Decreasing dependence on critical raw materials – strengthening the solar supply chain

Dr Maria Wallin
Senior Researcher
maria.wallin@ntnu.no

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NTNU

ReSilex

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Reducing the EU dependence on critical raw materials for solar panel production

Development of technological solutions for reusing silicon, recycling PV modules and designing new products

RESiLEX is a Horizon Europe project aiming to improve the resilience and sustainability of the entire silicon value chain in the EU

Reducing EU dependence on silicon imports from third countries

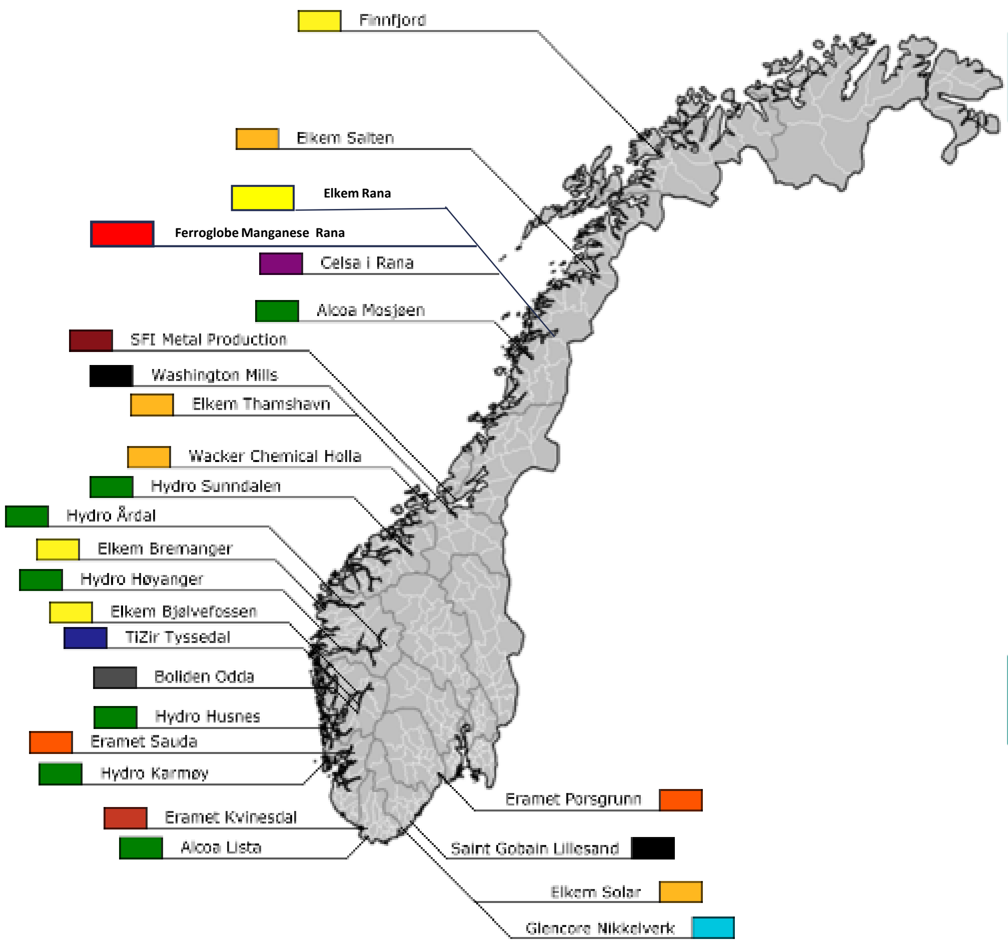
Developing a new carbon-free and more efficient process for the production of silicon

Recycling end-of-use solar PV panels for producing Li-ion battery cells

My background

Metal production in Norway

- Industrial high-temperature metallurgical processes
  - Chemical reactions, Mass and energy transfer, Phase transitions
- Traditional materials: steel, aluminum, ferroalloys/silicon and titanium slag.
- Recycling
- Development of new and improved processes
  - New raw materials, products and properties, reduced emissions of gases and particles (dust) and energy efficiency
- Advanced materials: silicon in solar energy, batteries, nanomaterials, intermetallic materials, rare earths and biomaterials



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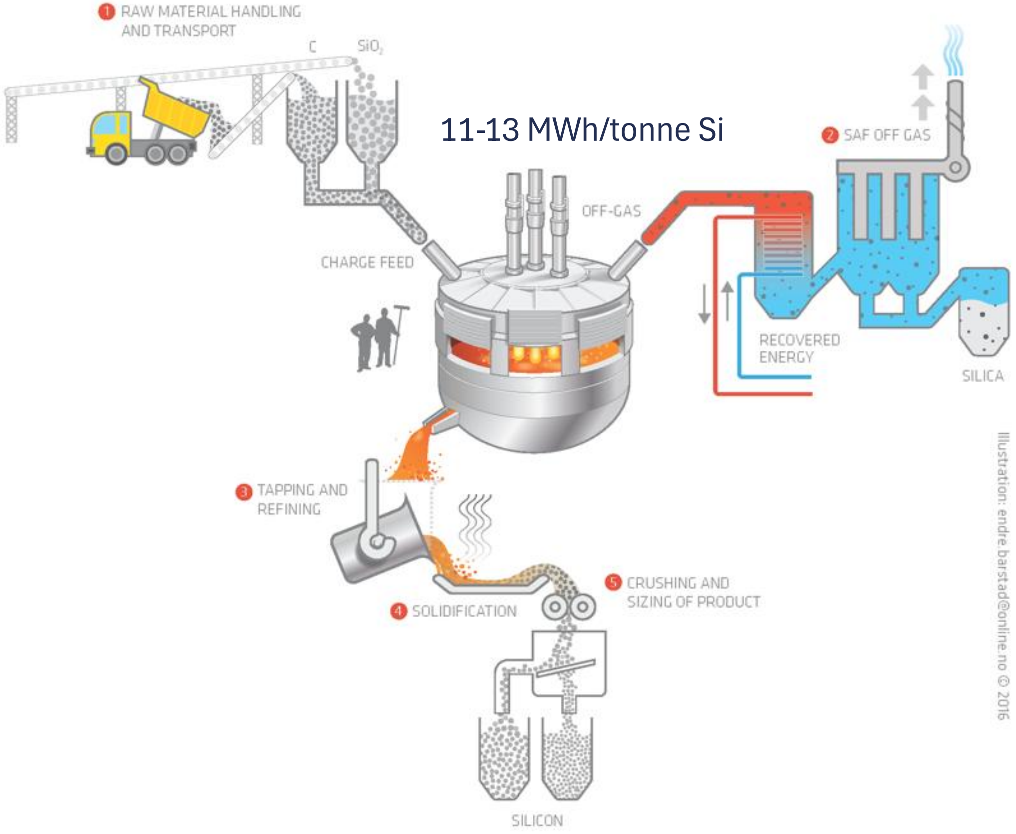
Industrial silicon production needs re-invention to meet sustainability demands!

