



D3.3 – Ingot-scale assessment of the PV properties of the produced silicon



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Index

Executive Summary	4
Introduction	5
Background and motivation	5
Objectives	5
Recycled silicon production	6
Directional solidification	6
Czochralski growth process	8
Reference	9
Cz silicon ingot for solar application	9
“Recycled silicon” ingot after additional directional solidification	10
Resistivity and type of conductivity	10
Glow Discharge Mass Spectrometry (GDMS)	11
Cz ingot characteristics	13
Simulation 1 : 100% recycled silicon	13
Simulation 2 : Blending - 1.8% of recycled silicon with 98.2% of EG silicon	14
Cz ingot from simulation 2	15
Brick squaring and characterization	17
Brick, grinding & wafering	17
“Recycled silicon” ingot from crystal pulling	19
Glow Discharge Mass Spectrometry (GDMS)	19
Conclusion	22

Executive Summary

The EU Horizon Resilex project aims to improve the resilience and sustainability of the silicon value chain in Europe. A sustainable method for silicon production and recycling of silicon from the solar industry are among the project's focus areas.

Here the first steps of the silicon value chain with the demonstration of a sustainable carbon-free process for the production of silicon compatible with solar applications are considered. From the purified silicon produced by NTNU, CEA assessed the suitability of alternative feedstock for the production of solar cells for two types of recycled silicon: one after additional directional crystallization and the other after Czochralski growth process.

With the additional directional crystallization, the recycled silicon reached a level of purity between 2N to 4N. According to these results CEA pulled one Cz ingot that simulates a blending condition (1.8% of recycled silicon with 98.2% of EG silicon) while retaining only the boron and phosphorus concentrations of the recycled silicon. From this Cz ingot, 700 M2 size wafers were prepared for cell processing.

With the additional Czochralski growth process, the recycled silicon reached a level of purity of 3N to 5N. According to these results, CEA will attempt, in the first quarter of 2025, to manufacture a Cz ingot from this recycled silicon under the following conditions: 10% recycled silicon after additional Czochralski growth process and 90% EG silicon.

This study is part of the EU Horizon Resilex project, which aims to increase the circularity in the PV industry. The project has received funding from the European Union's HORIZON Action Grant program under Grant Agreement N° 101058583.

Introduction

Background and motivation

Silicon has been characterized as a critical raw material (CRM) by the European Union since 2014. Even though silicon is abundant in the earth's crust, it is part of the EU's CRM list due to its high supply risk and economic importance. Silicon has high economic importance in the EU due to its irreplaceable presence in numerous products that our society relies on. The supply risk of silicon metal is also categorized as above the limit for a critical raw material, because of the risks of disruption in supply. The main factor is the high dependency on imports. The import reliance of silicon in the EU is 63%, and China controls 66% of the global market share.

Silicon (Si) for solar applications has experienced rapid growth in demand for the past decade. State-of-the-art production of Si and solar panels are linear, carbon-intensive processes that produce several low-value by-products and wastes. A shift away from waste generation and carbon, towards circularity, is necessary to create a sustainable industry.

The EU Horizon Resilex project aims to address these sustainability challenges by demonstrating different industry-driven technological solutions covering the full silicon value chain. The overall project goal is to improve the resilience and sustainability of the silicon value chain in Europe. A sustainable method for silicon production and recycling of silicon from the solar industry are among the project's focus areas.

Objectives

In WP3, RESiLEX will focus on the first steps of the Silicon value chain with the demonstration of a carbon-free sustainable process for production of Silicon compatible with solar applications. From the purified silicon produced by NTNU, the CEA will evaluate the suitability of this alternative feedstock for the production of solar cells and will define in particular the best way to produce Czochralski monocrystalline ingots, which will be transformed into wafers and then solar cells, tested in WP4.

Keywords: Recycled silicon, Cz pulling process, Solar PV wafers

Recycled silicon production

The production of recycled silicon was carried out by NTNU according to the process presented in deliverable D3.1. The silicon reaches a purity level of 98.91% in case of medium scale slag refining process and 97.79% in case of large scale slag refining at ELKEM. After a vacuum refining step, to increase the purity level, NTNU studied two additional steps: directional solidification or crystal pulling which are presented in the following paragraphs. For the additional directional solidification step, silicon with a purity level of 98.91% was used. For the additional crystal pulling step, silicon with a purity level of 97.79% was used.

Directional solidification

The directional solidification experiment was carried out using the Crystalox DS250 furnace under conventional crystallisation settings. The furnace is induction-heated with a graphite susceptor around the crucible. The crucible rests on a water-cooled pedestal with a heat-leak valve between the pedestal and the crucible. To control the solidification speed and shape, the set-point temperature on the susceptor is gradually reduced, and at the same time, the heat-leak valve is progressively opened to control the heat flow downwards. The crucible used was a standard silica crucible. Before charging the crucible, it was coated with silicon nitride (Si_3N_4), which was then heated according to standard procedure to 1100 °C in air. This heat treatment causes the silicon nitride to undergo oxidation, resulting in its non-wettability towards molten silicon. The crucible was filled with 12 kg of Si refined by the slag refining technique (Deliverable D3.1) and subsequently placed inside the furnace. The temperature profile was chosen to be the same as for a regular cast of PV-silicon. In Figure 1, the logged temperature and power measurements are shown.

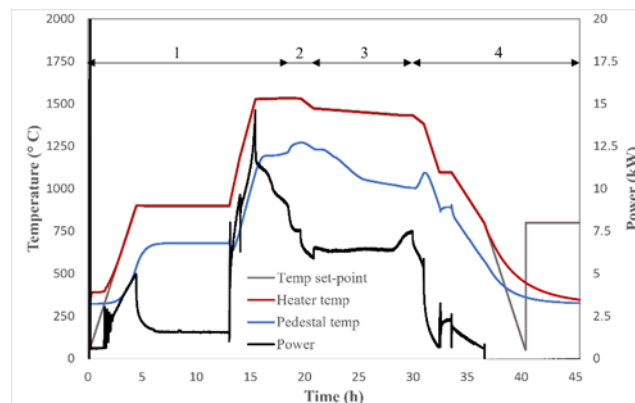


Figure 1: Logged data from the Crystalox furnace.

