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Slurry grit evolution

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ABSTRACT

In slurry-based wafering of silicon bricks using multi-wire saws, the slurry is subject to significant evolution with time as the grits become worn and the silicon kerf accumulates. A good understanding of this evolution would allow wafer producers to make better-informed decisions on when and how to replenish slurry during wafering. This paper summarizes certain important slurry properties and presents some experimental results regarding their evolution. Sampling the slurry at the front and rear of silicon bricks during wafering has allowed the effect of a single pass through the sawing channel to be studied. The wear on the slurry grit is interpreted in terms of identifying what portion of the particle-size distribution plays the most critical role in wafering, and how this critical region changes as the slurry ages. It is found that in a relatively fresh slurry, the particles around the median size and slightly larger are the most active, while particles more than a few µm below the median play only a small part. As the slurry ages, the active region of the particle-size distribution becomes narrower, and shifts towards larger particles even though there are fewer such particles available. This leads to less slurry–brick interaction and poorer material removal properties.

Introduction

The overall goal of the photovoltaic industry is to lower the cost at which energy can be delivered to the end users. In particular, the aim is to reduce this cost to a level comparable to that of conventional energy sources, in order to achieve the long-sought grid parity. Excluding political intervention, this can be done by increasing the power output of the photovoltaic cells, and by decreasing the energy, labour and material costs of their production.

For cells based on silicon wafers, the process of cutting bricks into wafers affects the end energy price mainly through its contribution to production costs (although poor wafering practices may lead to reductions in efficiency). This is connected to the silicon material lost as kerf, the energy necessary to run the wafering process, the waste of wafers and half-completed cells as a result of inferior mechanical strength, the consumables of the wafering process and so on.

In the present economic climate, rapidly changing prices of materials and products make it difficult to gauge the exact cost contribution of the wafering step, but it has previously been estimated to be 22% of the cell cost [1]. However, that the cost is significant cannot be debated, and the nature of the global market situation makes even minuscule cost reductions worthy of pursuit.

Amid the evolution of fixed abrasive wafering and exotic kerf-free wafering techniques, the industry workhorse remains slurry wafering with multi-wire saws. In this process, a single wire is passed in many hundreds of windings around a set of guide rolls to create a wire web. The wire moves continuously in a single direction at speeds of around 15m/s, necessitating wire lengths of several hundred kilometres. The bricks are forced down onto the moving wire web while an abrasive slurry of polyethylene glycol (PEG) and silicon carbide (SiC) particles is being homogeneously applied to the web in front of the brick. The wire and the carrier fluid transports the abrasive particles to the brick, where the forces from the wire lead to a three-body abrasion process between the particles, the wire and the silicon brick.

Naturally, the properties of the abrasive slurry will influence the wafering process and the resulting wafers. While the exact microscopic details of the wafering, and thus the role of the slurry grit parameters, are not fully understood, the choice of slurry parameters, in the experience of academic and industrial players, is frequently a matter of compromise between optimizing different outputs, for example productivity versus wafer surface quality.

> "A better understanding of slurry grit evolution would allow industry players to make better-informed choices on how and when best to replace the slurry in their operations."

As the slurry grit passes through the sawing channel, the forces acting upon it lead to the breakage of particles, and thus changes in both size distribution and shape. As a constant volume of slurry is being circulated in the machine throughout the cut, these changes accumulate. In addition, the silicon kerf and steel particles from the wire build up, changing the composition of the dry matter. The slurry is thus subject to significant evolution, and the lifetime of a batch of slurry is limited. Industrial practices vary, but after a given exposure, all or part of the slurry is dumped and replaced with freshly mixed materials. A better understanding of slurry grit evolution would allow industry players to make better-informed choices on how and when best to replace the slurry in their operations.

As the wafer quality is determined by a combination of slurry and machine settings, a perfect understanding of slurry evolution would allow operators to change the machine parameters to match a non-static optimum point; this is the ultimate goal of slurry evolution studies. Whereas slurry evolution also involves changes in the carrier fluid (such as pH and water absorption), this discussion will focus on the SiC particles.

Slurry grit parameters

The influence of some key slurry properties has already been summarized by Anspach & Lawerenz in 2010 [2], and only a brief description of the most important parameters – particle size and shape – will be given here.

