

Design of Critical Experiments for Scale-up

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SUMMARY

Commercial-scale equipment capable of producing large area photovoltaic modules with high throughput can only be effectively designed, operated, and controlled if a set of critical laboratory experiments has been planned, carried out and creatively interpreted. The panel and audience discussed these topics in relation to manufacturing today's thin film devices and modules..

INTRODUCTION

Critical laboratory-scale experiments are required to obtain information for the design, operation, and control of commercial scale equipment for profitable PV module manufacturing. A logical procedure must be followed in designing these experiments, or the pertinent information will not be obtained and resources will have been wasted. Figure 1 shows a logic diagram useful to help assure the effective use of resources. The iterative execution of these steps will be illustrated by examining aspects of the design of a reactor needed to produce 50-100 W Cu(InGa)Se₂ (CIGS) PV modules.

An interactive workshop was held at the Thin Film Photovoltaic Symposium at the University of Delaware on May 1, 1997. The workshop on the "Design of Critical Experiments for Scale-up" was lead by T.W.F. Russell of the Institute of Energy Conversion (IEC). Panel members and their topics were: T.W.F. Russell who discussed applying the logic diagram (Figure 1) to experiments identifying CIGS film formation and selenization kinetics; R. Gay who presented a comparison of CIGS module results from two differently sized and operated reactors; V. Dalal who described the complexity of plasma-CVD processing of a-Si; and S. Guha who compared the difference in priorities between research and manufacturing of triple junction a-Si solar cells. The key issues outlined by each panelist is given below.

CRITICAL EXPERIMENTS FOR CIGS ABSORBER

FORMATION

Proof-of-concept laboratory scale experiments must be completed before any step in the logic diagram can be carried out. There is over a decade of such experiments showing that high efficiency CIGS (15 to 17%) can be successfully made at the laboratory scale. There are today at least four different processes that have been extensively investigated. Two physical vapor deposition type reactors, one using three or four sources to produce CIGS from the elements and one using selenium to selenize copper indium layers, are commonly employed. CIGS has also been successfully made in CVD reactors using hydrogen selenide to selenize copper indium layers and in a rapid thermal processing reactor which rapidly heats a copper indium selenium sandwich to over 500°C.

To make a decision as to which process will be the best for commercial scale manufacture requires preliminary design sufficient to estimate capital and operating costs for the commercial scale equipment.

Any such engineering analysis must begin with a reaction and reactor analysis. The reaction analysis should quantify the film growth process by specifying the chemistry, the kinetics and the appropriate constants with their temperature dependence. It should be reactor independent. The reactor analysis is reactor specific and must deal with species delivery to the substrate and with heat, mass and momentum transfer issues for any specified reactor geometry. The mathematical models are used to present the reaction and reactor analysis quantitatively.

The results of a reaction analysis for CIGS is presented in Figure 2. The concentration time data were collected in a CVD reactor using hydrogen selenide and verified in a PVD reactor using a single selenium source. The solid and dashed

lines on the figure are mathematical model predictions.^{1,2} A reactor analysis which quantifies delivery of the species to the substrate in a PVD reactor has been developed and used to successfully design and build pilot scale units which grow semiconductor on a moving substrate. This has been reported in detail elsewhere.^{3,4,5}

Using the presently available analysis for CIGS deposition allows a preliminary commercial scale, four-source PVD reactor to be partially designed and identifies the next set of critical experiments needed to complete the design and to set the optimum operating conditions. A three or four source PVD reactor in which one can obtain concentration vs. time data is required. The Institute of Energy Conversion is in the process of building such a system to obtain this information.

CIGS MINI-MODULE FABRICATION FROM TWO DIFFERENT REACTOR SYSTEMS

Siemens Solar Industries makes CIGS by selenization of copper indium layers with hydrogen selenide. This presentation describes problems encountered in our scale-up of the reactors to make CIGS. This is illustrated by Figure 3 which plots efficiency of mini-modules for two reactors, one which produces 100 cm² modules (baseline) and one which produces 900 cm² modules (large). There are significant differences in average efficiency and in deviations from the average. More detail on the differences in device properties are given in Table 1.

