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# Applying $\text{LaPO}_4$ Phosphor via Spinning for BetaPhotovoltaic Devices

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*Sensors and Electron Devices Directorate, ARL*

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<b>13. SUPPLEMENTARY NOTES</b>					
<b>14. ABSTRACT</b> The semiconductor fabrication technique of spinning photoresist was modified and applied to the problem of creating a uniform coating of phosphor on a betaphotovoltaic (BPV) device. A mixture of phosphor was applied to 3 samples (Si, GaN, and a GaN die with devices fabricated on its surface), and all 3 samples were spun at various speeds using a spinner. A uniform coating of phosphor was not achieved for 2 reasons: the phosphor does not fully dissolve in water, and the phosphor mixture does not stick well to the samples.					
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## 1. Introduction

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Demand is increasing for microscale energy systems that are lightweight and have a capability to produce electricity over long periods of time. Applications of such power systems include micro-electro-mechanical systems (MEMS), sensors in remote and hostile environmental conditions, medical devices (e.g., a pacemaker), among others. The US Army has need to ease the burden on Soldiers in small units by providing lighter, wearable, and more compact power.

The working principle of a betaphotovoltaic (BPV) device is similar to a solar cell, except that the electron hole pairs (EHPs) are generated in a BPV by photons emitted from a phosphor instead of natural light. The phosphor emits photons when beta rays, emitted by a beta source material, are incident on it. In order to generate maximum EHPs, it is important to have a uniform coating of phosphor so beta rays are incident on a smooth surface of phosphor for a maximum photon generation.

In semiconductor device fabrication, uniform coatings of photoresist are routinely achieved via the technique of spinning. Our objective was to modify this technique to apply a uniform coating of phosphor on our devices. The general process steps of this technique are: mix phosphor and water, apply mixture to the sample, spin, and bake. The details of this experiment are discussed below.

## 2. Experiment

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A mixture was made with  $\text{LaPO}_4$  phosphor and water (0.15 g of  $\text{LaPO}_4$  was added to 2.35 g of water). The mixture was sonicated and stirred for 2 min. I observed that the phosphor does not fully dissolve in water. If the solution is not stirred for approximately 2 min, the phosphor powder settles on the bottom of the beaker. The mixture was applied to the sample with a dropper and the sample was spun using a spinner. The sample was baked on a hot plate to dry off the water.

I performed the following experiments to see if a uniform coating of phosphor is achieved via the spinning technique:

1. Mixture applied to a silicon sample, spun at 2000 rpm for 30 s, and baked at 110 °C for 30 s.
2. Mixture applied to a gallium nitride sample with fabricated devices on the surface. Spun at 2000 rpm for 30 s and baked at 110 °C for 30 s.

3. Mixture applied to a gallium nitride sample with no devices on the surface, spun at 1000 rpm for 20 s, and baked at 110 °C for 15 s.
4. Hexamethyl disilazane (HMDS) solution applied to the sample (g3123p-2), spun the sample at 1000 rpm for 15 s and baked at 110 °C for 15 s. The phosphor mixture is applied to the sample, spun at 1000 rpm for 15 s and baked at 110 °C for 15 s.

The parameters of the experiment were changed because we wanted to observe how the uniformity of phosphor is affected when the substrate is changed, how the sample is spun with or without fabricated devices on the surface, the spinner speed, or the use of HMDS (adhesion promoter) before phosphor application. The results of this experiment are discussed below.

### 3. Results

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Figure 1 depicts the optical microscope image of the silicon surface before the phosphor was applied, while Fig. 2 shows the surface after phosphor application. As shown in Fig. 2, a uniform coverage of phosphor was not observed on the surface.