The graph's upper section displays arrows and numbers to represent distinct stages. These are:

1. Heating and melting of charge,
2. Time to secure complete mixing of the melt,
3. Crystallisation,
4. Cooling.

Figure 2 represents the product of Si purification by using the Directional solidification technique.



Figure 2: The ingot obtained in the Directional solidification process.

ICP-EOS analysis performed by NTNU along the ingot (Erreur ! Source du renvoi introuvable. Figure 3 & Figure 4) show:

- a maximum silicon purity of 3N is reached at the bottom part of the ingot (sample 6 in the figure). In this case, the silicon purification allows to go from a level of 98.91% after the "slag refining" process to 99.98% after directional solidification.
- a low silicon purity of 2N (~99.50%) in the central part of the ingot (samples 3, 4, 7, 8 & 9).

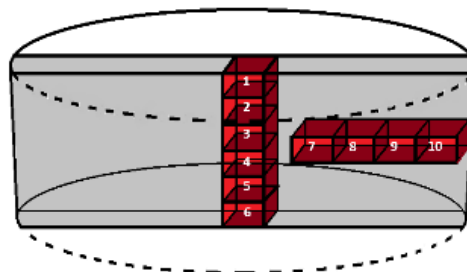


Figure 3: Position of the ICP-EOS analysis measurements.

Sample		Vertical						Horizontal			
		1v	2v	3v	4v	5v	6v	7h	8h	9h	10h
Al	%	0.0032	0.0068	0.0119	0.0115	0.0051	0.0026	0.0104	0.0085	0.0069	0.0027
B	%	0.0012	0.0012	0.0013	0.0014	0.0013	0.0011	0.0014	0.0014	0.0013	0.0012
Ca	%	0.0042	0.0089	0.0143	0.015	0.0066	0.0028	0.0133	0.0108	0.0087	0.0032
Cr	%	<0.0005	0.0012	0.0024	0.0027	0.001	<0.0005	0.0022	0.0019	0.0015	<0.0005
Fe	%	0.0525	0.149	0.301	0.332	0.115	<0.001	0.264	0.238	0.198	0.058
Mn	%	0.026	0.068	0.128	0.136	0.043	<0.001	0.111	0.093	0.078	0.024
P	%	0.00054	0.00073	0.0009	0.00093	0.00079	0.00041	0.0009	0.00085	0.00081	0.00067
Ti	%	0.01	0.0285	0.0565	0.063	0.0215	<0.001	0.0505	0.044	0.037	0.0115
V	%	0.00305	0.0084	0.01715	0.0196	0.00675	<0.001	0.01545	0.0135	0.01135	0.00365
Cu	%	0.00255	0.00575	0.0096	0.0092	0.0028	0.001	0.008	0.0069	0.00565	0.00175
Si	%	99.89626	99.72152	99.45695	99.40867	99.79616	99.98759	99.52285	99.58115	99.65079	99.89283

Figure 4. ICP-EOS analysis measurements

Czochralski growth process

The crystal-pulling experiment was conducted in the Czochralski furnace. 15 kg of starting material was blended with 15 kg of electronic grade Si to reduce the impurity concentration due to the limited furnace tolerance for the initial total impurity content. During the early phase of the Cz process, feedstock was melted in a quartz crucible. The growing process commenced with the seed dipping (in this particular experiment, an n-type seed) and involved multiple sequential stages. Following the dipping process, a thin section known as the neck was grown at a low growing speed of 1-5 mm/min until it reached the desired length, whereas the final section experienced a significantly faster growth rate. After the necking phase, the so-called "fishing" process began. This process involves nucleating the impurities visible to the naked eye on the surface of the melt by controlling the speeds of the seed and crucible rotation. Despite deviating from the usual protocol, a decision was made to halt the procedure and eliminate the grown section that had amassed a significant quantity of contaminants. Subsequently, the process's progression resumed; after a certain time, the necking phase started again, followed by the so-called "crown" growth, in which the diameter gradually increased. The crown shape was modified by manually adjusting the crystal pulling speed and the melt temperature. Once the crown reached a satisfactory diameter of about 170 mm, the pulling speed was increased, and the "shoulder" was made. The ingot's body is usually grown at a constant speed (typically 60-100 mm/h) until the target length is reached. Nevertheless, in this experiment, parameters had to be manually controlled, which resulted in an uneven shape of the ingot. Once the ingot achieved the desired mass, the final stage of the procedure, known as tailing, concluded the experiment.

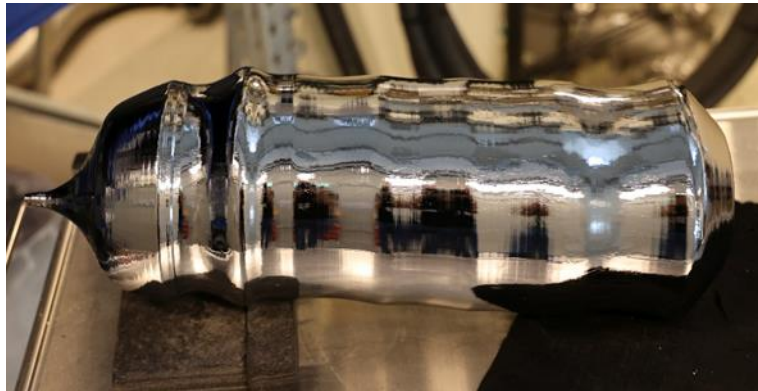


Figure 5: The ingot obtained in the Czochralski growth process

In this case, the silicon purification allowed to go from a level of 99.52% (Ratio: 97.79 % Si after the “slag refining” 15kg : electronic grade Si 15kg) to a purity level of 5N on the first 70% of the final Cz ingot.

