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(54) Title: METHOD FOR RECOVERING ELEMENTAL SILICON FROM CUTTING REMAINS

(57) Abstract: This invention relates to a method for recovering elemental silicon cutting remains containing silicon particles, wherein the method comprises manufacturing solid anodes from the cutting remains, arranging one or more manufactured anode(s) in an electrolytic cell with an electrolyte and one or more cathode(s), and applying a potential difference between the one or more anode(s) and cathode(s) to obtain an oxidation of metallic silicon in the one or more anode(s), transportation of dissolved silicon in the electrolyte, and reduction of the dissolved silicon to a metallic phase at the one or more cathode(s).



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Method for recovering elemental silicon from cutting remains

This invention relates to a method for recovering elemental silicon cutting remains containing silicon particles. The invention may be employed for salvaging high-purity silicon suitable as a feedstock for production of photovoltaic cells from residues from production of photovoltaic wafers.

Background

Photovoltaic (PV) technology is emerging as a major source of clean electrical energy. The most common base material for photovoltaic cells is silicon, in the vast majority of cases produced either as a multicrystalline ingot by a directional solidification technique, or as a single crystal by the Czochralski process.

In the multicrystalline process, a large ingot is first cut into smaller blocks. The amount of material removed in block cutting may be up to several per cent of the amount later processed into solar cells.

The block cutting process can be carried out with conventional equipment, such as diamond band saws. The ingot cutting remains will be in the form of a relatively coarse, wet powder contaminated by a small amount of metal and abrasive particles from the cutting process. This material is presently discarded.

Alternatively, the block cutting can be done using a multiple wire saw. In the wire cutting process, a steel wire running over guide spindles is placed under tension and pushed onto the silicon block while an abrasive slurry comprising abrasive particles (about 10 – 20 μm in diameter) in a cutting fluid, is fed to the cutting zone between the wire web and the block. The abrasive material is most commonly silicon carbide, and the cutting fluid is normally either polyethylene glycol or oil. This process can be referred to as free abrasive wire cutting. The material removed from the cutting remains is presently discarded.

The cutting of wafers is done by multiple wire cutting. In the state of the art of wafer cutting processes, the wafer may be 160 – 240 μm thick, and the width of the kerf may be 180 – 220 μm . Thus, an amount of silicon comparable to the amount processed into solar cells is transported away by the abrasive slurry. The used slurry will contain polyethylene glycol or other cutting liquid, silicon carbide or other abrasive material, silicon, and metal from the steel wire. The cutting liquid and the coarse portion of the abrasive particles are usually reclaimed by various recycling processes, such as the process disclosed in US 6,113,473 or the one disclosed in US 6,231,628. At present, the slurry residue containing abrasive fines, silicon, metal from the cutting wire and residual cutting liquid is discarded or sold as a low grade material.

In another process variant, the steel wire is diamond impregnated, and the liquid fed to the cutting zone is acting only as a coolant. This process can be referred to as fixed abrasive wire cutting. The cutting remains then consists of silicon particles and a small amount of metal and abrasive suspended in the cooling liquid. This residual material is presently discarded or sold as a low grade material.

Monocrystalline silicon is squared and cut to wafers. The cutting is usually done with free abrasive multiple wire saws, although fixed abrasive wire cutting or other cutting techniques may be used. The cutting remains are presently discarded or sold as a low grade material.

Silicon feed stock for photovoltaics is a high cost material, and a process that can salvage the silicon content of the residual material as a useful feedstock for photovoltaic applications, from either or all of the above sources, is in great demand.

Prior art

The need for a recycling process has been recognized for at least ten years (see, e.g., Tsuo et al. [1]) but no commercially or technically viable process has so far been developed.

US Patent 6,780,665 discloses a method of producing thin film solar cells from silicon recovered from cutting remains. The silicon is separated from wire saw slurry by conventional separation techniques such as centrifugation, decanting or filtration, followed by froth flotation or electrostatic precipitation. No examples were given, and it is unlikely that the required purity of silicon was obtained.

Production of silicon by molten salt electrolysis has a long history, as reviewed by Elwell and Rao [2]. However, no electrolytic silicon production process has found commercial application.

WO 02/099166 discloses a process wherein solar grade silicon is obtained by electrolysis of silica in an electrolyte of CaO and CaCl₂ at about 800 °C. One problem with this process is that elements such as boron and phosphorous deposit at a similar potential to silicon. Very pure and costly silica sources therefore have to be used.

Electrolytic refining in order to upgrade metallurgical silicon to electronic grade was disclosed in US 3,254,010. In this process current is passed through a molten salt electrolyte containing a fluoride, from a cathode to an anode consisting of impure silicon or germanium, or an alloy of these elements with nobler metals. The deposited silicon is at least 99.9 % pure. The electrolyte is a fluoride from the class consisting of alkali metal fluorides and alkaline earth metal fluorides, with up to

about 10 % of an oxide of the metal being refined. It was stated that silicon of 99.99 % purity was produced, but no chemical analysis was reported.

