Understanding the role of abrasives used in the multi-wire sawing process

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ABSTRACT

Multi-wire sawing is currently the most efficient slicing technology for silicon wafers in the photovoltaic industry. Nevertheless, the wafer producers are faced with major cost reductions in the production process and the demand for high-quality, very thin wafers with a total thickness variation less than 10% of the average wafer thickness. One approach to this is the understanding of the role of slurry, specifically the abrasives used in the multi-wire sawing process. In the past few years, more and more scientific investigations have been conducted and are summarised in this article.

Introduction

The main goal for the entire solar industry is to achieve grid parity for solar power – and soon. This is only possible by minimising the cost per Watt peak and ultimately the cost per kWh. The industry realises this task by reducing the production cost throughout the manufacturing chain on the one hand and by increasing the efficiency of photovoltaic systems on the other hand. Within the wafering part of the value chain for crystalline silicon-based solar cells, which will play a dominant role in the years to come, a main goal is to increase the number of wafers produced per kilogram of silicon. Reducing the wafer thickness and minimising kerf loss are two approaches that have emerged with the development of the wire-sawing process. In the last five years, wafer thickness decreased from 300µm down to 160µm in wafer production and wafers with thicknesses of around 100µm have been achieved by R&D demonstrations [1]. In the same way, kerf loss (sawing slot width) decreased from 240µm early this decade to around 140µm. Again, slot widths below 100µm have been achieved by several research groups [2,3]. In any case, next generation solar cells with exact adapted production processes (e.g. thermal process steps) will require very thin wafers with well-defined geometries.

Among other factors, the total thickness variation (TTV) of a wafer is important. The TTV depends only on the way the kerf is removed and is independent of the wafer thickness reduction [4,5]. The mechanism of abrasion in the slot during the wire-sawing process remains the same. Today the TTV is in the tens of micrometers range. As the thickness of a wafer decreases, it becomes clear that compared with its thickness the wafer's TTV plays a more and more important role. Fig. 1 shows the percentage of the total thickness variation compared to wafer thickness for four given TTV values.

For future reference, the percentage of TTV compared to the wafer thickness should not exceed 10%, implying a TTV below 10µm for wafers with a thickness of 100µm.

Reduction of the dimensions of the slots between the wafers and efforts to run the wire-sawing process more efficiently still relies on a 'trial and error' approach of adjustment of sawing parameters. The role of abrasives – and more importantly, carrier fluids – as the slurry formulation in this complex and highly dynamic multi-wire sawing system is still underestimated and underlies special interest at R&D at PV Crystalox Solar.

The following section presents an overview of the role of abrasives, which, with carrier fluid form one part of the slurry formulation, on the dimensions of the slots and the wire-sawing process.

Silicon carbide abrasives

Within the multi-wire sawing process, the use of loose abrasives in a carrier fluid is well established. Nevertheless, the process is not fully understood and is probably not at its optimum level of operation. Silicon carbide powder is by far the most cost effective choice for abrasives. Other abrasives are diamonds or minerals, which are harder than silicon. The carrier fluid consists of polyethylene glycol (mostly common in industry), oil or water with additives.

Production

Silicon carbide is typically a synthetic mineral. Minor quantities occur naturally in the mineral Moissanite. Silicon carbide is used mainly as abrasive powder, but also in ceramic materials and more and more in semiconductor applications.

The production for abrasive powder starts with the Acheson process, where quartz sand and petroleum coke are reacted in a graphite electrical resistance furnace at temperatures up to 2500°C. After the chemical reaction to SiC crystals, the product is crushed, magnetically separated, sieved and classified. The final product, which is suitable for the wire-sawing process, is a green or black silicon carbide powder with different particle sizes. The green SiC has a higher purity

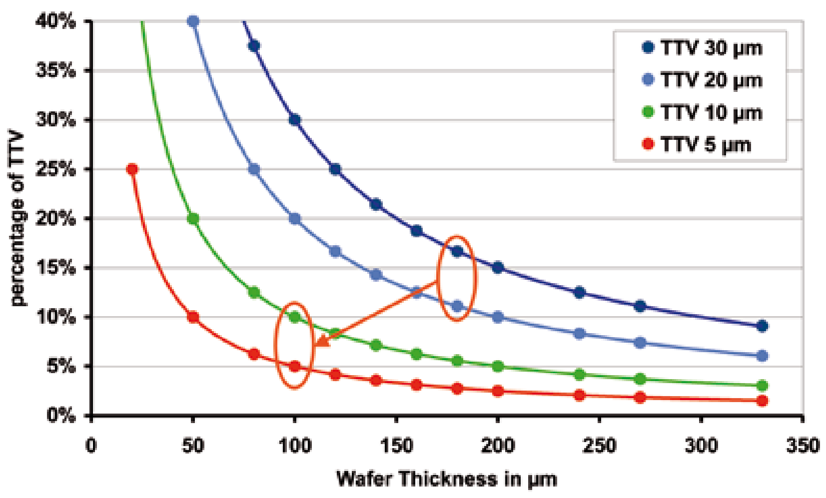


Figure 1. Percentage of TTV versus wafer thickness. The decrease in percentage TTV for wafers of 180µm to 100µm is marked in red [12].

than the black one, which contains a higher impurity level mainly of nitrogen and aluminium. Green SiC is at this time commonly used by wafer producers in Japan, while the black SiC is used in Europe and America. There is a lack of scientific investigations to highlight the differences in the use of black and green SiC in the multi-wire sawing process.