Reference

K. Jakovljevic, E. J. Øvrelid, N. S. Ganesan, P. Tetlie, C. Van der Eijk, M. Wallin, G. Tranell, From waste to high-purity Silicon: Refining of Silicon by directional solidification and crystal pulling method (accepted for inclusion in the proceedings REWAS 2025: Sustainable Practices in Strategic and Critical Raw Materials: Exploring Supply Chain Resilience and Recycling Innovations).

Cz silicon ingot for solar application

NTNU sent to CEA two “recycled silicon” ingots:

- One after additional directional solidification. CEA has performed analyses first to evaluate the suitability of this alternative feedstock for the production of solar cells and then to define the best way to produce ingots in order to get M2-size silicon wafers that will be used to fabricate solar cells.
- One after Cz crystal pulling. CEA received the ingot in October 2024 and has performed a first set of measurements. The evaluation of the suitability of this alternative feedstock for the production of solar cells is still on going.

The following paragraphs present the results obtained for both “recycled silicon” ingots.

“Recycled silicon” ingot after additional directional solidification

For this ingot CEA has carried out various measurements:

- Resistivity along the height and width of the ingot
- Type of conductivity of the ingot
- Glow Discharge Mass Spectrometry (GDMS) on 5 samples along the ingot height.

From the results obtained, resistivity simulations were carried out to define the best way to produce the Cz ingot. Then one Cz ingot was pulled, squared into a M2-size brick and wafered.

Resistivity and type of conductivity

From the recycled silicon ingot, resistivity measurements were performed along the height and the width of the ingot (Figure 6). To perform the measurements CEA used the SEMILAB resistivity-meter RT1000 with the low resistivity range probe (0.01 Ω .cm to 0.5 Ω .cm).

It can be observed that the resistivity measurements are low, in the range of ~ 0.032 Ω .cm to- 0.043 Ω .cm with no lateral measurement variation. Moreover, the variation of the resistivity from top to bottom of the ingot do not match with a segregation profile.

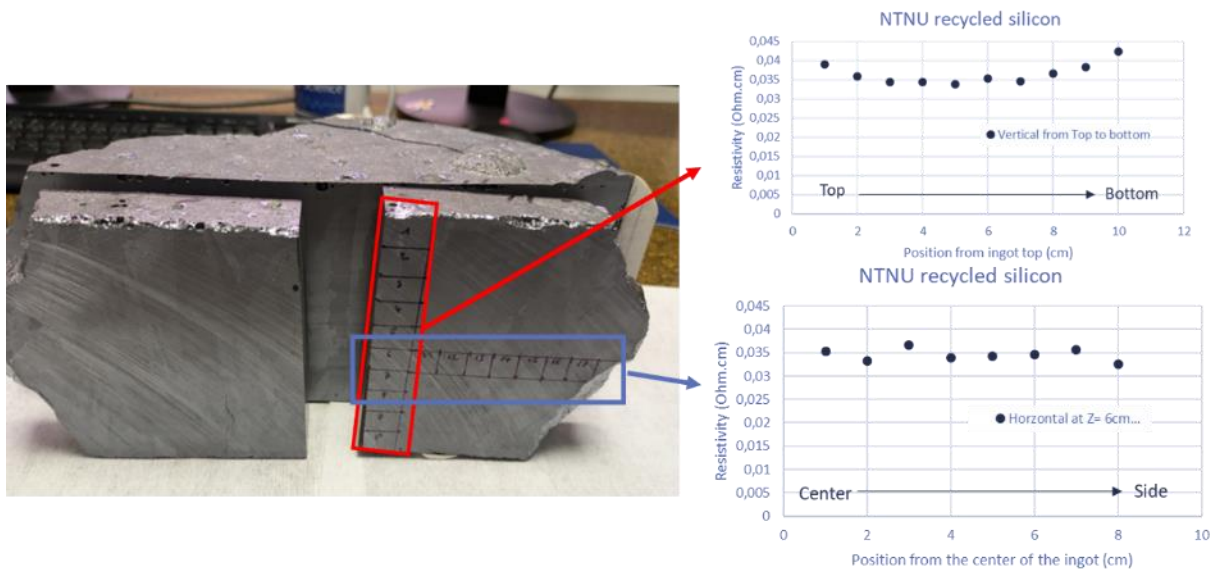


Figure 6: Resistivity measurements

The type of conductivity was checked from the top to the bottom of the ingot and from the center to the edge: all the ingot is p-type.

Glow Discharge Mass Spectrometry (GDMS)

To complement the ICP-OES measurements carried out by NTNU, CEA subcontracted GDMS measurements. Five samples were taken by CEA from the height of the ingot in its central part (Figure 7). The dimensions of the samples taken are: 2 x 2 x 2 cm³.



Figure 7: "Recycled silicon" ingot after additional directional solidification - Five GDMS samples.

On each sample, 25 elements were quantified and are presented in Figure 8.

The conclusions of the GDMS measurements are similar to those obtained by NTNU from ICP-EOS measurements: the silicon purity is in the range of 2N to 4N. The best purity level (4N) is reached at the bottom part of the ingot close to the crucible. The lower purity level (2N) is reached in the center part of the silicon ingot.

The GDMS measurement show:

- For the 5 samples the boron concentration is similar between 11 ppmw and 14 ppmw. But it can be noted that the absolute boron concentration values are very important compare to the boron concentration usually used in solar PV industry (0,05 ppmw). It is ~200 times higher.
- For phosphorus concentration the average concentration value for samples RESILEX1 to RESILEX4 is ~5.2ppmw. For sample RESILEX5 the phosphorus concentration is two times lower: 2.6 ppmw. However, it can be noted that the absolute concentration value is also important compare to phosphorus concentrations usually used in solar PV Industry (~0.2ppmw). It is ~10 times higher.
- Except for sample RESILEX5, the concentrations of metallic elements (Al, Mg, Fe, Mn, Ti, Cr, Ca, Co, Ni and Cu) are also significant for samples RESILEX1 to RESILEX4.