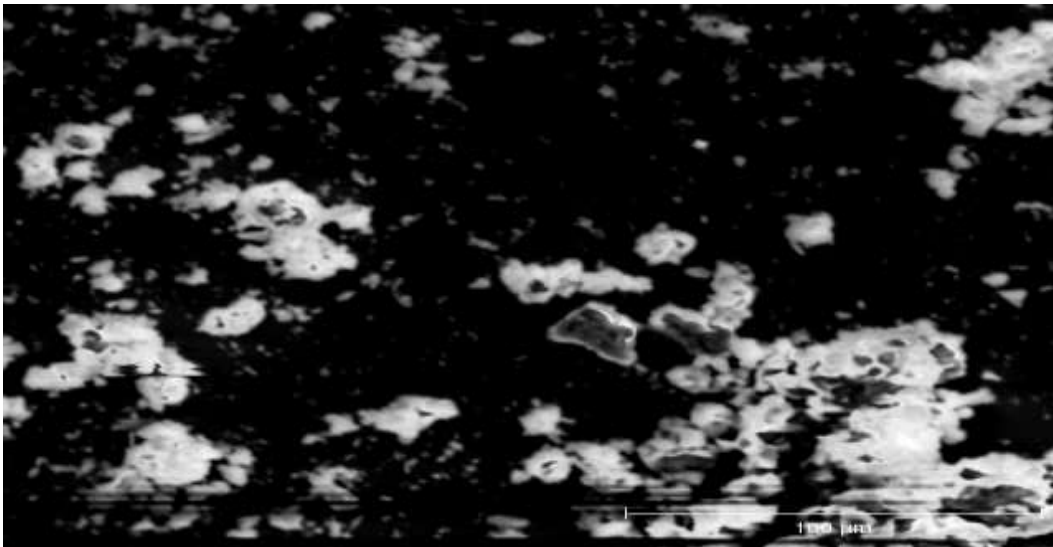


**Fig. 1** Optical microscope image of sample 20141120\_si before phosphor spin (20141120-1116\_20141120\_si\_om\_mrk.png)



**Fig. 2** Optical microscope image of sample 20141120\_si after phosphor spin (20141120-1208\_20141120\_si\_om\_mrk.png)

Secondary electron images were captured on the sample (20141120\_si) using the scanning electron microscope (SEM). When an SEM image is taken at a higher resolution, the grains of LaPO<sub>4</sub> are visible on the surface. Grain size is on the order of 100s of nm, as shown in Fig. 3.

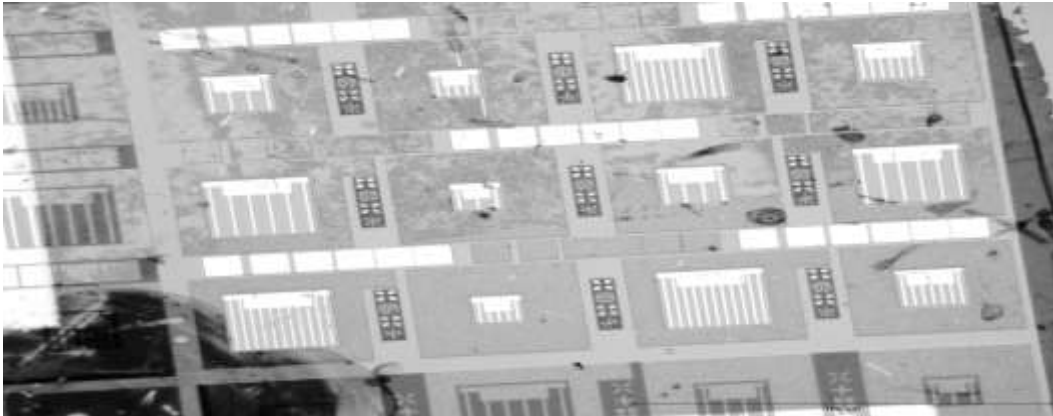


**Fig. 3** Secondary electron image of sample 20141120\_si (20141124-1532\_sei\_20141120\_si\_kwk.png)

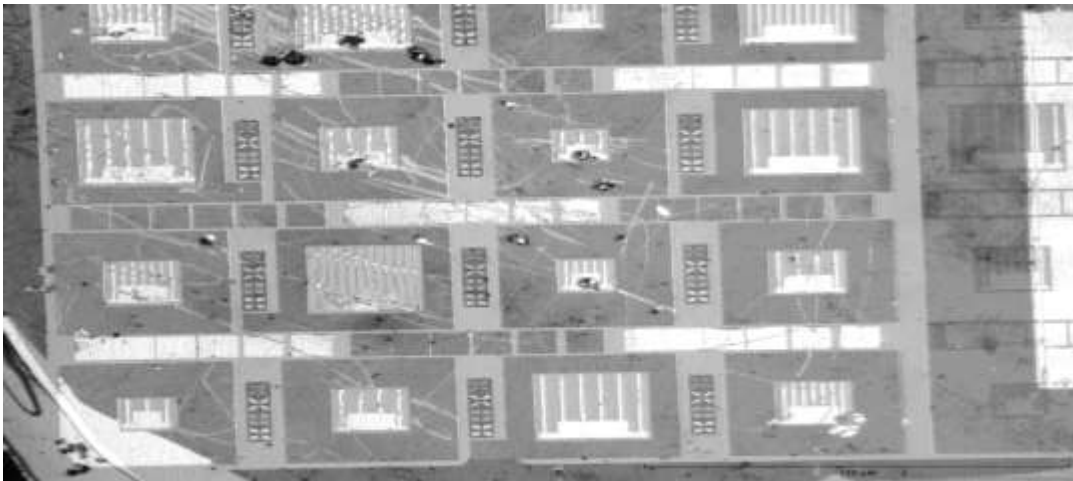
I observed that the LaPO<sub>4</sub> phosphor does not stick well to the surface. When the sample is baked to dry off the water, the powder remains on the surface but it comes off easily during handling. This is akin to what would have happened had

we not had the mixture and just applied the  $\text{LaPO}_4$  phosphor directly to the surface.

