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SLURRY SAWING OF MULTICRYSTALLINE SILICON WITH LOW-VISCOSITY CARRIER LIQUID

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ABSTRACT: The wafering of crystalline silicon is industrially realized by wire sawing with loose abrasive particles dispersed in a carrier medium (slurry process) or by fixed diamond particles embedded on the wire (diamond wire sawing - DWS). DWS demonstrates cost advantages by the usage of a cheap cooling fluid, which consists of water with about 2% of additives. In the slurry process, SiC particles are used as abrasive typically dispersed in polyethylene glycol (PEG200) as carrier liquid. A cost reduction of this process by saving electrical energy can be achieved by the usage of a lower-viscosity carrier liquid. In this contribution, PEG200 is substituted by the lower-viscosity organic substance propylene carbonate. In four consecutive sawing processes on multicrystalline silicon bricks, the cutting performance of this liquid was tested. The results are compared with the standard sawing process using PEG200. It was found that the average power of the wire saw main drive can be reduced by about 60%.

Keywords: Silicon, Wafering, Mechanical properties, Recycling

1 INTRODUCTION

Silicon wafers for photovoltaic applications are nowadays mainly produced by the so-called diamond wire sawing (DWS) process. Compared to the slurry process, which uses a free SiC particles dispersed in an organic carrier liquid, typically polyethylene glycol (PEG200), DWS offers cost advantages because water can be used as a cheap cooling liquid. Further advantages are the usage of diamond particles fixed on the wire, allowing for shorter process times and the availability of very thin wires (60-70 µm core diameter), which increases the productivity significantly. [1]

In this contribution, a measure to reduce costs in the slurry sawing process was tested. The standard carrier liquid PEG200 was substituted by a low-viscosity organic liquid. The results are relevant for remaining capacities in the photovoltaic wafer production that are based on the slurry sawing process as well as other industries like the microelectronic wafer production with a high share of slurry-based processes.

2 EXPERIMENTAL APPROACH

PEG200 has a dynamic viscosity of about 60 mPa·s at room temperature. On the one hand, this could be beneficial to produce wafers with a high quality, for example in terms of low thickness variations or a low number of saw marks on the wafer. On the other hand, with such high viscosity the shear stress in the sawing channel is increased leading to significant higher machine power consumption [2] and furthermore the cyclic pumping of PEG200 in the wire saw requires a significant amount of energy.

For the substitution of PEG200 with a low-viscosity cooling liquid, possible organics liquids were pre-evaluated in terms of chemical and physical properties, non-toxicity and reactivity with silicon.

Propylene carbonate (PC) was identified as an appropriate substance. PC has the sum formula $C_4H_6O_3$ and a molecular weight of 102 g/mol. Relevant properties of PEG200 and PC are compared in Table I. While the density of PC is slightly higher, the dynamic viscosity of 2.8 mPa·s is significantly lower compared to PEG200. The specific heat capacity of PC is lower, possibly affecting the temperature regulation of the slurry in the process.

Table I: Properties of polyethylene glycol (PEG200) and propylene carbonate

Liquid	**Density [kg·m^{-3}]**	**Dynamic Viscosity [mPa·s]**	**Specific heat capacity [kJ·kg^{-1}·K^{-1}]**
Polyethylene glycol (PEG200)	1.124	60-67	2.43
Propylene carbonate (PC)	1.205	2.8	1.71

Four consecutive sawing processes on multicrystalline silicon (mc-Si) were done using the same propylene carbonate based slurry. Bricks from the edge and center ingot positions were used. Selected process parameters are shown in Table II. A sawing process with the same parameters using PEG200 was used as the reference process.

The forces on the mc-Si ingot, the average power of the slurry saw main drive and the slurry temperature were measured during the processes. The resulting wafer quality was assessed in terms of total thickness variation (TTV) and roughness of the wafer surface.

