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A NOVEL APPROACH TO DETERMINE THE DIAMOND OCCUPANCY OF DIAMOND WIRES FOR OPTIMIZED CUTTING PROCESSES FOR CRYSTALLINE SILICON

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ABSTRACT: The production of wafers from crystalline silicon for solar cells is increasingly realized by diamond wire sawing. A steel core wire is used on which diamonds are fixed in a layer of nickel or resin. The cutting efficiency of the wire is determined by the interplay of the material properties of the crystal that is cut, the cooling fluid and the characteristics of the diamond grit on the wire. Hereby, the density of diamonds with respect to the wire length, the shape of the diamonds and the grain size distribution of the diamonds are important. A new approach is presented to remove diamonds from nickel bonded diamond wires and subsequently analyze their density and grain size distribution. The gravimetrically determined mass of diamonds on the wire shows the same trend as the number of diamonds counted manually on microscope images. Thus, a quality control of diamond wires prior to the wafering process as well as a characterization of the wire wear during the cutting process becomes possible with higher accuracy.
Keywords: Wafering, diamond wire, wire wear, silicon, characterization

1 INTRODUCTION

The wafering of silicon for photovoltaics is currently converted from slurry sawing to diamond wire sawing (DWS). While this technology transition is well advanced for monocrystalline silicon, the share of the DWS technology for the wafering of multicrystalline silicon is currently lower, which is partly due to the material properties of mc-Si. The cutting efficiency in DWS is determined by the properties of the material that is cut [1], the coolant that is used and in particular the wire, which has a distinct diamond occupancy.

A standard diamond wire for wafering consists of the wire core, the bonding layer and the embedded diamonds. The wire core is made from steel, core diameters in silicon wafering range from 60 to 120 µm. It is surrounded by the bonding layer with a thickness of 2 to 9 µm that is usually composed of nickel or resin with the abrasive particles, i.e. the diamonds, embedded. The number, the size and the distribution of the embedded diamonds can vary. Thereby the number of diamonds per meter wire length is called occupancy and has a major influence on the cutting speed and the resulting wafer surface quality, which is a topic of current research [2].

The size of the diamonds has a direct influence on the cutting efficiency. With up to 20% of the total cost the diamond wire also represents one of the main cost driving factors in the wafering process. Therefore it is important to get the diamond wire wear lifespan as close to the maximum as possible. Thus, it is important to know how variations of the coolant or material that is cut influence the diamond wire wear.

Therefore, a benchmarking of new diamond wires needs to include a detailed analysis of the diamond particle occupancy. Usually this is done by manually counting the number of diamonds on a microscopic image (see FIG. 1). Since this method uses only a limited wire length, it implies a high inaccuracy when predicting the diamond occupancy of the whole diamond wire with a length of typically 60 km on one wire spool.

Furthermore, it is not possible to accurately measure the size of the diamonds, since these are embedded in nickel (or resin) layers of varying thickness for different wire manufacturers. The SEM/EDX-images of wire cross sections in FIG. 2 illustrate this actuality.

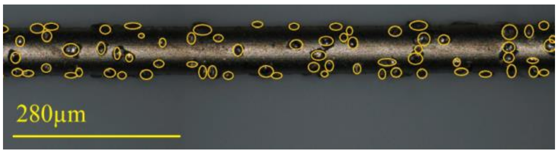

Figure 1: Manual counting of the diamond occupation in a microscope image on a wire length of 1 mm.

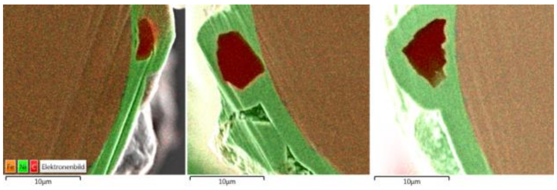

Figure 2: SEM/EDX-images of cross section of three different diamond wires. The varying thickness of the nickel layer (green) is demonstrated.

