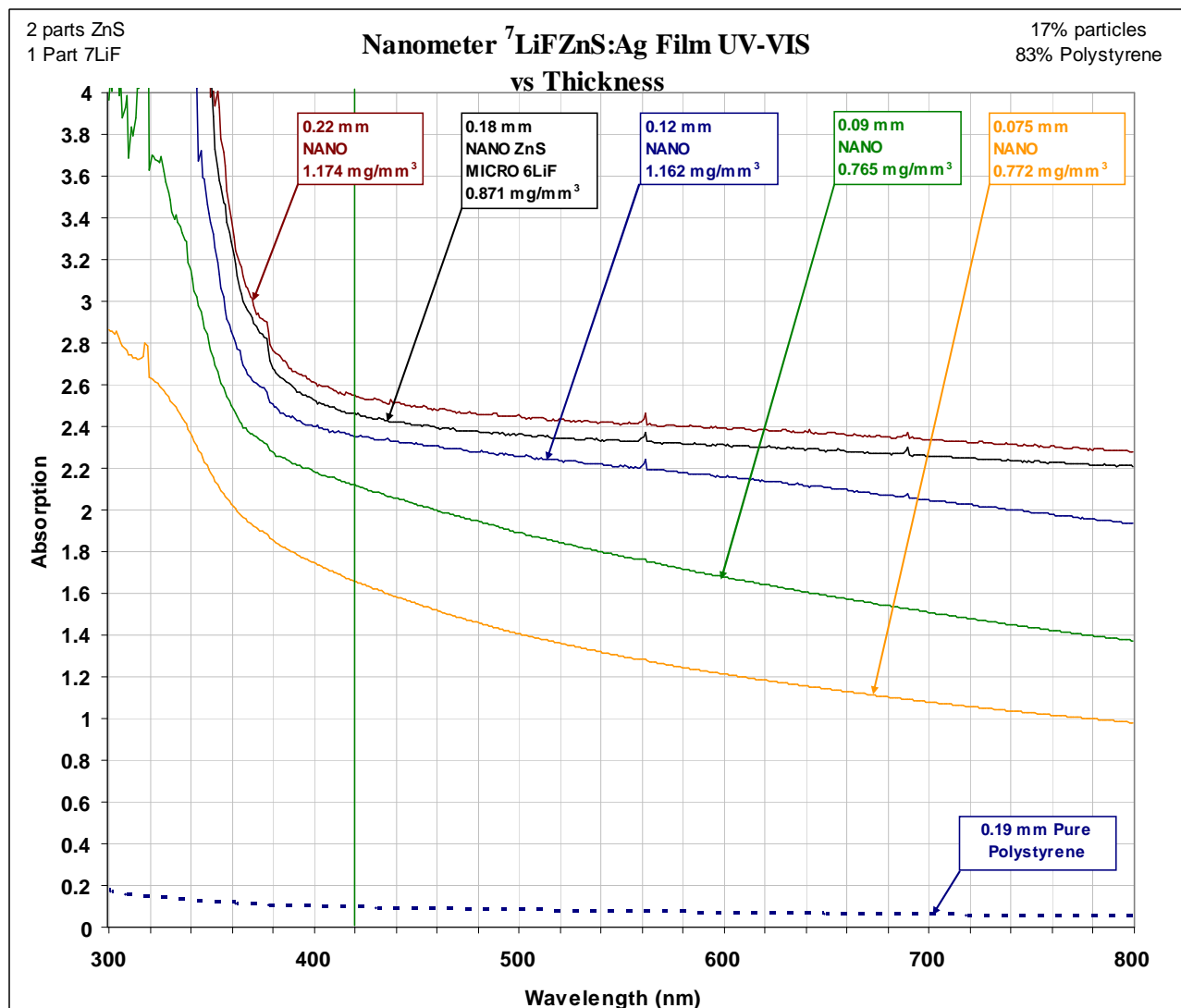
Batches 0 and 3 were made from <sup>7</sup>LiFZnS:Ag micro-particles and Batch 1 is made from <sup>7</sup>LiFZnS:Ag nano-particles. The micro-particles were bulk doped ZnS:Ag obtained from StGobain, and is believed to be the same material used in their commercial neutron scintillators. Batch 0 was the first prepared and proved to be largely a learning experience that resulted in a reproducible technique to produce useable foil samples. The first thing to notice about the Batch 0 micro-foils is that the spin coated foils have smaller density than the cast foils by a factor of 2 to 3. It was observed during spin coating that most of the batch solution was spun off the slide onto the sides of the spin coater, leaving negligible amounts of material on the glass. Attempts to obtain uniform foils by spin coating at various speeds from 500 to 3000 rpm and various mixture viscosities resulted in either very non-uniform foils at low rpm or very thin and low density foils at high rpm. This led to the development of

the casting technique that worked well for the Gd<sub>2</sub>O<sub>3</sub> foils. Spin coating was therefore dropped in favor of the casting technique to produce scintillator foils.

Light absorption at 420 nm as a function of scintillator thickness shows statistically no difference for the <sup>7</sup>LiFZnS:Ag nano- and micro-particle prepared foils. Multiple UV-VIS measurements, which proved to be very reproducible, were made on all foils to eliminate anomalous results. The absorption curves for the micro-particle foils from batches 0 and 3, fabricated 1 month apart, agree quite well with one another as shown in Fig. 9. The UV-VIS data for the nano-particle <sup>7</sup>LiFZnS:Ag foils produced from Batch 1 are shown in Fig 10.



**Figure 9.** UV-VIS measurements for various thickness <sup>7</sup>LiFZnS:Ag foils made using micro-particles cast in Polystyrene from Batches 0 and 3.



