



# High Efficiency Concentrating Photovoltaic Power System

## Cooperative Research and Development Final Report

**CRADA Number: CRD-07-229**

NREL Technical Contact: Mowafak Al-Jassim

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Contract No. DE-AC36-08GO28308

**Technical Report**  
NREL/TP-5K00-73939  
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## Cooperative Research and Development Final Report

April 10, 2019

In accordance with requirements set forth in the terms of the CRADA agreement, this document is the final CRADA report, including a list of subject inventions, to be forwarded to the DOE Office of Science and Technical Information as part of the commitment to the public to demonstrate results of federally funded research.

**Parties to the Agreement:** Spectrolab/The Boeing Company (“Boeing”)

**CRADA number:** CRD-07-229

**CRADA Title:** High Efficiency Concentrating Photovoltaic Power System

**Joint Work Statement Funding Table showing DOE commitment:**

<b>Estimated Costs</b>	<b>NREL Shared Resources a/k/a Government In-Kind</b>
Year 1	\$50,000.00
Year 2	\$50,000.00
Year 3	\$50,000.00
<b>TOTALS</b>	<b>\$150,000.00</b>

### **Abstract of CRADA Work:**

Using NREL’s characterization capabilities, NREL will conduct electrical, optical, and materials characterization on semiconductor samples for concentrator solar cells, grown by both NREL and Boeing-Spectrolab. NREL will also apply its solar cell fabrication expertise to develop advanced, potentially low-cost concentrator solar cell fabrication processes, e.g. improved etching, metallization, coating deposition, isolation, and other processes. NREL will extend its experience with transparent conductive coatings (TCC) to develop terrestrial concentrator cell contacts using these materials. As required, NREL will carry out metalorganic vapor phase epitaxy (MOVPE) growth development of inverted metamorphic multi-junction solar cells and compare with Boeing-Spectrolab’s MOVPE growth processes.

### **Summary of Research Results:**

The primary benefit of this CRADA was the enduring relationship built with a primary U.S. manufacturer over the 11-year life of the CRADA. The U.S. partner invested in the technology over more than a decade, an example of effective technology transfer, at a time when the market for concentrator solar cells and the MOVPE technology was just being proven. NREL carried out MOVPE cell growth experiments over the duration of the CRADA to improve concentrator cell voltage, current, and efficiency and compared that with Boeing-Spectrolab MOVPE growth processes. Particular emphasis was placed on interface morphology, chemistry, and structure. These were found to play a major role in the generation and propagation of stacking faults and dislocations in these devices.

### **1. Introduction**

NREL developed metamorphic and lattice-matched multijunction cells and novel cell architectures based on the III-V system, an existing NREL technology, developed previously by DOE Program investment.

NREL was able to use its unique and vast expertise to assist Boeing-Spectrolab to conceive more robust cells. A variety of III-V alloys was grown and optimized with much of the efforts focused on GaAs/GaInP structures for high efficiency. The partners in this CRADA were able to resuscitate MBE growth capabilities for dilute nitride solar cells, focusing on wide-depletion-width, p-i-n structures that could current match 5- and eventually 4-junction devices. NREL characterized the performance of two different rf plasma sources to understand the incorporation of different nitrogen species (atomic, ionic, excited molecular, etc.) into Ga(In)As and determine their effect on device performance. Those efforts produced several series of GaInNAs(Sb) solar cells to understand the effects of Sb/In ratio on background doping and device performance, and shared these results with Spectrolab. NREL performed modeling studies of concentrator solar cell efficiency and energy production.

## **2. Re-establishing dilute nitride growth capabilities at NREL**

Although NREL has previously grown high quality dilute nitrides by MBE, that capability has not existed since 2008 when the plasma source used for the production of active nitrogen species was removed and given to another group at NREL. In order to grow dilute nitrides again, we needed to purchase a new rf plasma nitrogen source. Spectrolab was also interested in the effects of antimony (Sb) in dilute nitrides, and the MBE system no longer contained an Sb source, so it was also necessary to purchase this source. The addition of these sources exceeded the source capacity of the MBE system. The solution to this was to combine the dopant sources (silicon and beryllium) into one source port using a dual-dopant source, which also needed to be purchased.