As can be seen in FIG. 3 the diamonds of wire #4 are located very close to each other appearing as conglomerates. However, the actual number of diamonds beneath the diamond wire coating can only be estimated and therefore an additional error in the inherently inaccurate counting method is bound to occur.

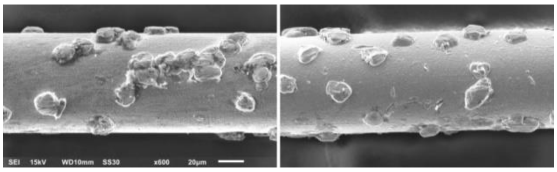

Figure 3: Comparison of SEM images of wire #4 (left) with apparent diamond conglomerates and wire #6 (right) with mostly separate diamonds.

In order to increase the accuracy of the diamond occupancy determination and thus enable for a prediction of the wire wear, a novel approach that offers a higher accuracy is shown in this work.

2 PROCEDURE

The new approach is to first remove the diamonds chemically from the diamond wire and to analyze them separately. First, the bonding nickel layer has to be dissolved from the steel core wire with a single etching step after an appropriate cleaning. It is important to dissolve the nickel layer without affecting the steel core. A mixture of HNO_3 (65%) and H_2O_2 (30%) has been proven to be successful. Due to the H_2O_2 the reaction rate is highly increased compared to when only using HNO_3.

FIG. 4 shows SEM and EDX images of an intact diamond wire before the etching, a diamond wire during the etching process, and a blank and intact diamond wire core after etching. After the etching process the resulting blank and intact core wire is extracted and discarded. By filtration of the thinned etching solution through a polycarbonate membrane with a low pore width (0.2 µm) the diamonds are caught on the filter membrane. The mass of the diamonds per meter of wire is then determined gravimetrically. If required the geometry of the diamonds on the membrane can be determined optically, e.g. with a laser confocal microscope.

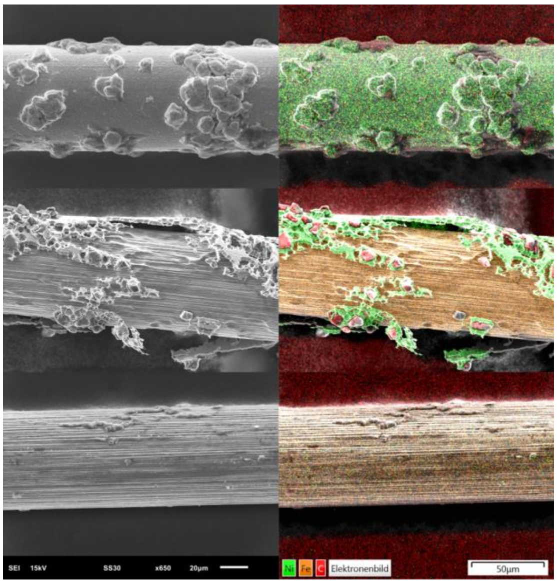

Figure 4: SEM (left) and EDX (right) images of a diamond wire before (top), during (middle) and after (bottom) the etching process.

Furthermore the grain size distribution of the diamonds on the wire can be determined by flushing the diamonds from the filter membrane. This is done by depositing the filter membrane in a centrifuge tube with ethanol and then applying a short ultrasonic treatment.

After the vaporization of the ethanol the diamonds are collected with a few drops of clean water and the grain size distribution is determined by laser diffractometry. By using the grain size distribution data and the gravimetrical data the total number of diamonds per meter wire length is calculated.

To be able to do the gravimetrical determination and the laser diffractometry even on used diamond wires from sawing processes or wires with a low diamond concentration it is recommended to use a diamond wire length of at least 3 - 10m.

3 RESULTS

3.1 Gravimetry and grain size distribution

Six diamond wires with a core diameter of 70 µm from different manufacturers were investigated. The etching solutions were filtered as described above and the masses of the diamonds were determined gravimetrically. Subsequently the grain size distribution was determined by laser diffractometry as shown for all six wire samples in FIG. 5.