Approx 5 tonne of CO_2/tonne Si

 +  → 

$$SiO_2 + 2C = Si + 2CO$$

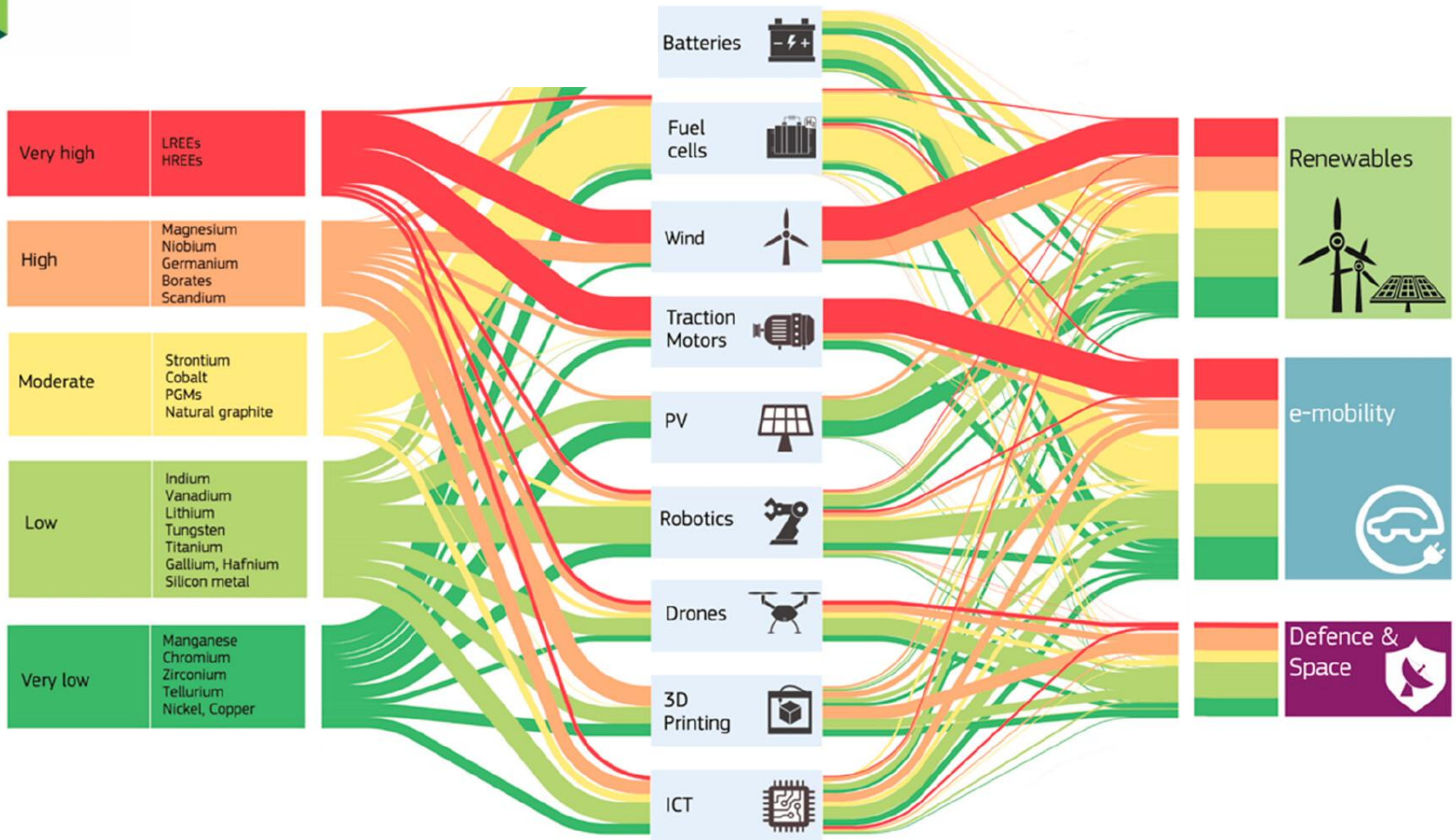
- High specific energy consumption
  - energy losses in off-gas partly remidied by energy recovery in some locations
- Significant specific GHG (CO_2, NO_x) emissions
- Can not use SiO_2 fines = raw material losses



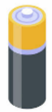
Industrial state of the art: Submerged arc – semi-open furnaces

Initiatives:

1. Net Zero Industry Act
2. European Critical Raw Material Act
3. Reform of electricity market design



Eco-designed PV

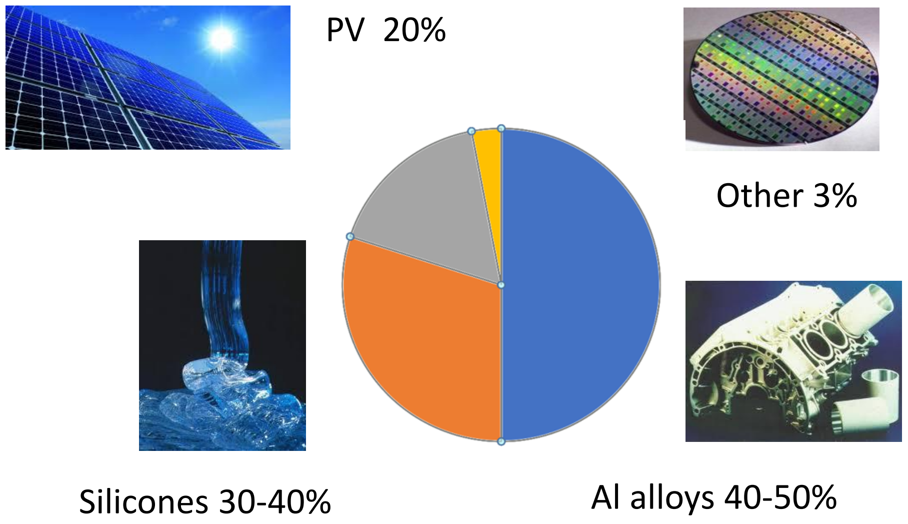


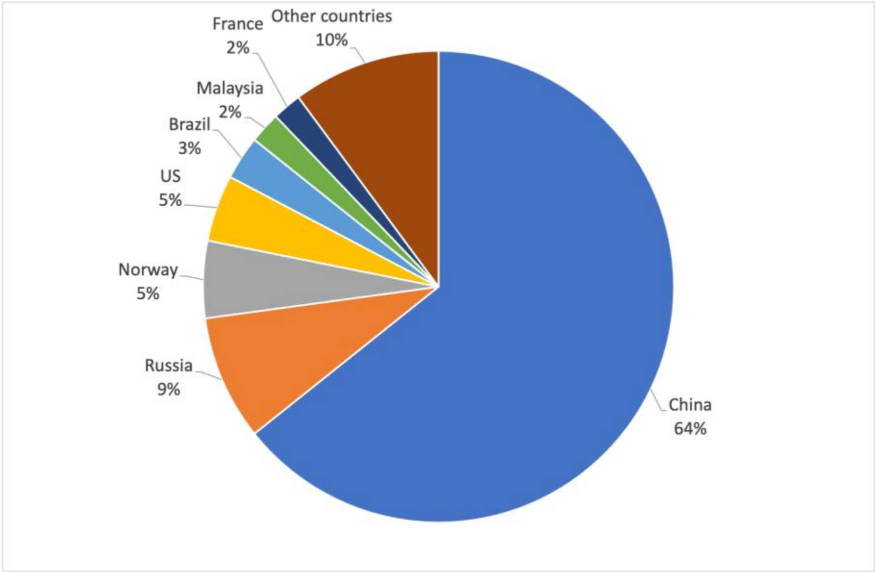
Carbon coated crystalline silicon nano-powder evaluated as composite anodes Li-ion batteries

Silicon – a European SRM and CRM!

- China dominating the world market
- Norway one of the worlds larger suppliers with approx 5% of the marketshare for MG-Si

- An opportunity for Europe to increase its self- sufficiency!



