# AREA II:

# PREPARATION OF NEW MATERIALS

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Our past research in this area consisted of expanding the reaction engineering methodology to describe the transport-kinetic interactions involved in manufacturing of new materials. Both modeling, based on fundamentals, and experimental work were pursued. Our goal has always been to facilitate the transfer of bench scale science to manufacturing and to improve the manufacturing processes. We have utilized this approach in the past in four areas:

i) preparation of semiconductor grade silicon,
ii) autoclave process for curing of high performance composites,
iii) microencapsulation and production of biomaterials,
iv) preparation of long carbon fibers.

Our research in the semiconductor silicon area has been dormant during the past few years (except for proprietary contract studies which cannot be reported here) but we may want to revitalize some new environmental aspects of it. A project was initiated in 1997 to quantify the effect of sonification on the reactivity, effectiveness and specificity of aqueous etching and in cleaning chemistries commonly used in the electronic materials industry. Unfortunately, the graduate student involved, Rebecca Smith, departed for personal reasons without completing her work.

We have perfected our models for devolatilization and resin flow during cure of long carbon fiber composites and we have extended their use to an increased number of polyimide systems with remarkable success. Using independently determined kinetic and mass transfer data, our model has the capability of relating operating procedure and composite quality, and is a useful tool in manufacturing. This success represents a good example of the virtues of team work involving specialists in composites (**J.L. Kardos**), reaction engineering (**M.P. Dudukovic´**) and control and optimization (**B. Joseph**). Work has been completed on modeling the manufacturing of carbon fibers and their carbonization in collaboration with the **Materials Research Laboratory** (MRL). This work is a joint effort of Professors **Kardos, Ramachandran** and **Joseph**.

Our microencapsulation project was completed several years ago and will not be reported on here. Electrophoretic purification of pancreatic islets of Langerhan has also been completed and provides a good quantification of the achievable degree of purification. This project was executed in the **Biological Transport Laboratory** (BTL) with input from CREL. The project of developing a new model and material for release system has been completed. This project also is executed in the BTL with input from CREL.

A brief report is included on the following project:

II-1. Semiconductor Grade Silicon: CREL know-how is reviewed. (**M.P. Dudukovic´** and **P.A. Ramachandran)**

# II-1. SEMICONDUCTOR GRADE SILICON

## A. Problem Definition

Many aspects of manufacture of semiconductor grade silicon can benefit by utilizing the reaction engineering methodology. Some of these are listed below through past accomplishments and suggested future work.

## B. Research Objectives

No specific objectives are set at present other than the overall goal to continue contributing via reaction engineering to improved and cleaner processes for silicon manufacture.

## C. Research Accomplishments

CREL contributed over the years to solution of the following problems in Si manufacture:

1. Polysilicon crystal growth in Siemens decomposers by silicon deposition via hydrogen reduction of chlorosilanes was modeled. Optimal operational window for industrial practice was identified.
2. Polysilicon crystal growth via silane pyrolysis was modeled. The model provided guidance for design and optimal operation of Komatsu decomposers.
3. Aerosol (free space reactor) for silicon production via silane pyrolysis was modeled. The model indicated that excessive staging would be needed to reach particle size of the order of 20 μm.
4. Fluidized bed for silicon production via silane pyrolysis was modeled. Model was used to identify operating and design conditions that minimized the formation of dust (fines) and maximized CVD growth.
5. A complete heat transfer model was developed for the Czochralski crystal puller. The model related the production rate and crystal quality to operating and design variables.
6. Improved model based CZ puller control was developed. Simultaneous crystal diameter and interface shape control was achieved. Innovative idea of jet cooling was introduced. Two NASA certificates of recognition were received for the work in this area.
7. A novel etcher for large wafers was developed and implemented in industry (contract work).
8. The analysis of IPA wafer drying has been completed and suggestions for improvement of particle removal made (contract work).

## D. Future Work

We are seeking opportunities to continue the work on the following topics:

A. Improved model based control of CZ pullers.
B. Novel acid etcher design.
C. Environmental control and contaminant elimination in various process steps of Si-manufacture.

## E. References


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