
# NyKoSi:
# New Composite for Silicon and Ferrosilicon Production

**Presentation by Bodil Monsen**
SINTEF Materials and Chemistry,
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## IPN project for FFF in cooperation with SINTEF

IPN = Innovation project with support from the Research Council of Norway
FFF = The Norwegian Ferroalloy Producers Research Association

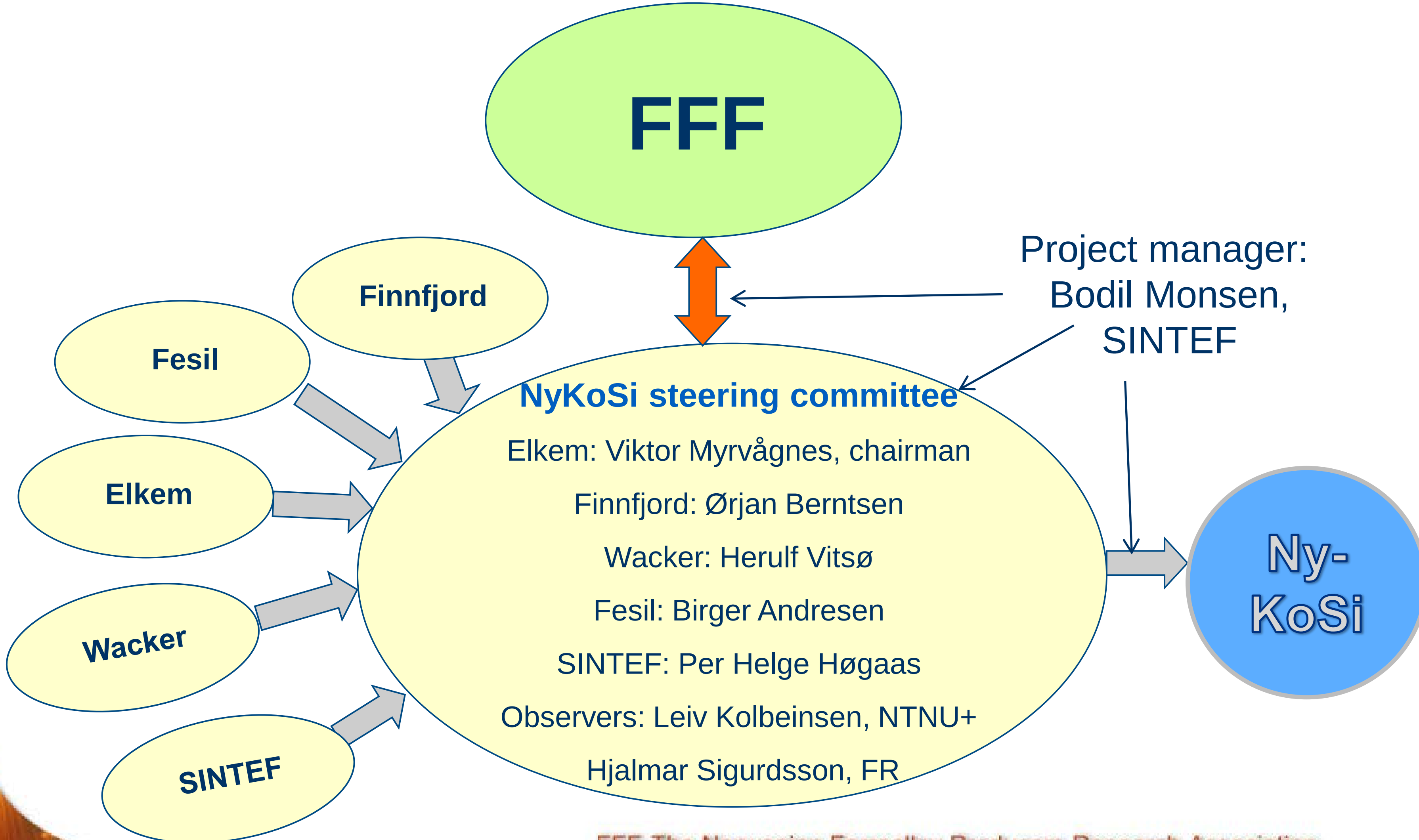
# NyKoSi:
# New Composite for Silicon and Ferrosilicon Production

Content:

- Project organization, main objective and sub goals, added value potential
- Raw material fines identified
- Chosen agglomeration method
- Briquetting machine and example of produced briquettes
- Briquetting in BRIKETTA, a video
- Testing briquettes and correlation between results
  - Cold crushing strength
  - Drop and Drum test
  - Porosity, Bulk weight
  - Thermal shock test
  - SiO reactivity test
- Summary of achievements

# NyKoSi – IPN project (2014-2017)

**2 steering committee meetings per year**

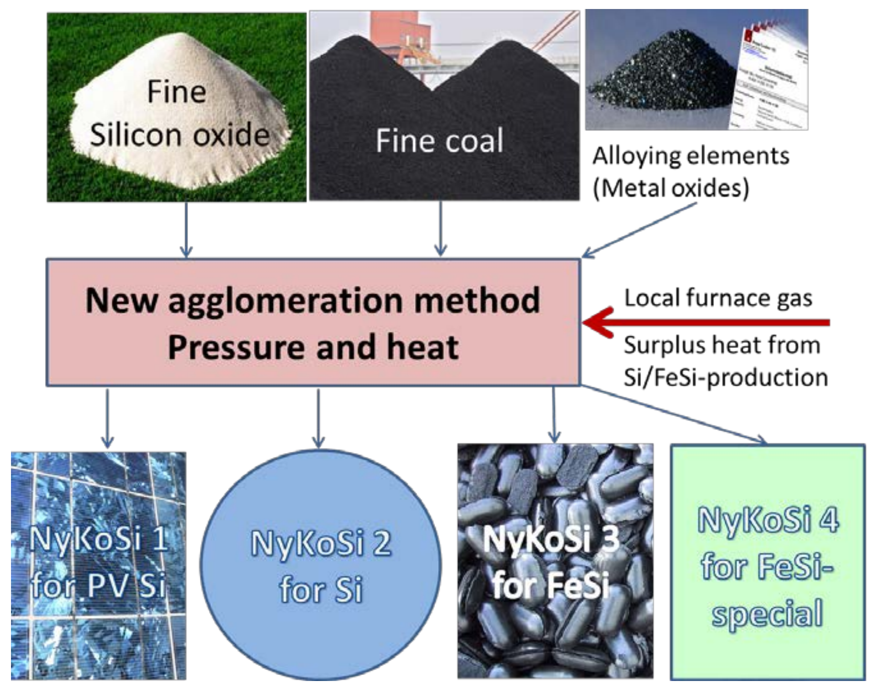


# NyKoSi – Main objective and sub goals

**Main objective: Develop a flexible agglomeration method for a new, reactive and environmental friendly composite as raw material for production of Si/FeSi**

Subgoals:

- Develop a cost effective agglomeration method that can utilize surplus heat and a broad specter of raw material fines.
- Achieve good bonding without the use of bonding agents, or only a small amount environmentally sound bonding agent.
- Develop a NyKoSi with good cold and hot strength and good reactivity towards SiO gas.