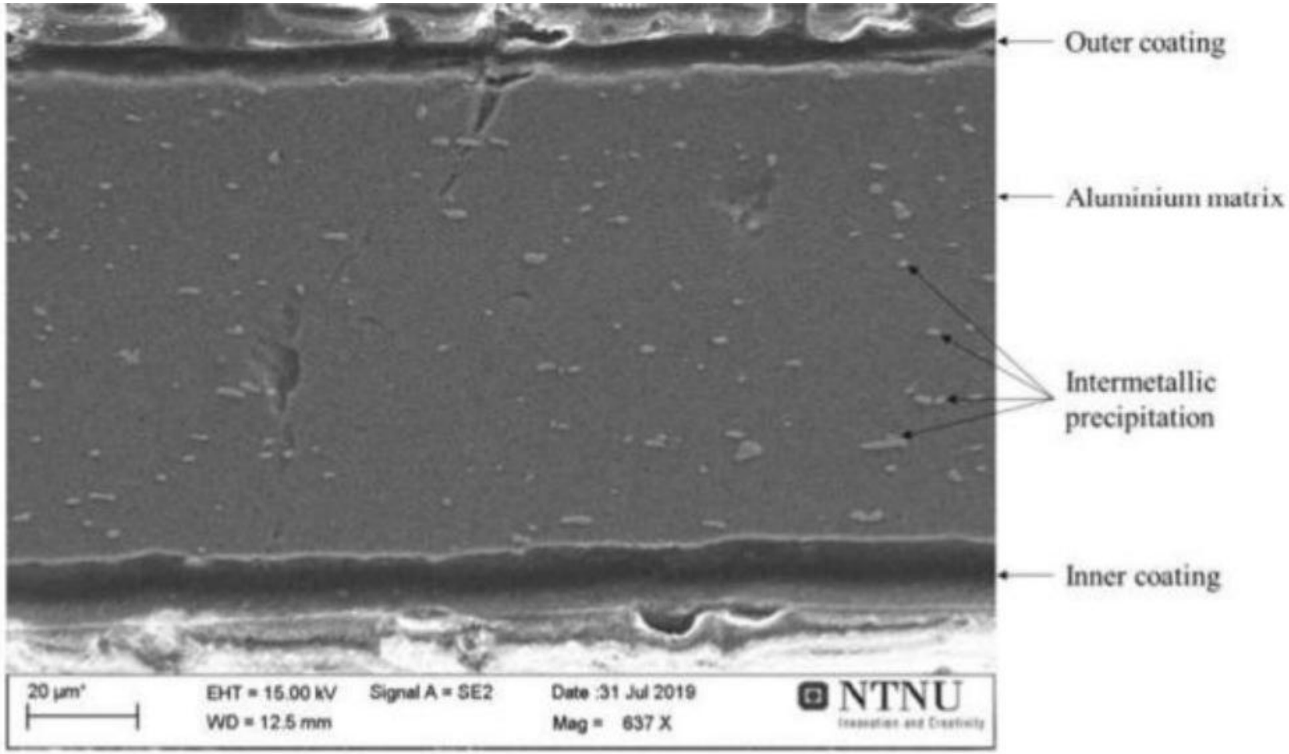


Global production of Si, source USGS

Different strategies to meet the climate goals for the silicon industry

- New reductants (e.g. charcoal, composites, metals, H_2)
- Plasma
- Electrolysis
- CCS/CCU

Si – a key alloying element for Al!



From cast alloys to foils…… Around 40% of Si produced used for alloying aluminium

The project has received funding from the European Union's Horizon 2020 research and innovation program under Grant Agreement N°869268.

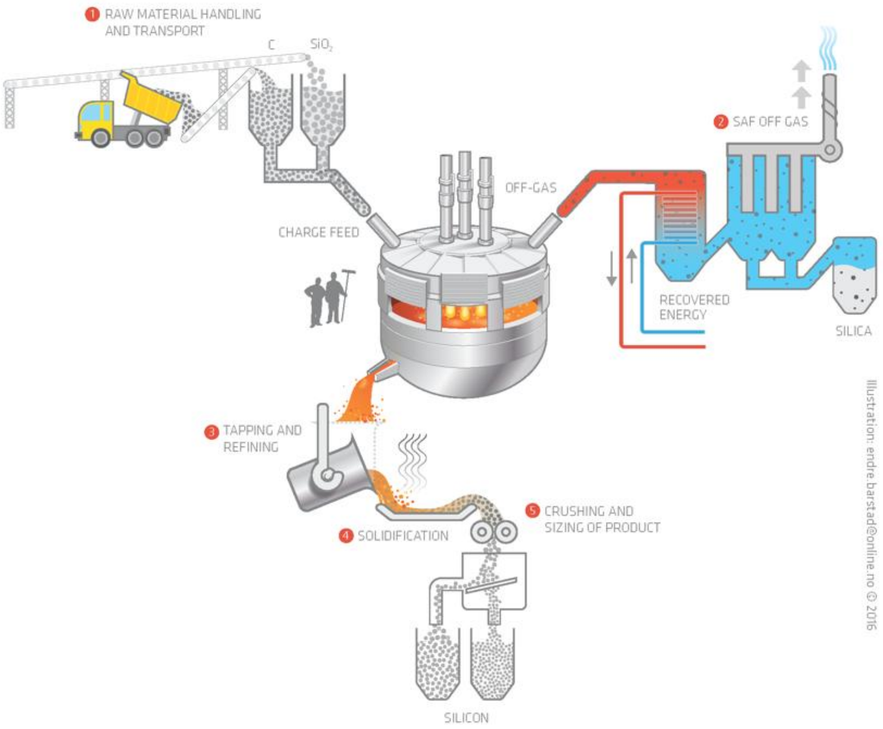
The Al industry also has its challenges

- Global annual Al production approximately 60 Mton, 20 * the size of Si and currently the largest user of Si
- > 600.000 tonnes Al scrap exported from Europe annually - **but new policies around the corner**
- > 80.000 tonnes of Al dross (>70% Al, rest oxide) generated per year in Europe and processed via salt treatment with varying Al yield and significant environmental footprint
- Industry puts increasing pressure on suppliers to fulfill/deliver low carbon footprint commitments/products - CO_2 free Si!

The project has received funding from the European Union's Horizon 2020 research and innovation program under Grant Agreement N°869268.

The SisAl Process – Our solution

Industrial Symbiosis between Si and Al industries!



CARBOTHERMIC REDUCTION

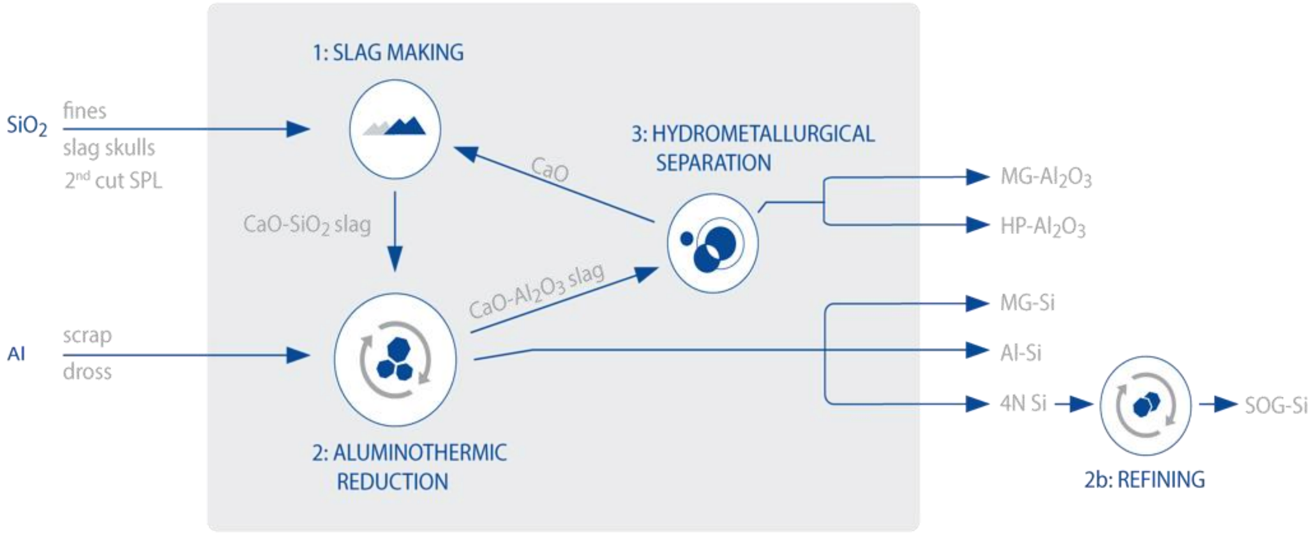
$SiO_2 + C \rightarrow Si + CO_2$

vs

$SiO_2 + 4/3Al = Si + Al_2O_3$

Aluminothermic reduction in slag!