The particle-size distributions of grits available on the market conform to specifications set out in FEPA, ANSI or JIS standards. As these standards are not uniformly defined, but rather give ranges of permitted values for certain size parameters, powders following the same standard definition may still have slightly different particle-size distributions.

Coarse particles unsurprisingly have a greater abrasive effect than finer ones, and are therefore desirable from a productivity point of view. However, they also lead to more surface damage and roughness, greater kerf loss and larger thickness variation across the wafer.

The particle shape can be described by a number of different parameters, such as aspect ratio or, most commonly, circularity. The circularity of a particle is defined as the ratio between the circumference of a circle and the perimeter P of the particle, where the area of the circle is equal to the maximum area projected by the particle in 2D (A): circularity = $(4A\pi)^{1/2}/P$. Although circularity is not a perfect parameter for describing the sharpness of particles, a low value correlates to a high sharpness, and is frequently used for this purpose. A certain level of sharp particles is desired in powders for silicon wafering, so while shape parameters are not part of any of the common standards, grit manufacturers often provide information about circularity.

Particles of low circularity, in other words high sharpness, in general have better material-removal properties than blocky and near-spherical particles of high circularity, and are thus preferable from a productivity point of view. However, such particles also give rise to a higher surface roughness and may lead to greater sub-surface damage.

It should also be noted that in a powder sample the particles with the very lowest circularities are often long, thin, needle-like particles. These have a large influence on the average circularity measurement of the sample, but are not believed by the present authors to play an important role in wafering.

Experimental work

At SINTEF Materials and Chemistry a Meyer Burger 265/4 wafer saw was used for studying slurry grit evolution and other wafering-related topics. The saw had been modified to provide access to the wire web during operation, allowing slurry samples to be extracted immediately at the front and rear of the silicon brick. This set-up was used with F800 slurry grit to perform three consecutive cuts. The slurry was not replenished during this period, and samples were taken throughout the process.

Slurry size and shape was analyzed using a Sysmex FPIA 3000 sheath flow particle imaging instrument. After the analyses, the FPIA software was used to extract more detailed information, in particular by looking at certain fractions defined by limits in size and/or circularity. The samples were chemically treated to completely dissolve iron and silicon fines prior to analysis.

> "There is a clear trend towards a reduction in particle size with slurry age."

Fig. 1 shows the circle equivalent (CE) diameter of the analyzed powders as a function of cutting time. There is a clear trend towards a reduction in particle size with slurry age, explained by the wear of the wafering process on the particles. There is also a clear difference between the slurry sampled at the front and at the rear of the

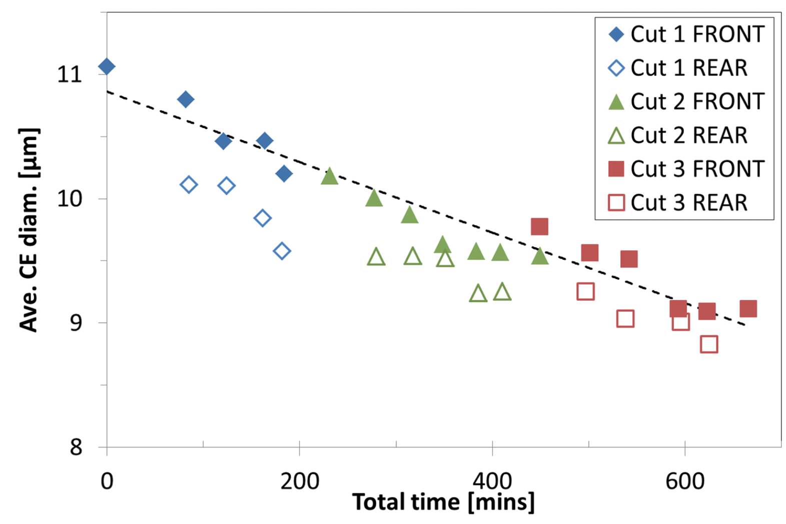


Figure 1. Average circle equivalent (CE) diameter of the analyzed powders when only particles between d50 and d3 are taken into account.