5 Sharma and Mukherjee [3] describe electrolytic refining of silicon in using an electrolyte of $\text{KF-LiF-K}_2\text{SiF}_6$. The process was carried in equimolar mixture of KF and LiF with 6 – 18 mol% K_2SiF_6 at temperatures between 650 and 800 °C. At cathodic current density 0.135 A/cm^2 , 10 mol% K_2SiF_6 and 750 °C, a current efficiency of 92 % was obtained. Evaporation loss of SiF_4 was noticeable at higher temperature. The silicon was upgraded from 97.5 % to 99.99 % purity, but the residual amount of boron was 6 ppm. This is too high for conventional silicon solar cells.

Objective of the invention

The main objective of the invention is to provide a method for recovering elemental silicon from cutting remains comprising particulate elemental silicon.

15 A further objective of the invention is to provide a method for recovering photovoltaic quality silicon content for use as a feedstock for photovoltaic applications from cutting remains from cutting processes in solar wafer production.

The objectives of the invention may be realised by the features as set forth in the description of the invention below, and/or in the appended patent claims.

Description of the invention

20 The invention is based on the realisation that the usual metal contaminants in cutting remains are nobler than silicon, such that it becomes possible to obtain an element specific transportation of silicon from the cutting remains by use of an electrochemically induced oxidation of elementary silicon in the cutting remains, transportation of the oxidised silicon in an electrolyte, and reduction of the oxidised silicon to elementary silicon in the form of a metallic phase at a site in a distance of 25 the cutting remains.

Thus in a first aspect, the invention relates to a method for recovering elemental silicon from cutting remains, which comprises:

- manufacturing solid anodes from the cutting remains,
- 30 - arranging one or more manufactured anode(s) in an electrolytic cell with an electrolyte and one or more cathode(s), and
- applying a potential difference between the one or more anode(s) and cathode(s) to obtain an oxidation of metallic silicon in the one or more anode(s), transportation of dissolved silicon in the electrolyte, and reduction of the dissolved silicon to a 35 metallic phase at the one or more cathode(s).

The electrolytic process according to the first aspect will be effective in removing elements more noble than silicon, as they will not be oxidized at the anode. These elements thus remain as solids in the anode.

5 The electrolytic refining process may optionally be followed by a directional solidification step. Common metal impurities are >1000 times more soluble in the melt than the solid, and a directional solidification effectively concentrates such impurities in the part of the material that solidifies last. This part of the material can then be discarded.

10 By the term "cutting remains" as used herein, we mean any solid fraction of the residual materials from cutting or sawing of elemental silicon and which contains solid particles of elemental silicon/saw dust. This solid fraction will typically be residual material from squaring, block cutting or wafer cutting, or from a mixture of materials from these sources, but may also be a solid fraction from other sawing/-cutting processes of elemental silicon. Any liquid remains, such as cutting fluid etc.
15 are thus expected to be separated as far as practically possible from the solid fraction of the residual material. The solid fraction of the residual materials, i.e. the cutting remains, will typically comprise particulate silicon contaminated by abrasive particles from the cutting process and a small amount of metal from the saw.

20 The cutting remains may advantageously be densified to increase the electric conductivity when being formed to the anode. Densification techniques known from the fields of ceramic and powder metallurgical technologies may be employed, including slip casting, uniaxial and isostatic pressing, injection moulding, tape casting, etc. In general it is advantageous to add certain processing aids in order to form the green body by the desired processing technique, although process variants
25 where no such additives are used fall within the scope of the invention. These processing aids may range from small additions of deflocculants in the case of slip casting, to substantial additions of waxes and binders in the case of injection moulding. Optionally, processing aids that promote sintering may be added to the cutting remains prior to green body formation.

30 One example of an anode forming process is manufacturing the anodes by mixing cutting remains with a suitable binder, drying the mixture and dry pressing the anode precursor powder. Polyvinyl alcohol, other water soluble polymers, or latex are suitable binder materials. The mixture may be dried with conventional drying equipment. Spray drying is particularly advantageous. A pressure in the range from
35 75 MPa to 250 MPa is suitable for densification of the anodes. The pressure may be applied with a conventional hydraulic press, or by an isostatic press.

Another example of an anode forming process is slip casting. The cutting remains are mixed with water, to form a pourable slip with high solids loading. Optionally a deflocculant, such as for instance 2-amino-2-methylpropanol, may be added in order

to promote higher solids loading of the slip. The anode is formed by casting the slip in moulds made from a suitable material, such as plaster.