To be achieved through choice of reactive carbon sources and combinations
– also with quartz fines.

# Added value potential for NyKoSi

Ny-
KoSi

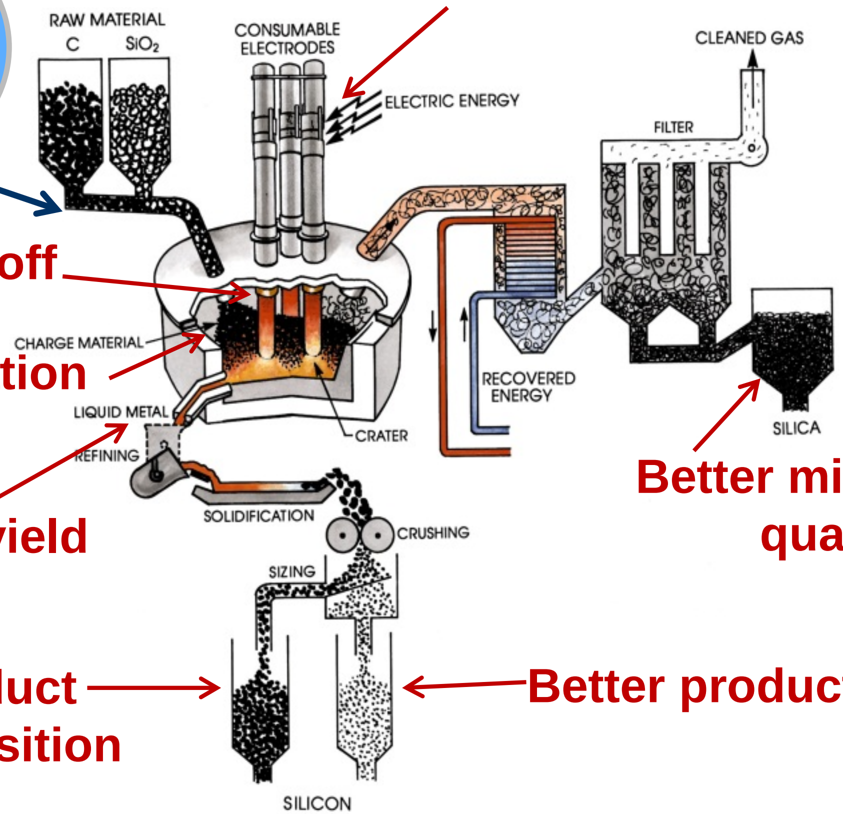
Reduced specific energy consumption

Reduced burn-off

More stable operation

Better yield

Better microsilica quality

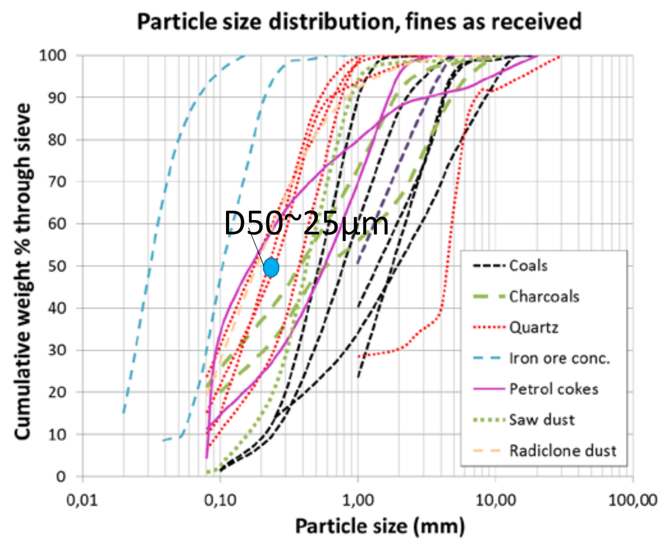



More pure product
Correct composition

Better product quality

# Raw material fines - agglomeration method

1. Identified and got hold of expected suitable fines (20) for FeSi/Si-production.
   - Charcoal (2), Coal (7), Petrol coke (2), Saw dust (1)
   - Quartz (5), Iron ore concentrate (2), Radiclone dust (1)
2. Characterized the fines:
   - Proximate- and ash analyses; Chemical composition, Free Swelling Index (FSI)
   - Grain size distribution; Sieve analyses, D50, D80, %< 1mm.




The fines are rather coarse:

- Coals: D50 = 0.5 - 2 mm
- Quartz: D50 = 0.2 - 5 mm
- Often enriched in impurities

3. Agglomeration methods considered:
   - Pelleting: Preferred D50 around 25 µm
   - Briquetting: Preferred < 1-3 mm (or maximum 6mm for big briquettes)
4. Briquetting chosen based on the rather coarse grain sizes of the fines, avoiding grinding/milling.