**Figure 10.** UV-VIS measurements for various thickness <sup>7</sup>LiFZnS:Ag foils made using nanoparticles cast in polystyrene from Batch 1.

*These data are plotted on the same scale as the Gd<sub>2</sub>O<sub>3</sub> plotted in Fig. 8. There are two obvious differences in the absorption curves for nano- and micro-particle foils shown in these figures. First, light absorption increases much more abruptly below 400 nm for the nano-particle foils than for the micro-particle foils. This phenomenon was not observed for the Gd<sub>2</sub>O<sub>3</sub> foils. Second, the absorption of light above about 500 nm is significantly smaller for nano-particle foils than for micro-particle foils, but only for foils of thickness less than about 0.1 mm. This result was anticipated to occur for all visible light at the beginning of this development work. Again, as the thickness of the foil increases to about 0.2 mm, this difference disappears. These results were observed in all LiFZnS:Ag micro/nano foil comparisons, including the foils made using micro-LiF and nano-ZnS particles. As a point of reference, a light absorption measurement for a 0.19 mm thick pure polystyrene foil is shown in all UV-VIS plots presented here.*

*The unexpected result from these data, however, is that the absorption of light between 350 and 450 nm is nearly the same for the same thickness nano- and micro-particle foils. Absorption of 420 nm light for 0.25 mm thick <sup>7</sup>LiFZnS:Ag is 2.551 for nano-foils and 2.508 for micro foils. For 0.10 mm thick foils the micro- and nano-foil absorptions at 420 nm average 1.77 and 1.89 respectively. If anything, the micro-particle foil absorption of light is slightly lower than observed for the nano-particle foils in this frequency range. Again this nano-/micro-foil absorption difference, and the absorption change with thickness decreases as they approach 0.25 mm. These observations demonstrate that there is a fundamental light scattering process occurring in these foils which mitigates the use of nano-particles in the LiFZnS scintillators*

Also notice that in Fig.10 there is a UV-VIS measurement of a <sup>6</sup>LiFZnS:Ag foil fabricated using 5 μm <sup>6</sup>LiF and 100 nm ZnS:Ag in house fabricated nano-particles (Batch 5 in Appendix C & D). This was done because the milled nano-particle LiF powders were gray in color as a result of the milling process at Powdermet, Inc, and it was thought that these color centers might contribute to higher light absorption values for the LiFZnS nano-foils. However, the absorption curve for this 0.18 mm thick foil is again nearly the same as a 0.22 mm thick foil made using all nano-particles. Although comparing <sup>6</sup>LiFZnS with <sup>7</sup>LiFZnS in this case, it will be shown that the light adsorption properties of <sup>6</sup>LiFZnS and <sup>7</sup>LiFZnS are very much the same, as one might expect.

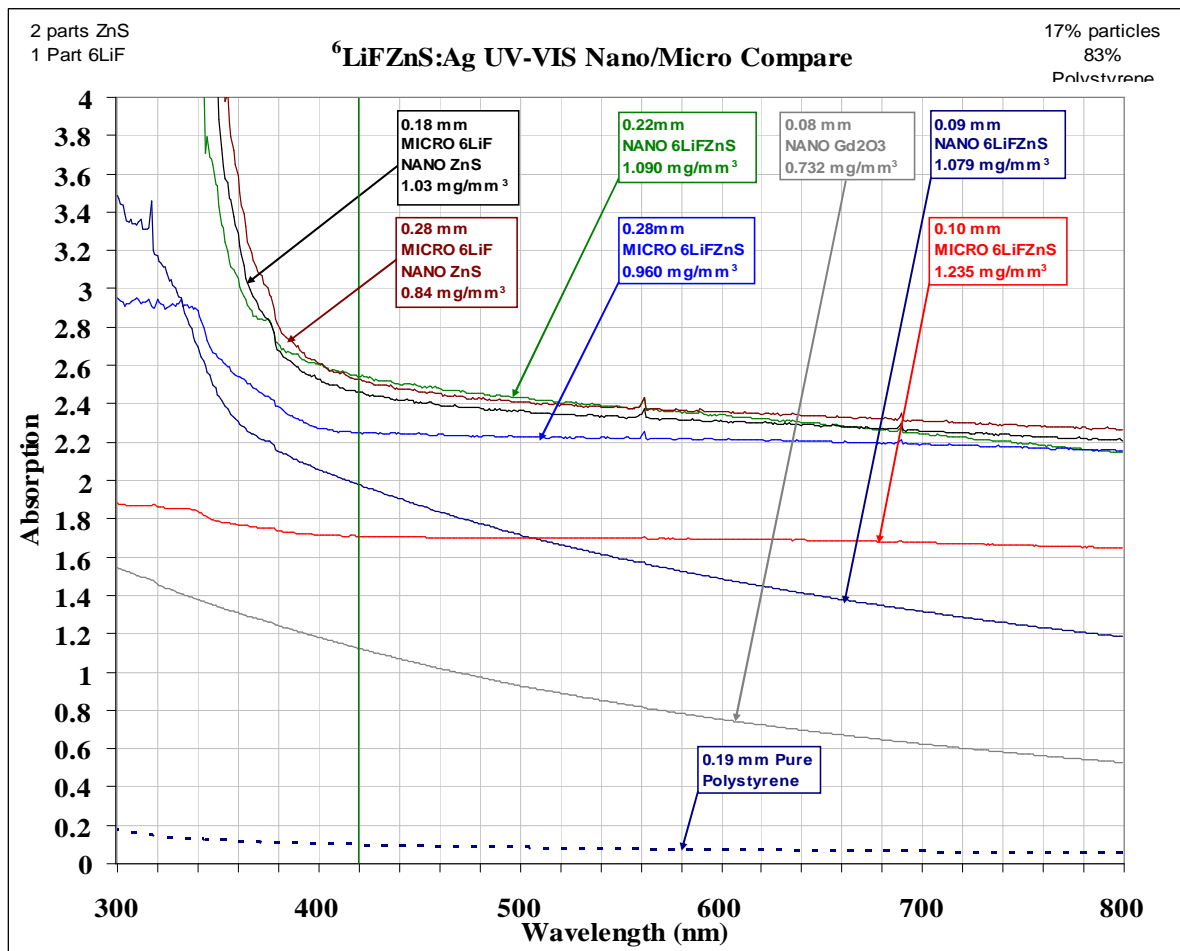
## **VII .2 <sup>6</sup>LiFZnS:Ag Foil Fabrication and Measurements**