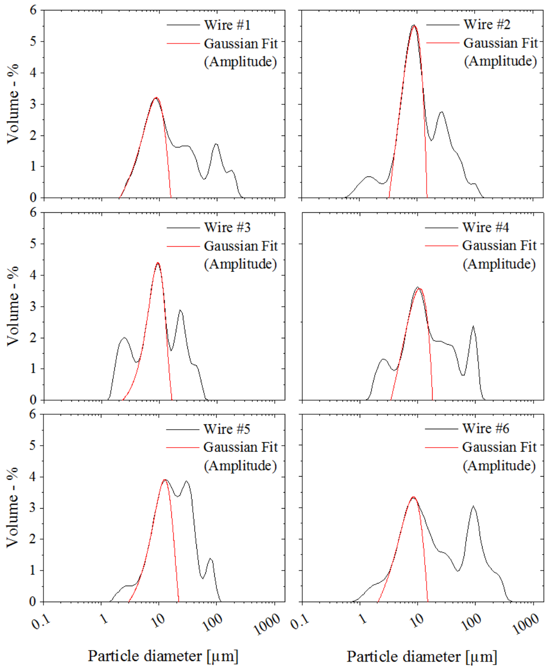

Figure 5: Grain size distribution of dissolute diamonds from six different wires. The peak related to the diamond particles is fitted with a Gaussian Amplitude function.

For comparison, the grain size distribution as measured with the same laser diffraction setup on a pure diamond powder is shown in FIG. 6.

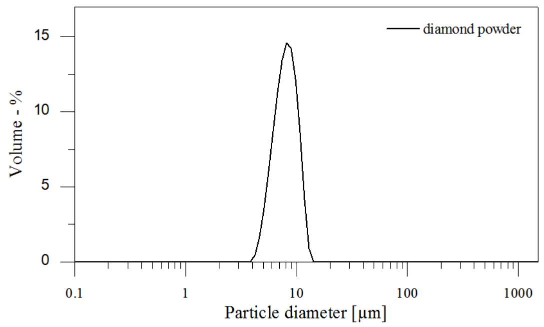

Figure 6: Grain size distribution of a pure diamond powder measured by laser diffractometry with a d50 value of 8.5 µm (specification: d50 = 8-9 µm).

The measured distribution shows only one sharp peak with a d50 value of 8.5 µm. This finding proves that the laser diffraction measurement is a reliable method to determine grain size distributions. In FIG. 5 it is obvious that further peaks next to the respective main peaks in the grain size distribution are present. Those peaks are supposed to be caused by metal residuals that are left in the etching solution and those should be dependent on the composition of the diamond wire coating. In addition the peaks at high particle diameters can be due to measurement artifacts, e.g. caused by bubbles in the measured solution.

The peak near the expected d50 value with respect to the manufacturers' specification was fitted with a Gaussian amplitude function. The d50 value of this peak is extracted and compared with the manufacturer specification in TAB. 1. The comparison shows that the measured and fitted d50 values are in good agreement with the given specifications of the different diamond wire manufacturers. The d50 value from the laser diffraction measurement is in all cases near the average of the distribution specified for the respective wire.

Table I: Measured d50 diamond diameter values compared to the specifications of the different wire manufacturers.

Diamond Wire Number	Manufacturer specifications [µm]	Measured d50 values [µm]
#1	6-12	8.9
#2	6-12	9.0
#3	6-12	9.4
#4	8-12	10.2
#5	8-16	13.3
#6	6-12	8.4

With the gravimetrically determined masses of the diamonds and the d50 values of the fits the number of diamonds per 10 m diamond wire length can be calculated assuming that the diamonds have a spherical shape. In FIG. 7 the so obtained values are compared to the values obtained by the original determination counting method. The depicted results of the different methods show a similar trend. However, the number of diamonds is systematically higher for the etching method. This should be due to the inherent inaccuracy of the manual counting method where only a side view of the diamond wire on a very short wire length is analyzed and then extrapolated. However with the above demonstrated new etching method the accuracy of the calculated number of diamonds per wire length is significantly increased by analyzing a total diamond wire length of 3-10 m.

