

METHOD AND SYSTEM FOR CLEANING CONTAMINATED SILICON CARBIDE PARTICLES

Technical Field

[0001] The invention relates to a method and a system for cleaning contaminated silicon carbide (SiC) particles, and in particular, the contaminated SiC particles are cleaned by removing fine grain particles adhering to the contaminated SiC particles after being used in suspension in a cutting medium for the cutting or sawing of silicon wafers for solar cells and electronic objects often called spent sawing sludge.

Background

[0002] When sawing thin silicon discs, commonly referred to as "wafers", particles of silicon carbide (SiC) of specific grit sizes, such as FEPA classes F500, F600, and F800, are dispersed in an organic liquid, thus forming a suspension, which is used as a cutting medium. The most common dispersing agents are organic glycolic liquids such as polyethylene glycol or di-propylene glycol. Sometimes surfactants that reduce surface tensions are added to the suspension.

[0003] The sawing is usually conducted by a wire saw to which a thin, hardened iron wire with brass on the surface, cuts the silicon (Si) block into a series of thin wafers while the particles from the sawing are suspended in the SiC containing suspension. During the sawing process, the suspension becomes contaminated with Si from the Si block, iron (Fe) from the cutting wire, and SiC fines (i.e. fine particles) from the breaking down of abrasive grains.

[0004] Typically, the Si wafers are used for the manufacture of electronic or microelectronic devices, or for the manufacture of solar cell panels for the production of electric power. The

cleanliness requirement of these Si wafers is typically so high that, in practice, only Si free and Fe free suspensions have been used for the cutting. Furthermore, the requirement for particle size distribution is precisely specified in order to obtain smooth surfaces on the Si wafers. SiC particles for sawing lies within a narrow grain size range, i.e. that there is little or minimal difference between the size of the largest and the smallest grains.

[0005] After having been used for some time, the cutting suspension becomes so contaminated with SiC fines, Si and Fe particles that it has to be replaced by a new suspension. In addition, particle size distribution of SiC particles is shifted out of the desired narrow range. When disposed off in special land fills there is a risk of glycol leak into the environment. Another alternative is to burn the spent cutting suspension whereby the liquid phase is burnt along with the solid particles. While glycol is burned and reduced to carbon dioxide and water, the solid particles form a heavy metal containing ash that presents another environmental problem.

[0006] From a resource, cost and environmental point of view, it is thus desirable to develop processes by which SiC fines, Si and Fe particles may be removed and recovered from the suspension. Recycling of SiC particles also means a reduction in the total energy consumption in, for example, manufacturing solar cells processes. Recycling also means less environmental strain with respect to both the production of the SiC and with respect to the burning or deposition of the carbide containing suspension in special land fills due to a lack of cleaning processes.

[0007] An economically and feasible process implies that it should give high yields of the recovered components for reuse, and that the waste contaminated with Fe, Si and SiC fines should be reused as valuable resources e.g. for use in the production of iron and steel, ferro-alloys or refractory materials. This in turn implies that the recycling process should be designed in a manner

such that organic dispersing agents are recovered in high yields and reused for the sawing of Si wafers.

[0008] FEPA F500, F600 and F800 microgrits are commonly used in the sawing of Si wafers and it is important that the grains of SiC conform to the standard to obtain a good result. The conventional practice is to dilute the suspension of particles and polyethylene glycol or di-propylene glycol with large quantity of water and treat the suspension in settling vessels. In such process, it is desirable to make use of the fact that the SiC particles have larger diameters than the contaminants of SiC fines, Fe and Si.

[0009] The rigid requirement that the grain diameter be within narrow limits leads to low yields. The settling process therefore has to be interrupted before finer particles of SiC fines, Si and Fe reach the settled phase of SiC with desired grain size. The settled particles of SiC obtained in the settling vessel are dried and screened according to known methods. Glycol based dispersing agents, highly diluted with water, are required to increase the settling velocity, which makes an economical and ecological recovery process for the dispersing agent difficult.