Once the green body has been formed by an appropriate technique, and dried if necessary, it is normally subjected to a heating cycle in air to remove the organic processing aids which were added to assist forming. This burn-out is generally performed by heating the green body carefully to a temperature in the range 300 - 400 °C.

After burn-out, the green body is consolidated (sintered) by heating in a non-oxidizing (i.e. vacuum or a noble gas) atmosphere to form the anode. The choice of consolidation temperature depends on the type of sintering aids added and the required degree of open porosity in the anode. Generally, temperatures higher than about 800 °C are necessary. With anodes manufactured from cutting remains with no sintering aids added, sintering temperatures in the range 1300 – 1450 °C and soak times at the sintering temperature of 0.5 to 24 hours have been found suitable.

Another way of obtaining increased density and electric conductivity of the anode is adding a metal more noble than silicon to the cutting remains. Suitable metals comprise Cr, Fe, Co, Ni, and Cu. In this case, the anodes may be sintered at a temperature close to the eutectic temperature of the system Si-M. A temperature within 100 °C of the eutectic temperature of the system has been found particularly suitable. For instance, if copper is used the optimal sintering temperature will be around 800 °C.

The concentration of silicon in the cutting remains may advantageously be increased in order to obtain anodes with increased silicon content, leading to increased productivity and reduced energy consumption of the process. The increased concentration of silicon may be obtained by removing a fraction of the abrasive particles in the cutting remains. For example by conventional separation processes for removal of abrasive particles in the cutting remains, such as those presently employed commercially for recycling of abrasive particles and cutting fluid, by optimizing said processes for a combination of high yield and high concentration of silicon in the residual material. Other known separation techniques, such as froth flotation, may be employed. Another method for increased silicon concentration in the cutting remains from free abrasive wafer cutting is mixing the material with cutting remains from other cutting processes having larger silicon contents than the cutting remains from free abrasive wafer cutting.

Anodes may alternatively be fabricated by a casting process wherein the cutting remains, having been subjected to additional processing steps to increase the volume fraction of silicon above about 70 %, is heated in an inert atmosphere directly to a temperature which is sufficient to cause melting of the metallic components of the processed material. The melting is either carried out directly in a

mould suitable for producing anode blanks, or it is performed in a crucible and the melt is subsequently transferred to a casting mould. The temperature is then decreased, causing the melt to solidify. After solidification the cast shape is subjected to a controlled cooling cycle to minimize the level of thermal stresses.

- 5 Sintered or cast anodes may optionally be machined to their final shape by grinding or milling.

The cathode may be manufactured from any material that is electrically conductive, resistant against the chemical environment of the cell, easily separated from the deposited silicon, and which has a low diffusion rate in silicon. The cathode may
10 advantageously be made of solar grade silicon, but other suited materials comprise high purity carbonaceous material, such as carbon, graphite or vitreous carbon, or transition or noble metals.

The electrolyte must be able to dissolve oxidized silicon as well as possessing high ionic conductivity. The metal constituents of the electrolyte must be significantly
15 less noble than silicon in order to avoid reduction at the cathode, as well as even in low concentrations dissolved in silicon not negatively affecting the properties of silicon relevant to solar efficiency. Suitable candidates for such electrolytes would be alkali metal halides, alkaline earth metal halides or mixtures of such. The halides should preferably be chloride, fluoride or mixtures.

20 The composition may advantageously be a mixture of an alkali metal fluoride selected from the group LiF, NaF and KF in a concentration of 10 – 90 mol% and an alkaline earth metal fluoride selected from the group CaF₂, SrF₂ and BaF₂, in a concentration of 10 – 90 mol%. Mixtures of several alkali metal fluorides and/or several alkaline earth metal fluorides may also be used. Addition of BaF₂, SrF₂, or a
25 combination of these has been found particularly effective in reducing volatilization. Optionally, K₂SiF₆ can be added in amounts up to 20 mol%. The applicants have found that avoiding oxide in the electrolyte is important in order to limit the electrical resistance of the cell, probably due to formation of electrically insulating layers on one or both electrode surfaces.

- 30 The total cell reaction for electrolytic silicon refining is:



The reversible cell voltage is thus 0 V. The cell voltage required for operating the process is that required to overcome resistance in circuitry, electrolyte and the electrodes, including overpotential at the cathode and anode. The optimum cell
35 voltage is thus a function of the exact features of the cell design, amongst other factors, the anode-cathode distance and the composition of electrodes and electrolyte. By the term “overpotential” we mean the potential difference between

the working electrode, i.e. the anode or the cathode, and a reference electrode placed in the immediate vicinity of the working electrode.

The most prevalent impurity element in the cutting remains is iron, but small amounts of other transition metals, such as chromium, nickel and copper, will normally also be present. Iron has 1.02 V higher standard reduction potential than silicon. It is estimated that the ratio of the thermodynamic activity of iron(III)fluoride to iron at the anode remains lower than 10^{-9} up to an anodic overpotential of about 350 mV. The other transition metals have standard reduction potentials in a similar range, and dissolution of these metals into the electrolyte can thus be avoided by operating the electrolytic refining process in such a way that the anodic overpotential is lower than about 300 mV.

