



Polysilizium geht online: Der Einsatz von Prozess- Gaschromatographen in der Polysilizium-Produktion

GDCh Arbeitskreis Prozessanalytik

6. Kolloquium

8. & 9. November, 2010

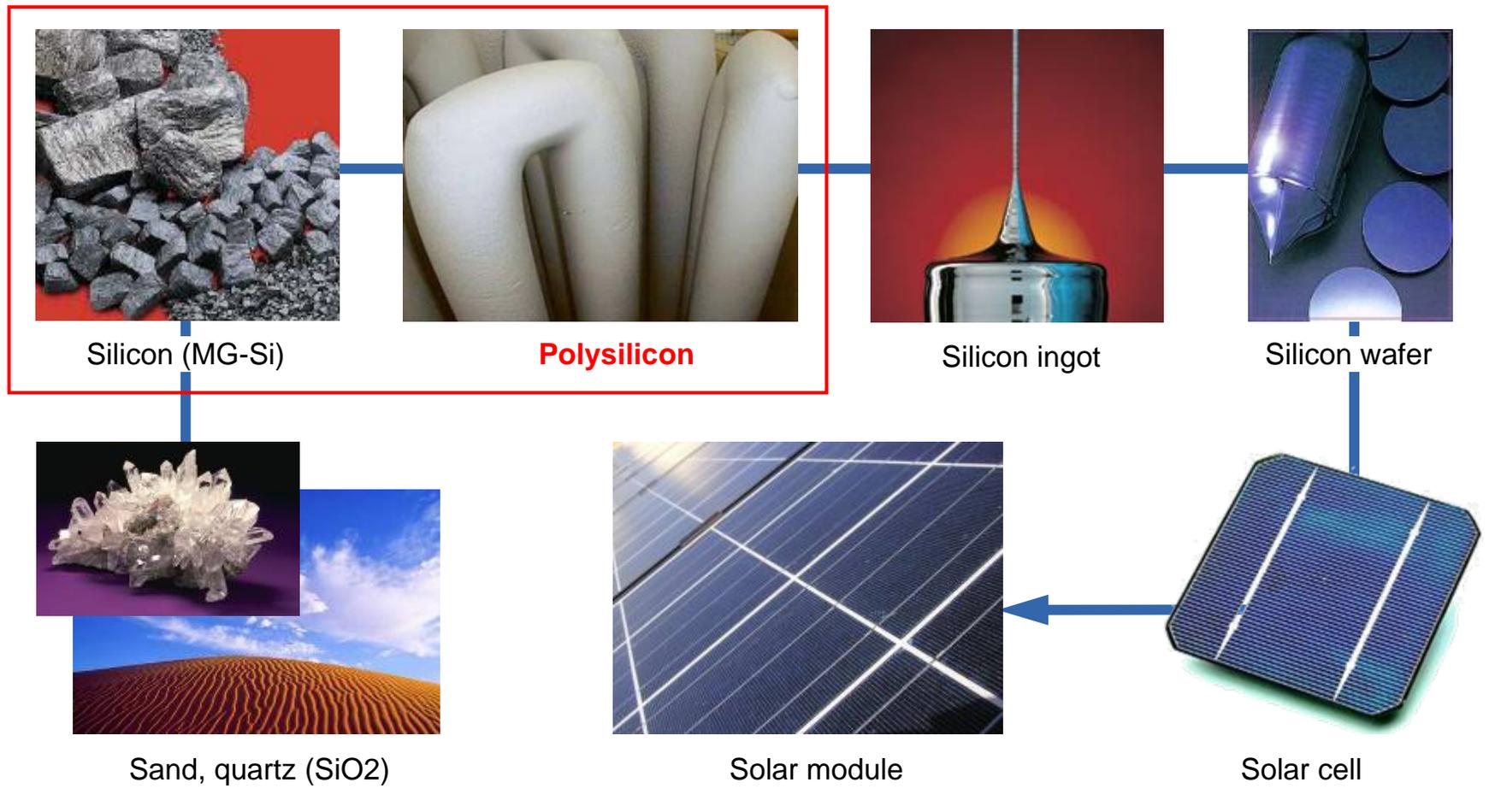
Waldbronn, Deutschland

Outline

1. Polysilicon Manufacturing
2. Examples: Purity Control & Process Control
3. Manufacturing Challenges
4. Value Propositions for Online Analytics
5. Analytical Challenges
6. Conclusion