[0010] In one study, in employing a direct settlement of particles from a standard SiC suspension, it took one year to settle 99 % of the finest particles. Separation according to this principle is thus not economically feasible in the industrial production of Si wafers, for example.

[0011] Therefore, there remains a need to provide for alternative methods and systems for cleaning contaminated SiC particles from a spent sawing sludge.

Summary

[0012] In a first aspect of the disclosure, there is provided a method for cleaning contaminated silicon carbide (SiC) particles by removing fine grain particles adhering to the contaminated SiC particles.

[0013] The method may include feeding the contaminated SiC particles to a jet mill to obtain a dispersed powder.

[0014] The method may further include feeding the dispersed powder to a classifier system to obtain the cleaned SiC particles. The classifier system may include more than one classifier.

[0015] In a second aspect of the disclosure, there is provided a system for cleaning contaminated silicon carbide (SiC) particles by removing fine grain particles adhering to the contaminated SiC particles.

[0016] The system may include a jet mill.

[0017] The system may further include a classifier system. The classifier system may include more than one classifier.

Brief Description of the Drawings

[0018] In the drawings, like reference characters generally refer to the same parts throughout the different views. The drawings are not necessarily drawn to scale, emphasis instead generally being placed upon illustrating the principles of various embodiments. In the following description, various embodiments of the invention are described with reference to the following drawings.

[0019] Fig. 1 shows a process flow of present method and system.

[0020] Fig. 2A shows the particle size distribution of the fine fraction from the first classifier of the system of Fig. 1.

[0021] Fig. 2B shows the particle size distribution of the fine fraction from the second classifier of the system of Fig. 1.

[0022] Fig. 2C shows the particle size distribution of the fine fraction from the third classifier of the system of Fig. 1.

[0023] Fig. 3A shows a SEM (1000x magnification) of new SiC particles before cutting of silicon wafer.

[0024] Fig. 3B shows a SEM (1000x magnification) of contaminated SiC particles after cutting of silicon wafer.

[0025] Fig. 3C shows a SEM (1000x magnification) of the cleaned SiC particles obtained by the present method.

[0026] Fig. 4A shows a SEM (2000x magnification) of new SiC particles before cutting of silicon wafer.

[0027] Fig. 4B shows a SEM (2000x magnification) of contaminated SiC particles after cutting of silicon wafer.

[0028] Fig. 4C shows a SEM (2000x magnification) of the cleaned SiC particles obtained by the present method.

Description

[0029] The following detailed description refers to the accompanying drawings that show, by way of illustration, specific details and embodiments in which the invention may be practised. These embodiments are described in sufficient detail to enable those skilled in the art to practise the invention. Other embodiments may be utilized and structural, logical, and electrical changes may be made without departing from the scope of the invention. The various embodiments are not

necessarily mutually exclusive, as some embodiments can be combined with one or more other embodiments to form new embodiments.

[0030] A method and a system for cleaning contaminated silicon carbide (SiC) particles that have been used in suspension in a cutting medium for the cutting or sawing of silicon wafers for solar cells and electronic objects often called spent sawing sludge are disclosed herein. The contaminated SiC particles are cleaned by removing fine grain particles adhering to the contaminated SiC particles after being used in the cutting medium suspension. Advantageously, the method allows recovery of SiC of a narrow grain size range by removing from the SiC particles smaller particles such as, but not limited to, iron (Fe), silicon (Si) and SiC fines. The method and system can be applied cost-effectively on an industrial scale and at the same time have minimal negative impacts on the environment.

[0031] In present context, recycled particles of SiC within the FEPA (Federation of European Producers of Abrasives) standards of microgrits are obtainable. FEPA is the international standard to which these kinds of materials have to comply. The relevant standard is FEPA standard 42-6B 1984, R 1993. (The same definition is incidentally defined by ISO 6344-3 1968, part 3: "Determination of grain size distribution of microgrits F230 to F1200").