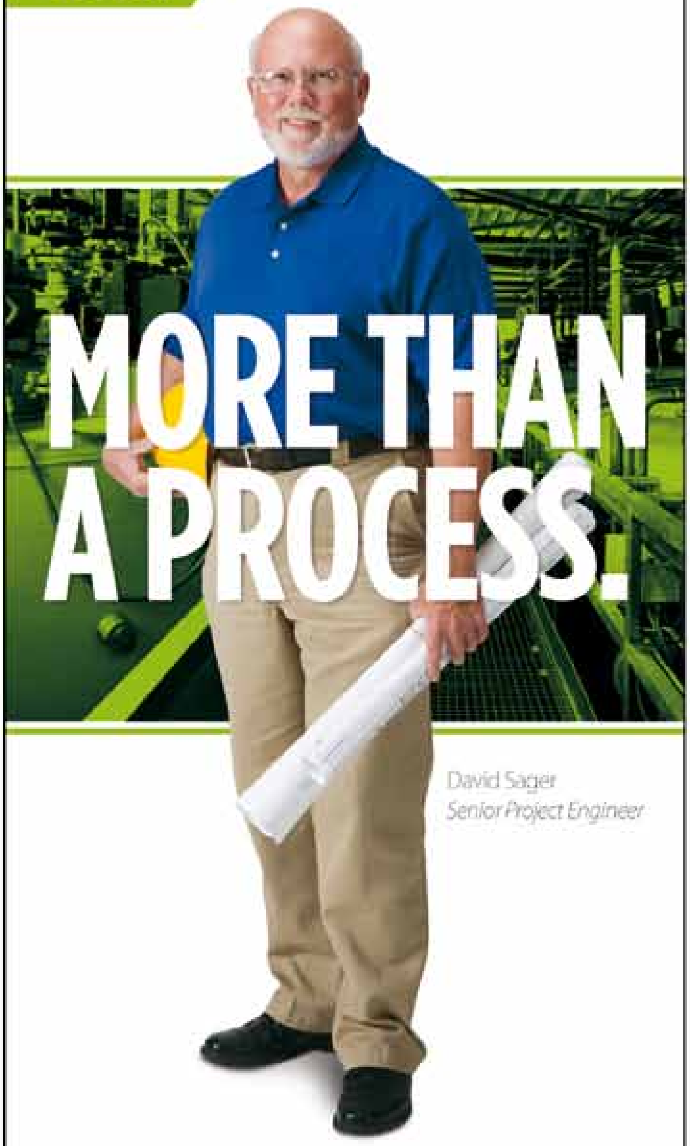
Size and shape characterisation methods

The most important property of abrasives that must be determined before the sawing process starts is their size distribution. To this end, there is a number of measuring methods available, some of which are also capable of measuring the shape of particles. Many of these methods serve to keep the abrasives in suspension, bringing with it the advantage that a suitable dilution can be adjusted to avoid an overlapping or even an agglomeration of particles. Some of the most widely used methods are discussed here.

Light microscopy is commonly used to analyse size and shape distributions of abrasives simultaneously. During measurement, the particles are illuminated and flow in a suspension in the focal plane of a camera. In the case of abrasives mixed with abrasive wear being measured in water suspensions, much care has to be taken to avoid the agglomeration of particles. The addition of special dispersing agents like sodium pyrophosphate and the application of ultrasound is helpful in such a situation (a special treatment is proposed in [6]). Depending on the pattern recognition, which takes place after particle image capture, a large variety of shape features can be analysed. Because these features are obtained from two-dimensional images, one must be mindful that the imaged particles are not oriented in a specific direction. Light microscopy cannot resolve particles that are much smaller than ca. 1µm. Because of its much better lateral resolution (down to ca. 5nm), scanning electron microscopy (SEM) might be an alternative to light microscopy. Scanning electron microscopes are used to get a qualitative picture of abrasives' sizes and shapes but until now there has not been an easy-to-handle preparation procedure which allows the quick analysis of large particle ensembles to obtain the size and shape distributions of abrasives, although efforts are being made to reach this goal. Because it is impossible to observe abrasives in suspension in electron microscopy, the particles have to be kept or transferred into a powder state. The main obstacles for the measuring of size and shape distributions of abrasives lie in the homogeneous and overlapping-free spreading of particles on the sample holder and in the scanning of a sufficiently large number of particles. If the abrasives are mixed with the abrasive wear containing a large amount of small particles, these obstacles will increase.