- **No direct CO_2 emissions, no NO_x**
- **Lower energy consumption**
- **Path to effective use of difficult scrap and dross**
- **No loss of SiO_2 fines**

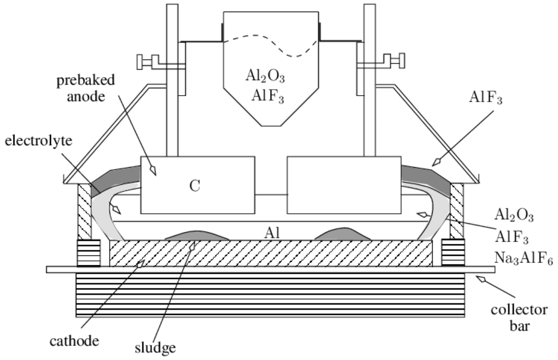


Tow products: Si alloys and CaO-Al_2O_3 based slag

+

Production of HP-Al_2O_3 to LED and battery separatores

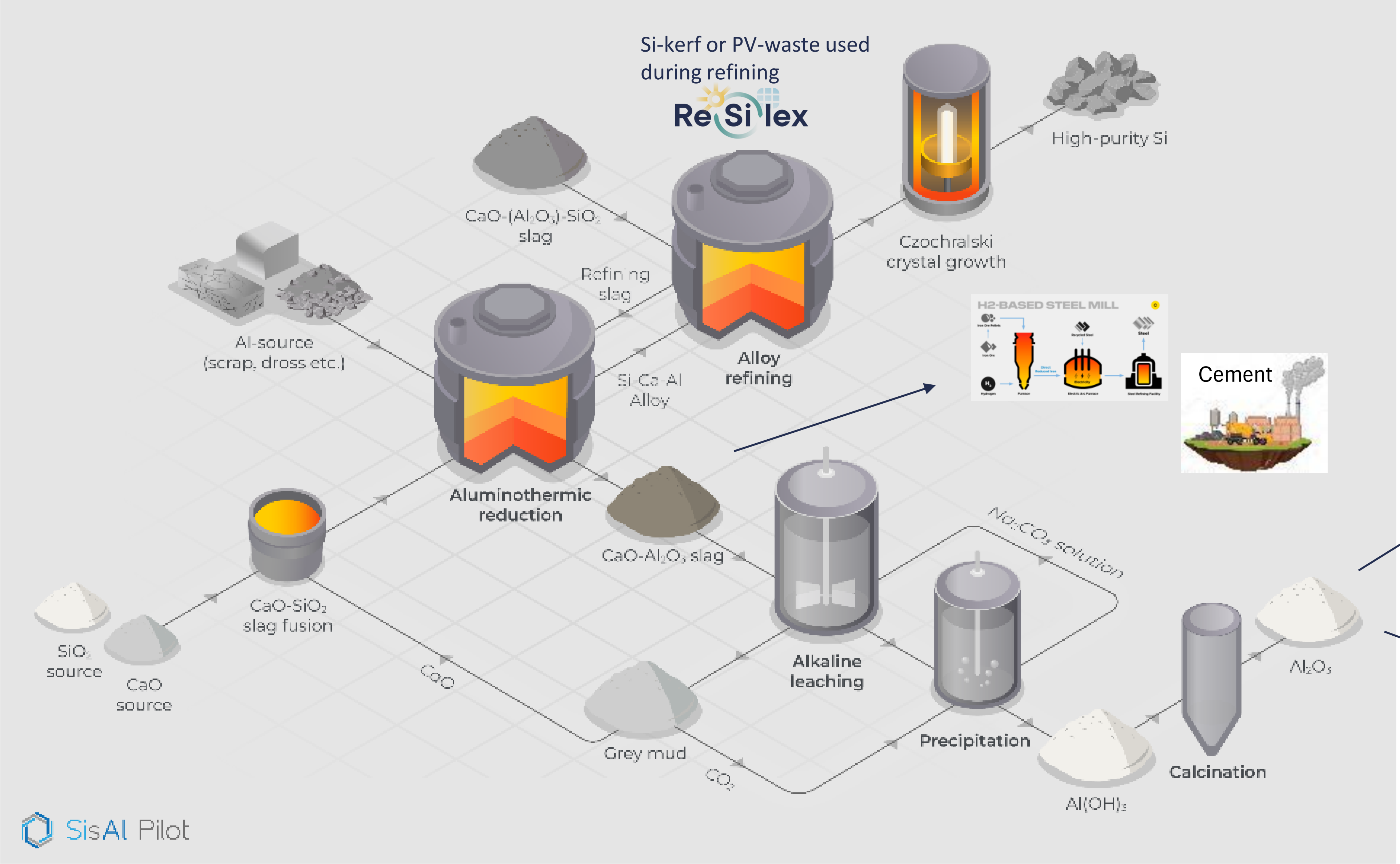
Production of MG-Al_2O_3 for Al electrolysis

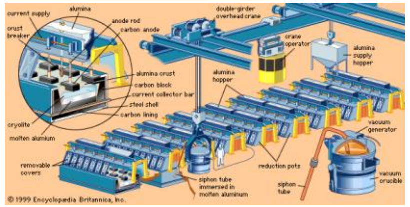


Additive in cement

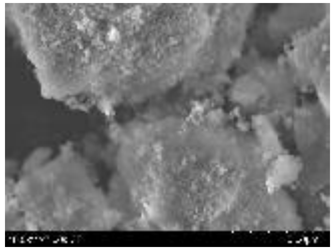
Refining slag in steel productoin

Resource efficiency





MG Al_2O_3
(alkaline leaching)

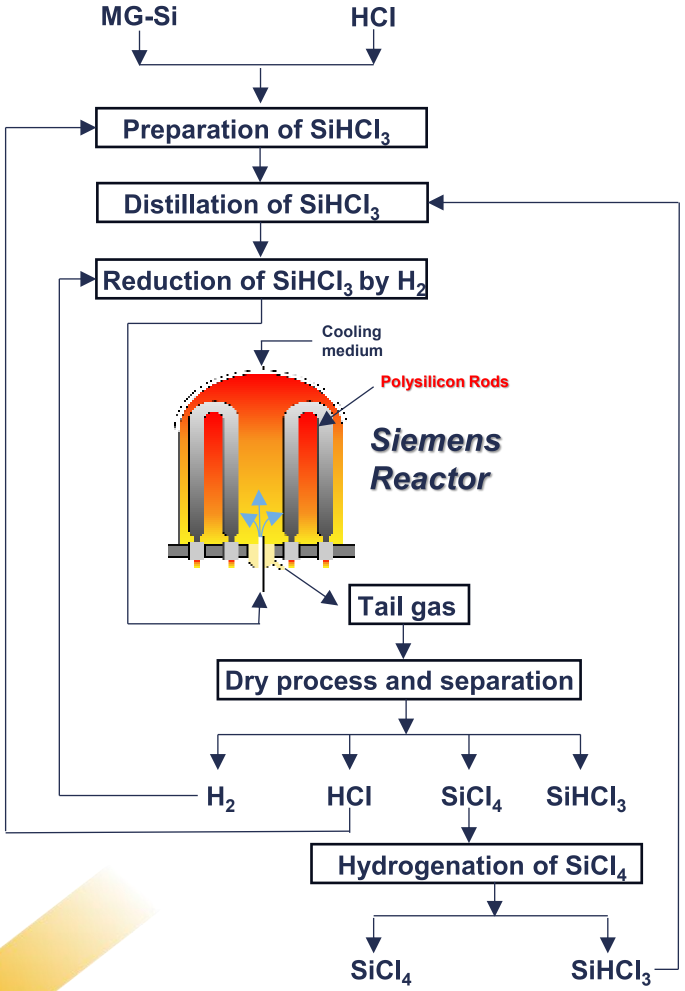


4N HPA
(acidic leaching)