A series of experiments were carried out in an attempt to resolve this problem. A critical experiment involved the selenization of the small area substrate in the large reactor. The devices produced from this experiment showed the same characteristics as the large area devices. This pinpointed the large reactor design and/or operation as the problem requiring attention. Selenium delivery to the substrate appeared to be

different between the two different size reactors with the large reactor being less effective.

Table 1. Differences in device properties.
Comparison of devices from baseline and large reactors.

	Baseline Reactor	Large Reactor	
Efficiency	11.6	7.8	(%)
Contact Resistance	2.9	9.2	(m ² -cm ²)
Bandgap	0.99	1.02	(eV)
SIMS sulfur profile	Sharp	Flat	
Adhesion over P1	Good	Unreliable	
Mo appearance	Shiny	Discolored	
Se/Mo in Mo	4	7.5	(%)
S/Mo in Mo	1.5	3.5	(%)

REACTION ANALYSIS OF PLASMA REACTORS

Amorphous silicon is made at the commercial scale by decomposing silane in a plasma CVD reactor. There are two firms producing product by such processing today but it is very much an art with almost no quantitative analysis available. A reaction analysis needs to consider the gas phase, the surface of the growing film, and the film growth itself. A reactor analysis must model species delivery to the substrate as a function of gas flow and reactor geometry. The reaction and reactor analysis should be able to predict silane conversion as a function of substrate temperature, reactor geometry and plasma properties. Similar modeling and analysis is needed to quantify film growth rate.

There are a number of critical experiments required:

- Key species in the plasma crucial to film growth need to be identified.

- The rate of delivery of these species to the substrate needs to be determined as a function of reactor geometry and gas flows.
- The influence of key species on film growth must be determined.

An engineering-type analysis is required. The issues are very complex so considerable judgment and creativity must be employed to concentrate the analysis on parameters which are of the most importance to the design, operation and control of the commercial scale equipment. This is not easy to do but the potential rewards are sufficient to justify the effort.

R & D VS. MANUFACTURING OF A-SI: ARE THE PRIORITIES THE SAME?

United Solar Systems Corporation (USSC) is presently producing product from a new 5 MW facility. The reactor to produce the a-Si modules is 140 feet long, has multiple chambers, and deposits a-Si continuously on a moving stainless steel substrate. USSC was able to design this system after many years of experimentation with smaller scale reactors operating in both a batch and continuous mode.

R & D efforts at USSC normally concentrate on raising the efficiency of a-Si devices and have been quite successful. They can readily make multi-junction devices at the laboratory scale with efficiencies over 13%. To attain this efficiency the growth rate is about 1 angstrom per second and in laboratory reactors they are not overly concerned with utilization of our raw material gases.

To operate a profitable business one must be concerned with both the capital and operating costs of production. A company needs to produce modules with the lowest possible cost per watt. While efficient modules are important, the most

critical issues for low cost production are growth rate and effective utilization of expensive gases (germane and disilane).

USSC feels that they must operate the commercial scale reactor at a film growth rate of three angstroms per second, approximately three times the rate used in the laboratory to make the high efficiency device. This is typical of the trade off that one must make in the production unit. A lower efficiency module is accepted because of the need for increased throughput. The critical experiments and analysis that need top priority action are those which will identify the reactor operating parameters that control rate of film growth while maintaining high module efficiency. This is not unlike the CIGS problem described by Robert Gay.

USSC operates their production unit in a regime where the flow rate of the active gases is low but still gives adequate deposition rates. Significant optimization using a "design of experiments approach" were carried out to determine these gas flow rates but they still need to design experiments which will permit higher utilization of raw material gases, particularly germane.

DISCUSSION

Photovoltaic research and development is carried out with the ultimate aim of providing society with inexpensive electric power. This can only be done if the manufacturing cost of power generation modules can be reduced by a factor of about three from today's cost. To do this requires a creative engineering effort that must be part art. The photovoltaic community needs to raise its consciousness with regard to laboratory scale experimentation devoted to issues other than maximizing efficiency. This workshop has attempted to do this by discussing the logical steps and analysis required to define the critical experiments and by considering some of the problems faced with the large scale production of both CIGS and a-si modules.

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FIGURE CAPTIONS

Figure 1. Logic diagram.

Figure 2. Reaction analysis of CIGS growth.

Figure 3. Baseline versus large area efficiencies.