Nearly identical results were obtained for micro- and nano-particle foils made using <sup>6</sup>LiF particles (Batches 2 and 4). These data are shown together in Fig. 11, which compares the micro- and nano- foil absorption curves for selected <sup>6</sup>LiFZnS:Ag foil thicknesses. In addition, two <sup>6</sup>LiFZnS:Ag foils(0.18 and 0.28 mm thick) made from Batch 5 (Micro <sup>6</sup>Li and Nano ZnS) and a 0.09 mm thick Gd<sub>2</sub>O<sub>3</sub> nano-particle foil (Batch 6) are shown for comparison. One immediately notices that the absorption of 420 nm light is significantly larger for the LiFZnS foils than for the Gd<sub>2</sub>O<sub>3</sub>, and that the absorption of light for LiFZnS nano- or micro-foils of thickness greater than about 0.2 mm are basically the same.

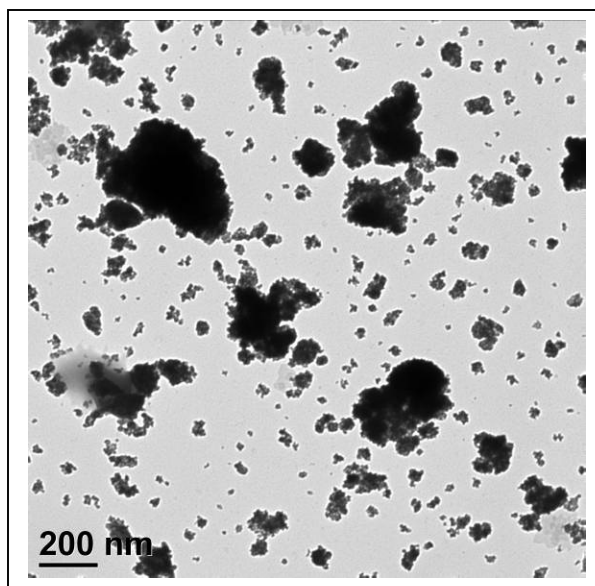
An initial reaction to the micro/nano LiFZnS comparisons was that perhaps the fabricated silver doped ZnS particles were not really nano-particles. TEM measurements of the fabricated ZnS were performed and are shown in Fig. 12. All particles are significantly smaller than 200 nm, with most smaller than 20 nm.

During fabrication of the LiFZnS mixtures, micro- and nano- ZnS particles were sonicated in Toluene for about 1 hour. The micro-particle ZnS remained suspended in the Toluene for no more than 3 minutes after stopping the sonicator, while the nano-particle ZnS remained suspended for the order of 15 to 20 minutes, and never really settled out completely. However, after adding the LiF to the ZnS in Toluene and sonicating again for about 1 hour, the LiFZnS settled in less than 20 seconds for both micro- and nano-particles, indicating that there was significant clumping of these particles. For this reason, the LiF and ZnS were sonicated separately in Toluene, and then added separately to the Polystyrene dissolved in Toluene while the Polystyrene solution was being magnetically stirred on the IKAMA hot





**Figure 11.** Comparison of micro- and nano-foils of <sup>6</sup>LiFZnS:Ag, <sup>6</sup>LiFZnS:Ag foils made from micro 6LiF and nano ZnS and nano Gd<sub>2</sub>O<sub>3</sub> foils.



**Figure 12:** TEM of Silver Doped ZnS in Toluene solution

plate stirrer. No TEM measurements of the mixture in polystyrene were possible to verify if this reduced the LiFZnS clumping. An indication, however, that the nano-particle solutions in polystyrene were truly different than the micro-particle solutions can be seen in the photo in Fig. 13. From left to right, the bottles contain nano-<sup>7</sup>LiFZnS:Ag, micro-<sup>7</sup>LiFZnS:Ag, nano-<sup>6</sup>LiFZnS:Ag and micro-<sup>6</sup>LiFZnS:Ag particles suspended in polystyrene and Toluene. These solutions were used to fabricate the foils listed in Appendix C and illustrated in the plots above. The photo was taken 8 days after the solutions were poured into the bottles. The micro-particle solutions show significant settling of the particle content, as seen by the ability to see the black background through the transparent Toluene in the upper portion of the solution, whereas the nano-particles remain well suspended in the Toluene.