Table II: Process parameters for the evaluation of propylene carbonate (PC) slurry

Parameter	**Value**
Wire	115 µm structured wire
SiC	F800
Slurry density at start	1.57 g·cm^{-3}
Wire speed	15 m·s^{-1}
Feed rate	0.6 mm·min^{-1}

3 RESULTS

3.1 Process results

For the evaluation of the sawing processes, the forces on the wire web have been measured in three dimensions throughout the whole process time. The force in the crystal feed direction (F_z) is a measure of the cutting efficiency; the force in wire direction (F_x) is proportional to the friction of the wire in the developing sawing channel. With a low-viscosity cooling liquid, a lower F_x is expected. As shown in Fig. 1, the force in wire direction is significantly lower when the PC slurry is used. Compared to PEG200 slurry, F_x is reduced by about 60%. Furthermore it can be seen that F_x increases in each subsequent process, which is due to the loading of the slurry with silicon kerf. In the fourth process, the maximum silicon load capacity is nearly reached, corresponding to 14 wt% silicon in the slurry at a density of 1.7 g/cm^3.

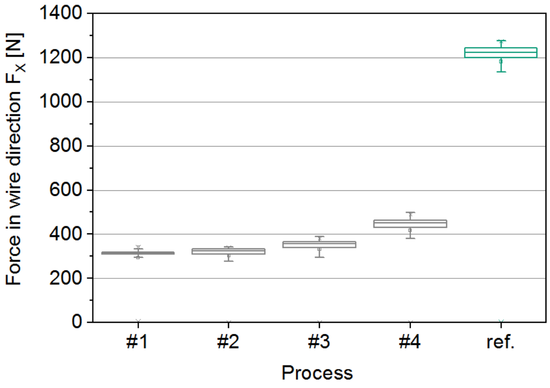

Figure 1: Force in wire direction (F_x) is significantly reduced with the PC slurry (processes #1 to #4) compared to the PEG200 slurry (reference)

A minor effect on F_z was observed, the force in feed direction with the PC slurry is about 10% higher than for PEG200 slurry, see Fig. 2. The higher F_z indicates a reduction of the cutting ability when PC slurry is used. An explanation can be found regarding the amount of free abrasive particles (SiC) that are transported into the sawing channel. With the lower viscosity it seems reasonable that a higher fraction of small SiC particles is transported by the wire into the sawing channel, excluding larger abrasive particles. Thus, the cutting ability is lowered as recognized by the increase of F_z. [3]

Within the four consecutive processes with PC, F_z does not clearly increase, which would be an indication for a reduction of the cutting ability. The observed variation in F_z is mainly due to the usage of bricks from different ingot positions that contain inclusions of silicon carbide and silicon nitride in different sizes and concentrations [4].

The lower friction force directly results in a significantly reduced average power of the wire saw main drive, see Fig. 3. In the first process with PC slurry, a reduction of 60% compared to the PEG200 slurry was found. The energy consumption increases with increasing silicon load of the slurry in the consecutive processes. The slurry density values measured at the end of each process support this result. While the density was 1.61 g/cm^3 after process #1, it was increasing to 1.71 g/cm^3 after process #4, which requires a higher pump performance.

It should also be mentioned that the slurry temperature was kept constant at 23°C in the PC processes; the lower Specific heat capacity did not affect the process stability.

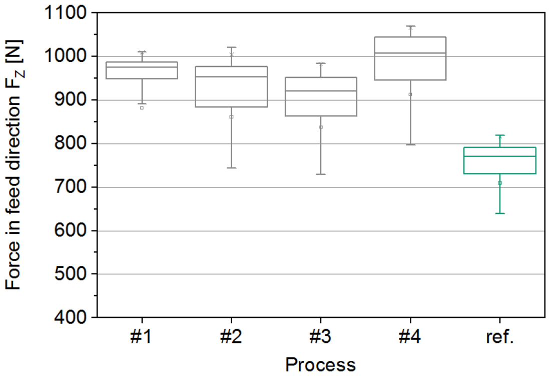

Figure 2: Force in feed direction (F_z) with PC slurry (processes #1 to #4) is slightly higher compared to the PEG200 slurry (reference), showing a minor reduction of the cutting ability

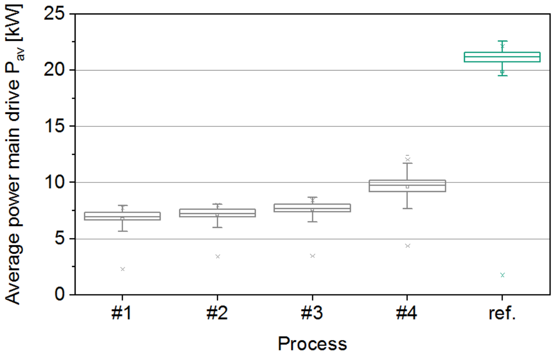

Figure 3: Average energy consumption of the wire saw main drive; a drastic reduction with the PC slurry (processes #1 to #4) is obtained in comparison to the PEG200 slurry (reference)

3.2 Wafer results

The wafers produced in the PC slurry processes were characterized in terms of geometry and roughness. The thickness of the PC slurry wafers exhibits median values of about 205 µm, which is equal to the wafers obtained from the PEG200 process, see Fig. 4.