Spectrolab purchased the following sources for use in the MBE system at NREL:

- SPECS PCS-RF-AN rf plasma nitrogen source
- Veeco LT-SUMO effusion cell for Sb
- Veeco Dual-dopant effusion cell for Si, Be

The sources were quoted in Jan. 2012 and ordered by Spectrolab in April 2012. The dual-dopant cell and the Sb cell arrived in early July 2012. The dual-dopant cell arrived with a damaged heat shield and was returned for repair. The repaired cell arrived at NREL in late Aug. 2012. In the meantime, it was determined by NREL management that NREL's ability to dope materials should not be dependent on a source that is borrowed from a collaborating institution, so NREL purchased a second dual-dopant cell that also arrived in late Aug. 2012. The Sb source and the NREL dual-dopant source were both installed on the MBE system during the next system vent in Nov. 2012. The Spectrolab dual-dopant cell was returned to Veeco for refund in Feb. 2013.

The SPECS nitrogen source arrived at NREL in Oct. 2012 and was also installed during the Nov. 2012 system maintenance cycle. We quickly discovered that an integral part of the source was broken. We obtained replacement parts, but continued to have part failures. After considerable trouble, we finally got the source back together with working parts, but we were unable to establish a plasma. It took three days of work with the source to get a plasma to fire, and then the plasma was not stable. We took the source out of the system again and found another part failure. We have experience with at least seven other plasma sources and are well aware of how to handle and operate them. It seemed clear to us that this source was simply not operating as expected, so after consultation with Spectrolab we decided to return the source for refund and order a plasma source from another vendor.

The new rf plasma source, an HD25 source from Oxford Applied Research, was quoted in Jan. 2013. During this time we became aware of an issue with borrowing equipment from outside organizations. There is an indemnity clause in the CRADA language that essentially says that if an injury occurs with

borrowed equipment, regardless of fault, the institution that owns the equipment will be held liable. After extensive efforts to remove or work around this provision, it became clear that it would remain in place for any equipment borrowed during the course of this CRADA. The return of the original plasma source and the ordering of a second source provided a way to avoid this indemnity clause by giving NREL the opportunity to purchase the source instead of borrowing it from Spectrolab, which is what occurred. The HD25 plasma source was ordered by NREL in Mar. 2013. Unfortunately, the Sb source that was originally purchased by Spectrolab was already installed on the MBE system and couldn't be returned in a similar way. This was finally remedied in Sept. 2013 when Spectrolab issued a quote to NREL and NREL purchased the source. There is no longer any borrowed equipment in this CRADA and the indemnity clause is no longer in effect.

The second plasma source arrived at NREL in April 2013, but the stainless steel rf shield was heavily pitted and was returned for replacement. The new rf shield arrived in early May 2013 and the source was installed during the next MBE system maintenance cycle in June 2013. After maintenance, repairs and system bake, the first growths using the plasma source commenced on July 30, 2013. Growth experiments continued until Sept. 25, 2013 when the III-V laboratory at NREL was shut down in order to replace ventilation in the lab space.

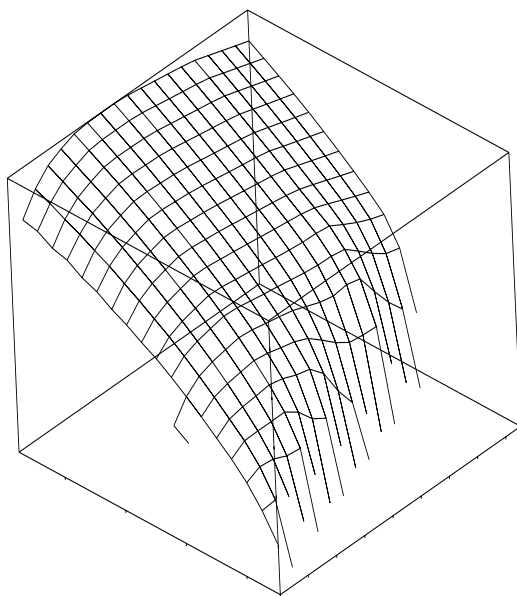


Figure 1. Optical emission from the HD 25 rf plasma source as functions of power and flow.