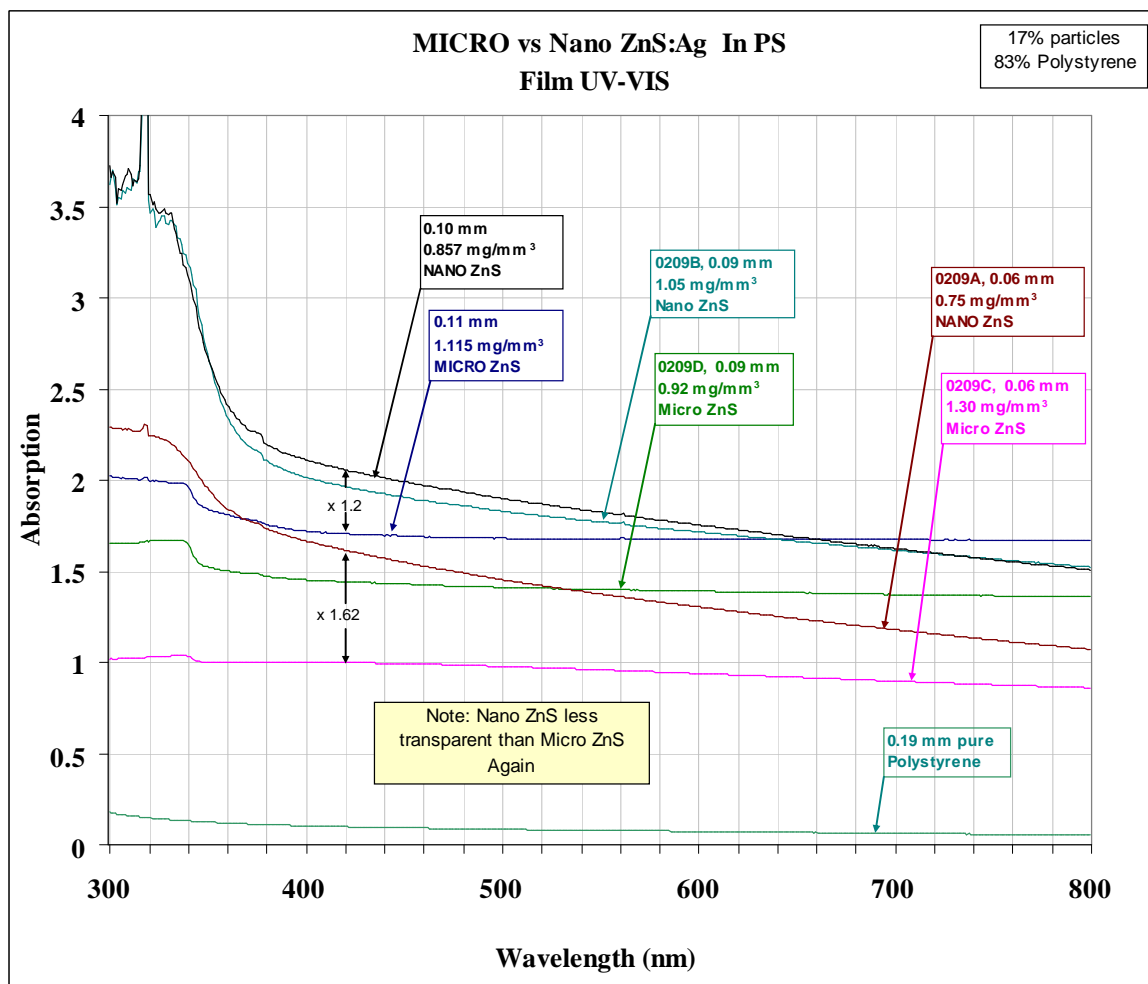


**Figure 13.** Bottles containing the LiFZnS/Polystyrene/Toluene solution fabricated in batches (left to right) 1, 3, 2, and 4 after 8 days of settling.

### VII. 3 ZnS Foil Fabrication

Another issue with the LiF nano-particles is their gray coloration caused by the milling of the 5  $\mu$ m particles to 76 nm using zirconia, which typically produces a 0.1% contamination of the powder being milled per 24 hrs of milling. The milling process took the order of 48 hours, causing a 0.2% contamination of the LiF. PartTec has no way of verifying this and we are

relying on the data provided by Powdermet for this estimate. However, because of this contamination, several foils of just ZnS:Ag nano- and micro- particles in polystyrene were fabricated to see if 420 nm light absorption by micro- and nano-particles would show the same improvements as the Gd<sub>2</sub>O<sub>3</sub> foils. Absorption measurements of these foils, fabricated from Batch 7b in Appendix C and D, are shown in Fig. 14. The unexpected result here clearly shows that nano-particle ZnS foils are very much less transparent than the micro-particle ZnS foils at all thicknesses. *Higher absorption values below 400 nm and lower absorption values above 500 nm for the nano-ZnS:Ag are also observed. These data appear to point toward the Silver doped ZnS as the primary scattering medium for the light in these scintillators.*