A category of impurities that may be found in the anode are elements which are less noble than silicon, such as alkaline earth metals and alkali metals. These will dissolve into the electrolyte, but will not readily be reduced at the cathode. For instance, it is estimated that the ratio of the thermodynamic activity of calcium fluoride to calcium at the cathode will exceed 10^9 up to a cathodic overpotential of more than 550 mV, so co-deposition can therefore be avoided by operating the refining process with cathodic overpotential less than about 500 mV. Less noble elements will, however, accumulate in the electrolyte as fluorides, and may in some cases build up to unacceptable levels. If a deleterious impurity is accumulating to an unacceptable level, the electrolyte may be partly or wholly replaced with fresh electrolyte.

Elements with a reduction potential that is similar to silicon cannot be refined by electrolytic techniques. Particularly relevant examples of this category of impurities are boron and phosphorous. These elements additionally have the disadvantage of a relatively small difference between solid and liquid solubility in silicon. In commercial metallurgical grade silicon and upgraded metallurgical silicon, the concentration of these elements is too great to obtain high quality solar grade silicon by electro-refining including subsequent directional solidification. Use of cutting remains from cutting of high purity silicon as a raw material overcomes this shortcoming, because of the inherent purity of the silicon in the cutting remains.

By appropriate adjustment of the process parameters, it is possible to obtain the refined silicon as a coherent deposit on the cathode. The morphology is a function of cathodic current density, but is also influenced by other process parameters such as electrolyte composition and temperature. Using higher cathodic current density than about $0.05 - 0.20 \text{ A/cm}^2$, a granular product is usually obtained. The granular product may be separated from residual electrolyte by known techniques, such as washing with aqueous AlCl_3 -solution. Both deposit morphologies are within the scope of the invention.

The electrolytic cell is operated at a temperature above the liquidus temperature of the electrolyte used. In order to minimize the volatilization of silicon species, it is advantageous to operate the cell at temperatures $< 50\text{ }^{\circ}\text{C}$ above the liquidus temperature. Depending on the electrolyte chosen, the cell temperature may be in the range $500 - 1200\text{ }^{\circ}\text{C}$.

The vessel containing the molten electrolyte may be made from a range of known materials that are resistant to the chemical environment of the process. Suitable materials include silicon oxide, silicon nitride, silicon carbide, carbon, graphite and composites thereof.

The residue in the anode compartment after the refining process (oxidation of metallic silicon) may typically contain silicon carbide, silicon oxide and metals more noble than silicon.

List of figures

Figure 1 is a linear voltammogram plotted on a Si electrode (sweep rate= 10 mV/s) in a $\text{NaF-BaF}_2\text{-NaSiF}_6$ (28-68-4 mass%) mixture at $900\text{ }^{\circ}\text{C}$.

Figure 2 shows a SEM micrograph of the cathode from test 2, with element analysis.

Figure 3 shows a photograph of obtained silicon in test 2.

Figure 4 is an x-ray diffractogram of the obtained silicon shown in figure 2.

Verification of the invention

The invention will be described in further detail by way of verification examples. These examples should not be considered constituting a limitation of the general idea of using electro-refining to extract elemental silicon from cutting remains.

Example of manufacturing of anode from cutting remains

Used wire cutting slurry was treated by a commercial process to remove and recycle the coarse fraction of silicon carbide and the majority of the polyethylene glycol. Approximately 365 g of the dried residue from this process was added to 300 ml of 1 % aqueous solution of the dispersant Dolapix A88 (2-amino-2-methylpropanol) and dispersed using an Ultra-Turrax disperser. The resulting slip, about 55 % solids by weight, was rolled overnight on a jar roller mill with a few milling balls. The slip was cast onto plaster moulds to a depth of 15-20 mm. After drying the anodes were sintered at $1415\text{ }^{\circ}\text{C}$ for 2 hours. The resulting anodes were machined to the desired size. The geometric density was 1.55 g/cm^3 .

Example of electro-refining silicon

Electro-refining tests were carried out in order to demonstrate the possibility of producing pure elemental silicon. During these tests, silicon was dissolved electrochemically at the anodes, and electrodeposited at the cathode. The electrolyte was a molten salt in which silicon ions were dissolved before the beginning of the electrolysis.

The molten salt was contained in a vitreous carbon crucible, placed in a graphite liner protecting the inside wall of a cylindrical vessel made of refractory steel. The cell was closed by a stainless steel lid cooled by circulating water. The atmosphere was U-grade (less than 5 ppm O₂) inert argon. Two anodes made of solar-grade silicon with a surface area of about 5 cm² were placed around the cathode. Two Si electro-refining tests were conducted in two different molten salts. The specific conditions applied in each tests are listed in Table 1.