### **3. Growth and characterization of dilute nitride materials and devices**

#### *3.1 Plasma characterization and initial GaNAs epilayers*

Initial experiments focused on understanding the operation of the new HD25 rf plasma source and understanding the incorporation of N into GaAs using this source. Rf plasma sources produce a complex soup of atomic nitrogen, excited molecular nitrogen metastable species, and atomic and molecular ions, all on a background of inert molecular nitrogen. The plasma source has an optical port that allows direct optical access to the plasma emission. This light is filtered by a line filter centered at 780 nm and is coupled through a fiber into a photomultiplier. The 780 nm emission from a nitrogen plasma comes from optical transitions related to atomic nitrogen and can be used as a relative measure of the amount of atomic nitrogen produced in the source for different operating conditions. Figure 1 shows the atomic

nitrogen optical intensity as a function of both the rf power and the N flow. Several features are visible. At low power, a transition from an inductively-coupled plasma to a capacitively-coupled plasma takes place yielding a much lower energy plasma. This defines the minimum operating power for the source for a given N flow. This transition occurs at higher powers for higher flows. Another general feature is increasing atomic N emission for higher rf power. The relationship with N flow is a bit more complicated. There is a transition from a sparse plasma at low flows where the plasma chamber density is too low for significant collision events to create atoms, to a collision-limited plasma where nitrogen molecules do not have sufficient time to absorb energy before losing their energy to collisions.

It was hoped that we could use these different plasma regimes to study the effects of different plasma conditions on the quality of dilute nitride materials. Indeed, a set of Si-doped GaNAs layers were grown with the same optical signal, but different flows (changing the rf power to keep the optical signal constant). Electrical data from Hall measurements and composition data from x-ray diffraction measurements are shown in Fig. 2. The first observation is that the samples have significantly different amounts of nitrogen incorporation despite having the same optical signal. This may indicate that atomic nitrogen is not the specie that is predominantly responsible for incorporation into GaAs. There are also trends in the mobility and carrier concentration versus the N flow, however it's likely that these data are more sensitive to the N incorporation rather than telling us anything useful about the effects of changing the nitrogen flow into the plasma source. As expected, the electron mobility increases and the electron compensation decreases for lower N incorporation.

Ideally, we would use a spectrometer or multiple line filters to determine the relative amounts of atomic and molecular nitrogen coming from the source and their effect on the quality of dilute nitrides. For example, do the data in Fig. 2 make more sense if we track the optical output due to metastable molecular nitrogen instead of atomic nitrogen? Answering questions like this will be important for understanding how to improve dilute nitride device performance.

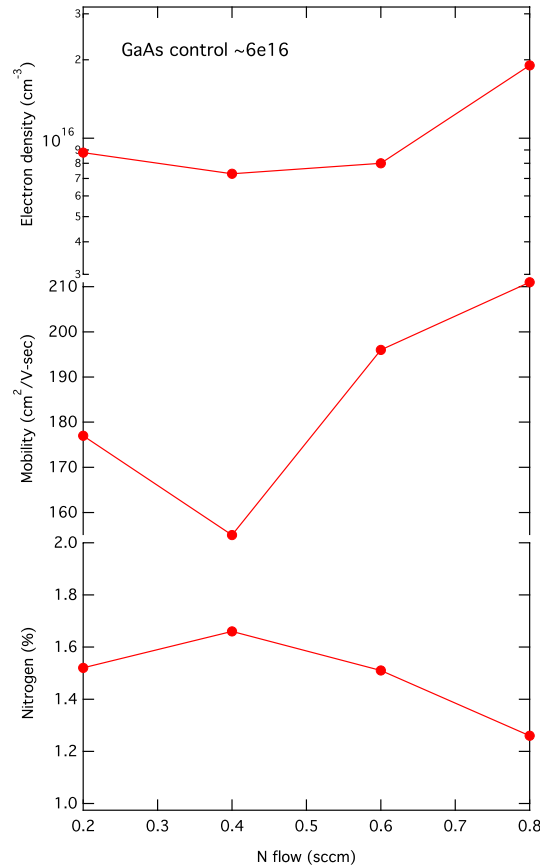


Figure 2. Electron density and mobility from Hall measurements for a series of GaNAs layers grown with constant plasma optical output and different N flows. Nitrogen incorporation was measured by symmetric XRD sca