SisAL Slag

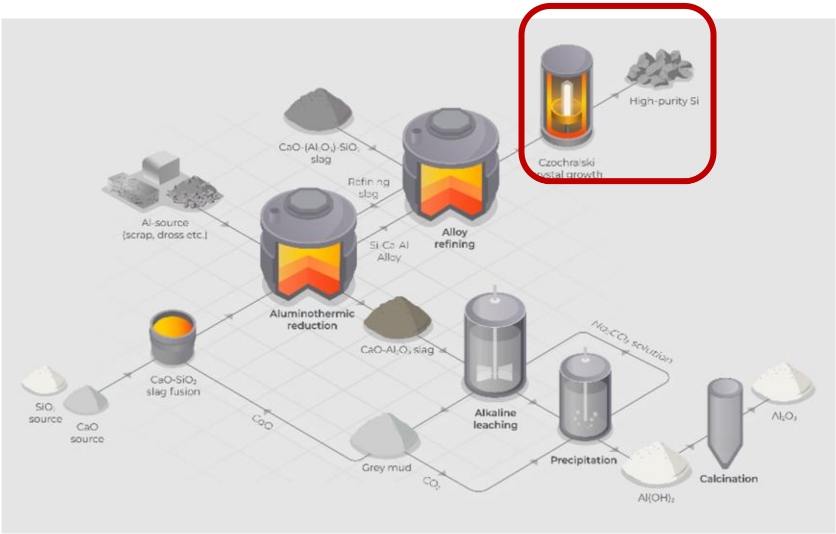
Techniques for obtaining SoG-Si from MG-Si

Siemens process



Alternative purification methods

- Slag refining
- Vacuum refining
- Acid leaching
- Crystallisation by **Directional solidification (DS)** or **Czochralski (CZ) growth method**



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The Resilex Process

SisAl process

SiO_2 source
+
Al source

SisAl
metal
(74 % Si,
10 % Al,
16 % Ca)

Slag refining

Kerf

Slag

MG-Si

Crystallisation

Directional Solidification

Czochralski Growth Process

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Medium and large scale refining trials

50kg (225 kW induction furnace) => 98,91% Si

400kg (600 kW induction furnace) => 97,79% Si

The hot metal in the mold

Slag and metal

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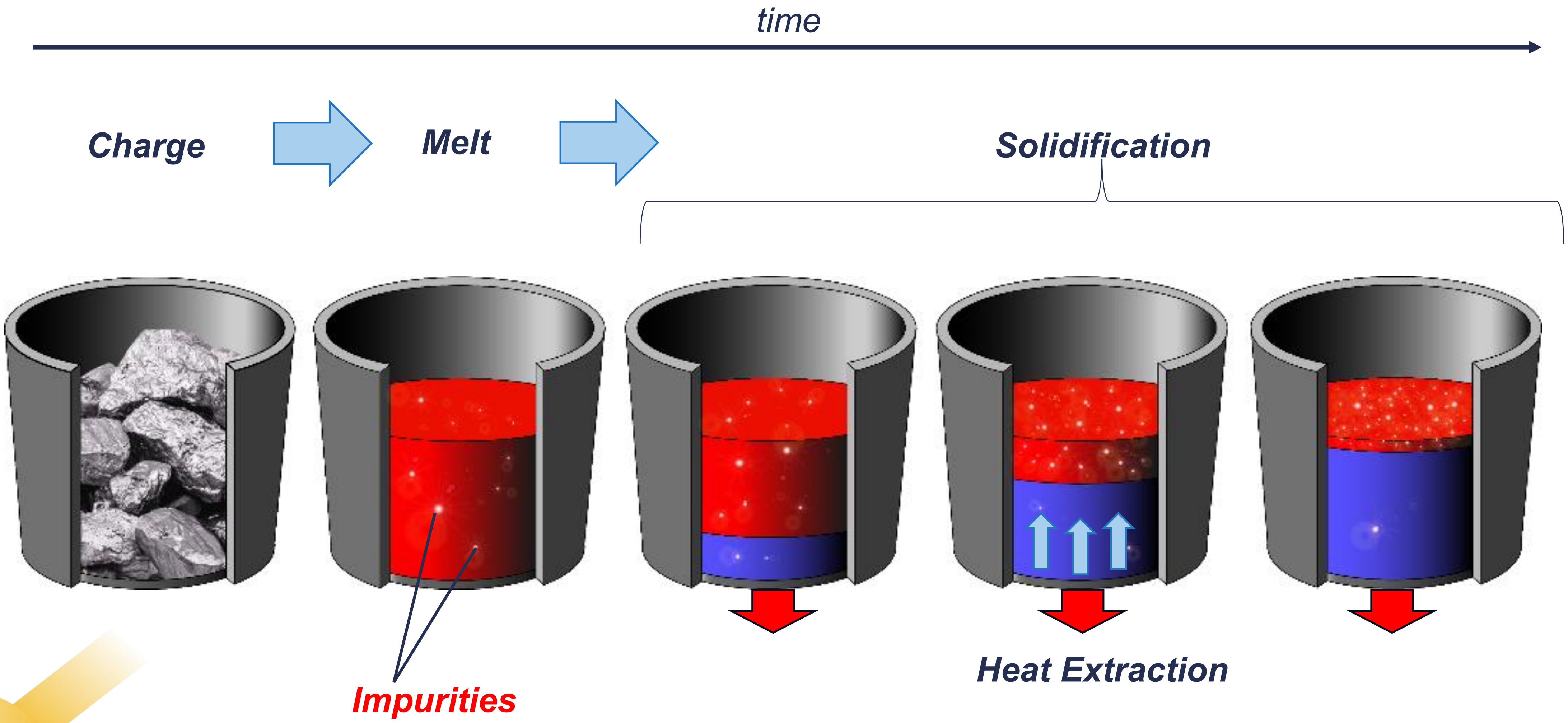
Directional Solidification



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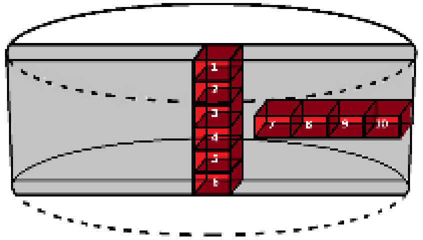
Directional Solidification

Re Si lex

Si metal obtained in slag refining (98.91 % Si)