	RESILEX 1	RESILEX 2	RESILEX 3	RESILEX 4	RESILEX 5
	Top				Bottom
	[ppm wt]	[ppm wt]	[ppm wt]	[ppm wt]	[ppm wt]
B	11	11	13	14	11
Mg	0,49	3,1	1,9	5,7	< 0.005
Al	14	61	34	56	0,39
Si	~99.96%	~99.82%	~99.90%	~99.84%	~99.998%
P	4,4	5,0	6,2	6,1	2,6
S	0,02	0,02	0,01	0,01	0,01
Ca	12	45	21	39	< 0.05
Ti	23	120	74	170	< 0.005
V	5,9	30	15	41	0,003
Cr	2,1	11	5,4	14	< 0.005
Mn	110	520	280	430	0,07
Fe	180	920	520	830	0,17
Co	0,10	0,53	0,29	0,62	< 0.005
Ni	7,9	31	21	42	0,11
Cu	13	50	26	46	0,35
Zn	0,06	0,15	0,05	0,05	0,36
Ga	11	28	15	25	0,41
Ge	0,11	0,08	0,06	0,06	< 0.05
As	0,08	0,12	0,08	0,03	< 0.01
Se	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Ag	< 0.01	0,06	0,07	0,11	< 0.01
In	< 0.01	< 0.01	< 0.01	< 0.01	0,02
Sn	0,05	0,48	0,14	0,39	0,05
Sb	0,08	0,38	0,21	0,32	< 0.01
Pt	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Au	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Pb	0,04	0,27	0,19	0,51	0,01

Figure 8: "Recycled silicon" ingot after additional directional solidification - GDMS measurements for the five samples.

Cz ingot characteristics

In the frame of the RESILEX project, the Cz ingots for solar PV application using recycled silicon should fulfill the following characteristics:

- n-type Cz ingot to be compatible with solar heterojunction cells process used at CEA INES pilot line.
- Ingot resistivity in the range of 0.8 Ω .cm to 1.6 Ω .cm
- Silicon load: 30kg
- Ideally, the recycled silicon purity used should target the electronic grade purity (9N).

The (4N) purity of the recycled material does not allow CEA to use this material in its Cz furnace due to high contamination risks. Nonetheless, simulations were made to assess if it is possible to reach the required ingot resistivity and type using the dopants concentrations (boron, phosphorus and aluminum) of this material. From the results of GDMS measurements of the RESILEX5 samples, two simulations were done:

- Simulation 1: 100% of the recycled silicon is used
- Simulation 2: Blending of 1.8% of recycled silicon with 98.2% of EG silicon

The results of the simulation are presented in the following paragraphs.

Simulation 1: 100% recycled silicon

Simulation parameters :

- Silicon load: 30kg
- Boron concentration: 11 ppmw
- Phosphorus concentration: 2.6 ppmw
- Aluminum concentration: 0.39 ppmw

Figure 9 shows the resistivity profile along the ingot height under these conditions.

It can be observed that:

- the resistivity range is very low 0.04 Ω .cm along the ingot (value in accordance with the RT1000 measurements),
- the whole ingot is p-type.

Even if partial compensation is made by the presence of phosphorus in the silicon, this resistivity profile can be explained by the boron concentration which is ~200 times higher compare to what is usually used in solar PV industry. To reach the conductivity type and the targeted resistivity, blending with electronic grade silicon is needed.

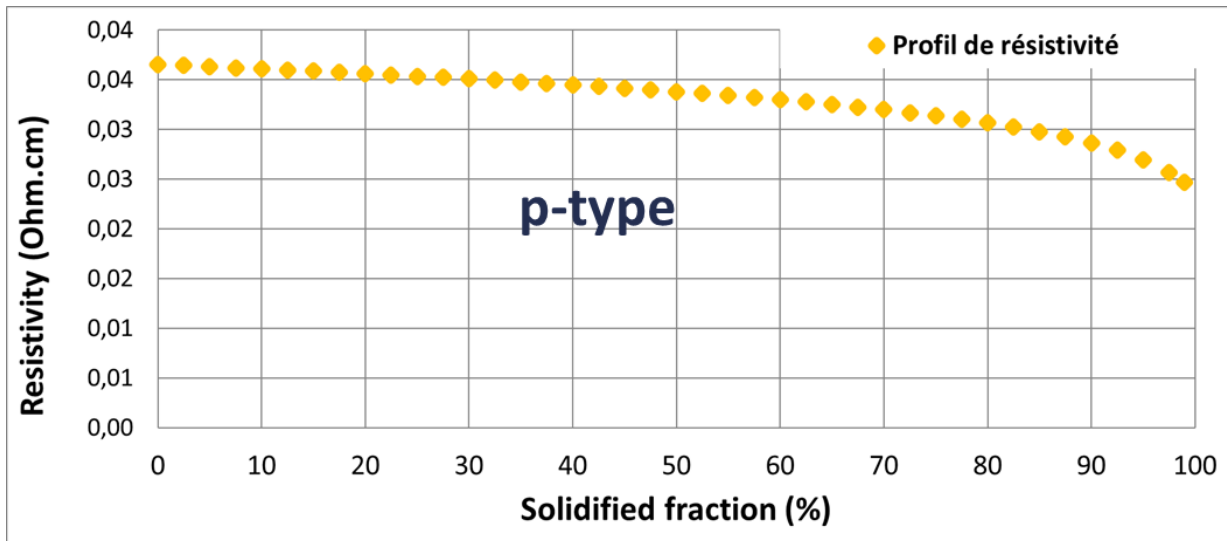


Figure 9: Resistivity profile simulation in case of 100% recycled silicon.

Simulation 2: Blending - 1.8% of recycled silicon with 98.2% of EG silicon

Simulation parameters:

- Silicon load : 30kg
 - o 1.8% of recycled silicon and 98.2 % of EG silicon
 - o Boron concentration: 0.2 ppmw
 - o Phosphorus concentration: 0.05 ppmw
- Additional concentration of phosphorus added in the silicon charge to aim for a resistivity of 1.6 Ω .cm at the top of the Cz ingot: 1.45 ppmw.

Figure 10 shows the resistivity profile along the ingot height under these conditions.