### 3.2 In segregation and surfactants

With understanding of the use of the new plasma source and control over nitrogen incorporation, we proceeded to determine conditions for the growth of lattice-matched GaInNAs. Specifically, the growth space shrinks considerable in MBE for In-containing materials due to surface segregation of the In at high concentrations and/or high substrate temperatures. Previous work at NREL showed that the depletion width in p-i-n solar cells is highly dependent on the substrate temperature, shown in Fig. 3. At the substrate temperature necessary for the widest depletion regions, In segregation occurs for In incorporation levels greater than approximately 5%. This limits the lattice matched GaInNAs bandgap achievable before segregation to no lower than ~1.15 eV. It is possible to mitigate In surface segregation by using a surfactant such as Sb or Bi. Previous work at NREL identified issues with using either Sb (increased dark current) or Bi (increased n-type background) as surfactants for GaInNAs solar cells, although the materials were certainly extremely smooth. The cause of the detrimental surfactant effects have not been determined. In the case of Sb, it is possible that a new defect, or an increase in the concentration of an existing defect, caused the increase in the dark current. For the case of Bi, Te impurities were observed in SIMS measurements and may account for some of the increase in the donor concentration, although SIMS Te levels did not seem to account for all of the increase in n-type conductivity. However, it may also be that the previous Sb and Bi source material was contaminated in some way and that starting now with “fresh” source material will show different results. Coupled with increased knowledge of the available parameter space for wide-depletion-width solar cells, we are hopeful that Sb and Bi surfactants will be useful in the production of high-quality dilute nitride solar cells.



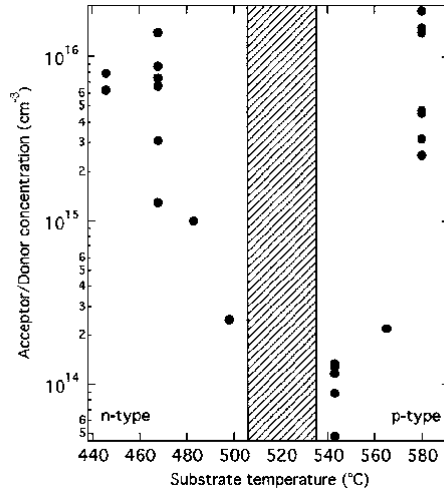


Figure 3. Compilation of CV measurements of the acceptor or donor concentration for GaInNAs epilayers. Samples are p-type to the right of the shaded region and n-type to the left of the shaded region. The shaded region represents material that is not clearly n- or p-type. From J. Vac. Sci. Technol. **B25**, 955 (2007).

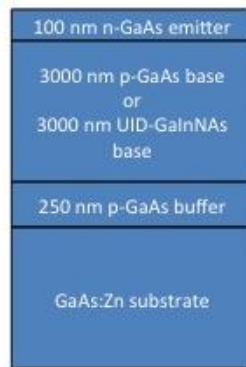


Figure 4. Schematic of the solar cell structure used here. Contacts are electroplated Au on both front and back.

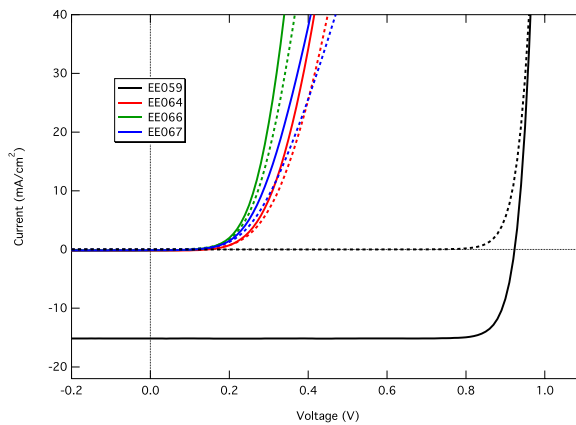


Figure 5. Light (solid) and dark (dashed) IV curves for a control GaAs solar cell (black) and several GaInNAs cells.