Crystalox furnace

~ 12 kg



10 samples

ICP-OES analysis

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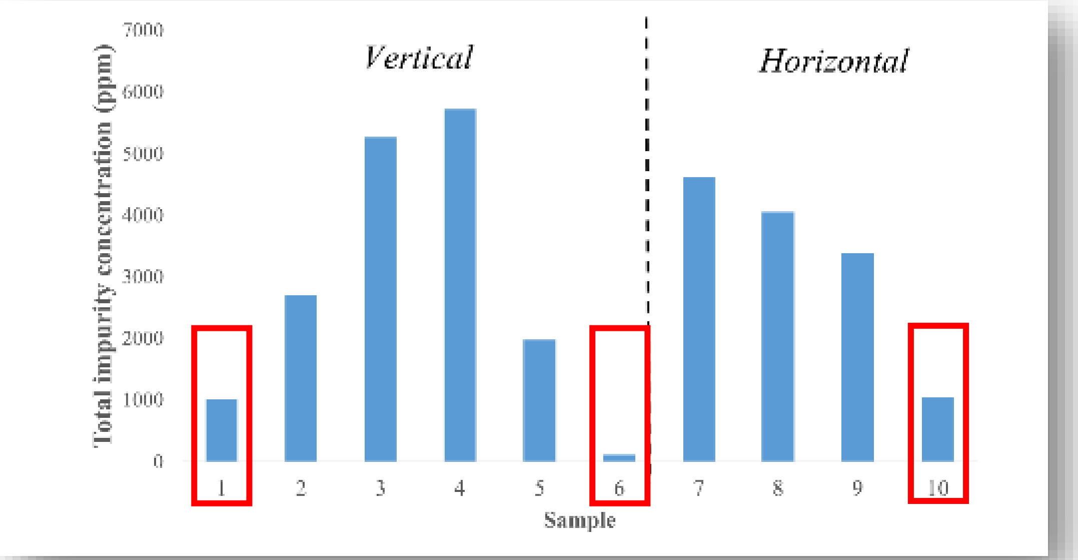
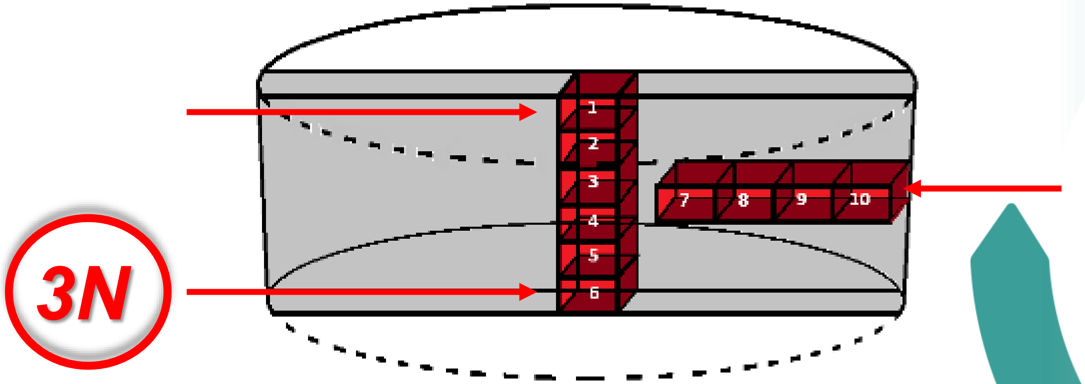
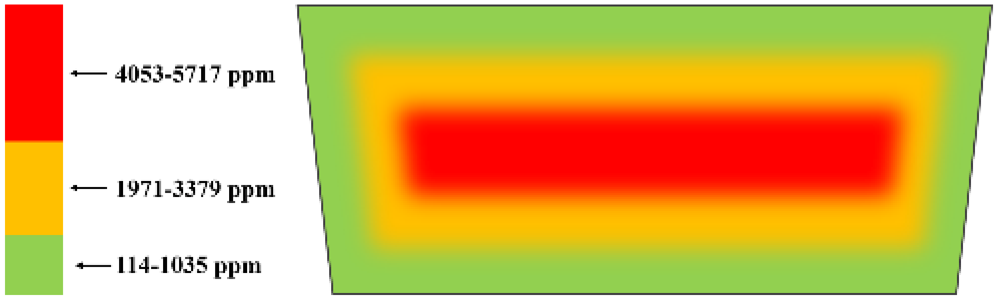
Directional Solidification

ICP-OES analysis results:







- The sample at the bottom of the ingot reached a purity level of 3N, followed by the sample up to the very edge of the ingot and the sample located at the top.
- The impurity distribution did not follow Scheil's equation.
  - Additional tests using different degrees of dilution for the secondary silicon to avoid collapsing the crystallisation front are necessary to have a more comprehensive understanding of the distribution of its impurities during directional solidification.

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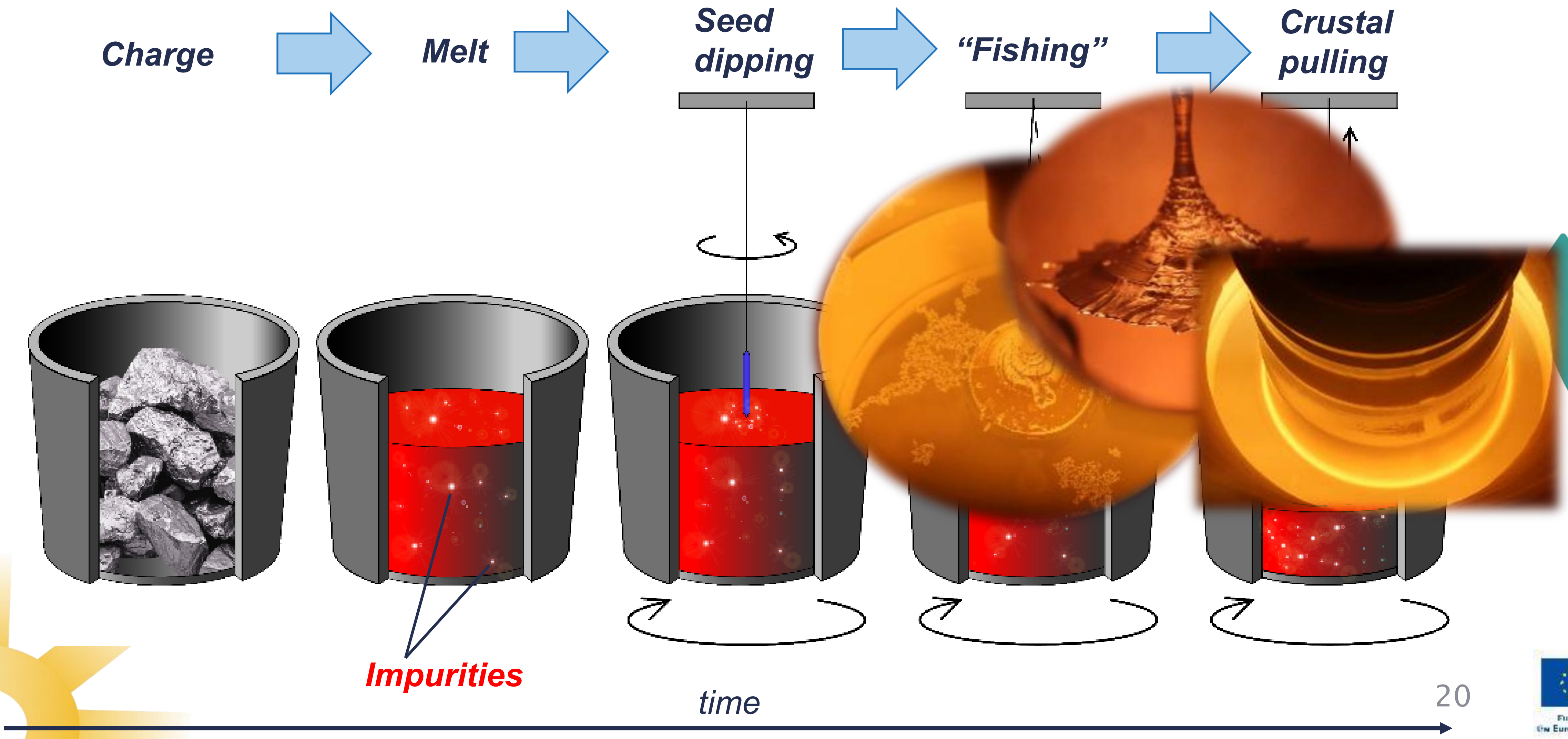
Czochralski Growth Process