Polysilicon Manufacturing

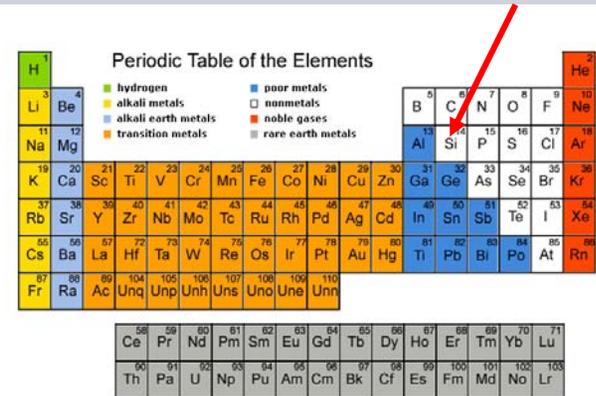


What is Polysilicon ?

- Silicon (Si) is a semi-metal and has semiconducting properties
- Polysilicon is highly purified, polycrystalline silicon

Elemental silicon of different purity:

- Metallurgical Grade Silicon (MG-Si)
98 - 99 %
- Solar Grade Silicon (SoG-Si)
99.9999 - 99.999999 % (6N - 8N)
Boron (B) < 3 ppm, Phosphorus (P) < 10 ppm, total metallic impurities < 300 ppm (preferably < 150 ppm)
- Electronic Grade Silicon (EG-Si)
99.9999999 - 99.999999999 % (9N - 11N)