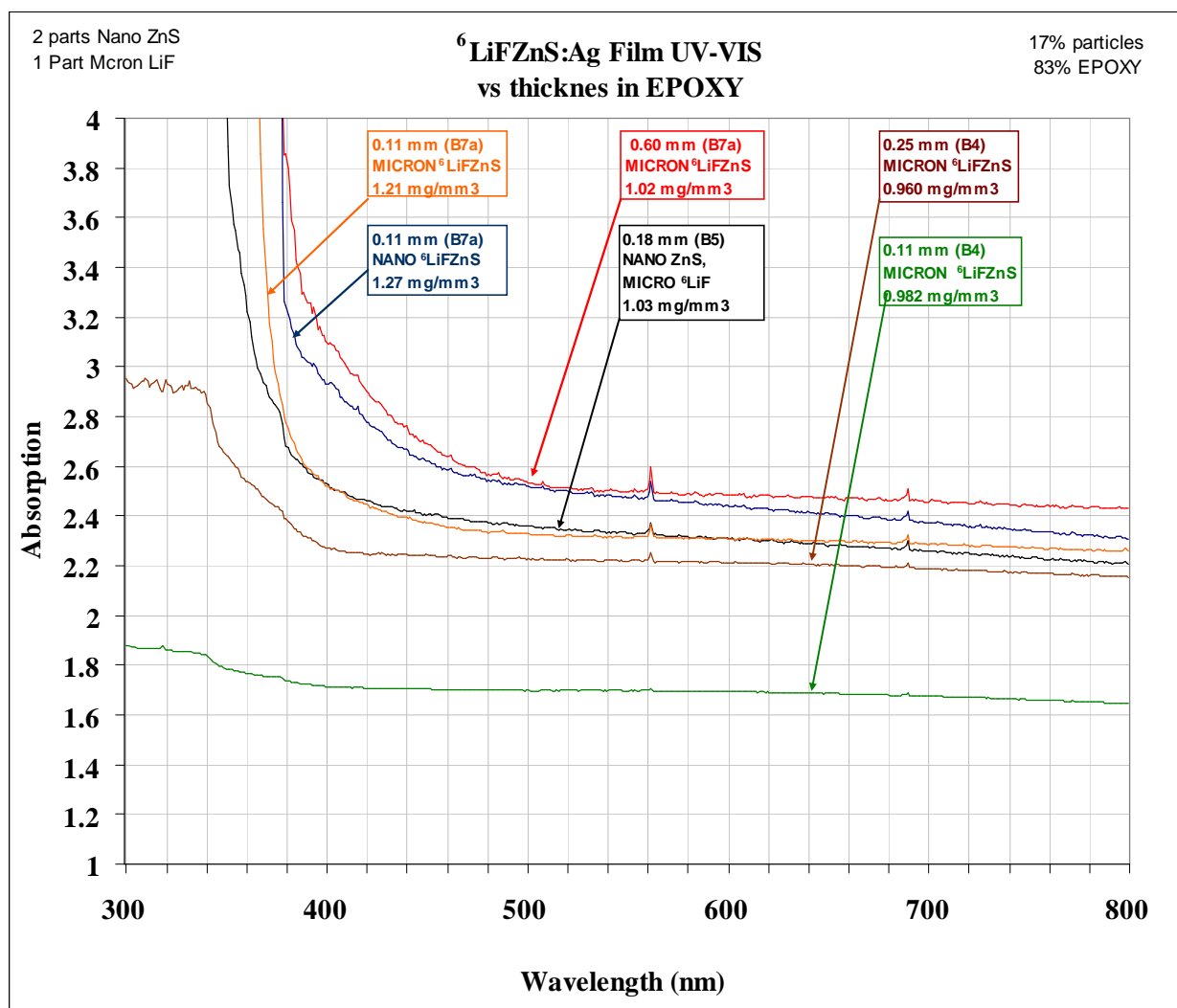


**Figure 14.** UV-VIS comparison of micro and nano foils of Silver Doped ZnS.

### VIII. LiFZnS(Ag) Fabrication in Epoxy

ZnS is a polar molecule, and will have a propensity to clump when mixed with LiF in Toluene, a phenomenon observed and described in Section VII above. In an attempt to avoid this problem, ZnS and <sup>6</sup>LiF particles were mixed separately in Acetone. These mixtures were

then immersed in a clear two-part epoxy solution also diluted with Acetone. This mixture was stirred until the solution had a viscosity appropriate for casting thin foils on glass slides, as was done for the Polystyrene based foils. The fabrication details and properties of these micro- and nano-particle foils in Epoxy are provided in Appendix C and D as Batch 7a. The UV-VIS measurements for the epoxy based foils are shown in Fig. 15, and compared with a 0.18 mm thick <sup>6</sup>LiF Micro- and ZnS nano-particle foil from batch 5 and two <sup>6</sup>LiFZnS micron particle foils from Batch 4. The particle/epoxy content of the foils was 17%, the same as used for all other foils produced using Polystyrene. The nano-particle foils had higher absorption than the micro-particle foils as was observed in the other measurements in section VII above. The foils using the Epoxy matrix also have similar light absorption at 420 nm as the micro and nano-particle foils produced in Batch 5. If anything, the epoxy foils were more absorbent to 420 nm light than the polystyrene based foils and the large increase in light absorption below 400 nm is also evident for the nano-particle foils. It is interesting that the 0.60 mm thick micro-particle foil in Epoxy also exhibits this behavior, unlike the thinner micro-particle foils shown in this figure.



**Figure 15.** Micro- and nano-particle foils in Epoxy matrix compared with foils mixed in Polystyrene

## IX. LiFZnS(Ag) Neutron Detection Efficiency Measurements

*Selected foils from those listed in Appendix C (marked with an asterisk) were taken to the Detector Development Group Lab at the Oak Ridge National Laboratory Spallation Neutron Source and tested with their in-house <sup>252</sup>Cf source. The source produced a flux of ~ 200 thermal neutrons per square cm from a 10 cm diameter aperture. The foil to be tested was mounted directly to the face of a PMT in a light tight manner, which was in turn connected to a PHA through a preamplifier. This setup was a standard test facility used by the Oak Ridge Detector Group to verify the performance of their newly developed detector systems. Two commercial neutron scintillators were used to verify the operation of the test facility, and provide a comparison for the new foils; a 0.25" thick Li Glass scintillator (Bicron GS20), and a 0.2 mm thick <sup>6</sup>LiFZnS(Ag) foil (Bicron BC704). A discriminator threshold was set to eliminate signal noise, and the total counts above this threshold for a 300 sec live time was collected for all samples. The results are listed in Table II below.*