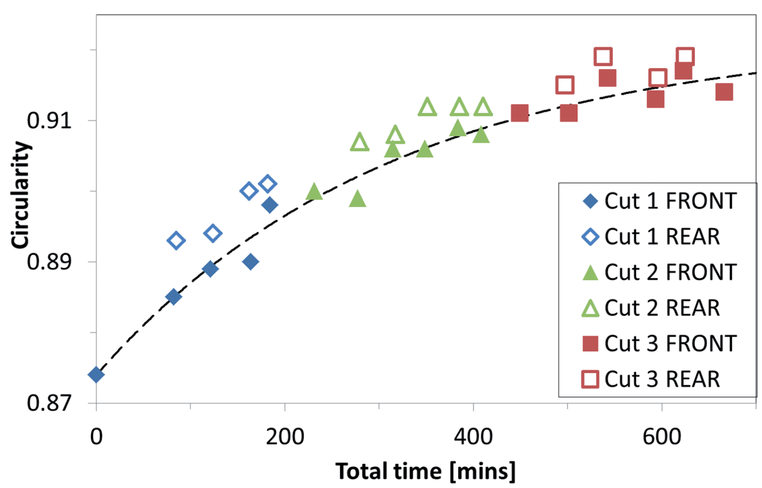


Figure 2. Circularity of the analyzed powders when only particles between d50 and d3 are taken into account.

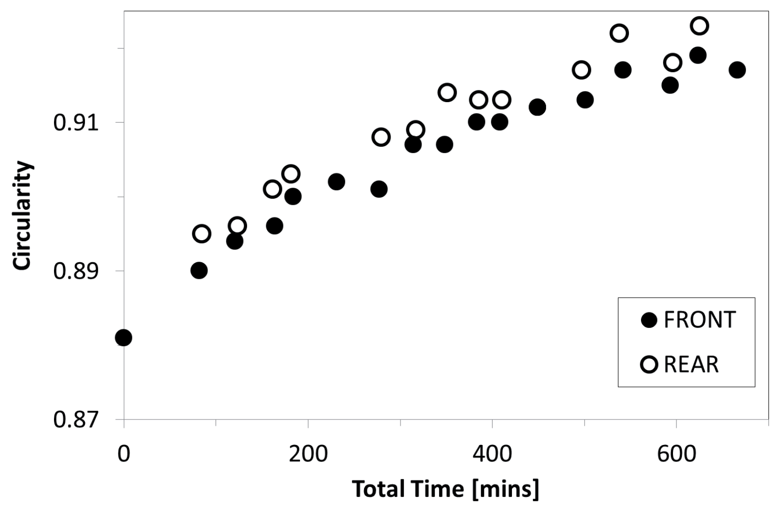
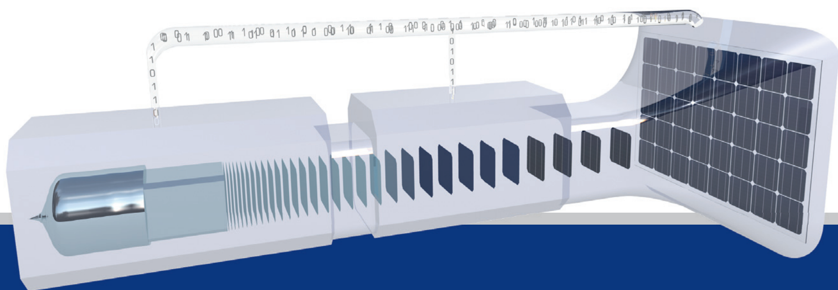


Figure 3. Circularity of particles with CE diameter between 9 and 10µm, extracted at the front and rear of the brick throughout three cuts.



brick: the powder at the rear of the brick contains much smaller particles on average.

Fig. 2 shows the circularity of the analyzed powders. Again, two observations can be made: there is a trend of increasing circularity over the course of three cuts, and the particles extracted at the rear of the brick are typically more rounded than particles extracted from the front of the brick at a similar point in the sawing process.

The intuitive explanation is that the silicon carbide is being worn down to smaller and more spherical particles by the sawing action: the changes seen between the front and rear represent the wear on a small sample of slurry during one pass through the sawing channel, and the cumulative effect of multiple passes results in the long-term trend. However, as reported in the literature [3], it is a known theory that the larger particles are more frequently expelled from the sawing channel than the finer grit; thus the size difference seen between two samples extracted at the front and the rear is not necessarily only due to the wearing down of particles. In addition, since the shape of particles varies with size, the same issue applies to the apparent changes in circularity.