> "Depending on the pattern recognition, which takes place after particle image capture, a large variety of shape features can be analysed."

Another popular method for ascertaining the size distribution is laser diffraction, where the abrasives – similar to light microscopy – are kept in suspension. The diffraction pattern of the scattered laser light is usually detected by an array of photodiodes. This method is capable of measuring small particles (down to ca. 50nm), but for small particles with diameters smaller than the laser wavelength the analysis of the particle size is dependent on the refractive index of this particle. Today's laser diffractometers are not able to measure the shape properties of particles (which is in principle possible by aligning the particles within the fluid using a two-dimensional array of photo detectors). It should be mentioned that the use of laser diffraction needs a careful calibration, otherwise results obtained with different diffractometers tend to diverge greatly. Obtaining the correct fine structure of size distributions is another challenging task. On the other hand, today's diffractometers are easy to use and do not require a complicated sample preparation.



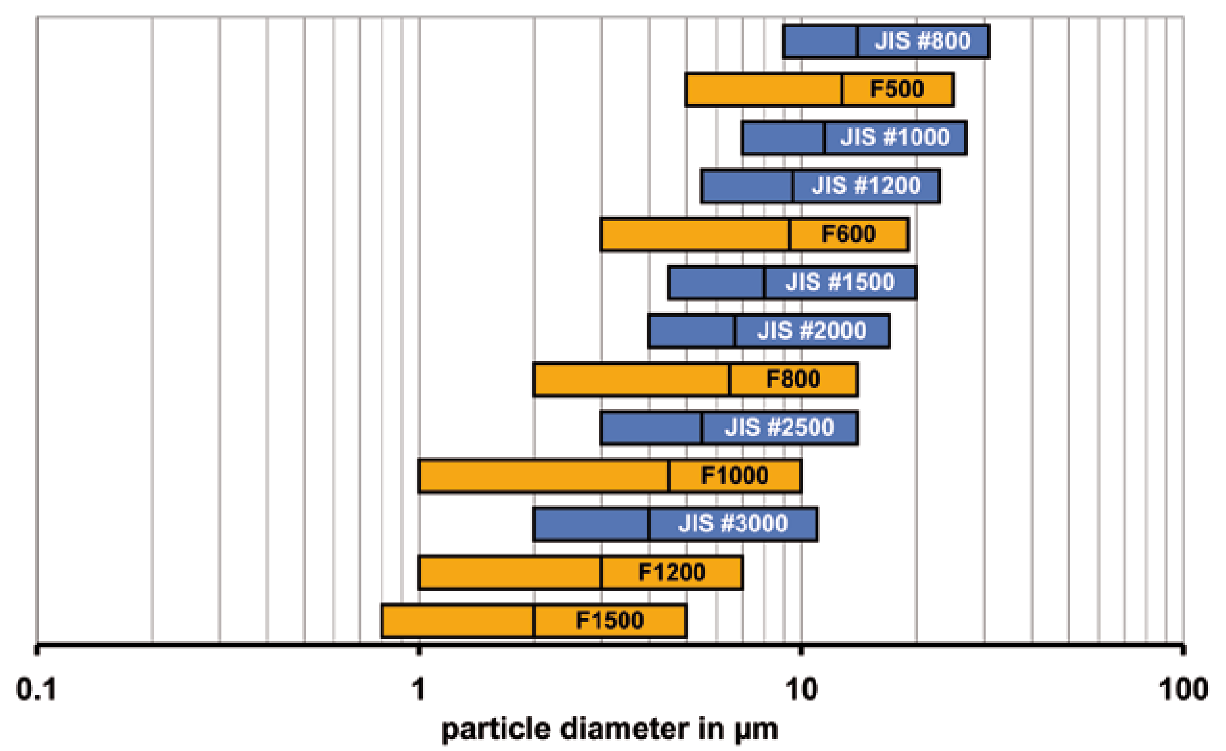


Figure 2. Overview of several classes of SiC powders according to the FEPA and JIS norm. The left end of the bar represents the ds94 value, the middle line the ds50 and the left end the ds3 value of the particle size distribution.

The electrical resistance method, often encountered as Coulter Counter, is also widespread partly because it has to be used for the specification of size distribution by the ANSI standard [7]. Particles are suspended in an electrolyte solution and have to pass a tube with a hole at its end (with an inner diameter somewhat larger than the particle's diameter). Whenever a particle passes the hole, a change in current measured between two electrodes occurs. This change is proportional to the volume of the particle. Therefore no shape distributions can be obtained.

> "The ratio of half-width to modal diameter is advantageous because it allows the comparison of the narrowness of the size distribution."