[0032] By ordinary classification, the smallest particles will be present as individual grains that may be separated from the larger particles by a convenient choice of process parameters. With respect to these SiC particles, however, subsequent to the removal and recovery of the dispersing agent from the used suspension, the small particles will adhere to the larger, say F500, F600 or F800 SiC grains that are typically applied for sawing silicon wafers.

[0033] Thus, in a first aspect of the disclosure, there is provided a method for cleaning contaminated silicon carbide (SiC) particles by removing fine grain particles adhering to the

contaminated SiC particles. An outline process flow of the method and accordingly a system implementing the method is illustrated in Fig. 1.

[0034] Used SiC containing suspensions from the sawing of silicon wafers are filtered according to prior art technology to separate the solid stream from the liquid stream. The solid stream is heated under agitation so that the contaminated SiC particles assume the form of a dry powder. The liquid stream is separately recovered according to a known technology.

[0035] The dry contaminated SiC particles (10) including SiC, Si (silicon) and Fe (iron) particles are then fed to a jet mill (12) to obtain a dispersed powder. During this process, any agglomerated powder is grinded and dispersed by compressed air. The agglomerated powder is grinded without crushing the SiC grains. The dispersed powder then leaves the jet mill via a set of rotors.

[0036] In exemplary embodiments, the jet mill (12) is operated at a rotor speed of about 800 to about 1,200 rpm. For example, the rotor speed of the jet mill (12) may be set at 800 rpm, 900 rpm, 1,000 rpm, 1,100 rpm, or 1,200 rpm.

[0037] Proper dispersion of powder in the air is critical for good particle separation in the subsequent processing steps. In this respect, compressed air pressure, rotor speed and rotor gap opening may be manipulated to ensure good particle separation.

[0038] The dispersed powder is then fed to a classifier system to obtain the cleaned SiC particles. The classifier system may include more than one classifier.

[0039] Classifier is a form of centrifugal separation of the particles into coarse and fine fraction. In operation, the dispersed powder is fed to a classifier via the bottom and is carried towards a set of rotors at the top of the classifier. Fine powder passes through the rotors and exit the classifier via the top, while coarse powder leaves the classifier via the bottom. The fine fraction then passes

through an air filter (dust collection system) whereby the fine powder is collected. To achieve effective separation, a triple classifier system consisting of three classifiers (14), (16), (18) illustrated in Fig. 1 is operated. Each of these classifiers has its own air filter to collect the fine powder. It is to be understood that any other number of classifiers may likewise be used, such as 4, 5, 6, or more classifiers.

[0040] In various embodiments, the classifiers may be arranged such that the classifiers are connected in series. In other words, in the illustration shown in Fig. 1, a first classifier (14) is positioned upstream of a second classifier (16), which in turn is positioned upstream of a third classifier (18).

[0041] Each classifier (14), (16), (18) produces a coarse fraction and a fine fraction. In the case where the classifiers are connected in series, each coarse fraction of an upstream classifier is fed to a downstream classifier and each fine fraction of a respective classifier is separated out from the classifier system.

[0042] Thus, in the embodiment shown in Fig. 1, the coarse fraction from the first classifier (14) is fed to the second classifier (16). The coarse fraction from the second classifier (16) is fed to the third classifier (18). Fine fractions from the first classifier (14), the second classifier (16) and the third classifier (18) may be individually collected.

[0043] Alternatively, fine fractions from the first classifier (14), the second classifier (16) and the third classifier (18) may be collectively collected.

[0044] Fine fraction usually contain significant portion of coarse powder. By recycling one or more fine fractions from the classifier system back to the jet mill (12), the yield of the cleaned SiC particles obtained from the present method improves by at least 3 %, for example. Thus, in various

embodiments, at least one of the fine fractions is recycled back to the jet mill (12). As illustrated in Fig. 1, fine fraction from the third classifier (18) may be recycled back to the jet mill (12).