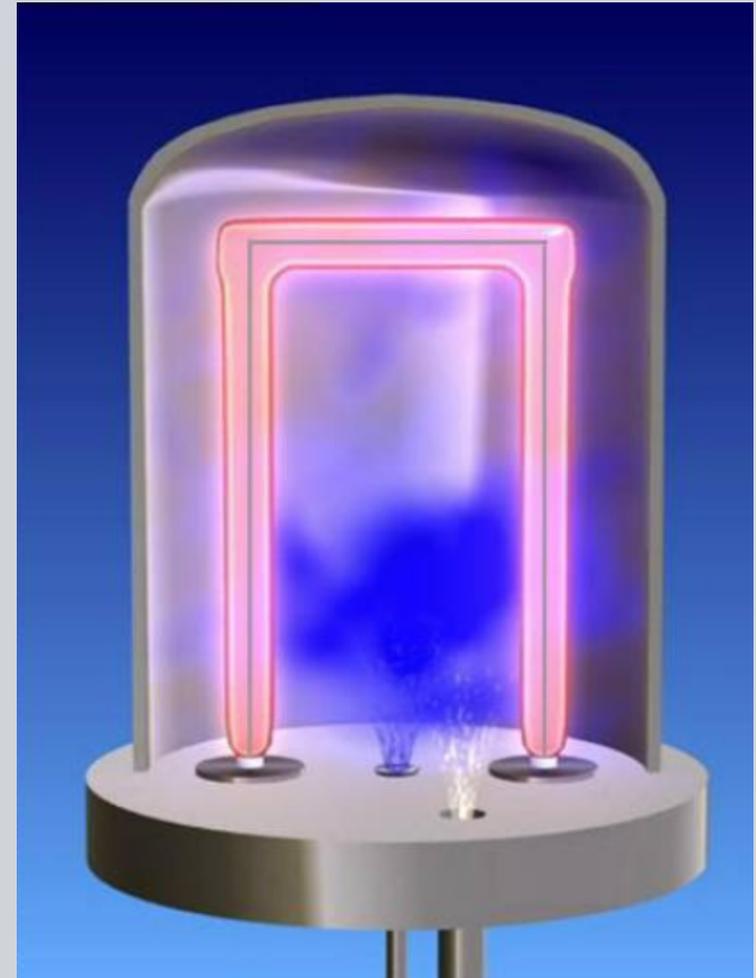
MG-Si



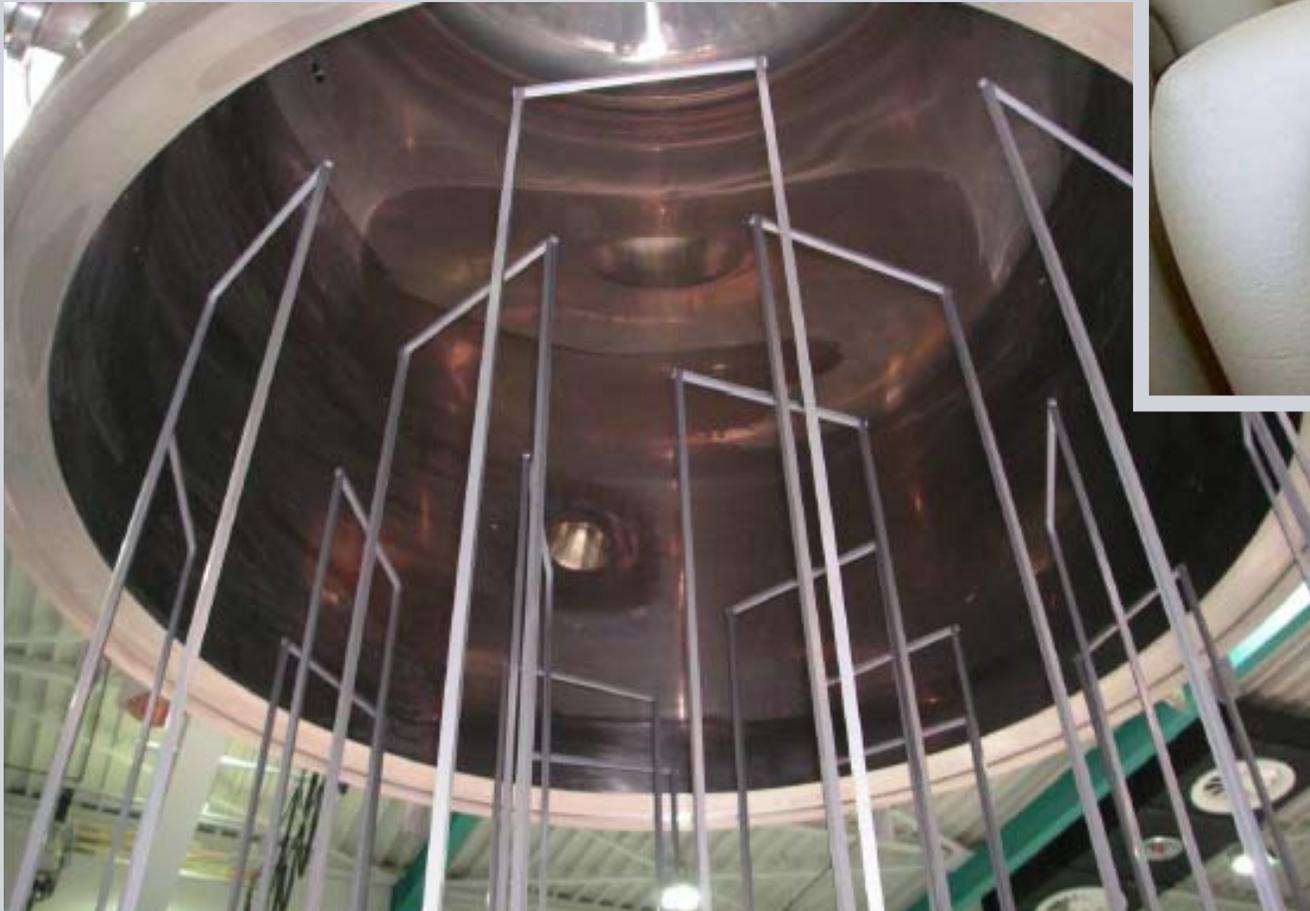
Polysilicon

The Siemens Process: Chemical Vapor Deposition

- Feed gases
 - Trichlorosilane HSiCl_3
 - Hydrogen H_2
- Decomposition of HSiCl_3 at $\sim 1000\text{ }^\circ\text{C}$:
$$4 \text{HSiCl}_3 \rightarrow \text{Si} + 3 \text{SiCl}_4 + 2 \text{H}_2$$
- Deposition of the Si-atoms on hot polysilicon slim rods
- Final rod diameter $\sim 200\text{ mm}$
- Recovery and recycling of by-products