In a sedimentometer the particles are filled into a vertically oriented tube, which is filled with a liquid. The measurement of the size distribution is based on the fact that larger particles settle to the bottom of the tube faster than smaller ones according to Stoke's Law. In the bottom of the tube, scaling enables the measurement of the amount of particles that have settled after a certain time. If small particles (in the micrometer range) are to be measured, this time can extend to several hours. This method needs a very careful preparation and filling of the abrasives into the sedimentation tube, otherwise a cluster sedimentation, where large and small particles are not separated, occurs – especially for abrasives with diameters below a few micrometers. Because of these drawbacks, other methods are replacing the sedimentation although sedimentation is still mandatory if the size distribution is to be measured according to the FEPA standard [8]. The tube must then be filled with methanol.

Measurement categories

Silicon carbide powders are most often specified by the FEPA, ANSI or JIS standards (FEPA: Fédération Européenne des Fabricants de Produits Abrasifs; ANSI: American National Standards Institute; JIS: Japanese Standardization Organization). The specified value of a certain powder (for example FEPA F600) is determined by the particle size distribution whereby a larger number indicates smaller particles. To be specified by a certain FEPA, ANSI or JIS standard value the size distribution of the powder

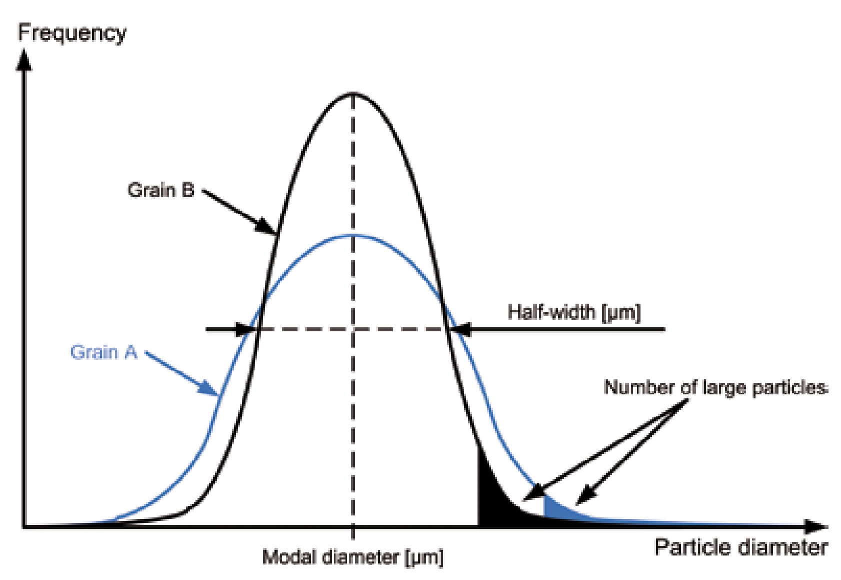


Figure 3. Sketch of a volume-based distribution of two abrasives, indicating the half-width and modal diameter.

has to fulfil some requirements, referred to as the ds3, ds50 and ds94 values. In a volume-based size distribution, a ds3 value is the diameter for which 3% of all particles are larger than the ds3 value (naturally, the ds50 and ds94 values are defined in the same manner). An example of the defined values for several particle size distributions according to the FEPA and JIS standards is depicted in Fig. 2.

It is difficult to compare different powders that are specified by different standards as these standards stipulate different measurement methods. In the ANSI standard, for example, the electrical resistance method is required [7] whereas in the FEPA standard, sedimentometers are used [8].

The ds3, ds50 and ds94 values are not the only useful values that can be taken to describe size distributions of abrasives. Other values include the mean diameter and the modal diameter (both measured in µm). The latter is especially beneficial if slurry particles, which contain abrasives as well as the much smaller abrasive wear particles, are to be characterised. In describing the narrowness of the size distribution, the full width of half-maximum (or half-width, measured in µm) can be used (see Fig. 3). It is also possible to normalise the half-width to the modal diameter. This dimensionless number is advantageous because it allows the comparison of the narrowness of the size distribution if abrasive particles with different modal diameters are to be compared. An example is shown in Fig. 4, where particles with the same normalised diameter lie on a line intersecting the origin. One of the SiC powders (F600 No. 2) has a smaller half-width (compared to powder F600 No. 1), although its normalised narrowness is larger. Another powder (F800 No. 4) has a larger modal diameter although its normalised narrowness is smaller in comparison with the F800 No. 3 powder.