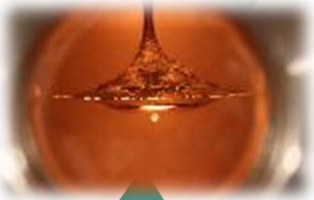


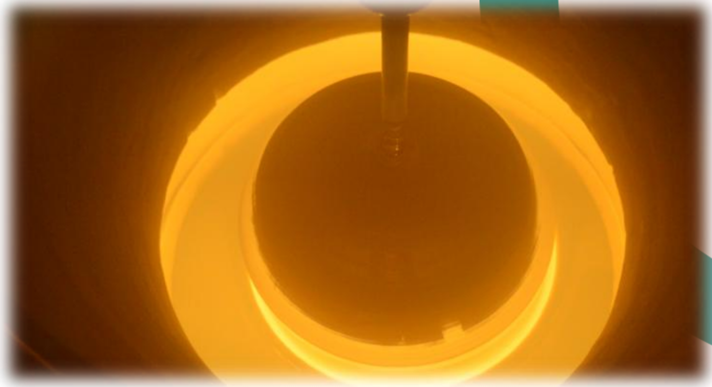
Czochralski Growth Process

Si metal obtained in large-scale slag refining

Blending with electronic grade Si

Purification by crystal pulling





97.79 % Si
(99.04 % Si)

- **Ratio**: 97.79 % Si : electronic grade Si = **1 : 1**
- **Mass**: 30 kg
- **Purity after blending**: 99.52 %

Czorchralski puller

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From Waste to High-Purity Silicon: Refining of Silicon by Directional Solidification and Crystal-Pulling Method

Katarina Jakovljevic, Eivind Johannes Øvrelid, Nagarajan Somi Ganesan, Pål Tetlie, Casper Van der Eijk, Maria Wallin, and Gabriella Tranell

Abstract Since 2014 silicon (Si) has been classified as a critical raw material by the European Union due to its high risk of supply disruption and significant economic importance. The SisAl process for Si production reduces direct CO_2 emissions and also promotes the utilisation of secondary raw materials such as quartz fines, aluminium dross, and scrap, enabling a shift from today's carbothermic reduction process to a process in line with circular economy goals. The present study focuses on the purification of a Si alloy acquired from slag-refined mixture of SisAl metal and kerf. 99.9% pure silicon was successfully obtained by utilising the directional solidification method, which relies on the different distribution of impurities during metal solidification. As an alternative, the Si alloy was also blended with high-purity Si and subjected to refinement by crystal pulling to assess its suitability for applications demanding very high Si purity. In this process, an ingot with 5N purity was obtained.

Keywords Silicon refining · Directional solidification · Czochralski process · SisAl process

Introduction

Metallurgical-grade silicon (MG-Si), typically containing 96–99% Si, is conventionally manufactured by using carbon to reduce silicon dioxide (SiO_2) in a submerged arc furnace (SAF) [1]. The process generates greenhouse gas (GHG) emissions and is highly energy demanding. Nevertheless, due to the substantial ramifications of global

K. Jakovljevic (✉) · M. Wallin · G. Tranell
Department of Materials Science and Engineering, Norwegian University of Science and Technology, Alfred Getz' Vei 2B, 7034 Trondheim, Norway
e-mail: katarina.jakovljevic@ntnu.no

E. J. Øvrelid · N. S. Ganesan · P. Tetlie · C. Van der Eijk
SINTEF Industry, Alfred Getz' Vei 2B, 7034 Trondheim, Norway

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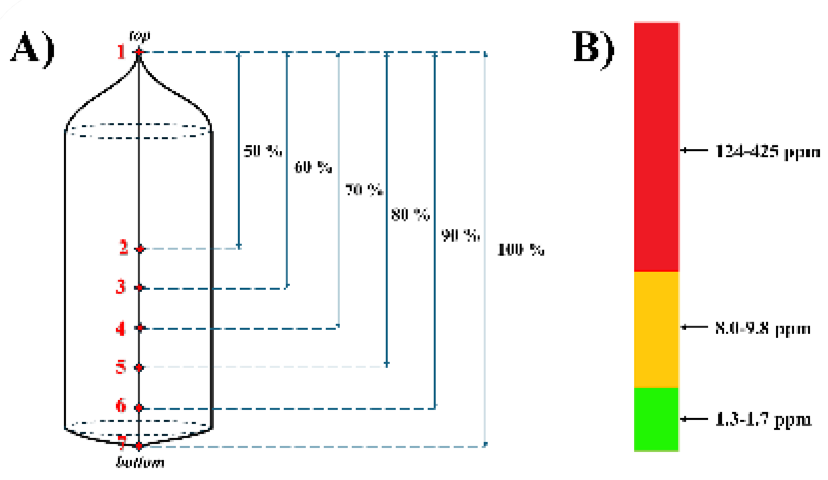
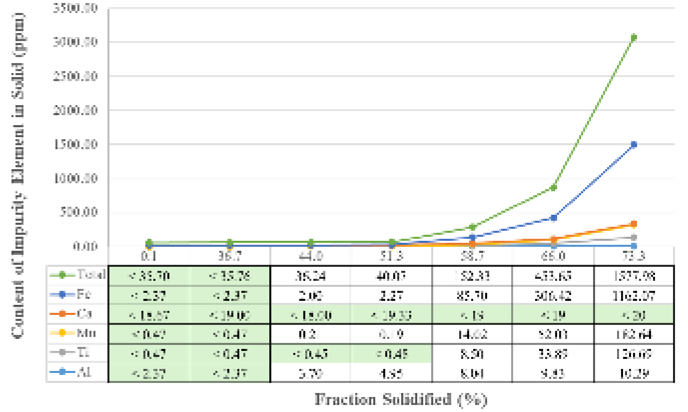
5N