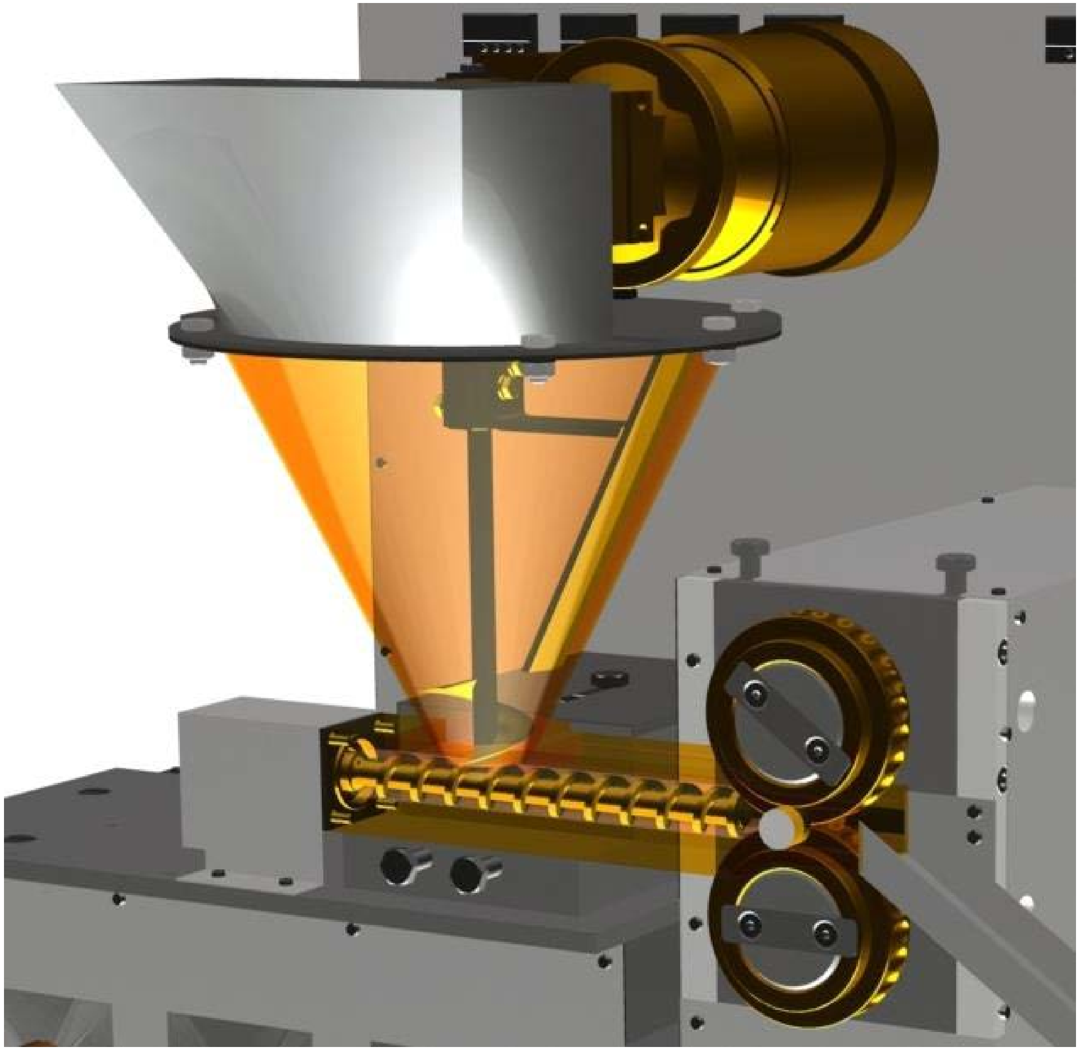
# New briquetting machine (B050): named BRIKETTA

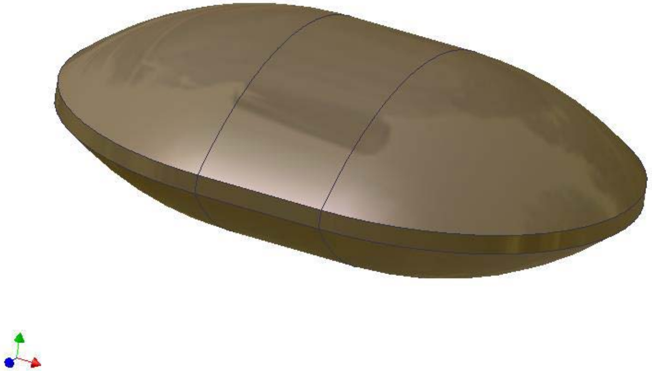
## Smallest lab scale Roller Press, from Euragglo/K.R.Komarek,US)


**Capacity/throughput : 1-45kg/h**

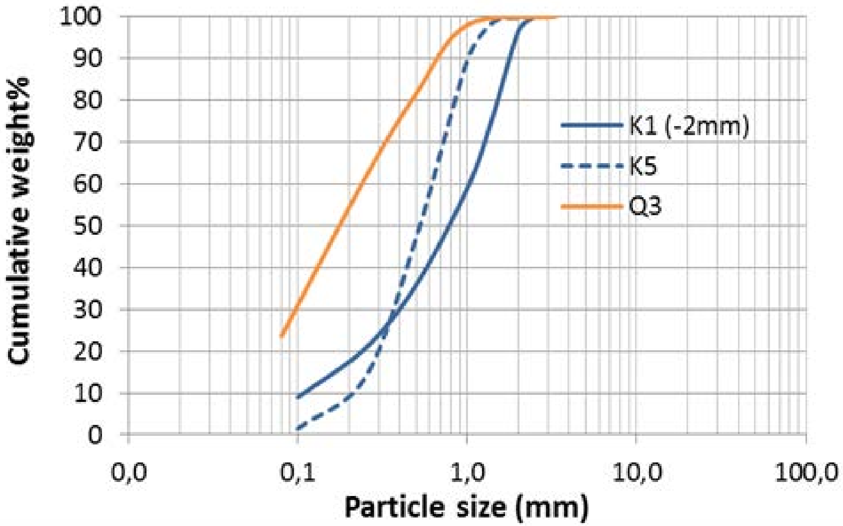
Roll Diameter & width: Φ100mm, 20mm (or 38, 16)
Total power installed: 1.5 kW
Roll speed: 0 - 6.5 rpm
Max roll separating force: 50 kN
Max roll torque: 2·420 Nm
Feed screw speed: 0-138 rpm
Max feed screw torque: 43 Nm
Feeder: Agitator speed: 0-22 rpm
Max briquette size: 34·18·12mm
Our briquette size: 17·11·7mm
Total power: 1.8 kW

- Designed specifically for research.
- Overall size 724 · 825 · 834 [mm]
- An inexpensive, versatile tool to provide data for improving existing or implementing a new roller press.
- Stainless steel, easily removable for quick setup.
- Integrated mixing system allows small batches
- Direct loading into the screw feeder.
- Continuous display of important process parameters.
- Output to data acquisition system.