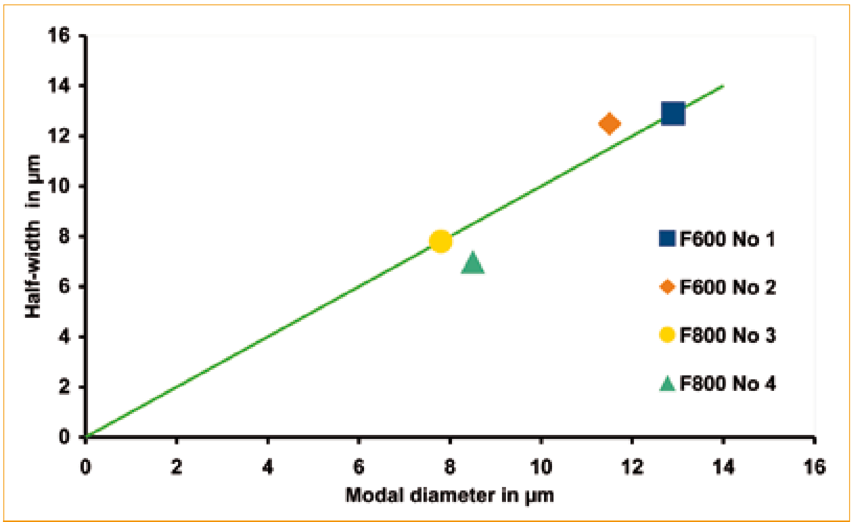


Figure 4. Half-width-modal diameter diagram. The green line indicates a reference line where all size distributions have the same normalised narrowness.

The shape of abrasives also influences sawing efficiency. All of the methods currently in use to measure shape properties are based on pattern recognition analysing only the two-dimensional images of the particles. The most common characteristic, which describes the shape of an abrasive, is the circularity, defined as the ratio of the

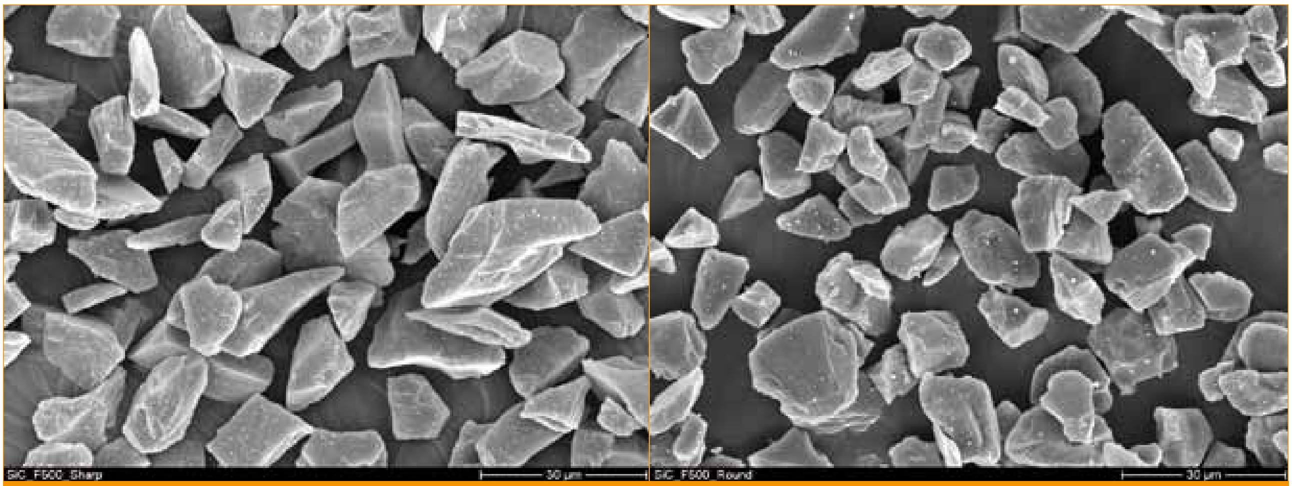


Figure 5. SEM images of SiC particles showing low circularity (SiC_F500_Sharp) and high circularity (SiC_F500_Round).

perimeter of a circle (with the same area as the respective particle) to the perimeter of the particle. The longer a particle is, the smaller the circularity. A perfectly round particle has a circularity of 1.00. Two samples of silicon carbide particles with a high and low circularity are depicted in Fig. 5. Today's instruments are capable of ascertaining a large number of shape features such as area, perimeter, circularity, feret diameter, aspect ratio, circularity, and convexity.

However, to our knowledge there is no single characteristic that specifies the sharpness of a particle. In contrast to the circularity or convexity, this demands the examination of the local proximity of the perimeter. A possible definition for the sharpness of a particle could be the minimum of all radii of curvature of the particle's perimeter.

Experimental setup

At PV Crystalox Solar, a standard industrial wire saw is set up to investigate the highly dynamic, fast and complex wire-sawing process. The wire web is separated to isolate a single wire winding to obtain single slots. An extra slurry tank provides the ability to manipulate and even replace small amounts of slurry during cutting (see Fig. 6). Temperatures of the silicon block and of the slurry can be set at constant in order to approach conditions, which are common in an industrial wire-sawing process. Furthermore, temperatures and forces that work on the silicon block can be measured [9].