To test uniformity of phosphor application to a sample with devices fabricated on the surface, I performed the same experiment as above on sample g3123P-2. Sample g3123P-2 was fabricated with betavoltaic devices on the surface, similar to the device structures for the betaphotovoltaic project. Figure 4 depicts the optical microscope image of the surface before the phosphor was applied, while Fig. 5 shows the surface after phosphor application. As shown on Fig. 5, a non-uniform patchy coverage of phosphor was observed.



**Fig. 4** Optical microscope image of sample g3123p-2 before the phosphor application (20141120\_1409\_g3123p-2\_OM\_mrk.tif)



**Fig. 5** Optical microscope image of the sample g3123p-2 after phosphor application (20141120\_1415\_g3123P-2\_OM\_mrk.tif)

To test the uniformity of phosphor application at a slower speed, I applied the  $\text{LaPO}_4$  phosphor mixture to sample, g3138a-13, and spun it at 1000 rpm for 20 s. Figure 6 shows the optical microscope image of sample before phosphor

application, while Fig. 7 shows the optical microscope image after phosphor application. Spinning at a slower speed didn't change anything. The same non-uniform patchy coverage of phosphor was observed, as shown in Fig. 7.

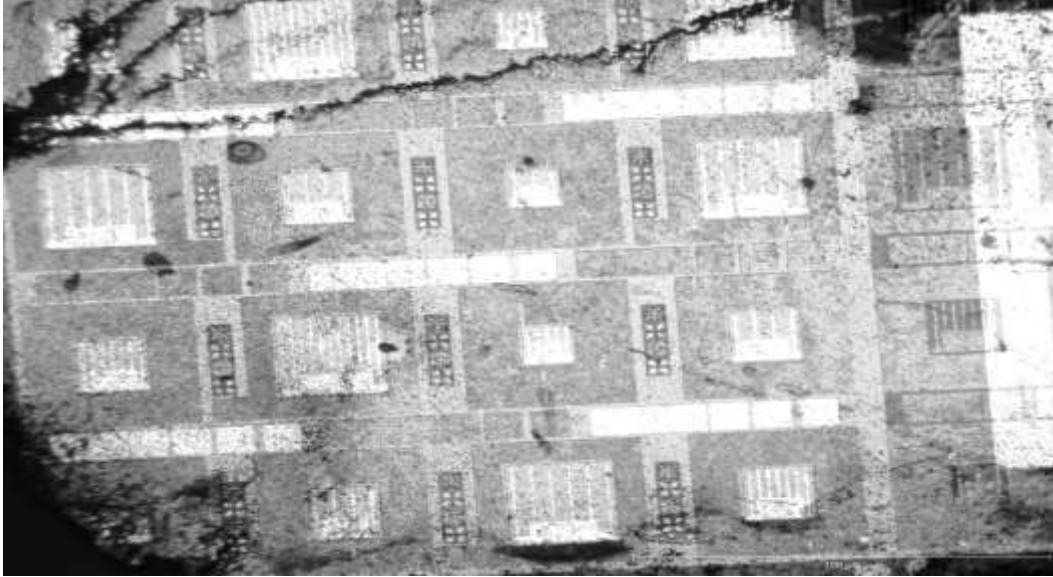


**Fig. 6** Optical microscope image of sample g3138a-13 before the phosphor application (20141120\_1350\_g3138a\_13\_OM\_mrk.tif)



**Fig. 7** Optical microscope image of sample g3138a-13 after phosphor application (20141120\_1407\_g3138a\_13\_OM\_mrk.tif)

I stripped off the patchy phosphor mixture from the g3123p-2 sample using acetone and applied HMDS on the surface before applying the phosphor mixture. HMDS is an adhesion promoter (helps in a better adhesion of photoresist to a substrate) and it is widely used in semiconductor fabrication. The use of HMDS before phosphor application did not help, either, as the same non-uniform patchy coverage of  $\text{LaPO}_4$  phosphor was observed, as shown in Fig. 8.



**Fig. 8** Optical microscope image of the sample (g3123p-2) after phosphor spin using HMDS and slower speed [20141120\_1423\_g3123p\_2\_OM\_mrk.tif]

#### **4. Conclusions**

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The typical photoresist spinning technique used in semiconductor fabrication does not yield a uniform coverage of phosphor on the devices and, therefore, is unsuited for this application. We experimented with several matrix parameters: changed the substrate, spun the sample at a slower speed with and without devices on the surface, and applied HMDS before the phosphor application. A uniform coating of phosphor was not achieved under any of these circumstances. Applying a uniform coating of phosphor is one of the most critical steps in the betaphotovoltaics project. The technique of spinning does not appear to be suitable for this purpose.

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