# Two examples:



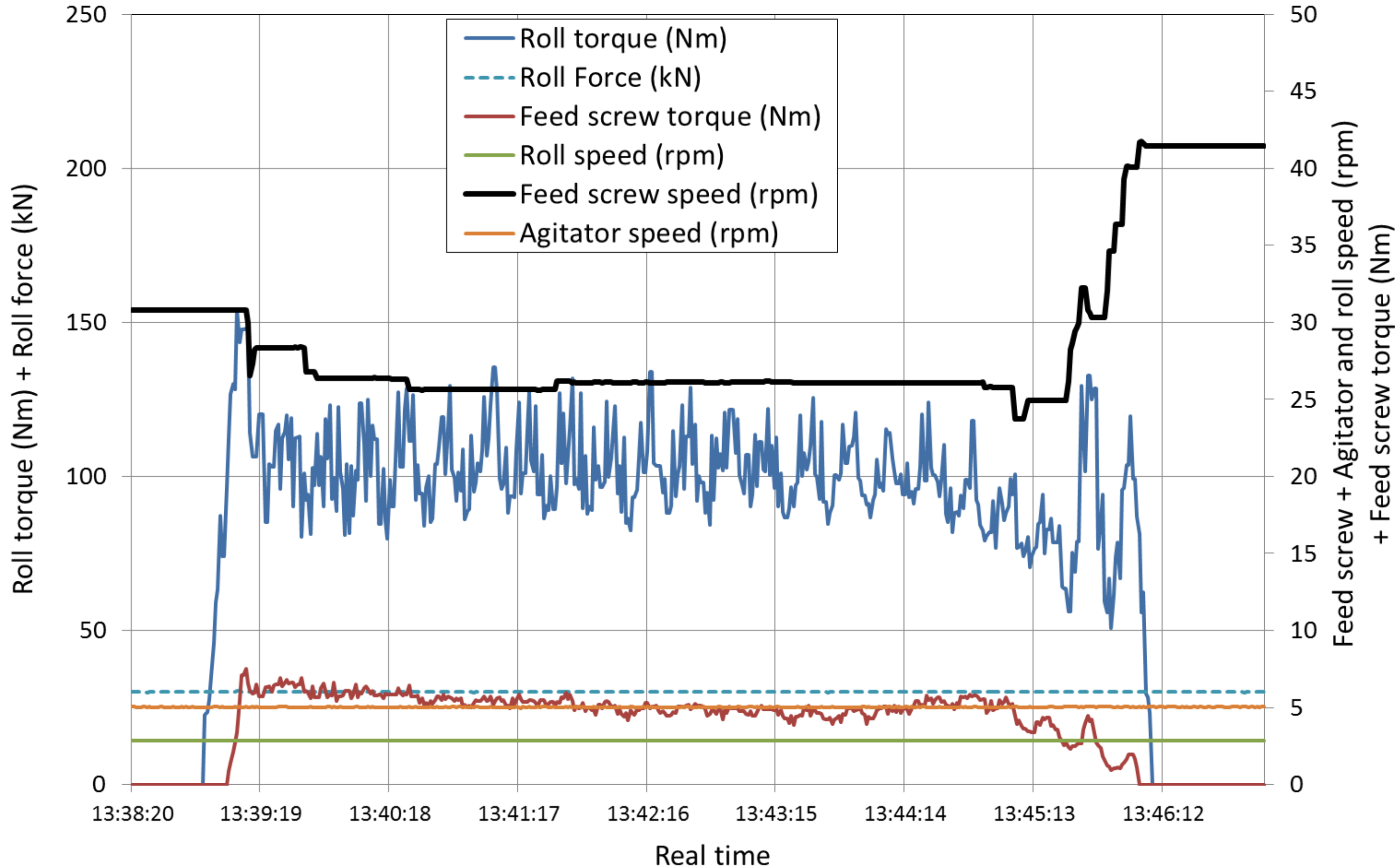

## NyKoSi-briquettes made from different fines in BRIKETTA

Briquettes directly from BRIKETTA

Cured hot briquetted briquettes

# Reporting template used for tests in BRIKETTA

## An example: Automatic curve generated



# Characterization, correlation and evaluation

Tests:

- Cold crushing strength
- Drop test
- Drum test
- Porosity, Bulk weight
- Thermal shock test (1200 °C)
- SiO reactivity test (1650 °C)

# Cold Crushing Strength (CCS)

Important for the ability to withstand to size degradation

- ISO 4700:2007 has a recommendation for pellets (D=10-12.5mm), which are close in size to our briquettes (7·11·17mm).
- The briquettes are compressed between 2 parallel steel plates at a speed of 10mm/min (ISO recommend 10-20 mm/min).
- Curve for force and displacement are recorded.
- The crushing strength is the maximum load applied (daN).
- Deviation from ISO: 15 briquettes used instead of 60 pellets.

Precision for 95% confidence levels:

$$\beta=\frac{2\sigma}{\sqrt{n}}$$

β = Precision
σ = Standard deviation
n = Number of briquettes.

Some precision lost using 15 briquettes instead of 60.


Hydraulic press

# Comparison of Cold Crushing Strength (CCS) for different briquette sizes

$$\text{CCS (MPa)} = \frac{Load\ (daN) \cdot 10}{A\ (mm^2)}$$

Large briquettes A1 = 40mm · 19mm = 760 $mm^2$
Small briquettes A2 = 17mm · 11mm = 187 $mm^2$

$$\text{Load (daN) small briquettes} = \frac{CCS\ (MPa) \cdot A2}{10}$$

| Cold Compressive Strength (CCS) Selected results | Load (daN) | CCS (MPa) |
|---|---|---|
| Komarek B100A roller press, large briquettes | | |
| Minimum target for this technique (Clark et al) | 26.5 | **0.35** |
| Coal A (FSI=9.5), Mangena and Cann, **VERY STRONG** | 84 | **1.1** |
| Coal B (FSI=3.5), Mangena and Cann, best result | 66 | 0.87 |
| Komarek B050A, BRIKETTA, small briquettes: | | |
| Corresponding minimum target this technique | **6.5** | **0.35** |
| Corresponding strength, coal A called very strong | 20.8 | 1.1 |

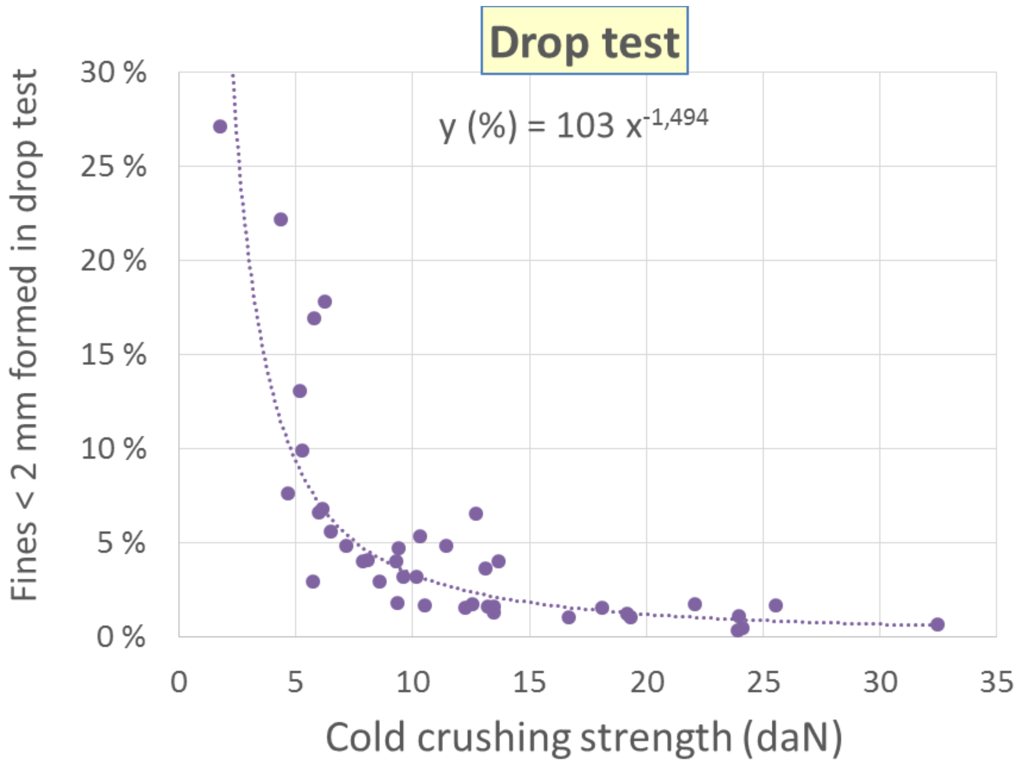
Clark et al, 1998, Brisbane, Australia, minimum target for this technique.
Mangena and Cann, binderless briquettes, International Journal of Coal Geology 71 (2007) 303-312.