In order to evaluate the performance of particular silicon carbide abrasives, slot width dimensions at the wire entry and exit side and the resulting TTV, roughness and surface damage of the side walls of the slot and the vertical force, measured during the experiment, are characteristic values [10-12]. The vertical force, which is parallel to the table drive direction and which occurs when the silicon block is forced through the wire web, indicates the 'abrasive performance' or how well the wire goes through the silicon during

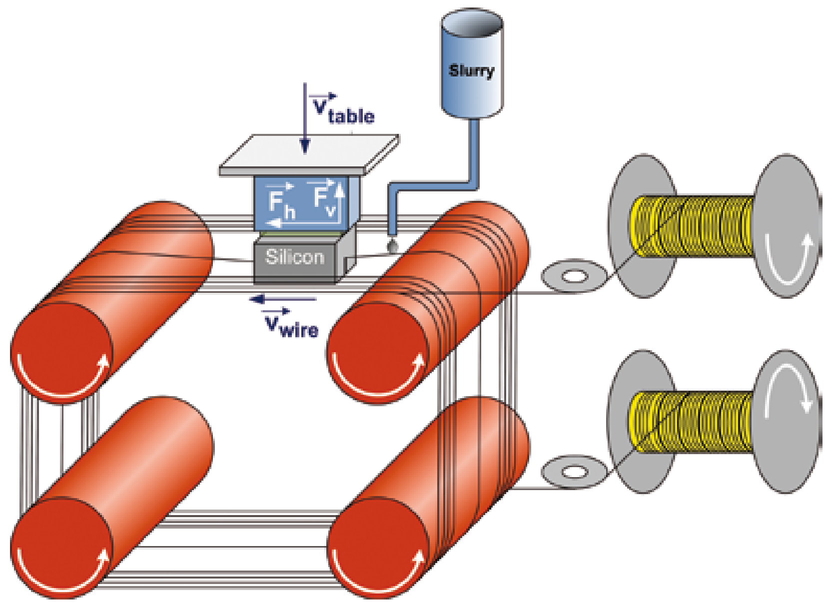


Figure 6. Sketch showing the single-wire experimental setup.

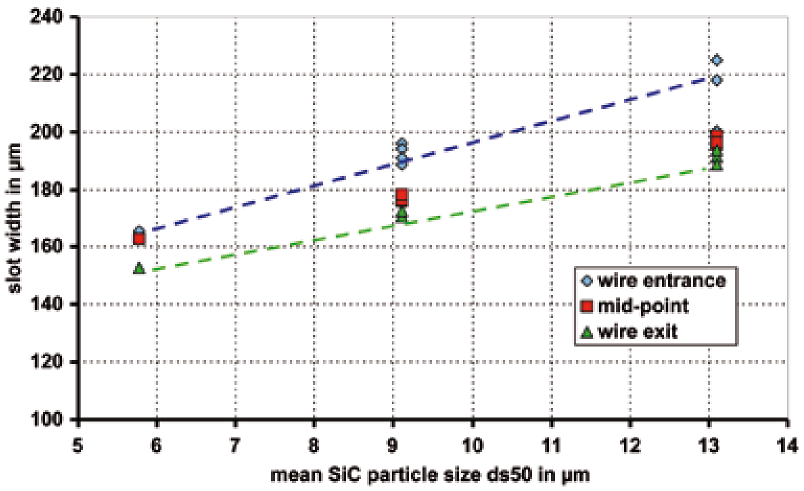


Figure 7. Slot width at the wire entrance and exit and at the mid-point of the Si block versus mean SiC particle size [11].

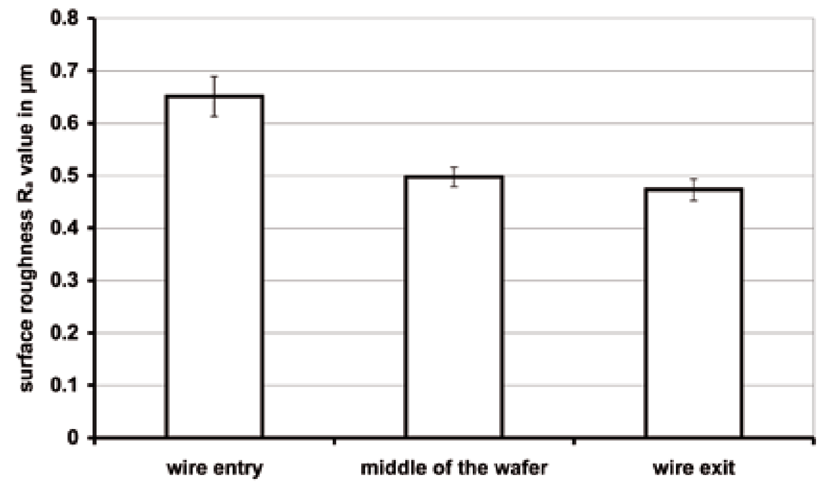


Figure 8. Mean results of the average surface roughness R_a given for the measurements at the wire entry and exit side and at the middle of the wafer [12].