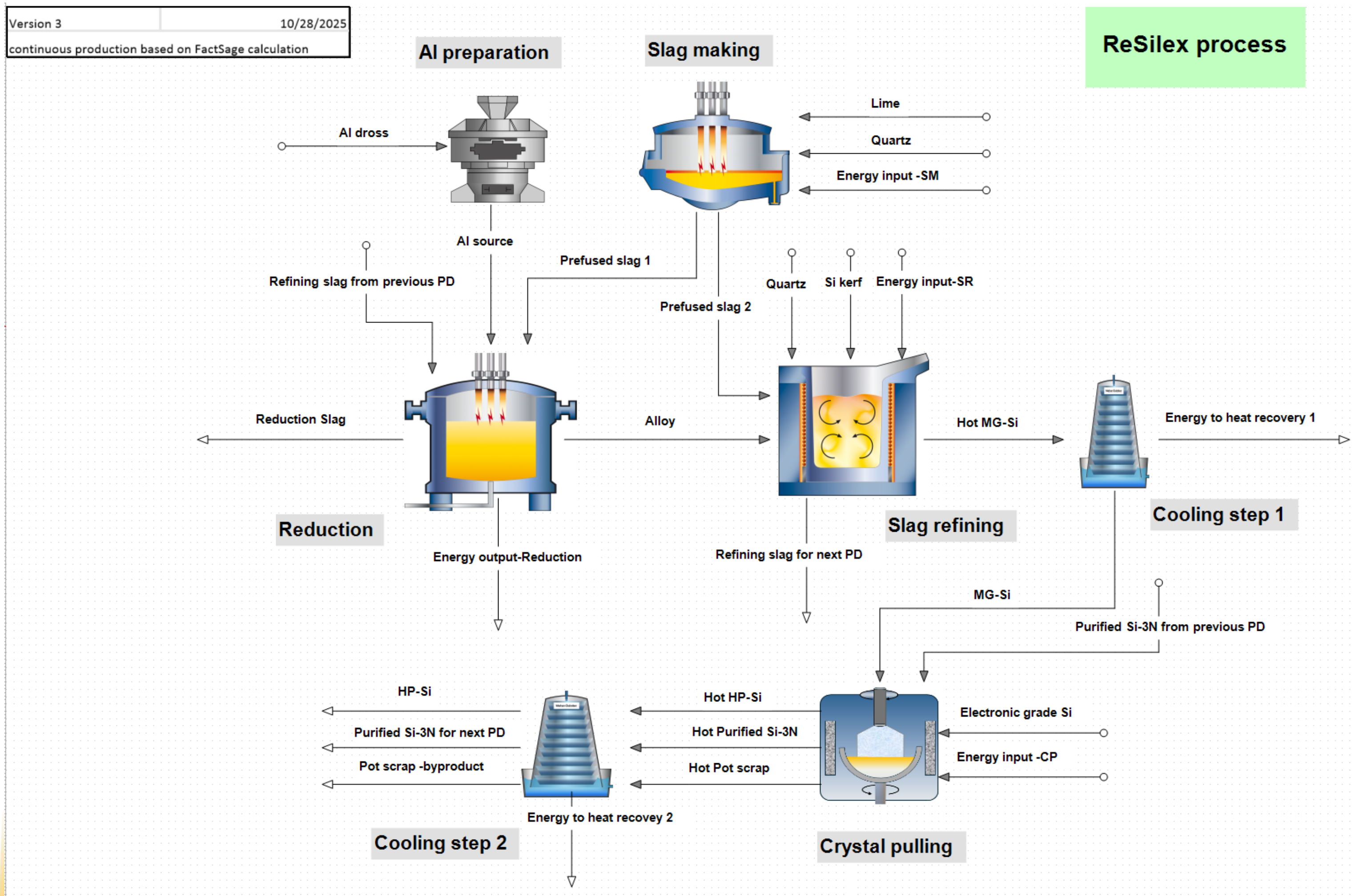


	0.1	36.7	44.0	51.3	58.7	66.0	73.3
Total	< 35.70	< 35.78	35.24	40.07	152.33	433.65	1577.98
Fe	< 2.37	< 2.37	2.02	2.27	85.70	306.42	1163.07
Ca	< 18.57	< 19.00	< 18.00	< 19.33	< 18	< 19	< 20
Mn	< 0.42	< 0.47	0.21	0.19	14.02	52.03	162.64
Ti	< 0.42	< 0.47	< 0.45	< 0.45	8.50	33.85	126.05
Al	< 2.37	< 2.37	5.79	4.65	8.04	9.83	10.29

Weight: 22 kg

Purity: 5N in the first 70% of the ingot if only the measured elements are considered

HSC process simulation



Materials Input Parameters	kg/h
Al dross	150.00
Lime	149.35
Quartz-Slag making	143.49
Quartz-Slag refining	30.11
Si kerf	141.13
Electronic grade Si	87.49
Refining slag from previous PD	245.34
Purified Si-3N from previous PD	122.11
Energy Input Parameters	**kwh**
Energy input-SM	138.02
Energy input-SR	99.57
Energy input-CP	359.81
Materials Output Parameters	**kg/h**
Al source	150.00
Prefused slag 1	104.66
Prefused slag 2	188.18
Alloy	94.09
Reduction slag	405.91
Hot MG-Si / MG-Si	209.60
Refining slag for next PD	243.90
Hot HP-Si / HP-Si	284.21
Hot Purified Si-3N / Purified Si-3N for next PD	121.86
Hot Pot scrap / Pot scrap-byproduct	13.14
Energy Output Parameters	**kwh**
Energy output-Reduction	14.89
Energy to heat recovery 1	184.14
Energy to heat recovery 2	357.90

compositions	wt%
Al source	
Al	70.00
Al2O3	30.00
Prefused slag 1 & 2	
CaO	49.73
SiO2	49.76
Al2O3	0.51
Refining slag from previous PD	
CaO	46.91
SiO2	47.60
Al2O3	5.49
Alloy	
Si(l)	74.97
Ca(l)	17.17
Al(l)	7.86
Reduction slag	
SiO2	4.42
CaO	35.61
Al2O3	59.97
MG-Si / Hot MG-Si	
Si	99.03
Ca	0.91
Al	0.04
Fe	0.01
Refining slag for next PD	
SiO2	47.31
CaO	46.61
Al2O3	6.08
Hot HP-Si-4N / HP-Si-4N	
Si	99.99
Ca	0.01
Al	0.00
Purified Si-3N from previous PD	
Si	99.95
Ca	0.05
Al	0.00
Hot Purified Si-3N / Purified Si-3N for next PD	
Si	99.95
Ca	0.05
Al	0.00
Hot Pot scrap / Pot scrap -byproduct	
Si	84.78
Ca	14.39
Al	0.65
Fe	0.18

This process represents for continuous production.

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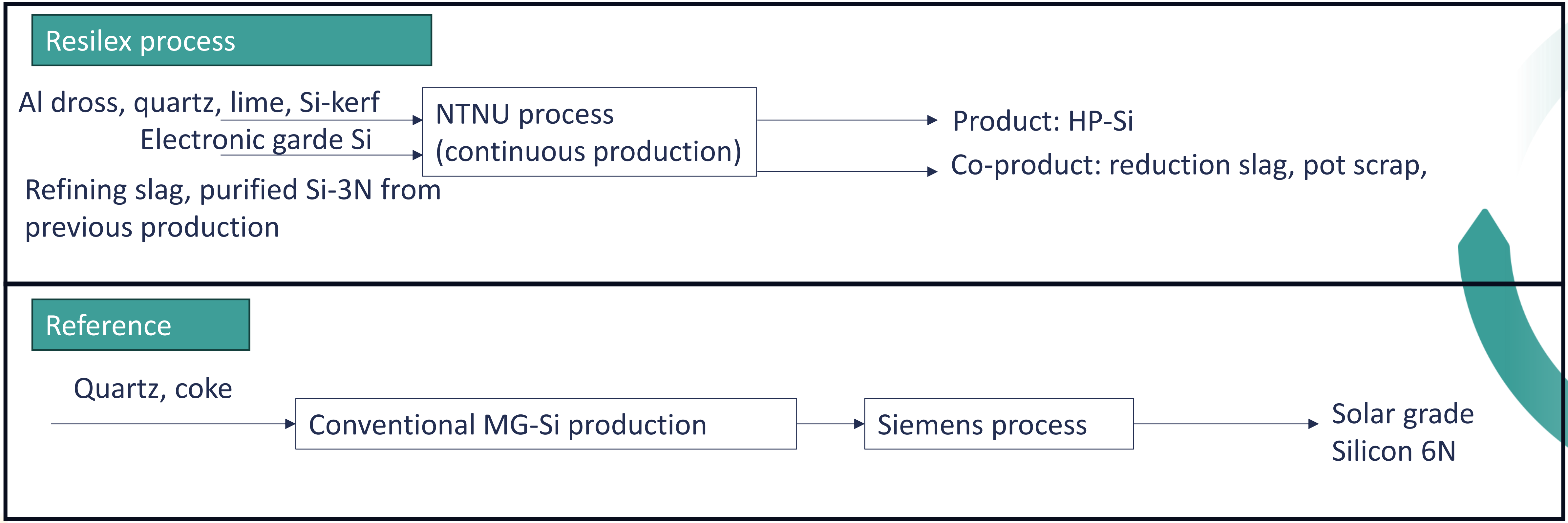
The system boundary for LCA and LCC



Conclusion

- NTNU produced approximately 200 kg of Si following the SisAl process with a **purity obtained reached 98.91%** Si. Major impurities were Ca, Al and Fe.

- Two ingots were produced by NTNU (directional soldfication => 3N and crystal pulling => 5N)

- The produced silicon was sent to CEA that produced three 30kg Cz ingots and evaluation for PV production.

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