[0045] In other examples, fine fraction from the first classifier (14) or the second classifier (16) may be recycled back to the jet mill (12).

[0046] In certain other embodiments, fine fractions from the second classifier (16) and the third classifier (18) may be collectively collected and recycled back to the jet mill (12).

[0047] The fine fractions from the respective classifier (14), (16), (18) may be individually collected and separated out from the classifier system. The fine fractions may be sold as useful products. For example, fine fraction from the first classifier (14) is collected and sold under the trade name SiSiCar®50. Fine fraction from the second classifier (16) is collected and sold under the trade name SiSiCar®70.

[0048] The coarse fraction of the last classifier, i.e. the third classifier (18) of Fig. 1, contains the cleaned SiC particles.

[0049] In various embodiments, the first classifier (14) is operated at a rotor speed of about 2,100 to about 2,800 rpm. For example, the rotor speed may be set at 2,100 rpm, 2,200 rpm, 2,300 rpm, 2,400 rpm, 2,500 rpm, 2,600 rpm, 2,700 rpm, or 2,800 rpm.

[0050] Fig. 2A shows the particle size distribution of the feed (Sample 1), fine fraction (Sample 3) and coarse fraction (Sample 4) from the first classifier of the system of Fig. 1.

[0051] In various embodiments, the second classifier (16) is operated at a rotor speed of about 2,100 to about 2,800 rpm. For example, the rotor speed may be set at 2,100 rpm, 2,200 rpm, 2,300 rpm, 2,400 rpm, 2,500 rpm, 2,600 rpm, 2,700 rpm, or 2,800 rpm.

[0052] Fig. 2B shows the particle size distribution of the feed (Sample 4), fine fraction (Sample 5) and coarse fraction (Sample 6) from the second classifier of the system of Fig. 1.

[0053] In various embodiments, the third classifier (18) is operated at a rotor speed of about 2,100 to about 2,800 rpm. For example, the rotor speed may be set at 2,100 rpm, 2,200 rpm, 2,300 rpm, 2,400 rpm, 2,500 rpm, 2,600 rpm, 2,700 rpm, or 2,800 rpm.

[0054] Fig. 2C shows the particle size distribution of the feed (Sample 6), fine fraction (Sample 7) and coarse fraction (Sample 8) from the third classifier of the system of Fig. 1.

[0055] Other than the classifier rotor speed, rotor gap opening and air filter rotor speed are important parameters which may be manipulated to optimize the cleaning process.

[0056] In accordance with a second aspect of the disclosure, a system for cleaning contaminated silicon carbide (SiC) particles by removing fine grain particles adhering to the contaminated SiC particles is disclosed.

[0057] The system may include a jet mill.

[0058] The system may further include a classifier system. The classifier system may include more than one classifier.

[0059] In various embodiments, the classifiers may be arranged such that the classifiers are connected in series. In such an arrangement, each classifier produces a coarse fraction and a fine fraction, whereby each coarse fraction of an upstream classifier may be fed to a downstream classifier and each fine fraction of a respective classifier may be separated out from the classifier system.

[0060] To demonstrate the effectiveness of the present method and system, comparison of SiC particles before and after cutting of silicon wafer is made: Fig. 3A shows a SEM (1000x

magnification) of new SiC particles before cutting of silicon wafer. Fig. 3B shows a SEM (1000x magnification) of contaminated SiC particles after cutting of silicon wafer. Fig. 3C shows a SEM (1000x magnification) of the cleaned SiC particles obtained by the present method. Fig. 4A shows a SEM (2000x magnification) of new SiC particles before cutting of silicon wafer. Fig. 4B shows a SEM (2000x magnification) of contaminated SiC particles after cutting of silicon wafer. Fig. 4C shows a SEM (2000x magnification) of the cleaned SiC particles obtained by the present method.