Table 1: Tests conditions

Test	Salt composition	Cathode	Cathode surface [cm ²]	Temperature [°C]	Duration [h]	Cathode current density [mA/cm ²]
1	NaF-LiF (37-63 mass%)	Vitreous carbon	2	800	5	75
2	NaF-BaF ₂ (29-71 mass%)	Silicon	5	900	24	16

15

An electrochemical technique (linear voltammetry) was performed on a silicon electrode before the beginning of run 2. Reference potentials were measured against a silicon electrode. The linear current-voltage response of the voltammogram in Figure 1 shows that there is no limitation either for the dissolution of the anode or for the deposition of silicon at the cathode for a current density up to 60 mA/cm².

20

The runs consisted in applying a constant current between the anodes and the cathode. At the end of the run, the cathode was removed from the cell in order to recover the deposited silicon. The cell voltage was found constant during the runs and close to 100 mV.

25

The deposit partly consisted of a coherent layer on the cathode substrate and partly of silicon in granular form. The deposit was washed in an aqueous AlCl₃ solution for 24 hours at room temperature, in order to dissolve the adhering salt. The

solution was finally filtered and dried. A SEM micrograph of the cathode from test 2 is shown in figure 2. A coherent deposit of silicon can be seen. An EDS-analysis of the deposited silicon shows only silicon and carbon, which is an artefact due to contamination by e.g. oil residues in the vacuum chamber of the microscope. The powder is shown in figure 3. An X-Ray diffractogram obtained from test 1 is shown in figure 4: No impurity was found in the silicon apart from oxide formed after separation from the electrolytes.

The current efficiency of the process was calculated according to the ratio between the mass of silicon recovered after washing and the theoretical mass that should be obtained for a given charge passed (calculated according to Faraday's law). Table 2 shows that in test 1 and test 2, the current efficiency is about 70%.

Table 2: Current efficiency

Test	Theoretical Si mass [mg]	Recovered Si mass [mg]	Current efficiency [%]
1	270	196	72.5
2	496	340	68.5

15

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1. Y. S. Tsuo, J. M. Gee, P. Menna, D. S. Strebkov, A. Pinov, and V. Zadde, "Environmentally benign silicon solar cell manufacturing," presented at 2nd World Conference and Exhibition on Photovoltaic Solar Energy Conversion; 6-10 July 1998; Vienna, Austria
2. D. Elwell and G. M. Rao, "Electrolytic production of Silicon," *J. Appl. Electrochem.*, 18 (1988) 15-22
3. Sharma and Mukherjee, "A Study on Purification of Metallurgical Grade Silicon by Molten Salt Electrorefining", *Metallurgical Transactions*, Vol. 17B, 1986, 395 - 397.

CLAIMS

1. Method for recovering elemental silicon from cutting remains, wherein the method comprises:
 - manufacturing solid anodes from the cutting remains,
 - 5 - arranging one or more manufactured anode(s) in an electrolytic cell with an electrolyte and one or more cathode(s), and
 - applying a potential difference between the one or more anode(s) and cathode(s) to obtain an oxidation of metallic silicon in the one or more anode(s), transportation of dissolved silicon in the electrolyte, and reduction
 - 10 of the dissolved silicon to a metallic phase at the one or more cathode(s).
2. Method according to claim 1,
wherein the applied potential is regulated to give a cathode overpotential of less than 500 mV and an anode overpotential of less than 350 mV.
3. Method according to claim 1 or 2,
15 wherein the anode is formed from cutting remains densified by:
 - mixing the cutting remains with a binder,
 - drying the mixture,
 - dry pressing the mixture, and
 - sintering the pressed mixture by heating it to a temperature above 800 °C in
 - 20 a non-oxidising atmosphere.
4. Method according to claim 1 or 2,
wherein the anode is formed from cutting remains densified by:
 - forming a slip by suspending the cutting remains in water with an optional deflocculant,
 - 25 - casting the slip in a mould made of plaster or another suitable material, and
 - sintering the slip by heating it to a temperature above 800 °C in a non-oxidizing atmosphere.
5. Method according to claim 3 or 4
wherein
30 -the formed anode is sintered by heating it to a temperature in the range 1300 to 1450 °C, and
- the time at the sintering temperature is between 0.5 and 24 hours.
6. Method according to claim 3 or 4,
wherein
35 - one or more of the following particulate metals; Cr, Fe, Co, Ni or Cu is added to and mixed with the cutting remains, and
- the sintering temperature is within 100 °C above or below the eutectic temperature of the system silicon and added metal(s).