To determine if the changes seen between the front and the rear of the brick are due to the wear in the sawing channel, the circularity of the powders was analyzed and compared in fixed intervals: 9–10µm, 10–11µm and 11–12µm. The results for the first of these intervals are shown in Fig. 3: it is evident that there is a visible and significant circularity change taking place in a single pass through the brick.

In an attempt to illustrate the slurry evolution in greater detail, a more thorough examination of four powder samples will now be presented. These powder samples are all from the same batch of slurry, used to perform three consecutive cuts. The samples represent two pairs of samples, each pair having one sample from the front of the brick and one from the rear, which are taken more or less simultaneously. The first pair of samples was extracted midway through the first cut (fresh slurry), and the second pair from midway through the third cut (worn slurry), with no replacement or replenishing of the slurry. The shape analysis was performed with the Sysmex FPIA 3000 as before.

The shape analysis data for each powder was grouped in 1µm-wide size intervals (plus a >15µm bin). Figs. 4 and 5 show the particle-size distribution at the front and rear of the brick for the fresh and worn slurry respectively. As expected, in both cases there is a change in the size distribution between the front and rear of the brick. The number of large particles is seen to decrease between the front and the rear in both cases, while

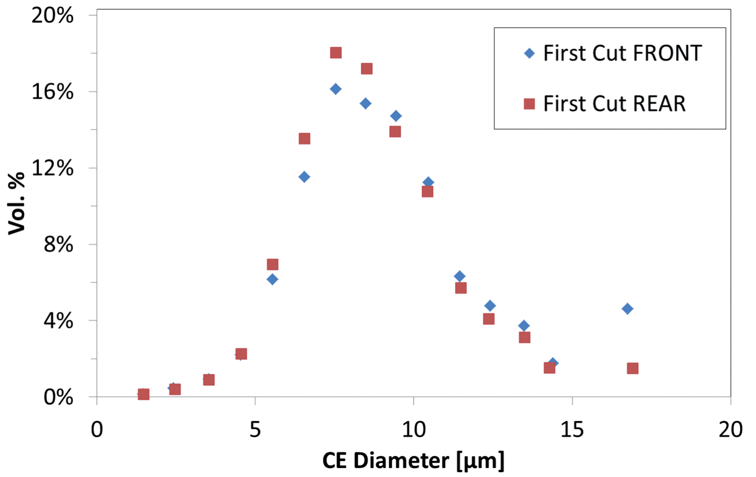


Figure 4. Particle-size distribution of samples extracted from the front and rear of the brick midway through the first cut.

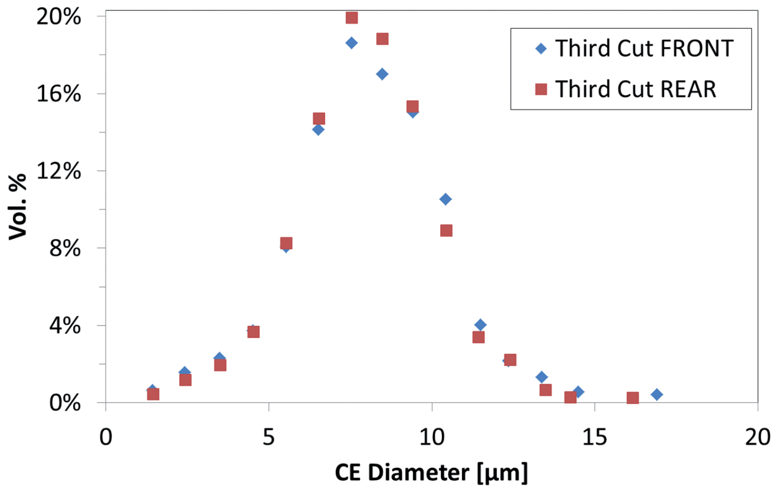


Figure 5. Particle-size distribution of samples extracted from the front and rear of the brick midway through the third cut.