[0061] In order that the invention may be readily understood and put into practical effect, particular embodiments will now be described by way of the following non-limiting examples.

Examples

Example: Cleaning of contaminated FEPA F800 Silicon Carbide Particles

[0062] Some users employ silicon carbide manufactured in accordance with FEPA F800 suspended in polyethylene glycol as the cutting medium. After usage, the suspension will be saturated with fine impurities that the particle size distribution would have shifted out of the desired narrow range.

[0063] By the process of the present invention, it is an intention to clean the contaminated silicon carbide particles such that it complies with the following Particle Size Distribution:

D3	< 18.00 μm
D50	7.00 μm to 9.00 μm
D94	> 4.00 μm

Test Method: Laser Diffraction using Malvern Mastersizer 2000

[0064] The used suspension is filtered according to prior art technology, and the solid is heated to become a dried contaminated silicon carbide powder with the following Particle Size Distribution:

D3	15.53 μm
D50	7.46 μm
D94	0.92 μm

This powder is heavily laden with fine impurities.

[0065] The contaminated silicon carbide powder is cleaned as described in the present invention, and shown in **Fig. 1**. Firstly, the material was fed into a COMEX JMX 200 Jet Mill, in which any agglomerates are grinded and broken up at a compressed air setting of about 4 bar. The dispersed powder then leaves the Jet Mill at a rotor speed of 1000 rpm.

[0066] Next, the dispersed powder is cleaned in a series of 3 COMEX ACX 200 Air Classifiers, described as follows.

[0067] The dispersed powder then enters the bottom of first Classifier and separates into a fine and coarse fraction at a rotor speed of 2,400 rpm. The Particle Size Distributions of these streams are shown in the following:

Feed	Fig.2A Sample 1
Fine	Fig. 2A Sample 3
Coarse	Fig. 2A Sample 4

[0068] The first fine fraction leaves the top of the first Classifier via the rotors, and is collected in an air filter (with a fan speed of 3,300 rpm) as SiSiCar@50. The composition of this first fine fraction may typically be:

Silicon Carbide Fines	> 50 wt%
Silicon Fines	< 40 wt%
Iron	< 6 wt%

[0069] The first coarse fraction then enters the bottom of second Classifier and separates into a second fine and second coarse fraction at a rotor speed of 2,700 rpm. The Particle Size Distributions of these streams are shown in the following:

Feed	Fig.2B Sample 4
Fine	Fig. 2B Sample 5
Coarse	Fig. 2B Sample 6

[0070] The second fine fraction leaves the top of the second Classifier via the rotors, and is collected in an air filter (with a fan speed of 3,400 rpm) as SiSiCar®70. The composition of this second fine fraction may typically be:

Silicon Carbide Fines	> 70 wt%
Silicon Fines	< 25 wt%
Iron	< 4 wt%

[0071] From Fig.2B Sample 6, the second coarse fraction has already met the FEPA F800 Particle Size Distribution requirement. However, in order to rid the fine impurities to a minimum, this second coarse fraction is again fed into the bottom of third Classifier and separates into a third fine and third coarse fraction at a rotor speed of 2,700 rpm. The Particle Size Distributions of the streams are shown in the following:

Feed	Fig.2C Sample 6
Fine	Fig. 2C Sample 7
Coarse	Fig. 2C Sample 8

[0072] From Fig.2C Sample 7, the third fine fraction contained a significant portion of coarse silicon carbide particles, hence it is routed back into the Jet Mill to increase the yield.

[0073] The third coarse fraction is collected as cleaned silicon carbide particles, with the following Particle Size Distribution which complied with those of FEPA F800:

D3	13.62 μm
D50	7.71 μm
D94	4.75 μm

[0074] As shown in Fig. 3C and Fig. 4C, the cleaned silicon carbide particles are very well "dedusted", that is, most of the fine impurities have been removed.