7. Method according to any of claims 3 to 6,
wherein at least a fraction of the abrasive particles in the cutting remains are removed before the cutting remains are formed into the anode.
- 5 8. Method according to claim 1 or 2,
wherein the cathode is made of one of solar grade silicon, high purity carbonaceous material, such as carbon, graphite or vitreous carbon, or transition or noble metals.
- 10 9. Method according to claim 1 or 2,
wherein the electrolyte is made of one or more of alkali metal halides and alkaline earth metal halides.
10. Method according to claim 9,
wherein the halides are one or more of chlorides and/or fluorides
11. Method according to claim 9,
wherein the halides comprises:
15 - a mixture of an alkali metal fluoride selected from the group LiF, NaF and KF in a concentration of 10 – 90 mol%, and
- an alkaline earth metal fluoride selected from the group CaF₂, SrF₂ and BaF₂, in a concentration of 10 – 90 mol%, optionally
- with addition of up to 20 mol% K₂SiF₆.
- 20 12. Method according to claim 1 or 2,
wherein the electrolytic cell is operated at a temperature above the liquidus temperature of the electrolyte used, but at less than 50 °C above the liquidus temperature.
- 25 13. Method according to any of the preceding claims,
wherein the vessel containing the electrolytic cell is made of silicon nitride, silicon carbide, carbon, graphite or composites thereof.
14. Method according to any of the preceding claims,
wherein the method also comprises a directional solidification step followed by discharging of the part of the material that solidifies last.

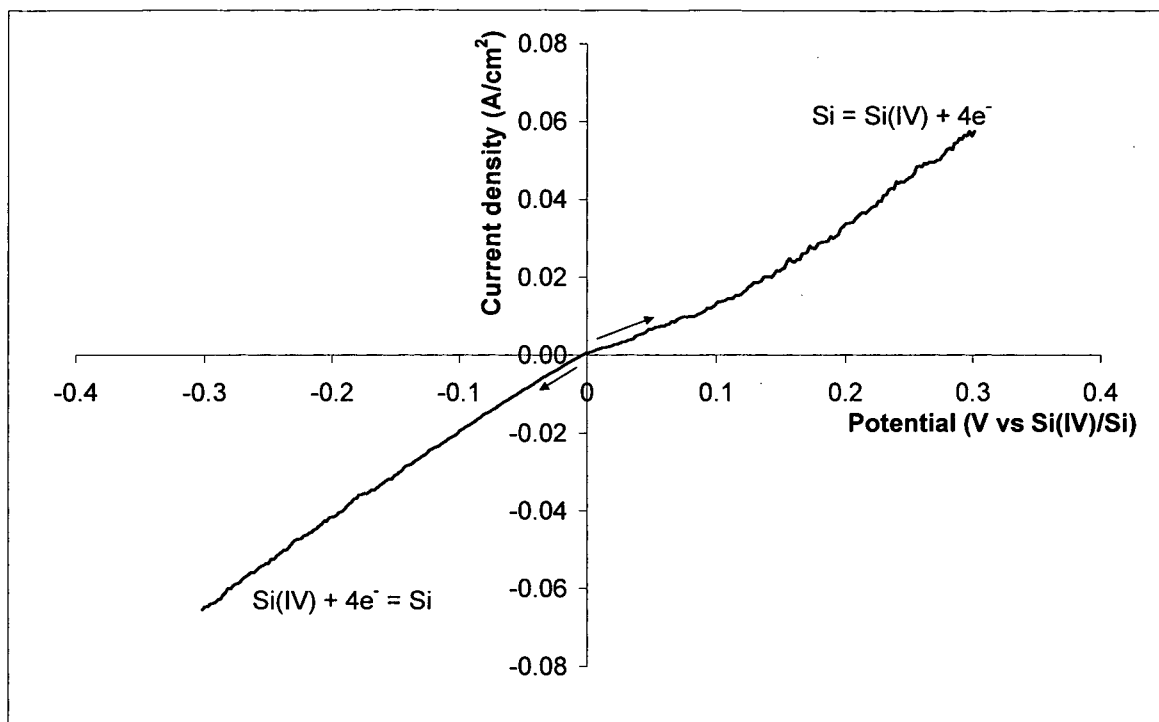


Figure 1 Linear voltammogram plotted on a Si electrode (sweep rate=10 mV/s) in a NaF-BaF₂-NaSiF₆ (28-68-4 mass%) mixture at 900 °C.