It can be observed that:

- the resistivity range is in the range of 1.6 Ω .cm to 0.2 Ω .cm at 70% of solid fraction,
- the whole ingot is n-type.

These results appear to be compatible with Cz ingot for solar PV applications. From the results of simulation 2, CEA decided to pull a Cz ingot under these “dopants concentrations” conditions. With this growth, CEA will simulate an ideal case corresponding to a recycled silicon where the other impurities (e.g, metals) were removed via known segregation methods for example.

This ingot will allow us to measure the impact of the dopants concentrations of the recycled silicon on the Cz growth and properties and then to measure their impact on the solar cell efficiency in WP4.

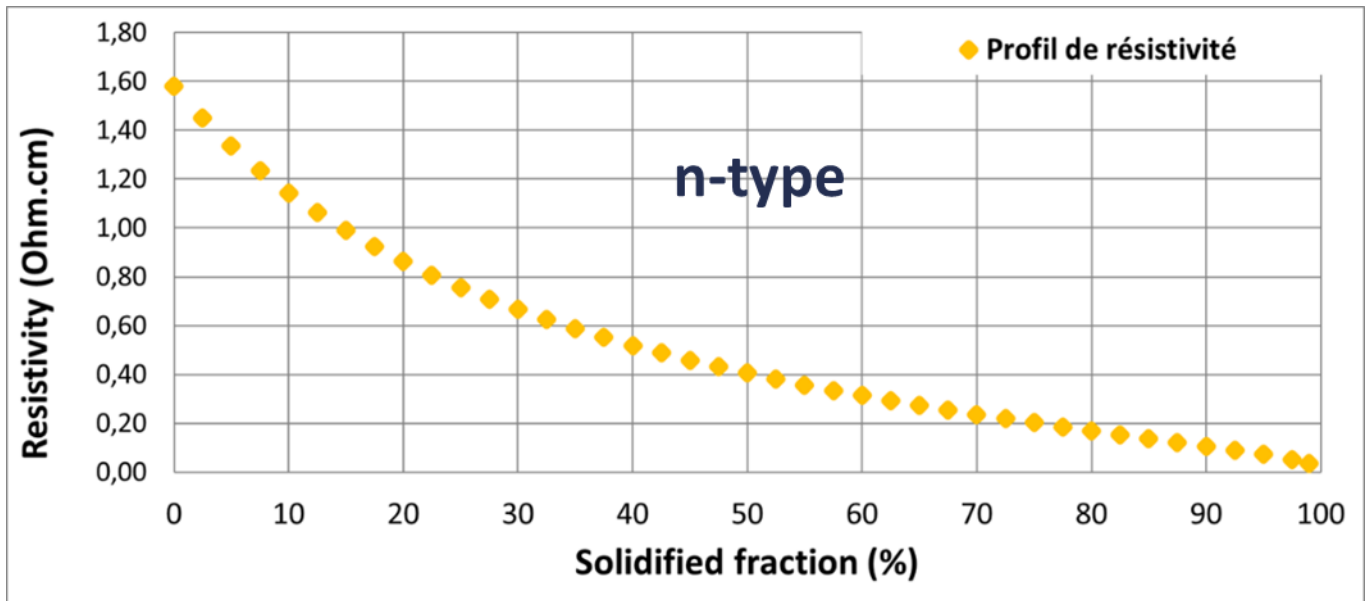


Figure 10: Resistivity profile simulation in case of blending - 1.8% of recycled silicon with 98.2% of EG silicon

Cz ingot from simulation 2

From simulation 2, the parameters used to pull the Cz ingot are:

- Silicon load: 30kg electronic grade
- Boron concentration : 0.2 ppmw
- Phosphorus concentration: 1.5 ppmw
- Ingot diameter target: 218 mm to be compatible with M2 size wafers

Heavily doped Cz wafers (Table 1) are used to add the required amount of dopants into the silicon feedstock. To achieve the doping targets, 7.44 grams of boron “silicon dopant” and 44.2 g of phosphorus “silicon dopant” wafers were added to the silicon feedstock (Table 1 and Figure 11 a.).

Dopant	“Silicon dopant” resistivity	Doping “silicon dopant”	Target concentration	Doping silicon weight added
	$\Omega.cm$	$at.cm^{-3}$	$at.cm^{-3}$	grams
Boron	0.0011	1.05E20	2.6E16	7.44
Phosphorus	0.0015	4.63E19	6.83E16	44.2

Table 1: Highly doped silicon Cz wafers and doping silicon weight added

The silicon was melted at around 1450 °C. After a temperature stabilization step of about 4 hours, the pulling process started with the necking step to eliminate the extended crystallographic defects generated by the thermal gradient in the seed when it touched the silicon melt. Then the pulling steps are the growth of:

- the crown to reach the target diameter of the ingot,
- the body (Figure 11 b.). It is the main part of the ingot used for squaring of the brick and cutting the wafers,
- and the tail, last part of the ingot before the ingot (Figure 12) enters the cooling chamber in the upper part of the puller.

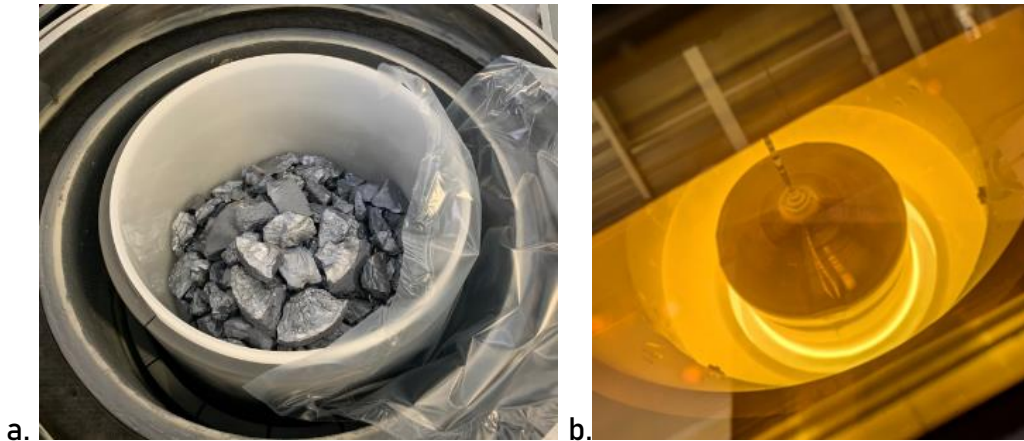


Figure 11: Cz ingot experiment: a. Quartz crucible loading, b. "Body" pulling step