# Drop test

Drop test by
Edith Thomassen

Drop test at SINTEF:
10 briquettes are dropped 4 times through a 2m long tube (D15cm)
Sieve analyses: 0.5, 1.0, 2.0, 4.0, 6.3mm

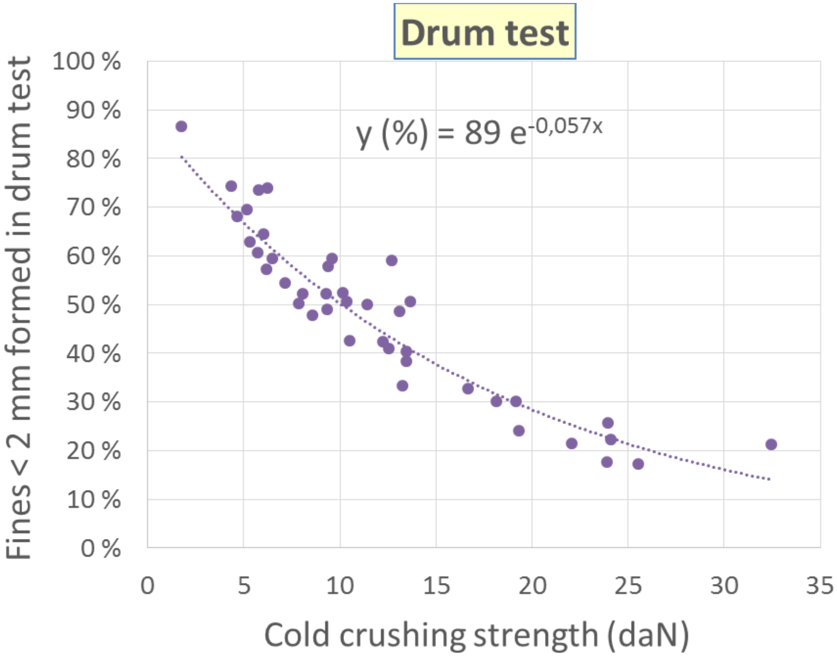
Evaluation based on fines formed: wt% < 2.0mm



# Drum test

A measure for the ability to withstand size degradation, especially during handling and transport.

Drum at SINTEF/NTNU, test by Edith Thomassen




120 g briquettes in a drum for 30 min at 40 rpm
Sieve analyses: 0.5, 1.0, 2.0, 4.0, 6.3mm
Evaluation based on fines formed: wt% < 2.0mm

| | Weight | Parallels | Sample size | Drum Diameter/length | Rate | Time |
|---|---|---|---|---|---|---|
| ISO 3271* (pellets) | 15 kg | 4 | D= 6.3 – 40 mm | 1000 mm / 500 mm | 25 rpm | 8 min |
| SINTEF briquettes | 120 g | 1 | 7·11·17 mm | 200 mm / 100 mm | 40 rpm | 30 min |

*Determination of Abrasion index (AI) wt%<0.5mm and Tumble Index (TI) wt > 6.3mm

The drum and test conditions are also used in tumble tests for other materials at SINTEF/NTNU.

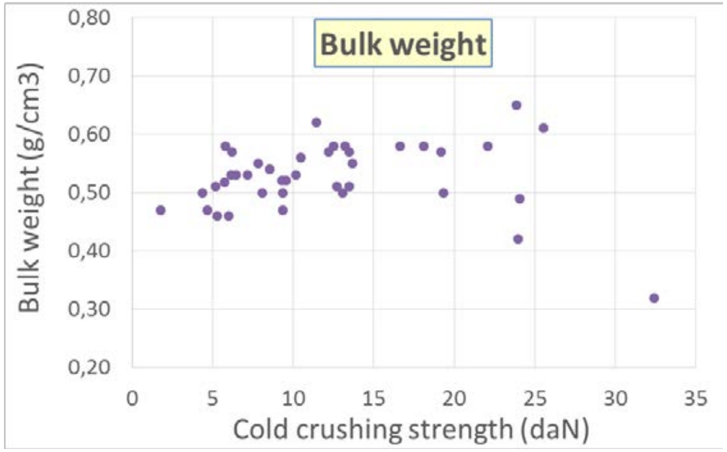
# Porosity

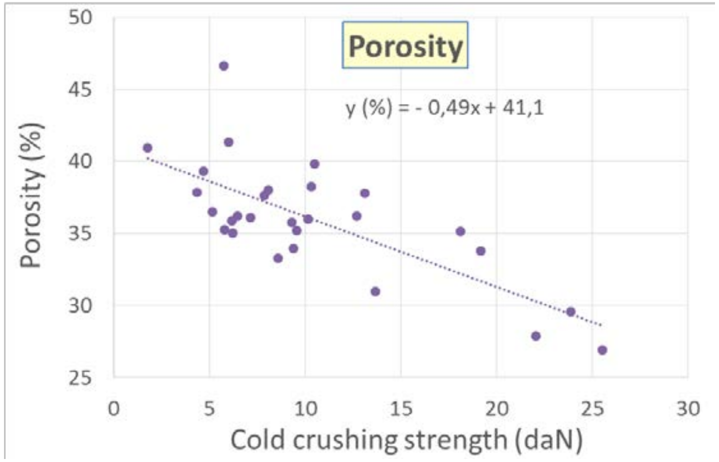
$$Porosity\ (\%) = \frac{\rho\ (absolute) - \rho\ (apparent)}{\rho\ (absolute)} \cdot 100$$

$\rho\ (absolute)$: Absolute density without open pores
$\rho\ (apparent)$: Apparent density by GeoPyc 1360.