[0075] The overall yield of the cleaned silicon carbide is about 73 %.

[0076] By "comprising" it is meant including, but not limited to, whatever follows the word "comprising". Thus, use of the term "comprising" indicates that the listed elements are required or mandatory, but that other elements are optional and may or may not be present.

[0077] By "consisting of" is meant including, and limited to, whatever follows the phrase "consisting of". Thus, the phrase "consisting of" indicates that the listed elements are required or mandatory, and that no other elements may be present.

[0078] The inventions illustratively described herein may suitably be practiced in the absence of any element or elements, limitation or limitations, not specifically disclosed herein. Thus, for example, the terms "comprising", "including", "containing", etc. shall be read expansively and without limitation. Additionally, the terms and expressions employed herein have been used as terms of description and not of limitation, and there is no intention in the use of such terms and expressions of excluding any equivalents of the features shown and described or portions thereof, but it is recognized that various modifications are possible within the scope of the invention claimed. Thus, it should be understood that although the present invention has been specifically disclosed by preferred embodiments and optional features, modification and variation of the

inventions embodied therein herein disclosed may be resorted to by those skilled in the art, and that such modifications and variations are considered to be within the scope of this invention.

[0079] By "about" in relation to a given numerical value, such as for temperature and period of time, it is meant to include numerical values within 10% of the specified value.

[0080] The invention has been described broadly and generically herein. Each of the narrower species and sub-generic groupings falling within the generic disclosure also form part of the invention. This includes the generic description of the invention with a proviso or negative limitation removing any subject matter from the genus, regardless of whether or not the excised material is specifically recited herein.

[0081] Other embodiments are within the following claims and non-limiting examples. In addition, where features or aspects of the invention are described in terms of Markush groups, those skilled in the art will recognize that the invention is also thereby described in terms of any individual member or subgroup of members of the Markush group.

Claims

1. A method for cleaning contaminated silicon carbide (SiC) particles by removing fine grain particles adhering to the contaminated SiC particles, the method comprising:
feeding the contaminated SiC particles to a jet mill to obtain a dispersed powder;
feeding the dispersed powder to a classifier system to obtain the cleaned SiC particles,
wherein the classifier system comprises more than one classifier.
2. The method of claim 1, wherein the classifiers are connected in series.
3. The method of claim 2, wherein each classifier produces a coarse fraction and a fine fraction, wherein each coarse fraction of an upstream classifier is fed to a downstream classifier, and wherein each fine fraction of a respective classifier is separated out from the classifier system.
4. The method of any one of claims 1-3, wherein the coarse fraction of the last classifier contains the cleaned SiC particles.
5. The method of any one of claims 1-4, wherein at least one of the fine fractions is recycled back to the jet mill.
6. The method of any one of claims 1-5, wherein the jet mill is operated at a rotor speed of about 800 to about 1,200 rpm.
7. The method of any one of claims 1-6, wherein the classifier system comprises a first, a second, and a third classifier.
8. The method of claim 7, wherein the first classifier is operated at a rotor speed of about 2,100 to about 2,800 rpm.
9. The method of claim 7 or 8, wherein the second classifier is operated at a rotor speed of about 2,100 to about 2,800 rpm.

10. The method of any one of claims 7-9, wherein the third classifier is operated at a rotor speed of about 2,100 to about 2,800 rpm.
11. A system for cleaning contaminated silicon carbide (SiC) particles by removing fine grain particles adhering to the contaminated SiC particles, the system comprising:
 - a jet mill; and
 - a classifier system, wherein the classifier system comprises more than one classifier.
12. The system of claim 11, wherein the classifiers are connected in series.
13. The system of claim 12, wherein each classifier produces a coarse fraction and a fine fraction, wherein each coarse fraction of an upstream classifier is fed to a downstream classifier, and wherein each fine fraction of a respective classifier is separated out from the classifier system.