**TABLE II**  
*Comparison of Thermal Neutron Detection Efficiency between Commercial and Fabricated Nanoparticle <sup>6</sup>LiFZnS Scintillators.*

<b>Scintillator ID</b>	<b>Material</b>	<b>Thickness (mm)</b>	<b>Counts/300 sec</b>
GS20	Micro-particle <sup>6</sup> Li-glass	10 mm	126,621
BC704	Micro-particle <sup>6</sup> LiFZnS	0.2 mm	24,203
0114C	Micro-particle <sup>6</sup> LiFZnS	0.28 mm	1775
0113C	Nano-particle <sup>6</sup> LiFZnS	0.22 mm	0
0117	Micro <sup>6</sup> LiF & Nano ZnS	0.10 mm	3
Sample 7	Nano-particle <sup>6</sup> LiFZnS	0.02 mm	0
E0207A	Micro <sup>6</sup> LiF & Nano ZnS	0.11 mm	1
E0206B	Micro-particle <sup>6</sup> LiFZnS	0.11 mm	113

The nano-particle scintillators fabricated under this SBIR failed to provide significant scintillation light when compared with the commercial scintillators. This is likely due to the fact that the ZnS nano-particles were surface doped rather than bulk doped, a process PartTec could not re-produce. This conjecture is supported by the scintillation counts observed in scintillators E0206B and 0114C, which were fabricated from commercially available micro-particle <sup>6</sup>LiF and Bulk doped ZnS(Ag) micro-particles from StGobain. If the particle density and thickness of the 0114C foil are accounted for, the 1775 counts for this foil would be about 1,500 counts when compared with the BC704. All foils fabricated with the surface doped ZnS failed to produce significant scintillation light from thermal neutrons.

## X. Summary

The development work conducted in this SBIR has so far not supported the premise that using nano-particles in LiFZnS:Ag foils improves their transparency to 420 (or other

frequency) light. This conclusion is based solely on the light absorption properties of LiFZnS foils fabricated from nano- and from micro-particles. Furthermore, even for the case of the Gd<sub>2</sub>O<sub>3</sub> foils, the transmission of 420 nm light gained by using nano-particles all but disappears as the foil thickness is increased beyond about 0.2 mm, a practical scintillator thickness. This was not immediately apparent from the preliminary study since no foils thicker than about 0.04 mm were produced. Initially it was believed that the failure to see an improvement by using nano-particles for the LiFZnS foils was caused by the clumping of the particles in Toluene due to the polarity of the ZnS particles. However, we found, much to our surprise, that nano-particle ZnS alone in polystyrene, and in Epoxy, had worse light transmission properties than the micro-particle foils for equivalent thickness and density foils. The neutron detection measurements, while disappointing, are attributable to our inability to procure or fabricate Bulk Doped ZnS nanoparticles.

The cause for the failure of nano-particles to improve the scintillation light, and hence improved neutron detection efficiency, is a fundamental one of light scattering within the scintillator. A consequence of PartTec's documentation of this is that several concepts for the fabrication of improved <sup>6</sup>LiFZnS scintillators were formulated that will be the subject of a future SBIR submission.

### References

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Web: [http://www.powdermetinc.com/Contact\\_us.html](http://www.powdermetinc.com/Contact_us.html)
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- [5] VWR Scientific products, Inc, West Chester, PA 19380; VWR Brand Crepe Fluted Filter Paper, 18.5 cm, 315 folded: Cat. No. 28331-060;
- [6] P. Calandra I, M. Goffredi, V. Turco Liveri, Study of the growth of ZnS nano-particles in water:AOT:n-heptane microemulsions by UV-absorption spectroscopy, *Colloids and Surfaces A: Physicochemical and Engineering Aspects* 160, 9–13 (1999)



## **Appendix A**

### **List of Equipment and Materials Used for the Nanoparticle Scintillator SBIR**

#### **Parttec Equipment List:**

1. Dr. Hielscher Ultrasonic Processor;  
Type UIP500 -115; 500 Watts Adjustable, 20 kHz, Continuous or Dry Ops
2. Cole Palmer Ultrasonic Processor;  
Type K-04711-60; 750 Watts Adjustable; 20 kHz
3. Cole Palmer Sound proofing Box  
Type K-04710-48
4. Spin Coater:  
Type SPS G3P-8; Variable Ramp and Dwell Cycles from 100 to 5000 RPM

#### **Chemistry Department Equipment Availability**

1. 50 kV SEM and TEM microscopes
2. Perkin-Elmer UV-VIS spectrometer
3. Various Hoods, Micro balances, Centrifuges, Hot Plates, magnetic stirrers and Glass Ware.
4. Schlank Tube Vacuum System.
5. Microwave and resistance ovens.