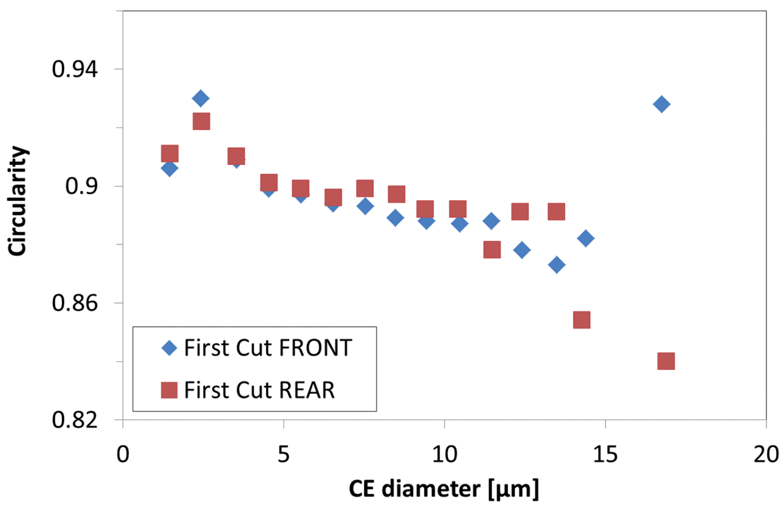


Figure 6. Circularity as a function of circle equivalent diameter: samples extracted at the front and rear of the brick midway through the first of three cuts.

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there is an increase in particles around the d50 and immediately below. Moreover, the particle-size distribution for the worn slurry, in addition to being shifted to the left as expected, is somewhat narrower when compared to the fresher slurry.

> "There are more fines in the samples extracted at the front than in those taken from the rear."

A curious fact is that for both slurries there are more fines in the samples extracted at the front than in those taken from the rear. This is the opposite of what would perhaps be intuitively expected, as the fines obviously originate from the sawing channel. However, comparing worn with fresh slurry, there is no doubt that the number of fines increases with time.

The average circularity of each 1μm-size bin is plotted versus the average CE diameter of those bins and shown in Figs. 6 and 7. For the largest particles, there is significant scatter in the data, mainly caused by a low number of particles in this range. The focus can therefore be put on the particles between approximately 7 and 11μm, where in Fig. 6 it is clear that there is significant rounding of particles at the rear compared to the front. For the worn slurry in Fig. 7, the difference is much slighter.

It should be noted that Figs. 6 and 7 do not in themselves justify the conclusion that the particles from 7 to 11μm are the most important for the cutting process. It must be remembered that these two figures give only the average circularity and the change in this measurement. The number of particles represented by each point in the graphs is not taken into account.

In an attempt to visualize the combined change in particle shape and size, a 'cutting potential' is defined for each size bin. This quantity takes into account the fact that rounder particles are less able to remove material, and also that a particle bin with a larger volume fraction of the total number of particles has a greater potential to affect the sawing process. A first attempt at defining such a cutting potential is then: potential = (1 – circularity) × vol%.

The cutting potentials at the front and rear for the fresh and worn slurry are shown in Fig. 8. The areas where there is a large change in this potential are the parts of the particle-size distribution which are the most changed in terms of vol% and/or shape. It can perhaps be assumed that these areas are therefore also the areas that have the greatest effect on the sawing process (and thus wafer quality).

Looking first at the change between the front and rear for the fresh slurry, one can see that there is a large decrease in cutting potential of large particles. This is hardly surprising, as these large particles are being ground down and, in the same process, rounded. It must also be remembered that this is an area of large uncertainty because of the low number of particles present. There is also an area between

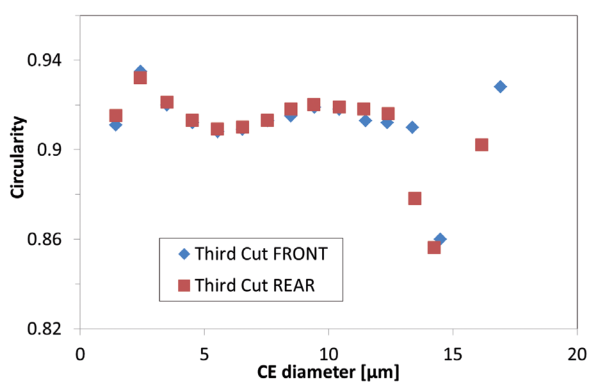

Figure 7. Circularity as a function of circle equivalent diameter: samples extracted at the front and rear of the brick midway through the last of three cuts.