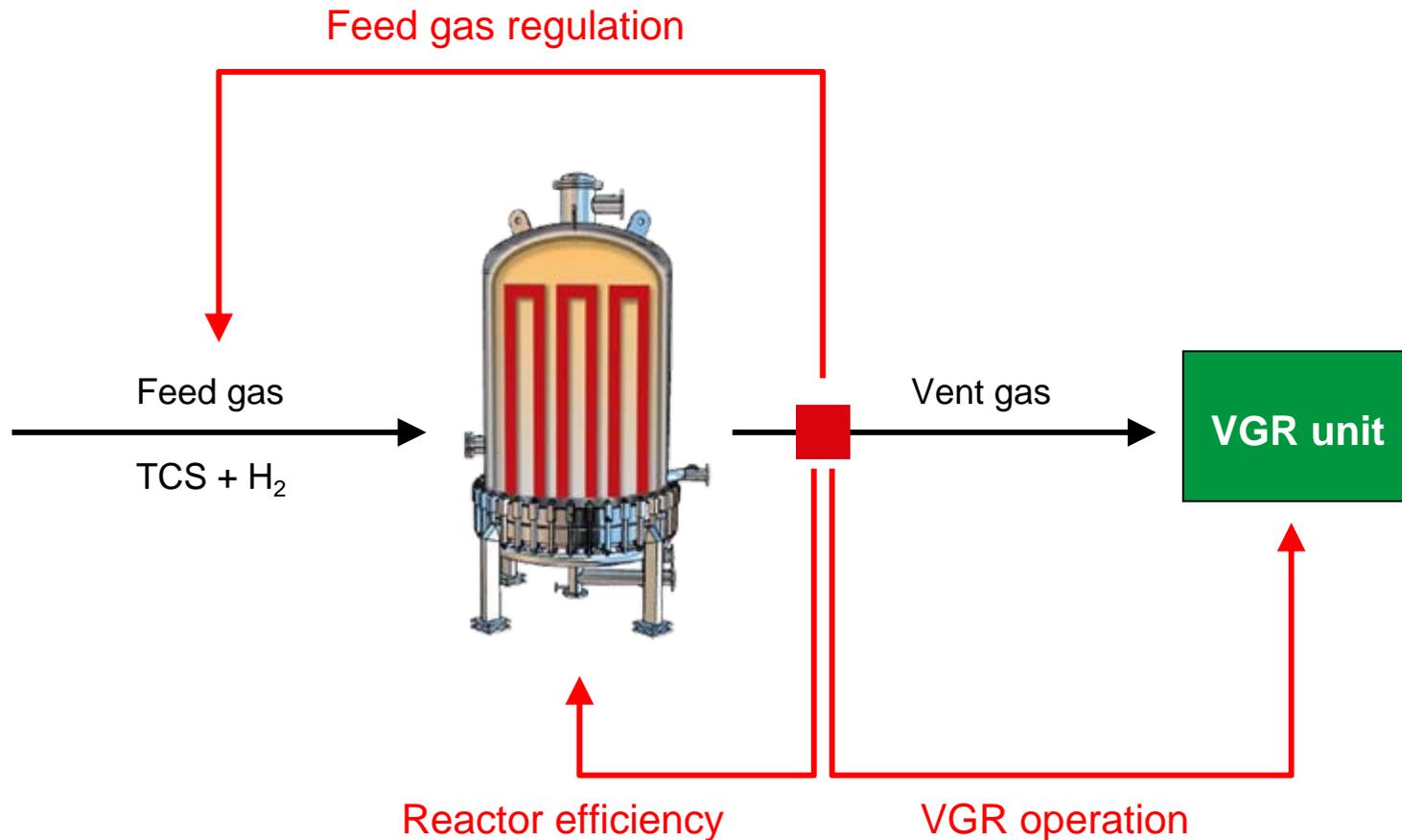


Siemens Reactor: Charging

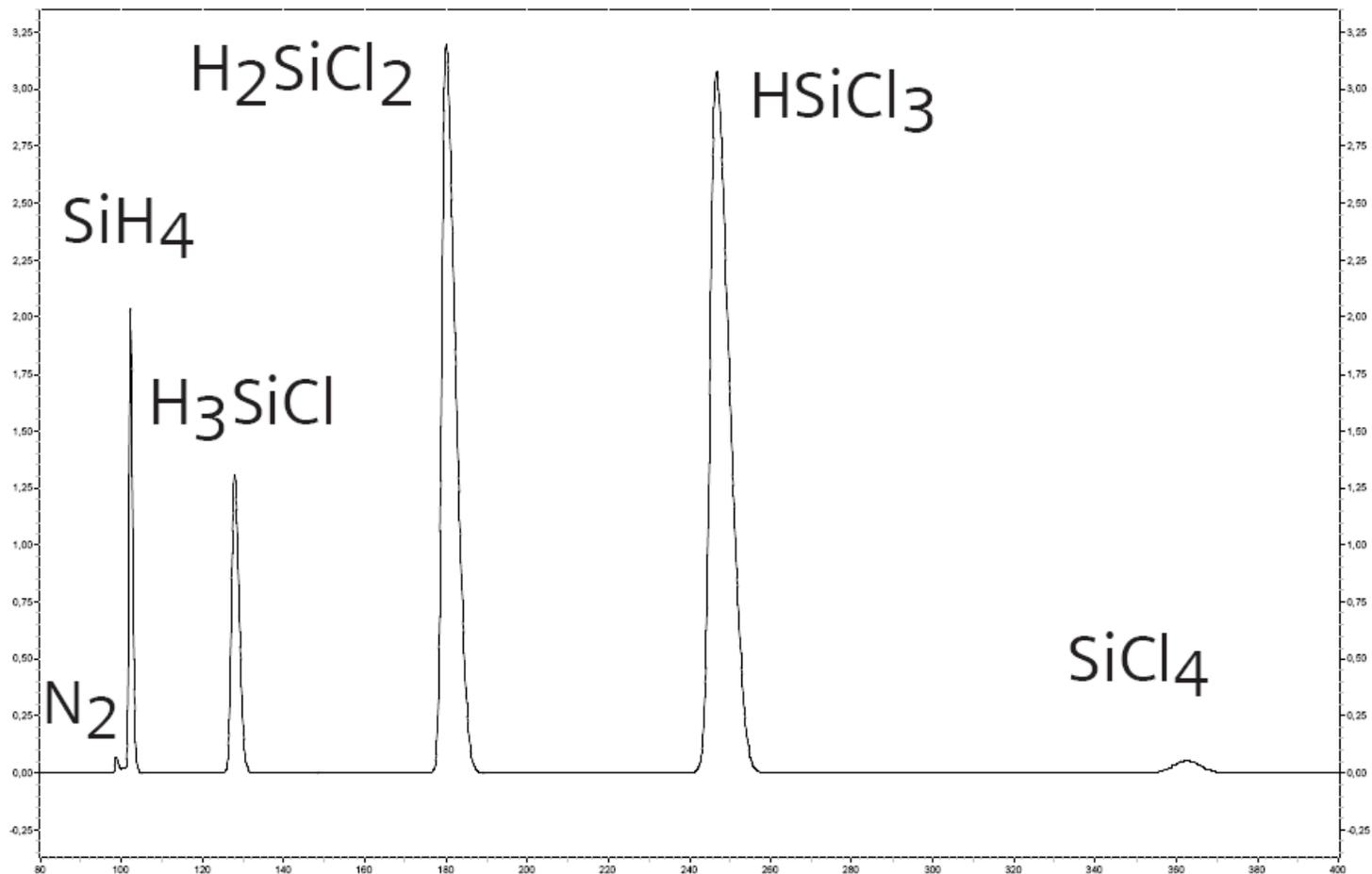


5 Days

Process Control & Process Optimization: e.g. Siemens Reactor



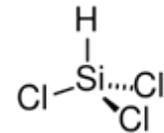
Chromatogram



Polysilicon Manufacturing: Challenges (I)

- Chemicals:
 - Highly flammable & explosive
 - Highly corrosive
 - Toxic
 - Air and moisture sensitive (forming HCl & SiO₂)
 - Violent reaction with water
- Manufacturing process is extremely energy consuming
- Low product yields (CVD reactor & converter)
- High turnover of materials (recycling)
- Stable product quality is crucial
- Products are extremely precious
- High degree of operational know-how required
- Very limited availability of experienced process engineers
- Operator companies very concerned about intellectual property

Trichlorosilane
(TCS), HSiCl₃:



C

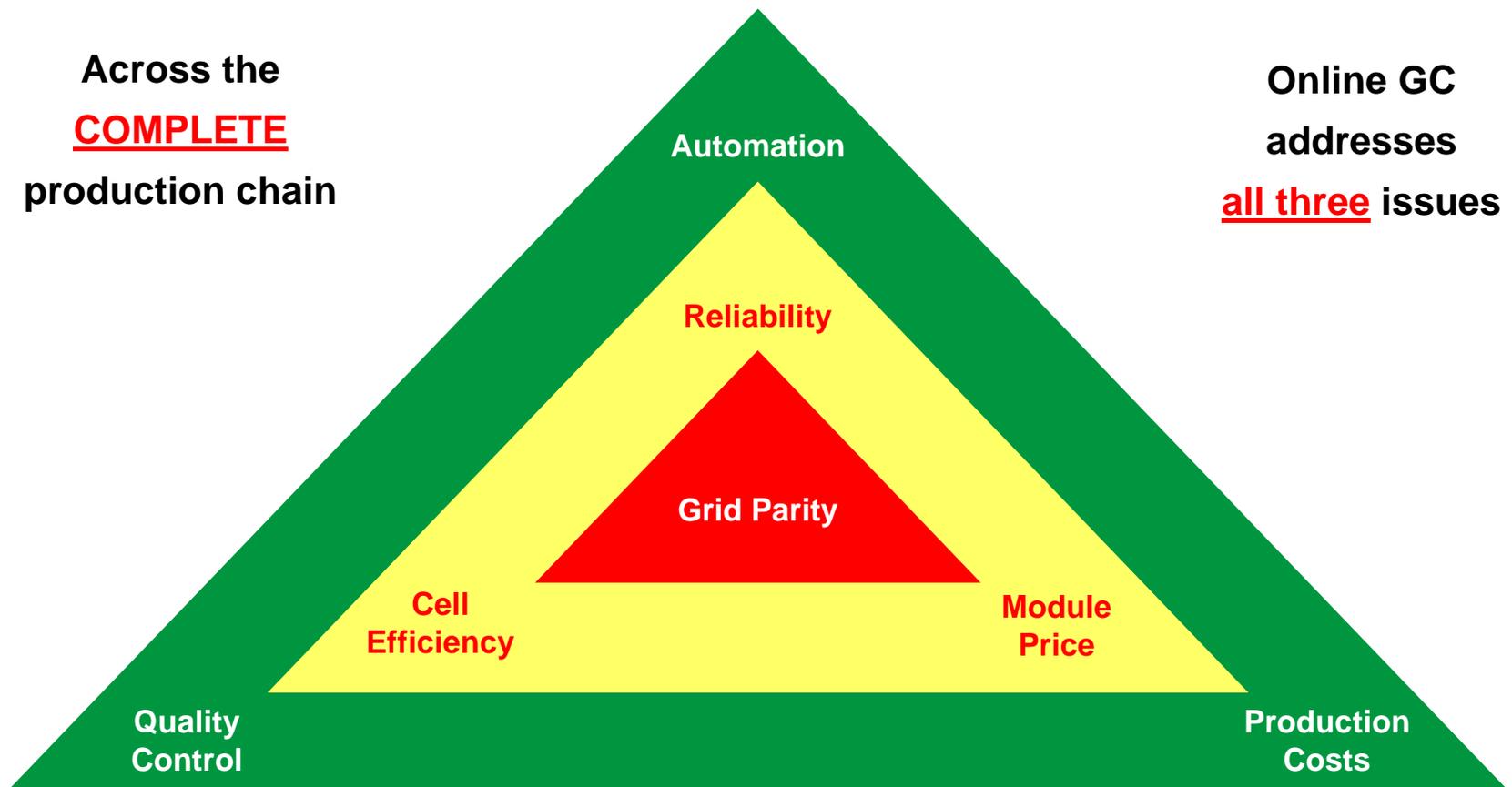
F+

Polysilicon Manufacturing: Challenges (II)

Cost pressure on polysilicon production is rapidly raising:

- Solar energy costs have to drop to be competitive in the future energy mix
Target spot price of 35 \$US / kg polysilicon
- Many newcomers are entering the solar business worldwide:
 - Lower energy costs in Asia
 - Lower labor costs in Asia
 - Environmental regulations less stringent
 - Health protection less stringent
- Established manufactures ramp up their polysilicon capacities to flood the market
Strategy to protect against newcomers to the market
- Innovative technologies are outpacing the traditional Siemens process:
 - Enhancement of the Siemens process
 - SiH₄ based technologies - less energy consuming
 - FBR - continuous process, no chlorine chemistry, simplified vent gas recovery, low energy
 - Metallurgical upgrading - more economic but 5N only

Polysilicon Manufacturing: Challenges (III)



Value Propositions for Online Analytics

- ✓ Shortening of **time to market** (first silicon out)
Fast ramp-up and stable operation of CVD reactors
- ✓ Increase of polysilicon **output**
- ✓ Reduction of **production costs**
Less feed gas consumption, heating & cooling, vent gas treatment, waste products
- ✓ Enhancement of polysilicon **quality**
Permanent control of the feed gas purity
- ✓ Total product **traceability** along the complete value chain
- ✓ Ensures **process safety** (personnel & plant)
- ✓ Reliability of **analysis results**
- ✓ **Comprehensive knowledge** based on sufficient process data



Analytical Challenges