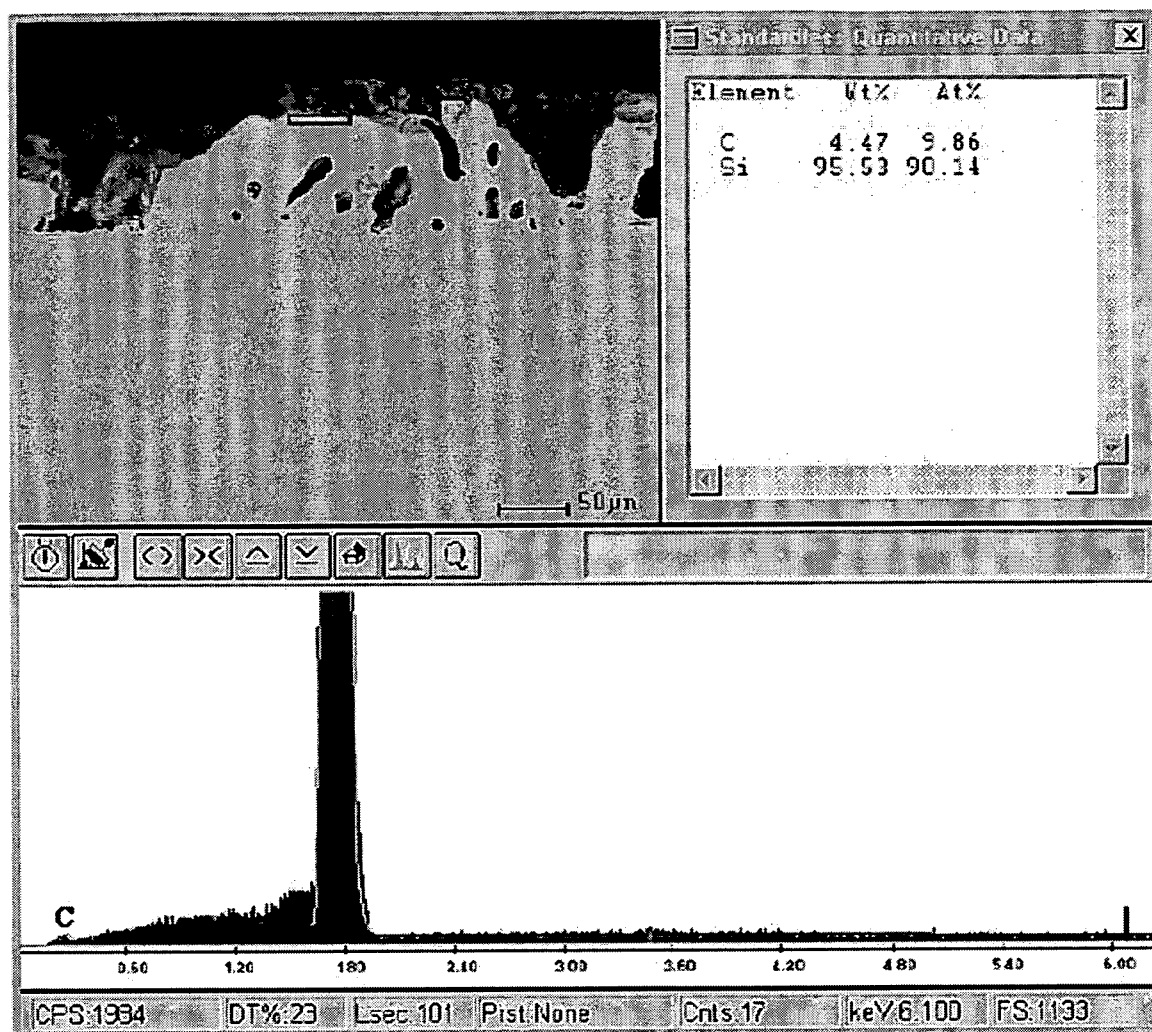


Figure 2 SEM micrograph of the cathode from test 2, with EDS element analysis.