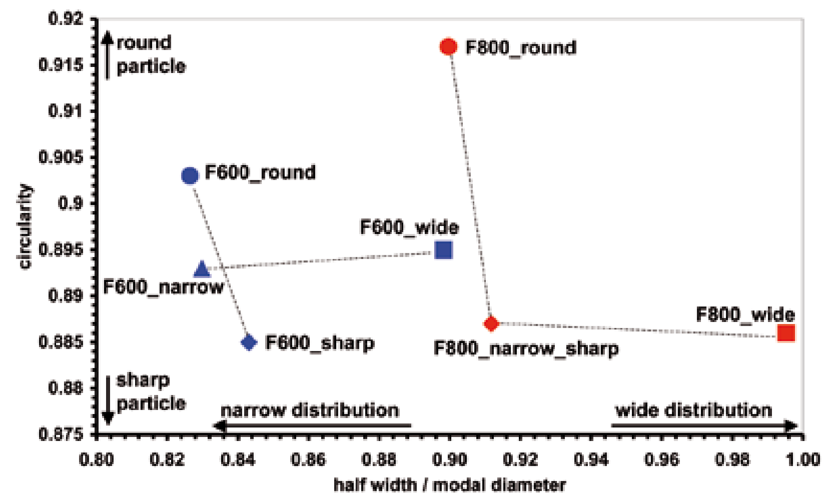


Figure 9. Circularity versus ratio between half-width and modal diameter. For the experiments chosen, F600 abrasives are marked in blue; F800 abrasives are marked in red [12].

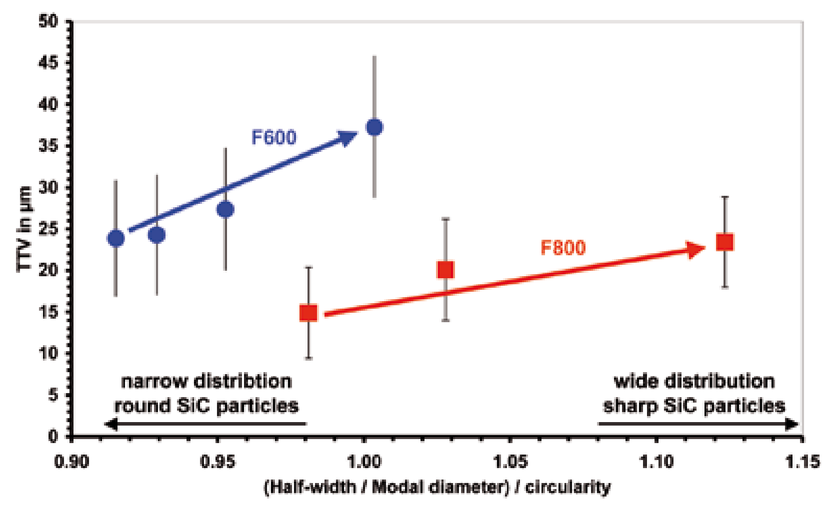


Figure 10. TTV versus ratio between half-width and modal diameter and the circularity [12].

cutting. The lower the vertical force, the 'better' the cutting process. Nevertheless, measurements of e.g. thickness, TTV, roughness and sub-surface damage can be also performed on the wafers cut with the multi-wire sawing process themselves, as found by other research groups [1,3-5, 13-25].

Influence of abrasives

Size

It is obvious that with increasing mean particle size of SiC in slurry, the slot width increases and therefore the wafer thickness decreases, a finding that has been well described in literature [1,3,11,20,26]. This correlation is also shown in Fig. 7, where the average of slot width at wire entry and exit and in the middle of the slot is plotted versus the mean particle size [11]. The slots measured were cut using three slurries, each with different mean SiC particle sizes. This diagram shows also that within the same slurry the slot width at the wire entry is larger than the slot width at the wire exit. Hence, this means that using a unidirectional wire-sawing process, the wafers on the wire entry side show a lower thickness than on the wire exit side.

Regarding the vertical force, it is known that the larger the mean particle size, the lower the vertical force on the silicon block, resulting in lower wear on the wire [2,19,20]. In other words, larger particles cut more easily through silicon and show a higher 'abrasive performance.'

> "Using a unidirectional wire-sawing process, the wafers on the wire entry side show a lower thickness than on the wire exit side."