Porosity is measured by Irene Bragstad at SINTEF.

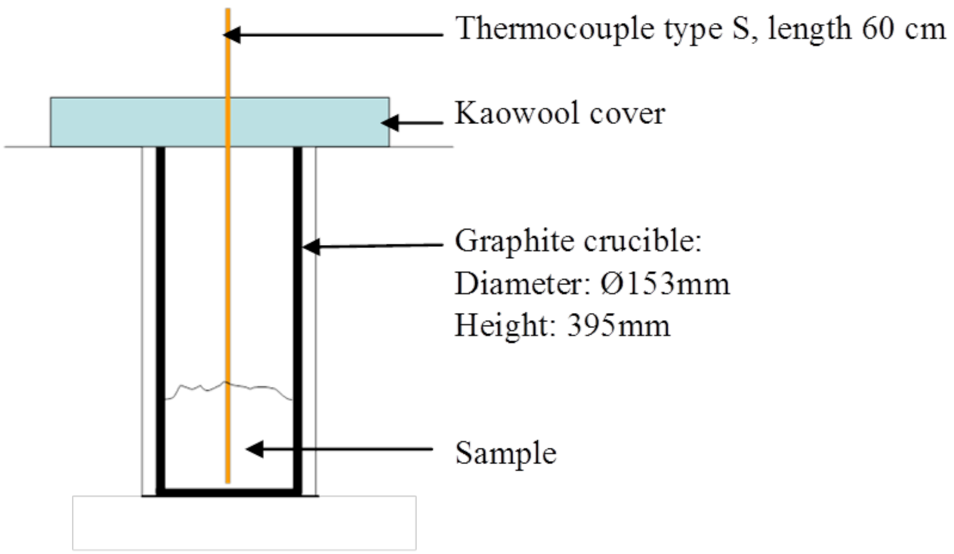
| **Briquette example** | | |
|---|---|---|
| Absolute density (on powder) | $g/cm^3$ | 1.54 |
| Apparent density (briquettes) | $g/cm^3$ | 1.10 |
| Porosity | % | 28.3 |
| Porosity average of 4 briquettes | % | 27.9 ± 0.5 |

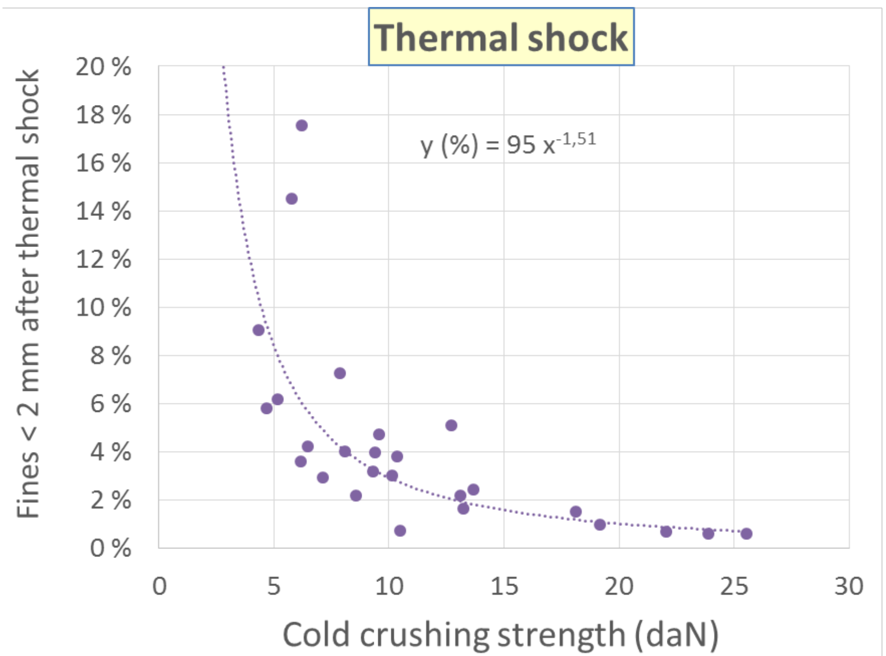



- Absolute density by gas pycnometer **AccuPyc II 1340**, best on powders. Crushed briquettes (g) are poured into a cylinder cup with known volume. Helium gas is filled into the cup and will fill open pores. The powder volume ($cm^3$) is the cup volume subtracting the He volume.
- Apparent density is the briquette weight divided by it's envelope volume determined by **GeoPyc 1360** that measures the volume of sand with and without briquette and calculates the porosity.

# Thermal shock test at 1200°C




Thermal shock test at SINTEF by Steinar Prytz:

100 g briquettes are poured into a graphite crucible at 1200°C.
They are kept there for 10 min, than poured into a metal container with lid.
Sieve analyses: 0.5, 1.0, 2.0, 4.0, 6.3mm, pictures of briquettes before and after the test

Evaluation based on fines formed: wt% < 2.0mm.

# SINTEF's SiO reactivity test

- Basis: reactivity with SiO gas
  - $SiO(g) + 2\ C(s) = SiC(s) + CO\ (g)$
- Operated since 1974
- Test to select reduction materials for Si production
- Reacted at 1650˚C with 13.5%SiO - 4.5% CO - 82% Ar,
  - producing 18% CO – 82% Ar, at start
- Average difference between repeated tests is less than 10%
- Determine the suitability of new reductant sources

