The SWIRL reactor

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Presented at Prometia Annual event
28th November 2017
Barcelona, Catalonia/Spain
Outline of talk:

- Background for development of concept
- Theoretical evaluation
- Experiments
- Results
- IPR
- Conclusions and future prospects
- Acknowledgements
Background for development of concept

• Initially, the need for a more energy efficient production of photovoltaic silicon was immanent

• High purity silicon is made through gaseous Si-compounds, i.e. $\text{HSiCl}_3 + \text{H}_2 \rightarrow \text{Si} + 3\text{HCl}$, in Siemens reactors
Background for development of concept

• To use silane, SiH$_4$, directly to form Si + 2H$_2$ by thermal decomposition would be an advantage

• One problem is the Si fines sticking to all surfaces and which is hard to handle

• Today REC uses fluidized bed reactor to solve this challenge
The Concept:

Requirements for process development:
• Based on thermal decomposition of silane
• Achieve a controlled mass transport of gas and fines
• An energy efficient technology compared to the currently used technology
• Possibilities for continuous production

⇒ Will a swirl flow reactor fulfil these requirements?
The swirl flow reactor

• Efficient energy transfer
  – Gas close to reactor wall at all times
• Controlled mass transport possible, needs carrier gas
  – Small particles swept along the gas flow
• Continuous process
Theoretical studies

• Swirl flow review
  – Some work done related to swirling flows and their applications, in particular on cyclones
  – No good analytical model describing swirl flow
  – Expanding swirl flow with reaction and heat transfer in concentrating cones has not been reported
  – No work using swirl flows to produce silicon
Theoretical studies

- Flow simulations – CFD
  - Software tool: ALSIM4
  - Simulations did not converge
  - Simulations still suggested that the swirl flow concept will work
Theoretical studies - CFD

• Interesting parameters are:
  – Gas flow, temperature, injection angle, reactor length, reactor diameter and nozzle size
Flow studies

• Experimental flow studies
  – Cold air + SiO$_2$, heated in reactor in order to simulate flow expansion due to reaction
  – LDA measurements (velocity components measured by Laser Doppler Anemometry)

• Influence of design parameters on flow
  – Reactor cone angle
  – Injection nozzle angle
  – Flow rate VS cone/cylinder diameter
  – Injection design
LDA – Laser Doppler Anemometry

• Measuring principle of LDA:
  – Doppler shift of light scattered from small particles
• Advantages of LDA
  – non-intrusive
  – no calibration required
  – sensitive to velocity magnitude and direction
  – measures single velocity components
  – high accuracy obtainable
  – no limit to measurable turbulence including reverse flow
• Disadvantages of LDA
  – particles in flow required
  – optical access to measuring point required
  – flow medium must be transparent
  – relatively expensive
LDA - Backscatter configuration

- Laser
- Bragg cell
- Colour splitter
- Single mode polarisation preserving fibres
- Fibre manipulators
- Multimode fibre
- Interference filters
- Colour splitter
- PM
- Back scattered light
- Single mode fibres
- Multimode fibres

Flow

www.pipas.no
Experimental setup
Measurements of swirl flow

SiO$_2$ in air, measured with LDA-equipment
Measurements of expanding swirl flow

Experimental results:

• Swirl flow preserved
• The swirl flow expands towards the middle of reactor
Isosurface plots – “sausage plots”
Conclusions from the initial tests

• Powder and inert carrier gas were shown to maintain the swirl flow
• Swirl flow was maintained
  – even when heating of gas made the flow expanding
  – And even when the reactor was conical, i.e. the flow was compressed
Experimental setup

• Initially, a quartz reactor was used
  – Transparent – possible to use Laser-Doppler Anemometry (LDA)
  – Conducts heat poorly
  – Much silicon stuck on the reactor wall
  – The reactor cracked, causing leaks
• Main results obtained with steel reactor
  – Conducts heat well
  – Less silicon stuck on the reactor wall
  – Possible contamination of Fe and other materials
• Injection of 5 % SiH₄ in Ar via an injection loop
• Ar used as carrier gas
• Si-fines collected at filter after cooling of mass flow
Experimental results

- The pattern on the filters depend on gas flow and temperature
- Reproducible system
Another set of operational parameters:
Experimental results - yield

• The yield of the reactor is 100 % in the sense that all silane reacts

• Collection of Si on filter challenging
  – Cooling effects at the end of the reactor due to water cooling
  – Turbulence at the end of the reactor due to the filter
  – Small Si particles seen on the far side of the filter

• Tested collection of Si in glycol
  – Loss of Si in filter housing and hose
Conclusions

• Swirl flow is stable given the flow and injection angle
• Powder or fines can be transported and controlled
• Conical reactor is possible, swirl maintained
• Temperature gradient can be applied
• Reactor is continuous and energy efficient since swirl at inner surface is maintained
IPR - Patents on swirl reactors

United States Patent [19]
Levin et al.