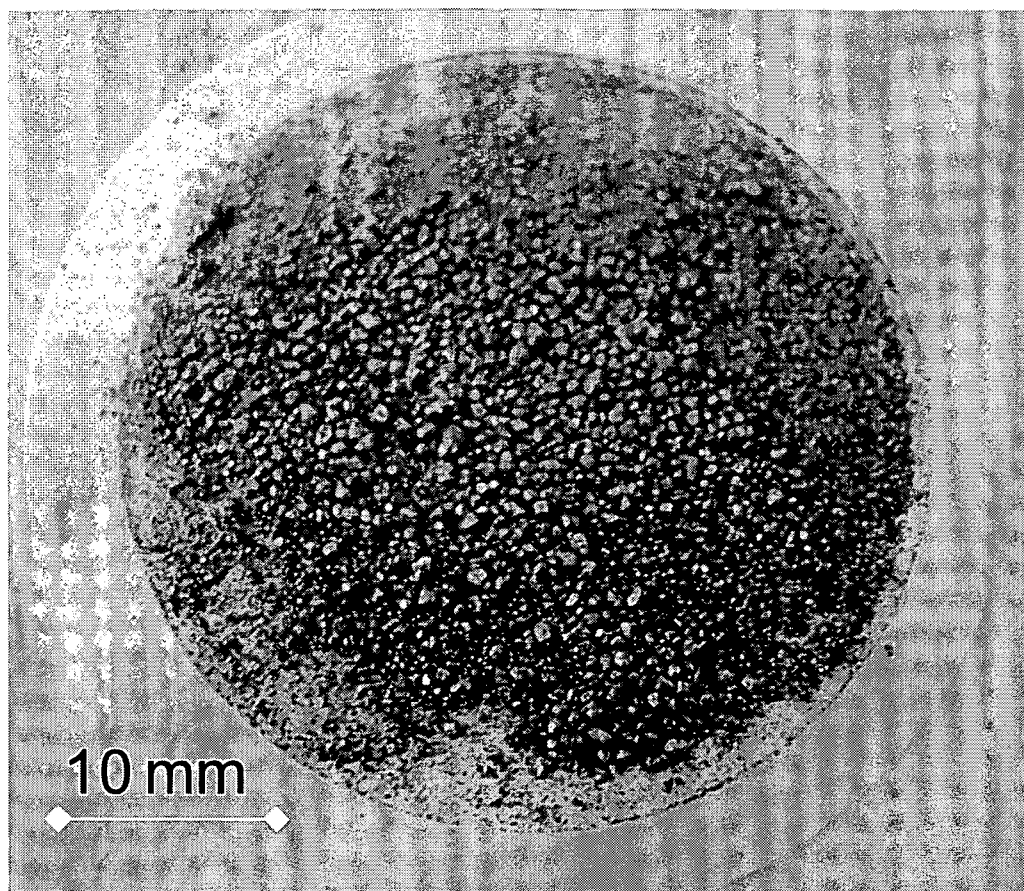


Figure 3: Picture of obtained silicon (test 2)

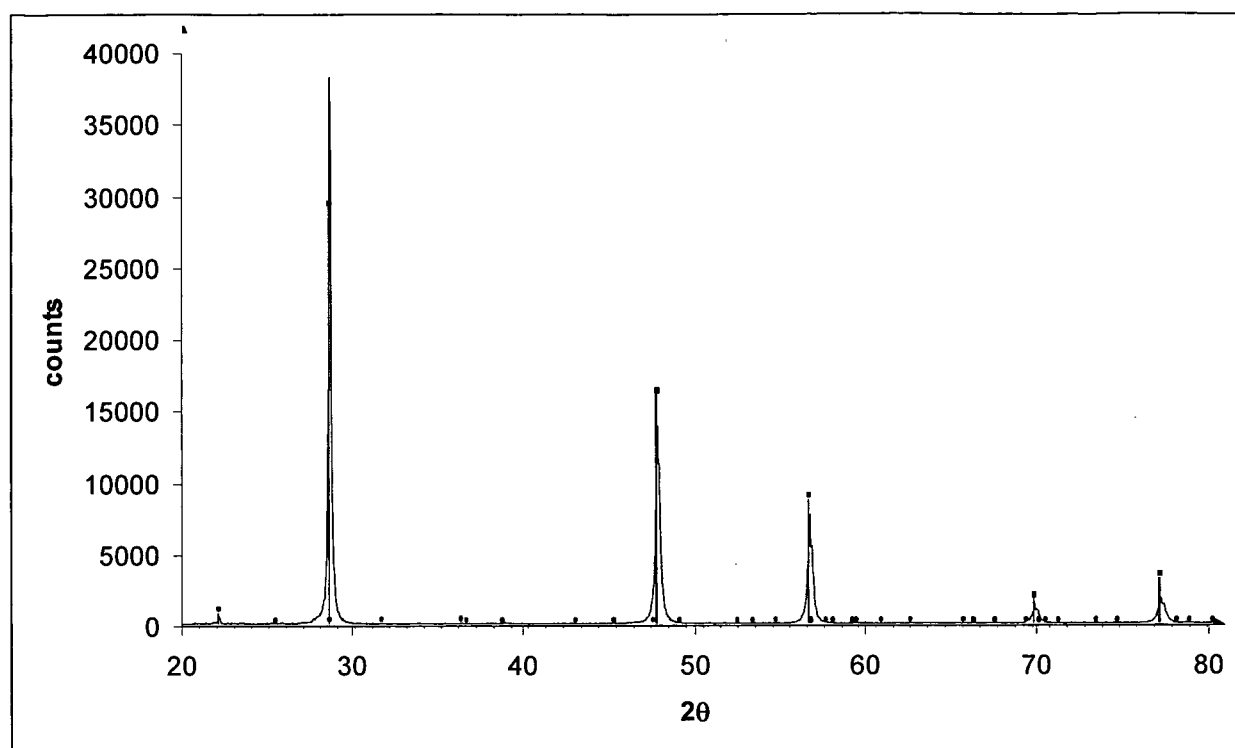


Figure 4: X-Ray diffractogram of the obtained silicon