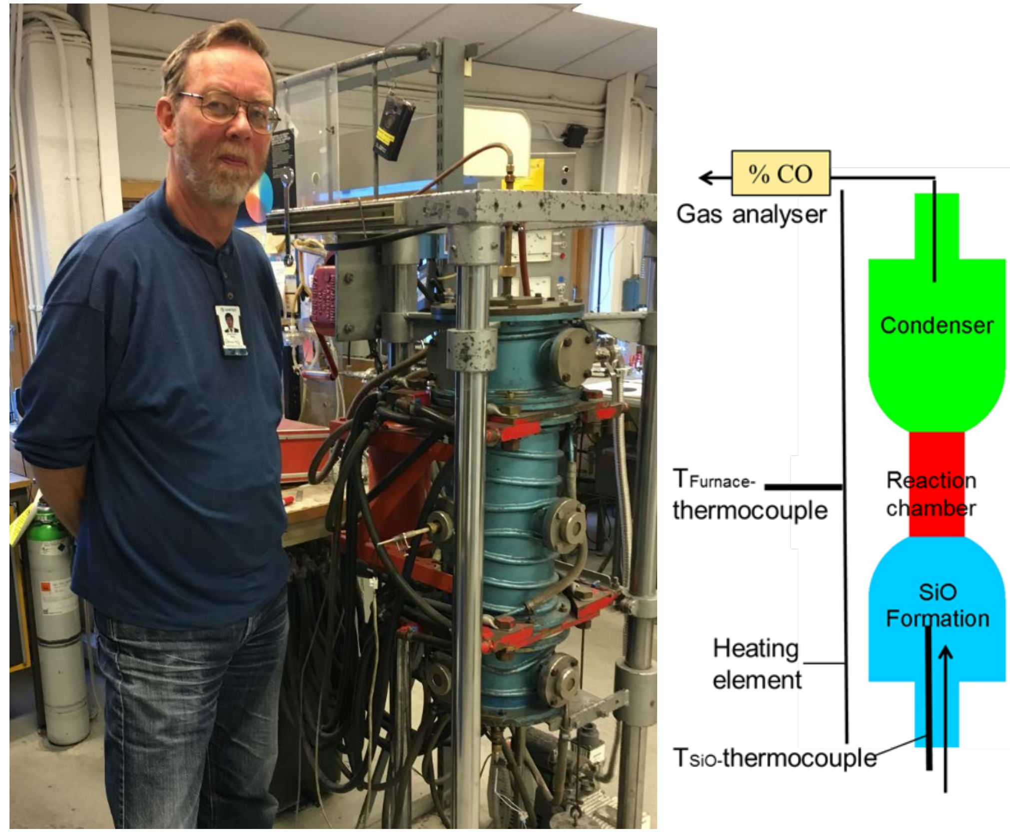
# SINTEF SiO gas reactivity test at 1650°C

## $SiO + 2C = SiC + CO$

SiO reactivity test:

1. Thermal shock test (1200 °C)
2. SiO reactivity test of grains 4-6.3mm (sample in the red reaction chamber).

- Vacuum graphite tube resistance furnace, see picture.
- Argon as an inert carrier gas.
- In-house made pellets produces the SiO gas together with some CO gas.
- Test sample reacts with SiO gas to SiC and more CO gas in the red chamber, no SiO gas pass through at start, Argon with 18% CO will pass.
- The CO content is recorded and decreases as the reaction slows down.
- Stops at 5.2 % CO, which corresponds to the max conversion of C to SiC.




The SINTEF SiO reactivity test by Steinar Prytz.

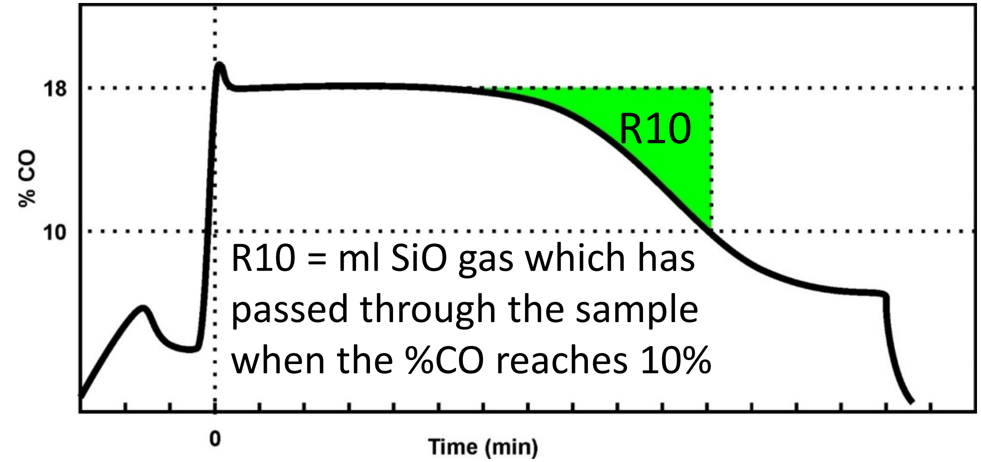
# SINTEF SiO gas reactivity test at 1650°C

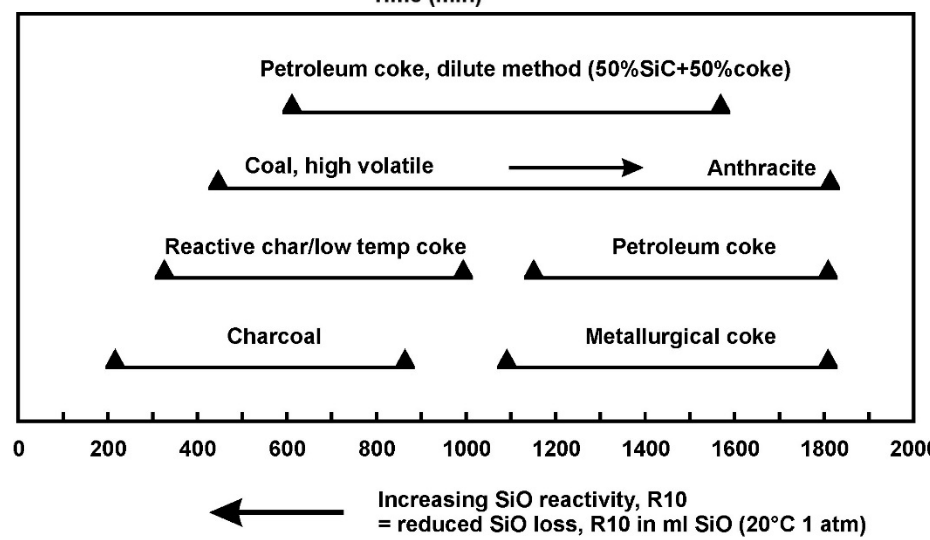
## $SiO + 2C = SiC + CO$

- The test is widely used by the silicon and ferrosilicon producers.
- It simulates this reaction that is occurring in the upper part of industrial submerged arc furnaces.