- Expertise in chromatographic separation of uncommon components
- Ability to handle highly reactive samples in the application laboratory
- Customer samples are not available for method development
- Commercial availability of calibration blends is very limited
- Shipping of calibration blends is cumbersome & expensive
- Special sample pre-treatment to remove low-volatiles (AlCl₃, polysilanechlorides)
- Highly leak-proof design of sample conditioning systems
Special fitting techniques, seals & hardware components
- Special layout of SCS to allow for purging procedures
- Special concept for SCS to minimize sample consumption
- Effective protection against corrosion
- Proper selection of analytical hardware (valves, columns etc.)
- Trace level detection at ppb levels
- Sample condensation

Conclusion

Polysilicon manufacturing:

- GC systems have been installed and commissioned in many polysilicon plants worldwide and are operating to the full satisfaction of customers
- Process GCs are increasingly replacing offline GCs
- The use of online analytics is becoming an accepted standard
- Online analytics is playing a significant role in establishing solar energy in the future energy mix

Generally:

- Expertise of Siemens Process Analytics in sophisticated chemical applications pays off
- Emerging markets and technologies offer new opportunities for established analyzer techniques

Thank you for your attention!



Dr. Akbar Tellenbach
Siemens AG

Östliche Rheinbrückenstr. 50
76187 Karlsruhe, Germany

Phone: +49 - 721 595 87 22
Cellular: +49 - 173 29 49 325

E-Mail: akbar.tellenbach@siemens.com