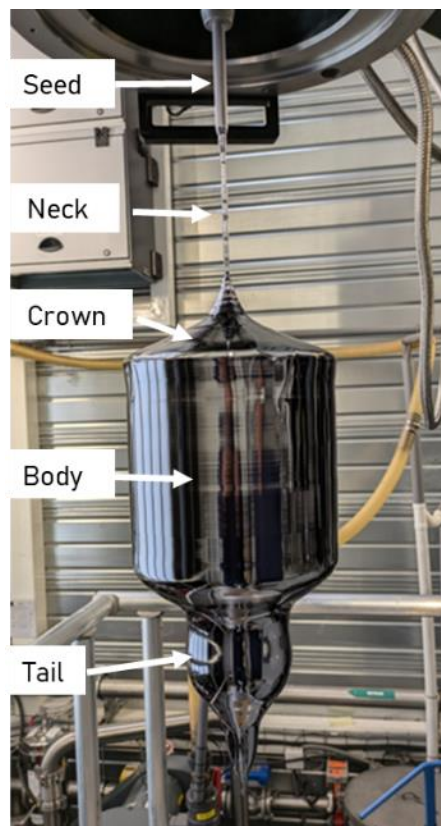


Figure 12: Cz ingot pulled according to simulation 2 parameters

Brick squaring and characterization

The squaring of the brick is performed at CEA with a “B.E.A” squarer (Figure 13 a.) using 0.6 mm diameter diamond wire. It is possible to cut two sides of the brick per cutting run, two cutting runs are thus necessary to square the brick.

The type of conductivity was checked from the top to the bottom of the brick: all the ingot is n-type as expected.

Resistivity measurements, with the SEMILAB resistivity-meter RT1000, were performed along the height of the brick. The resistivity of the brick varies from 0.4 Ω .cm at the bottom to 0.7 Ω .cm at the top. The resistivity at the top of the brick is two times lower as expected. Here are some points that could explain this difference:

- 1- boron concentration. Using the simulation tool and under the experimental conditions presented in the previous paragraph, if the quantity of boron in the silicon melt is reduced by 1.4 grams (-18%) then the resistivity at the top of the ingot goes from 1.6 Ω .cm to 0.7 Ω .cm. A loss of dopant can occur during the melting step through evaporation before the silicon is fully melted.
- 2- “silicon dopant” resistivity. Heavily doped Cz wafers (Table 1) are used to add the required amount of dopant into the silicon feedstock. If the “silicon dopant” resistivity of the boron is reduced by 10^{-4} Ω .cm (-9%), then the resistivity at the top of the ingot goes from 1.6 Ω .cm to 1 Ω .cm.
- 3- Quantity of silicon load. If the silicon load in the quartz crucible is 1.6% lower than expected (-0.5 kg) then the resistivity at the top of the ingot goes from 1.6 Ω .cm to 1.4 Ω .cm.

These simulations show the sensitivity of the co-doping on the final resistivity of the ingot. A combination of these points can explain part of the observed shift in the resistivity at the top of the brick.

Brick, grinding & wafering

In this paragraph, the brick grinding and wafering are presented. It is worth noting that the methodology presented here is in line with the equipment available at CEA: It is a “semi-manual” R&D mode compared to what is done today in industry.

The grinding of the brick is performed with an “Arnold” grinding equipment (Figure 13 b.). The grinding equipment allows the brick to be adjusted to the exact M2 size (Table 2). At each pass, it is possible to remove up to 300 μ m of silicon. Once the final size of the brick is reached, two “finishing” passes at 20 μ m and 10 μ m are carried out on each face of the brick (Figure 14 & Figure 15 a.).

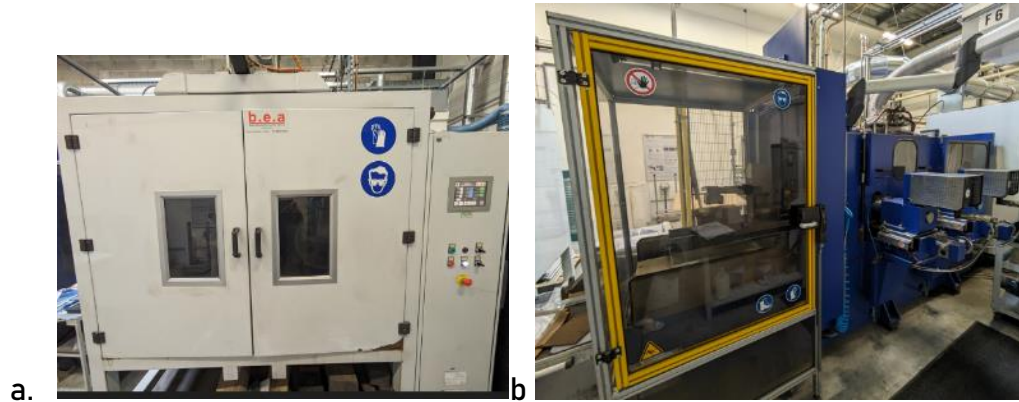


Figure 13: a. BEA squarer equipment (CEA), b. Arnold grinding equipment (CEA)

Brick size	Lateral side (mm)	Hamfer (mm)
M2	156.75 x 156.75 ± 0.25mm Diameter: 210mm ± 0.25mm	11.68 ± 0.25mm