However, the thickness of the PC slurry wafers shows a broader distribution, which is also reflected in the total thickness variation (TTV), see Fig. 5. Parameter optimizations were done within the four processes; with the parameter set used in process #3, the best wafer quality was obtained so far.

Further process optimization is necessary in order to produce wafers with a TTV of about 10 µm as demonstrated in the reference process with PEG200. Especially the slurry nozzle system needs to be adapted for the low-viscosity coolant.

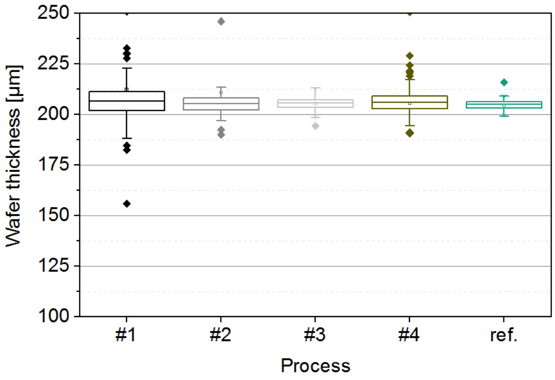

Figure 4: Wafer thickness obtained from four PC slurry processes and the PEG200 process as reference

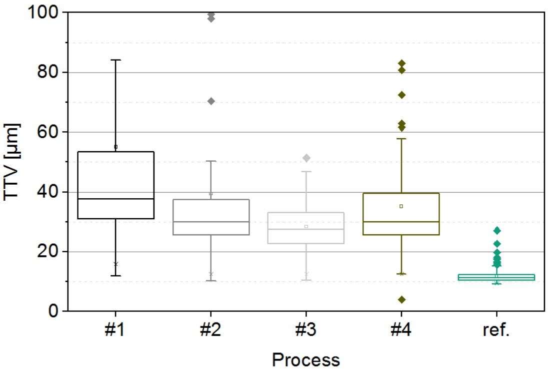

Figure 5: Total thickness variation for wafers obtained from four PC slurry processes and the PEG200 process as reference

As shown in Fig. 6, the roughness R_a of PC slurry-sawn wafers is about 15 % lower compared to wafers sawn with PEG200-based slurry, which could turn out to be beneficial for the mechanical strength of the wafers [5].

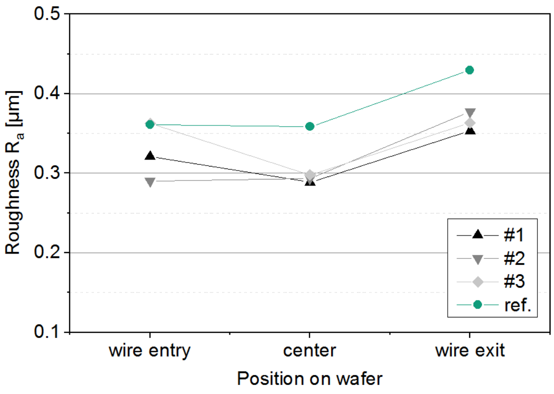

Figure 6: Roughness R_a at wire entry side, center and wire exit side of the wafer

The mechanical strength was determined by a ball on three ball (B3B) test on wafers of the same average thickness. The B3B test concentrates the load on the center of the wafer and excludes fracture effects on the wafer edges. Assuming a Weibull distribution of the fracture forces, the PC slurry sawn wafers exhibit a slightly higher critical fracture force of 3.68 N compared to 3.53 N for the PEG200 wafers, see Fig. 7. This result is in good agreement with the lower roughness of the PC-slurry sawn wafers.