THERMAL REACTOR

Inventors: Robert A. Froesch, Administrator of the National Aeronautics and Space Administration, with respect to an invention of Harry Levin, Woodland Hills, Larry B. Ford, Pasadena, both of Calif.

[21] Appl. No.: 126,863

[51] Int. Cl. 3 C22B 26/00
[52] U.S. Cl. 422/200; 422/202;
[58] Field of Search 422/198, 200, 202, 224;
422/230; 53/204, 201, 459 C, 459 F; 423/349

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14 Claims, 5 Drawing Figures

Primary Examiner—Hiram Bernstein
Attorney, Agent, or Firm—Paul F. McCaul, John R. Manning

[57] ABSTRACT

A thermal reactor apparatus and method of pyrolytically decomposing silane gas into liquid silicon products and hydrogen by-product gas is disclosed. The thermal reactor (1) has a reaction chamber (21) which is heated above the decomposition temperature of silane. An injector probe (106) introduces the silane gas tangentially into the reaction chamber (21) to form a first, outer, forwardly moving vortex (22) containing the liquid silicon product and a second, inner, rearwardly moving vortex (23) containing the by-product hydrogen gas. The liquid silicon in the first outer vortex (22) deposits onto the interior walls (28) of the reaction chamber (21) to form an equilibrium equilibrium layer (26) which flows to the forward or bottom end of the reaction chamber within the liquid reaction stage where it is removed. The by-product hydrogen gas in the second inner vortex (23) is removed from the top or rear of the reaction chamber by a vortex finder (30).

The injector probe (106) which introduces the silane gas into the reaction chamber (21) is continually cooled by a cooling jacket (110) having water circulating through it to keep the temperature of the silane gas below its decomposition temperature prior to being introduced into the reaction zone.

[19] United States
Eriksen et al.

[45] Pub. Date: Nov. 27, 2008

[54] METHOD AND REACTOR FOR CONTINUOUS PRODUCTION OF SEMICONDUCTOR GRADE SILICON

Inventor: Dag O. Eriksen, Kongsberg, Norway

[30] Foreign Application Priority Data

Jul. 16, 2004 (NO) ........................................... 20043187

Publication Classification

42/1004 E01J 16/00
425/354; 422/129

ABSTRACT

This invention relates to a method and reactor for continuous production of semiconductor grade silicon by decomposition of a silicon containing gas of ultra-high purity to particular silicon and other decomposition products in a free-space reactor and in which the gaseous stream of decomposition gas is set into a swirl motion. Optionally, the method and reactor also includes means for removing the formed particulate silicon to obtain a continuous phase of elementary silicon, and then casting the liquid silicon to form solid objects of semiconductor grade silicon.

IFE later withdrew the application, i.e. the technology is free
Future prospects - technical

• The swirl reactor is working most effectively when the diameter is small, but many may be stuck together, i.e. “spherical honeycomb”.
• One reactor may have more than one inlet
• Heating devices and material may be challenges.
• Swirl reactor should be well suited for induction heating.
Future prospects - chemical

- Heating of gases together with powder works. Possible uses of the swirl reactor:
  - Thermal purification, e.g. heating of zeolites
  - Carbochlorination, e.g. $\text{TiO}_2 + \text{C} + 2\text{Cl}_2 = \text{TiCl}_4 + \text{CO}_2$
  - Reactions generating $\text{H}_2$, e.g. reforming of methane: catalyst + $\text{CH}_4 + \text{H}_2\text{O} = \text{CO} + 3 \text{H}_2$.
  - ......
Acknowledgements

• The project was supported by the Research Council of Norway as a Strategic Institute Project at Institute for Energy Technology (IFE), Kjeller, Norway

• Thanks to research scientist Oddvar Gorset and post doc. Håvar Gausemel. Both contributed significantly.

• Thanks to IFE for releasing the patent rights

• Thanks to Prometia for letting me present the Swirl reactor concept

• Thank you for your attention
INVITATION

The 4th Seminar on Hydrometallurgy will take place at Thon Hotel Opera in Oslo
March 6. & 7. 2018

Please visit www.hydromet.no to see details and program