The interaction between abrasive particles and silicon during the sawing process leaves a certain roughness at the wafer's surface. The literature states a correlation between the mean particle size of the used abrasives in the slurry and the surface roughness on wafers: with larger particle sizes comes more surface roughness [13,14,20,24-28]. Again, however, it became obvious that the surface roughness varies within one wafer depending on whether the measurement is carried out at the wire entry, the wire exit side or at the middle of the wafer. Fig. 8 shows the mean results of average surface roughness measurements on 42 wafers at three positions [12]. The fact that the surface roughness is higher at the wire entry side than at the middle of the wafer and at the wire exit side is in good agreement with the results of [2,27]. A correlation between the mean particle size and sub-surface damage, which is indicated

Figure 11. Collage of a wire beneath silicon and SiC particles in between the wire entry side (left) and the wire exit side (right) [12].

by the crack depth distribution on wafers, is discussed in the literature [13,14,19,28]. Larger slurry particles result in deeper sub-surface damage.

After all, the data in the literature show dependencies on the mean particle size of SiC in slurry. But one must bear in mind that correlations between different particles should also take into account that the correlations shown above also depend on particle size distribution and shape. Hence, we performed wire-sawing experiments at different table forward and wire speed conditions using slurries containing silicon carbide particles with extreme shape and size distributions. Fig. 9 shows the chosen SiC samples with narrow and wide size distribution (nearly the same circularity) and sharp and round particles (nearly the same particle size distribution) within two size classes, F600 and F800 [12].

Particle size distribution and shape

The results of the slot width measurements show that the wider the particle size distribution and the sharper the abrasives in slurry, the larger the slot width at the wire entry side. The slot widths at the wire exit side show no dependency on shape nor on size distribution [12]. A very interesting fact is observed when the difference between the slot width at the wire entry and exit side (respectively the TTV) was plotted versus the ratio between half-width to modal diameter and circularity (see Fig. 10). On the one hand, the slot widths cut with slurries with smaller particles (F800) show lower TTV than slots cut with slurries containing larger particles (F600), which is in good agreement with [11,20]. On the other hand, a direct correlation between the ratio of half-width to modal diameter and circularity can be observed in this case. In other words it can be stated that the narrower and rounder the particles in slurry, the lower the TTV of the slot cut with this slurry. It was also observed that the vertical forces, which were applied to the silicon block using slurries with sharper particles (low circularity), were lower than the forces with other slurries [12]. This means that the 'cutting performance' of sharper particles is higher than for round particles. Such a dependency intensifies with higher ratios between table forward and wire speed.

> "Larger particles are cutting at the wire entry, smaller particles at the wire exit side."

Bearing in mind that a) larger particles cause higher surface roughness, deeper sub-surface damage and larger slot widths, and that b) this can be observed not only on wafers cut with different particle sizes but also along one slot cut with one particular slurry, a new approach to classification of the participating particles along the whole length of the slot was pursued. In other words, larger particles were cut at the wire entry side and smaller particles were cut at the wire exit side regardless of the mean size of the particles (e.g. F600 vs. F800). Fig. 11 gives an idea of these dimensions. This mechanism of classification along the slot was observed by [29], where the researchers captured large particles pushed aside in a large-scale experiment. Nevertheless, although the mechanism remains unclear so far, a complex interaction between wire, particles and slot wall (silicon) can be presumed.

Silicon versus silicon carbide particles

During the wire-sawing process, silicon particles are abraded. The mean particle size of these silicon particles ranges between 2 and 5µm. Scientific experiments are required to investigate the correlation between the mean particle size of silicon carbide and the mean silicon particle size resulting from the wire sawing process.

Nevertheless, it becomes clear that with increasing the silicon load of the slurry, more and more silicon particles, among the silicon carbide abrasives, enter the slot. Since silicon particles compete against the silicon carbide particles in the very narrow space between wire and slot walls, at a certain silicon content the slurry is unable to cut the silicon block, as observed in the industrial wire-sawing process.

Recycling

For this reason, the slurry needs to be replenished and should also be recycled by separating the larger, still useable, silicon carbide particles from the smaller silicon particles (fines). Two main processes have been established in the past years: the centrifuge and the hydro-cyclone technique. Both techniques separate more or less efficiently the larger silicon carbide particles from the fines. However, it should be made clear that these fines also contain small silicon carbide particles, which results from the abrasion and breaking of larger silicon carbide particles. The abrasion rounds the edges of the SiC particles; the breaking minimises the amount of larger particles. Both processes result in a higher amount of fine fraction, which is an indicator for the breakability of SiC. This breaking and abrasion of silicon carbide particles during the wire-sawing process needs to be subject to further investigations.

Conclusion

Besides the carrier fluid, abrasives are a crucial component for the improvement of wire-sawing conditions and the subsequent production of high-quality wafers. However, to obtain such quality wafers (low TTV, low surface roughness) with high cutting rates (low vertical force per wire), a balance between slurries containing small and round particles with narrow distribution on the one hand and larger and sharp particles with narrow distribution on the other hand must be found. In fact, the 'narrowness' of the particle size distribution plays an important role in minimising the total slot width variation and therefore in achieving the goal of TTV less than 10% of the wafer thickness. Nevertheless, even the best silicon carbide particle suitable for multi-wire sawing shows its properties only after the recycling steps and usage in several wire-sawing runs.

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