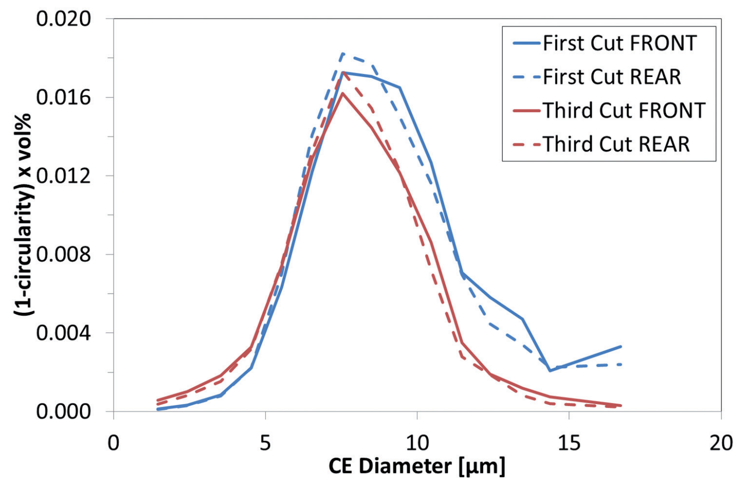

Figure 8. 'Cutting potential', defined as the product of (1 – circularity) and vol%, plotted as a function of circle equivalent diameter. The curves show the situation at the front and rear of the brick, halfway through the first of three cuts and halfway through the last.

~5 and 8µm where there is a considerable increase in the cutting potential, meaning that the number and sharpness of particles in this range increase. This could perhaps be caused by the larger particles being broken down, leading to an increase in the number of particles, and perhaps these particles are also sharper than the 'original' particles in this size range. These two last suppositions agree with Figs. 4 and 6.

It would be natural, then, to assign the 'active region' of the cutting potential to the region that sees the largest decrease in cutting potential. For the fresh slurry, this is the region from the 9–10µm bin to the 13–14µm bin.

For the older slurry, the first observation is that the cutting potential has a narrower and lower peak, indicating a decline in material-removal properties. The cutting potential is all but gone for large particles, while there is a significant build-up of fines on the right side. There is a significant shift of the rightmost edge of the main 'bell curve' towards smaller particles, while the leftmost edge is much less affected. Particles in the 9–10µm range in the worn slurry do not, on average, have their cutting potential much changed between the front and the rear. For the fresh slurry, this had been the particle bin with the greatest reduction in cutting potential. Below this, as for the fresh slurry, there is a region down to about 5µm where the cutting potential is increased. The 'active region' has moved towards larger particles, with its lower bound now limited by the 10–11µm bin. This would mean that the cutting is now due to slightly larger particles than to the fresh slurry. As such particles are far less plentiful than in the fresh slurry, one would expect a significant reduction in material-removal properties. This is reflected in the fact that the total change in cutting potential in the active region has dropped to around half of what was observed for the fresh slurry, which – if slurry wear correlates to silicon wear – would also suggest that the action of the slurry upon the silicon block is less powerful.

"Particles around d50, and a few µm larger, are the ones most influenced by the sawing process, while particles below d50 are not affected so much."

Summary

The effect of the sawing process upon slurry grits is significant enough to be observable after one pass through the sawing channel. Accumulated over time, this leads to significant evolution of the slurry grit properties, and in how the grits interact with the silicon brick.

For a relatively fresh slurry, the large particles still play a role, while these are almost insignificant for an older slurry. Particles around d50, and a few µm larger, are the ones most influenced by the sawing process, while particles below d50 are not affected so much.

As the slurry ages and the particles are reduced in size and sharpness, the interaction with the silicon brick also changes. The region of greatest change narrows with slurry age, but the region of greatest decrease in cutting potential actually shifts towards larger particles, even though there are fewer such particles available. However, the total change of the powder is much less in an old slurry, indicating less interaction and poorer material-removal properties, as expected.

Acknowledgements

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