Table 2: M2 brick size

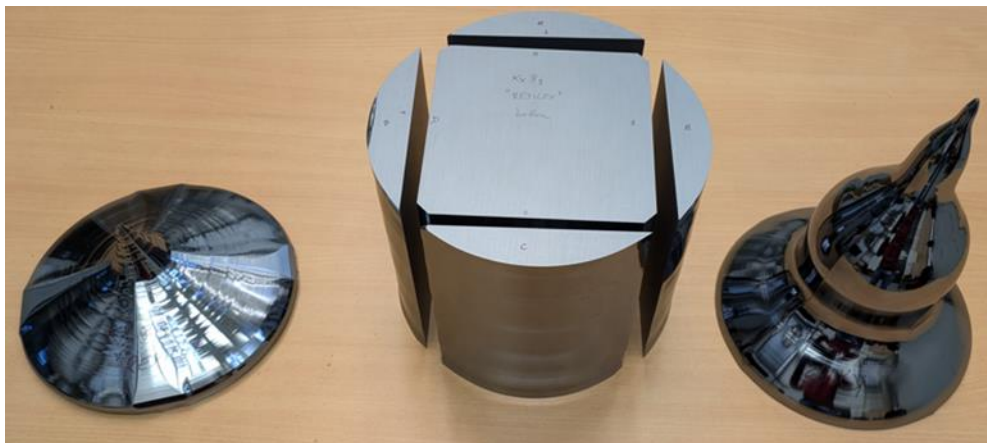


Figure 14: Cz ingot squaring and M2 size brick grinding

The brick was cut at CEA with the Meyer Burger DW288 sawing machine (Figure 15 b.). The diameter of the diamond wire used was 60 μm and the target thickness of the wafers was 160 μm . During the wafering step, no problem was observed and the wafer topology values are within the specifications (Table 3). The brick was cut into 700 wafers available for WP4.

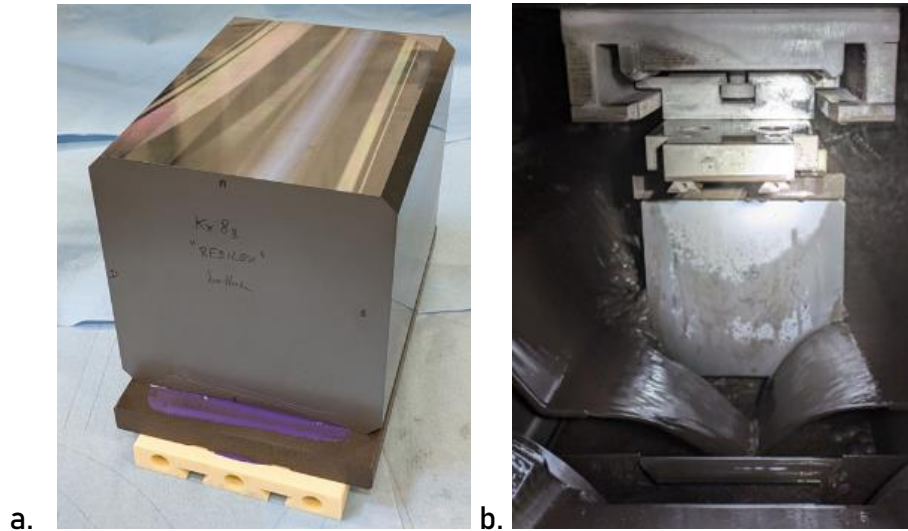


Figure 15: a. Brick glued on the beam before wafering, b. Wafering

	M2 wafers (μm)
Average thickness	159.4
TTV	13.5
Bow	5.2

Table 3: Wafer topology. Values averaged over 90 wafers along the brick (top, middle and bottom)- CEA.

“Recycled silicon” ingot from crystal pulling

For this ingot, CEA has carried out Glow Discharge Mass Spectrometry (GDMS) measurements on 5 samples along the ingot height. From the results obtained, resistivity simulation was carried out to define the best way to produce the Cz ingot.

Glow Discharge Mass Spectrometry (GDMS)

CEA subcontracted GDMS measurements. Five samples were taken by CEA from the height of the ingot in its central part (Figure 16). The dimensions of the samples taken are: $2 \times 2 \times 2 \text{ cm}^3$.



Figure 16: Recycled silicon" ingot from crystal pulling – Five GDMS samples along ingot height

On each sample, 25 elements were quantified and are presented in Figure 17. The silicon purity is in the range of 3N to 5N. The best purity level (5N) is reached in the main part of the silicon ingot for samples RESILEX2_1 to RESILEX2_4. The low purity level (3N) is reached at the bottom part of the silicon ingot (sample RESILEX2_5).

The GDMS measurements show:

- For the 5 samples, the boron concentration is similar between 1.5 ppmw and 2.7 ppmw. Compared to the previous silicon ingot the boron concentration has been reduced by a factor of about 7
- For the phosphorus concentration the average concentration value for samples RESILEX2_1 to RESILEX2_4 is ~2,1 ppmw. For sample RESILEX2_5 the phosphorus concentration is two times higher: 5.8 ppmw. Compared to the previous silicon ingot the phosphorus concentration has been reduced by a factor of about 1.4
- Except for sample RESILEX2_5, the concentrations of metallic elements are very low, which is a significant improvement compare to the previous silicon ingot.