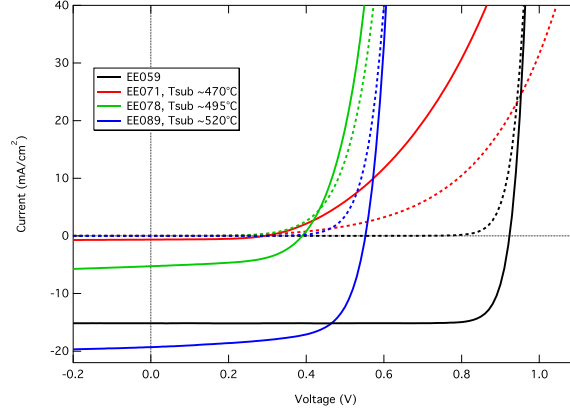


Figure 6. Light (solid) and dark (dashed) IV curves for a GaAs solar cell (black) and several GaInNAs cells grown at different substrate temperatures.

### 3.3 Initial device results

We grew a GaAs solar cell as a test of the operating state of the MBE system. The structure for this cell, and subsequent GaInNAs cells is shown in Fig. 4. The light and dark IV curves for the initial GaAs cell are shown in Fig. 5. The cell lacks a back-surface field and a window, but still shows respectable  $V_{oc}$  and  $J_{sc}$  values. Early lattice matched GaInNAs cells are also shown in Fig. 5. These cells show essentially no photocurrent. Scanning Kelvin Probe measurements showed that the junction in these devices was at the back of the structure and that the base layer was n-type. These samples were grown at a substrate temperature of  $\sim 440$  °C. Referring to Fig. 3, it is easy to see that an n-type base layer should be expected. The layers also had bandgaps near 0.98 eV. In surface segregation is negligible at this growth temperature, even with enough incorporated In to lattice match a cell at 0.98 eV.

The next set of GaInNAs cells incorporated two changes. The nitrogen source was decreased to the minimum operating conditions (before transitioning to a capacitively-coupled mode) and the substrate temperature was sequentially increased. Figure 6 shows the IV curves for several GaInNAs solar cells, showing increasing  $J_{sc}$  for increasing substrate temperature. Again, referring to Fig. 3, this is expected as the material becomes more depleted at the higher temperatures. Table I contains solar cell metrics for the devices shown in Fig. 6. The GaInNAs solar cell with the highest  $J_{sc}$  only has a current density of  $\sim 5.4$  mA  $cm^{-2}$  available below a GaAs filter, so additional work is required to achieve the necessary  $\sim 13$  mA  $cm^{-2}$  to current match a 5J structure.

Table I. Solar cell metrics for the samples shown in Fig. 6.

Sample	Material	Base doping	$E_g$ (eV)	$V_{oc}$ (V)	$W_{oc}$ (V)	$J_{sc}$ (mA/cm <sup>2</sup> )	FF (%)	J (>880nm)
EE059	GaAs	2e16	1.41	0.923	0.487	15.2	86.5	--
EE071	GaInNAs	UID	1.19	0.293	0.897	0.65	54.2	0.43
EE078	GaInNAs	UID	1.19	0.390	0.800	5.3	55.4	2.0
EE089	GaInNAs	UID ( $\sim 2e15$ )	1.20	0.552	0.648	19.3	66.9	5.4

These cells represent the highest bandgap (lowest N incorporation) that we can reach with the current plasma source configuration, so GaInNAs bandgaps between  $\sim 1.2 - 1.4$  eV are inaccessible. We have a smaller conductance aperture plate that we will install during the next maintenance cycle to allow for more range in our investigations. Also, the substrate temperatures that yield wide depletion widths are also where In surface segregation begins, and we are limited to bandgaps no lower than  $\sim 1.15$  eV before significant segregation occurs. We are therefore limited to a range between  $1.15 - 1.20$  eV bandgap materials that can achieve wide depletion widths. Clearly we need to continue to improve the depletion width, but in order to go to lower bandgaps and achieve higher current collection, we will need to invoke surfactants or find significantly different growth conditions.

**Subject Inventions Listing:**

None

**ROI #:**

None

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Office of Energy Efficiency and Renewable Energy (EERE) Solar Energy Technologies Office (SETO)