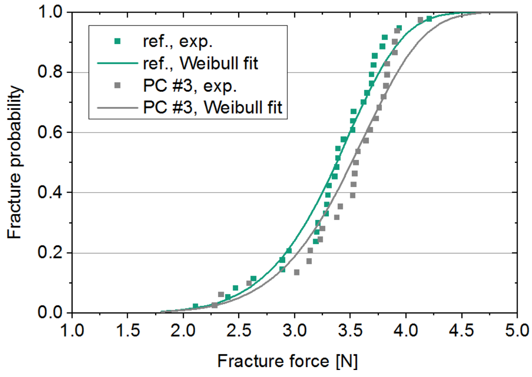

Figure 7: Weibull plots of the fracture forces in B3B test

4 RECYCLING OF USED SiC/PROPYLENE CARBONATE SLURRY

Propylene carbonate (PC) has been separated from solids by means of filtration. The moisture content of the used PC amounts to 1.8 wt%. While other carrying fluids, as for example PEG200, are prone to partial oxidation and formation of small amounts of carbon acids under conditions of the wafering process and, consequently, to higher corrosion rate of wire metals, PC remains completely stable.

This stability is reflected in the relatively high pH value (8.13) and low conductivity (13 µS/cm) of the used PC, as well as low amount of solved wire metals (Fe < 20 ppm, Cu < 5 ppm; Zn < 20 ppm). As the result, the recycling of PC is essentially simplified, limited to the filtration and drying to the required moisture content.

The recycling process of the SiC from the used propylene carbonate slurry is quite similar to the recycling of SiC from the conventional SiC/PEG200 slurry and comprises classification of particulate solids by means of hydro cyclones, as well as chemical removal of remained metals.

The yield of recycled SiC corresponded to 53.1%. There are indications of higher mechanical degradation of Si and SiC grains under conditions of wafering process in propylene carbonate, compared to PEG200, as shown in Fig. 8 by the shift of the particle size distribution after wafering to smaller particle sizes.

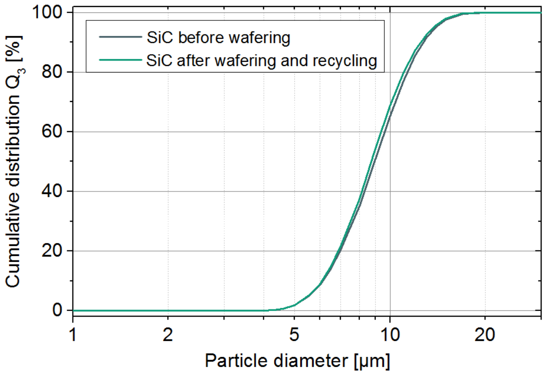

Figure 8: Particle size distribution of silicon carbide abrasives before wafering and after wafering/recycling

Beside the lower yield of the recycled SiC, this results in much lower retention rates of solids in the standard filtration step and in the higher content of fine Si particles in the underflow of the hydro cyclones, resulting in 1.7 wt% elemental silicon in the recycled SiC.

5 CONCLUSION

The substitution of the standard carrier liquid PEG200 in slurry sawing of crystalline silicon with a low-viscosity organic substance was investigated in this work. We found propylene carbonate (PC) to be a promising substance having a viscosity, which is reduced by a factor of 20 compared to PEG200.

In four consecutive sawing processes the suitability of propylene carbonate as carrier liquid was demonstrated. The lower viscosity resulted in a reduced force developing in wire direction and, consequently, in a reduction of energy necessary for the wire sawing process. The average power of the wire saw main drive could be reduced by about 60% compared to PEG200, which is a measure to reduce electrical energy costs.

Compared to wafers produced in slurry sawing processes with PEG200, wafers propylene carbonate based processes show the same thickness with a higher variation and thus with higher values of the total thickness variation. This may be improved by adapting process parameters as for example the slurry volume flow or the geometry of the slurry nozzles. Furthermore, a lower roughness and higher mechanical strength of the wafers produced with PC slurry were obtained. Although this is generally advantageous it is also a hint that the larger abrasive SiC particles are partially not transported into the sawing channel and thereby the cutting efficiency is slightly reduced. This was proved by an increase of the force measured in feed direction by about 10%.

Propylene carbonate remained absolutely stable during the wire sawing process which reduces the recycling procedure to filtration and drying steps. Advantageous is the content of dissolved metals in the used slurry. However, the recycling yield of SiC particles is lower compared to PEG200 which may be due to a higher mechanical degradation of SiC.

6 ACKNOWLEGMENTS

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7 REFERENCES

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