#### **Chemical Material Acquisition:**

##### **1. Gd<sub>2</sub>O<sub>3</sub>**

- a. 50 grams Gd<sub>2</sub>O<sub>3</sub>, 99%, 100nm Powder (*Sigma Aldrich*)
  - o TEM Verify Gd<sub>2</sub>O<sub>3</sub> → NOT 100nm, measured 2 um
- b. 100 grams, 99%, 10 -30 um Powder(*Am. Elements*)
- c. 10 grams Gd<sub>2</sub>O<sub>3</sub>, 99%, 100nm Powder (*Sigma Aldrich*)
  - o TEM Verify Gd<sub>2</sub>O<sub>3</sub> → Measured < 100 nm
- d. 25 grams, 99%, 20-40 nm Powder(*ALFA AESAR*)
  - o Same Vendor and Lot Number as used for preliminary work
  - o TEM Verify Gd<sub>2</sub>O<sub>3</sub> → Measured < 40 nm

##### **2. Li<sup>6</sup>F, Li<sup>7</sup>F**

- a. 25 grams <sup>6</sup>LiF, 95%, 5um Powder (*Sigma Aldrich*)
  - o 2 grams kept at parttec, 23 grams sent to Powdermet for milling [3]
  - o 9.7 grams 35 nm Li<sup>6</sup> delivered to Parttec, (TEM Verify 35 nm Powder)
- b. 150 grams <sup>7</sup>LiF, 99%, 5 um Powder (*Sigma Aldrich*)
  - o 100 grams <sup>7</sup>LiF kept at PartTec, 50 grams sent to Powdermet for milling
  - o 35 grams 76 nm Li<sup>7</sup> Delivered to PartTec (TEM Verify 76 nm Powder to be < 100 nm)

##### **3. ZnS:Ag**

- a. 100 grams Undoped ZnS, 99% 10 um Powder
- b. 100 grams ZnS:Ag, 99%, 10-30 um (*from StGobain*) [4]  
(TEM Verify ZnS:Ag, Measured 0.080 - 4um)
- c. ZnS:Ag; 20 nm Powder FABRICATED in I.U. Chem. Lab as needed  
(TEM Verify particle Size Measured 20 - 200 nm)

##### **4. Miscellaneous**

Various Solvents (i.e., Toluene, etc) and chemicals available through Indiana University Chemical Stores facility.

## **Appendix B**

### **Batch Preparation Details for fabrication of Silver Doped ZnS Nano-particles.**

Nanoparticle Silver doped ZnS was prepared via a known preparation that followed the prescription below;

Prepare:

- a. 0.1 mole solution of Zinc Acetate in water (dissolve 2.195 g Zn(Ac) in 100 ml water)
- b. 0.1 mole solution of Sodium Sulfide in water (dissolve 2.44 g Na<sub>2</sub>S in 100 ml water)
- c. 0.02 mole solution of PVP in water (dissolve 0.222 g PVP in 100 ml water)
- d. 0.0002 mole solution of Silver Nitrate in water (dissolve 0.005g AgN<sub>2</sub> in 100 ml water)

Dilute:

- a. 50 ml Zn(Ac) solution in 500 ml water, and add 50 ml PVP solution and 25 ml AgN<sub>2</sub>
- b. 50 ml Na<sub>2</sub>S solution in 500 ml water

Mix:

Mix solutions a. and b. together in a single beaker. Nanoparticle ZnS(Ag) particles immediately start to precipitate out of the solution. Let settle for several hours until all particles precipitate to the bottom of the beaker

Recover Nano-particles:

- a. Drain beaker of all but 150 to 200 ml of Na(Ac) solution leaving nano ZnS(Ag) particles.
- b. Stir remaining contents and pour into a standard crepe fluted filter[5]
- c. When fluid has drained through filter leaving precipitate, wash with ~ 150 ml water to dissolve and remove the Na(Ac), then with Mythl alcohol, leaving the ZnS precipitant on the filter paper. Let dry for several hours.
- d. Scrape ZnS off the filter paper and into a small test tube.
- e. Place test tube in a schlank tube until there is no further weight reduction with time under vacuum. (about 12 hours)

This process produced nearly 1 g of silver doped ZnS nano-particles per batch. The TEM measurement of the resulting particles sonicated in Toluene verified that the particle size ranged from 20 to a maximum of 200 nm. Most particles were less than 50 nm as shown in Fig. 12 in Section VII.