	RESILEX2_1	RESILEX2_2	RESILEX2_3	RESILEX2_4	RESILEX2_5
	Top				Bottom
	[ppm wt]	[ppm wt]	[ppm wt]	[ppm wt]	[ppm wt]
B	1,5	1,7	1,8	2,1	2,7
Mg	< 0.005	< 0.005	< 0.005	< 0.005	< 0.005
Al	0,24	0,24	0,26	0,28	15
Si	~99.9996%	~99.9996%	~99.9995%	~99.9994%	~99.91%
P	1,6	1,9	2,2	2,8	5,8
S	0,02	0,01	0,01	0,02	0,02
Ca	< 0.05	< 0.05	< 0.05	< 0.05	28
Ti	< 0.005	< 0.005	< 0.005	< 0.005	56
V	< 0.001	< 0.001	< 0.001	< 0.001	16
Cr	< 0.005	< 0.005	< 0.005	< 0.005	16
Mn	< 0.005	< 0.005	< 0.005	< 0.005	130
Fe	< 0.05	< 0.05	< 0.05	< 0.05	580
Co	< 0.005	< 0.005	< 0.005	< 0.005	0,38
Ni	< 0.01	< 0.01	< 0.01	0,04	18
Cu	0,05	0,06	0,05	0,08	12
Zn	0,07	0,04	0,06	0,05	0,07
Ga	0,05	0,05	0,09	0,13	7,4
Ge	0,12	0,07	0,11	0,07	0,23
As	0,03	0,04	0,03	0,05	0,13
Se	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Ag	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
In	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Sn	0,11	0,14	0,11	0,14	0,27
Sb	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Pt	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05
Au	< 0.01	< 0.01	< 0.01	< 0.01	< 0.01
Pb	0,02	0,02	0,01	0,02	0,01

Figure 17: Recycled silicon* ingot from crystal pulling - GDMS measurements for the five sample

From the results of GDMS measurements, a resistivity simulation of the Cz ingot that could be grown using this material was performed using boron, aluminum and phosphorus average concentrations from the four samples RESILEX2_1 to RESILEX2_4. The simulation takes into account a 10% blending.

Simulation parameters:

- Silicon load : 30kg
 - 10% of recycled silicon and 90% of EG silicon
 - Boron concentration: 0.18 ppmw
 - Phosphorus concentration :0.21 ppmw

- Additional concentration of phosphorus added in the silicon charge to aim for a resistivity of 1.6 Ω .cm at the top of the Cz ingot: 1.15 ppmw.

Figure 18 shows the resistivity profile along the ingot height under these conditions. It can be observed that:

- the resistivity is in the range of 1.6 Ω .cm to 0.2 Ω .cm at 70% of solid fraction,
- the whole ingot is n-type.

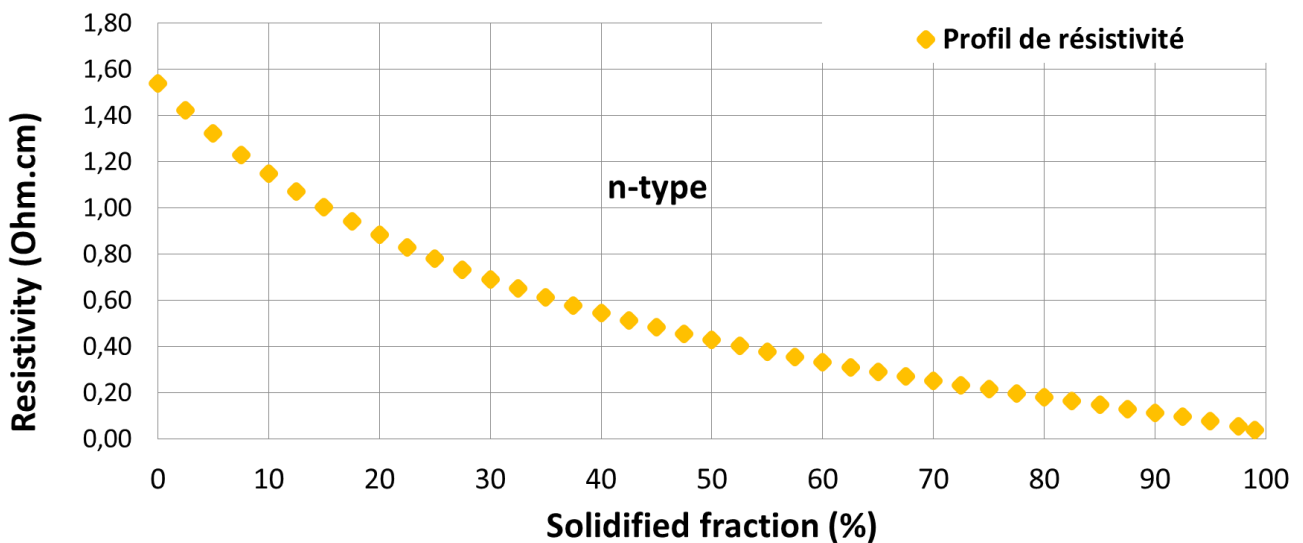


Figure 18: Resistivity profile simulation in case of blending - 10% of recycled silicon from crystal pulling with 90% of EG silicon

These results appear to be compatible with the Cz ingot for solar photovoltaic applications. In addition, the low concentration in metallic impurities of this recycled silicon (5N) allows CEA to use this material in its Cz pulling furnace. Thus, CEA will attempt, in the first quarter of 2025, to manufacture a Cz ingot from this recycled silicon under the following conditions: 10% of NTNU second recycled ingot and 90% EG silicon.

Conclusion

From the purified silicon produced by NTNU, CEA assessed the suitability of the alternative feedstock for the production of solar cells for two types of recycled silicon: one after additional directional crystallization and the other after Czochralski growth process.

From the additional directional crystallization, GDMS measurements on five samples along the ingot height show that the recycled silicon reached a level of purity between

2N to 4N. Based on these results, CEA carried out a resistivity simulation to verify the suitability of this silicon for photovoltaic applications.

Two simulation were done:

- Simulation1: 100% of the recycled silicon is used
- Simulation 2: Blending of 1.8% of recycled silicon with 98.2% of EG silicon

Simulation2 results appear to be compatible with Cz ingot for solar PV applications. CEA pulled a Cz ingot under blending condition (1.8% of recycled silicon with 98.2% of EG silicon) while retaining only the boron and phosphorus concentrations of the recycled silicon. From this Cz ingot, 700 M2 size wafers were prepared for cell processing.

From the additional Czochralski growth process, GDMS measurements on five samples along the ingot height show that the recycled silicon reached a level of purity of 3N to 5N. According to these results, CEA will attempt in the first quarter of 2025, to manufacture a Cz ingot from this recycled silicon under the following conditions: 10% recycled silicon after additional Czochralski growth process and 90% EG silicon.



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