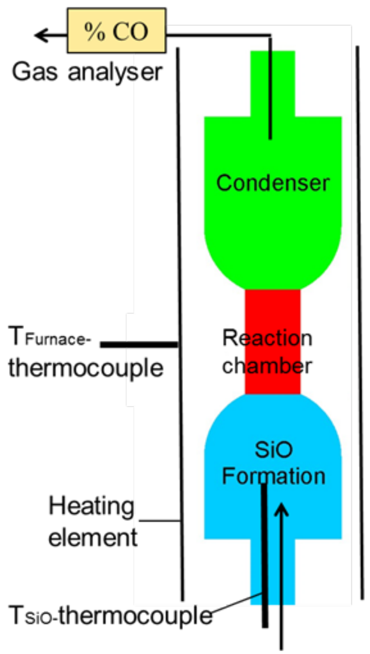
SiO reactivity evaluation:

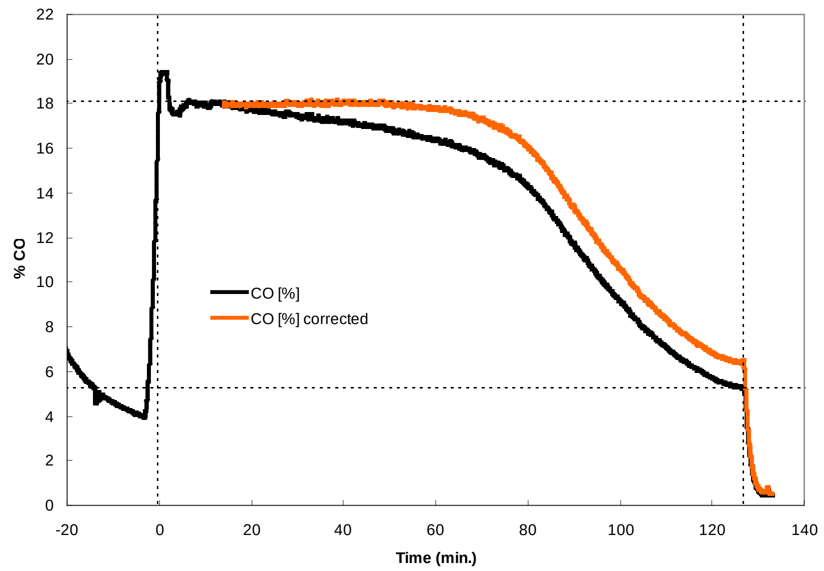
1. Good reactivity: Low numbers (for instance R10, R10corr, R5.2) meaning that a low amount SiO gas (ml) has passed the sample without reacting.
2. High degree of conversion to SiC. It is essential that carbon react completely to SiC so the reacted sample should not contain un-reacted carbon.



# Condensation chamber reactions




- Condensation, main reaction: $SiO \rightarrow Si + SiO_2$
- May be followed by: $CO + Si \rightarrow SiC + SiO_2$
- Alternative (pellet back reaction): $CO + 3SiO \rightarrow SiC + SiO_2$

- "Back-reactions" consumes CO, corrections made, see orange curve
- Reaction increases as test progresses

# Degree of Conversion

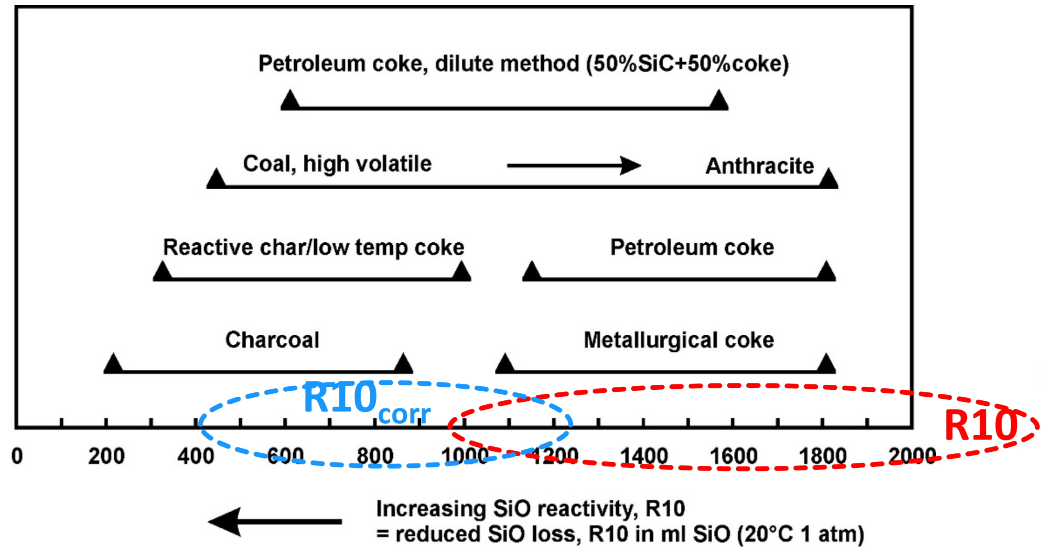
- Degree of conversion is important
  - An outer dense rim of SiC product may be formed, preventing further SiC formation.
  - Unconverted carbon is then found in the grain center
  - Unconverted carbon reaching into the lower part of FeSi/Si furnaces will reduce the Silicon yield

- Samples analyzed for:
  - Initial %C (fixed carbon in the calcined sample)
  - Total carbon and unreacted carbon in SiO-tested sample

- Material balance for carbon in each test; Conversion to SiC is calculated

# SiO reactivity of briquettes

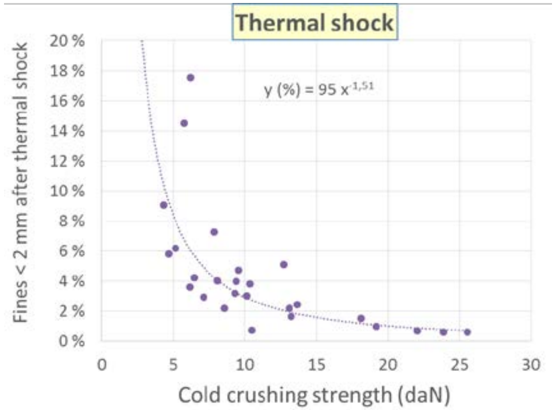
- The SiO reactivity varies from good to poor for the briquettes tested
    - See ranges for R10 and R10corr in the diagram below.
- Probably dependent on several parameters such as:
    - Raw materials used and their composition
    - Grain sizes (max particle size in the raw materials used for briquetting)
    - Porosity/density etc.
- The degree of conversion has usually been good for our briquettes.
- A modified SiO reactivity test has been developed for briquettes with quartz.




# NyKoSi: Summary of achievements

- We have identified, analyzed and tested 20 raw material fines.
- Briquetting chosen based on the coarse grain sizes of the fines, avoiding to raise the price by grinding/milling needed for pellets
- SINTEF invested in a laboratory roller press, called BRIKETTA.
- We have carried out more than 50 tests in BRIKETTA.
- A briquette test program has been developed, including;
  - Thermal shock, strength, drop and drum tests, porosity
  - SiO reactivity, or modified SiO reactivity for briquettes with quartz.
- Screened briquette properties for a broad specter of raw material combinations.
- Good correlation between compressive strength, and the amount of fines created in drop, drum, and thermal shock tests.
- High briquette yield above 95 % can be obtained without the use of a binder in cold briquetting by recirculating some fines.
- Strength improves by curing the briquettes.

It will probably be possible to design a flexible briquette with the desired properties by combination of raw materials, without the use of binder, but further testing is required.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**Thank you for your attention!**