**Appendix C**  
**Nano- and Micro-Particle 17% LiFZnS:Ag Foils Fabricated for this Study**

Batch No.	Foil ID	Particle Type	Particle Size	Thickness (mm)	Density (mg/mm <sup>3</sup> )	Absorption @ 420 nm	Comments
0	1130	<sup>7</sup> LiFZnS	5-10 um MICRO	0.10	0.796	1.954	Cast on glass, 05 ml
	1203			0.01	1.13		Cast on glass
	1203A			0.28	0.858	2.503	Cast in mold, very uniform
	1204			0.06	0.242	-	Spun on glass @ 2000rpm, 2ml
	1207			0.22	0.779	2.366	Cast on glass, 1ml
	1208			0.11	0.296	-	Spun on glass @ 2000rpm, 2ml
	1210			0.05	0.326	-	Spun on glass @ 2000rpm, 2ml
	1211			0.05	0.739	-	Cast on glass, 1 ml
	1213			0.18	1.194	-	1 ml Cast on glass
1	0109	<sup>7</sup> LiFZnS	5-100 nm NANO	0.10	0.772	1.660	All foils cast on glass. Foils have 0.01 to 0.5 mm thick meniscus on edges, but uniform thickness in center
	0110			0.10	0.765	2.120	
	0111			0.28	1.174	2.551	
	0112			0.16	1.162	2.352	
2	0113A	<sup>6</sup> LiFZnS	5-100 nm NANO	0.075	0.831	1.975	Uniform thickness
	0113B			0.085	1.079	1.977	0.01 mm meniscus on edges
	0113C*			0.22	1.090	2.544	0.05 mm meniscus on edges
3	0115A	<sup>7</sup> LiFZnS	5-10 um MICRO	0.075	1.061	1.653	O115B Thickness varies from 0.085 to 0.12 mm top to bottom. 113A & C uniform
	0115B			0.10	0.917	1.773	
	0115C			0.25	1.072	2.295	
4	0114A	<sup>6</sup> LiFZnS	5-10 um MICRO	0.11	0.982	1.437	Uniform Thickness
	0114B			0.10	1.235	1.709	
	0114C*			0.28	0.960	2.247	Thickness varies 0.25 to 0.3 top to bottom
5	0116	<sup>6</sup> LiFZnS	5 um <sup>6</sup> LiF + 100 nm ZnS	0.075	0.729	2.231	Uniform Thickness
	0117*			0.10	1.235	2.402	
	0118			0.05	0.960	2.139	Thickness varies top to bottom
	0119			0.18	0.982	2.463	Uniform Thickness
	0120			0.09	1.235	2.403	
6	GdOum17	Gd <sub>2</sub> O <sub>3</sub>	10 um	0.03	0.982	1.664	Spin Coated foils from Preliminary work reported in proposal
	GdOnm17		40 nm	0.03	1.235	0.635	
	GdOum20		10 um	0.04	0.960	0.960	
	GdOnm20		40 nm	0.04	0.982	1.222	
	020108		40 nm	0.08	1.235	0.757	Cast foils fabricated in I.U. Chemistry Lab 2008
	020208		10 um	0.08	0.960	0.671	
	020308		40 nm	0.20	0.982	1.258	
	020408		10 nm	0.22	1.235	1.212	
7a	E0206A	<sup>6</sup> LiFZnS	10 um	0.60	1.021	2.898	Micron <sup>6</sup> LiFZnS Mixed with Epoxy and Acetone
	E0206B*		10 um	0.11	1.211	2.452	
	E0207A*	LiF Micro ZnS Nano	5-100 nm	0.11	0.779	2.777	Nano <sup>6</sup> LiFZnS Mixed with Epoxy and Acetone
	E0207B		5-100 nm	0.38	1.129	-	

7b	0209A	<b>ZnS</b>	5-100 nm	0.06	0.757	1.616	All cast on glass, Last batch fabricated and tested.
	0209B		5-100 nm	0.09	1.063	1.966	
	0209C		10 um	0.06	0.734	0.998	
	0209D		10 um	0.09	0.921	1.442	
	0210A		5-100 nm	0.10	0.857	2.058	
	0210B		5-100 nm	0.14	1.304	2.338	
	0210C		10 um	0.04	1.377	1.383	
	0210D		10 um	0.11	1.115	1.706	

**Appendix D**  
**Batch Preparation Details for Micro- and Nano-particle scintillator foils produced for the Organic Scintillator SBIR.**

Batch No.	Date	Particles Used	Weight g	LiFZnS/PS %	Fabrication Method/Comments
0	11/28/07	5um 7LiF	0.500	16.7%	- Used St Gobain ZnS(Ag) - Sonicate 7LiF & ZnS in 300 ml Tu for 2 hrs. - mix with PS & Stir solution at 70°C down to 60 ml
		StG ZnS:Ag	1.000		
		Polystyrene	7.500		
1	1/9/08	Nano 7LiF	0.424	14.8%	- ZnS:Ag Fabricated in 500 ml Batches. - TEM ZnS 20-200 nm & Powdermet 7LiF 76 nm, - Sonicated ZnS in 200 ml Tu 1Hr, ZnS+ LiF 1hr -stirred down to 75 ml
		Nano ZnS	0.848		
		Polystyrene	7.310		
2	1/10/08	Nano 6LiF	0.456	16.9%	- Used Powdermet 6LiF nanometer, - ZnS:Ag Fabricated in 500 ml Batches. - Sonicated ZnS in 200 ml 1Hr, ZnS+ LiF 1hr, - stirred down to 55 ml
		Nano ZnS	0.912		
		Polystyrene	6.730		
3	1/10/08	Micro 7LiF	0.500	16.7%	- Used St Gobain ZnS(Ag) - Sonicated ZnS in 200 ml 1Hr, ZnS+ LiF 1hr, - stirred down to 55 ml
		Micro ZnS	1.000		
		Polystyrene	7.500		
4	1/10/08	Micro 6LiF	0.500	16.7%	- Used St Gobain ZnS(Ag) - Sonicated ZnS in 200 ml 1Hr, ZnS+ LiF 1hr, - stirred down to 55 ml
		Micro ZnS	1.000		
		Polystyrene	7.500		
5	1/15/08	5 um 6LiF	0.351	17.0%	- ZnS Fabricated in 500 ml Batches. - Sonicated ZnS in 200 ml 1Hr, ZnS+ LiF 1hr, - stirred down to 80 ml
		Nano ZnS	0.703		
		Polystyrene	5.150		
6	1/29/08	Micro Gd2O3	1.000	17.0%	-Used American Elements 325 mesh Gd2O3 in TU sonicate and stir into PS
		Polystyrene	4.880		
		Nano Gd2O3	1.000	17.0%	-Used Alfa Aesar 40 nm Gd2O3 in TU sonicate and stir into PS
		Polystyrene	4.880		
7a	2/6/07	Nano 6LiF	0.080	16.4%	- ZnS Fabricated in 500 ml Batches, dried - Sonicated ZnS in 50 ml Acetone 1Hr, ZnS+ LiF 1hr, - Mix epoxy in Acetone & Stir
		Nano ZnS	0.160		
		EPOXY	1.220		
7b	2/9/08	Micro ZnS	0.500	17.0%	-Used StGobain ZnS in Tu sonicate and stir into PS
		Polystyrene	2.440		
		Nano ZnS	0.500	17.0%	-Fabricated ZnS, mixed in Tu sonicate and stir into PS
		Polystyrene	2.440		