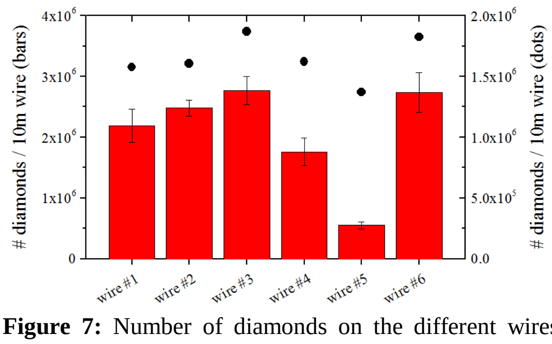

Figure 7: Number of diamonds on the different wires determined with the new etching method (bars) and the manual counting method (dots).

3.2 Analysis of diamond grain size distribution in microscope images

As a next step, the grain size distribution of diamonds can directly be measured in microscope images of the diamonds on the filter membrane. An exemplary image is shown in FIG. 8 for wire #1. The manual analysis of the diamonds present in the microscope image yields the grain size distribution in the right graph of FIG. 8 with a d50 value of 12.1 µm. This value is higher as measured by the laser diffraction method which may be due to the low number of diamonds that were counted. In the future a higher number of diamonds will be incorporated in the analysis by an automated counting of the diamonds in a set of microscope images.

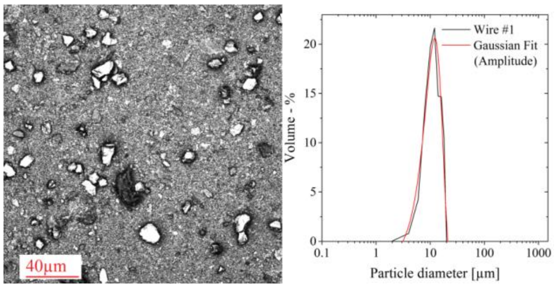

Figure 8: Laser confocal microscope image of diamond particles on a filter membrane extracted by dedicated etching of diamond wire #1 (left), the grain size distribution (right) shows a d50 of 12.1 µm.

3.3 Shape of diamond grains

As additional information the diamond laden filter membranes can be analyzed by laser confocal microscopy before the rinsing step with ethanol. Thus the shape of single diamond grains can be evaluated (see FIG. 9) and it could e.g. be analyzed if the shape of the diamond grains has an influence on the cutting efficiency and the resulting geometry and quality parameters of the wafer.

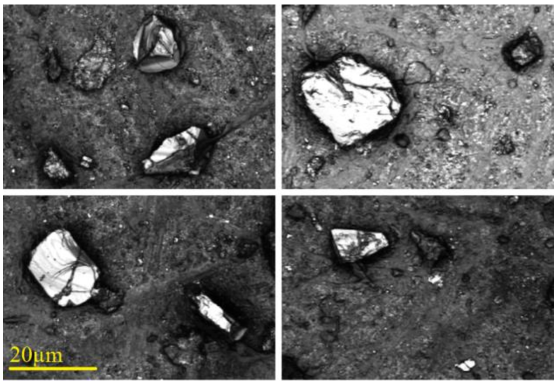

Figure 9: Laser confocal image of single diamonds on a polycarbonate membrane.

4 CONCLUSION

In this contribution, a new method was presented to extract diamonds from nickel coated steel wires used in multi wire sawing of crystalline silicon. The method was applied to state-of-the-art diamond wires with core diameters of 70 µm and diamond grit sizes of 6-12 µm up to 8-16 µm. The d50 values determined by laser diffraction are in good agreement with the mean values of the diamond grit sizes according to the manufacturers' specifications. It was also found that the gravimetrically determined mass of diamonds on the wire shows the same trend as the number of diamonds counted manually on microscope images but offers higher accuracy. A still existing difference of the methods will be investigated further by developing an automated counting of the extracted diamonds on the filter membrane and taking into account the shape of the diamonds.

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