Research on suppressing brittle fracture and implementing ductile mode cutting for improving surface quality at silicon wafers manufacturing

A M Kovalchenko¹*, E O Pashchenko² and D O Savchenko²

¹Institute for Problems of Materials Science, Kyiv 03142, Ukraine
²Institute of Superhard Materials, Kyiv 04174, Ukraine

E-mail: andrii.kovalchenko@gatech.edu

Abstract. Single crystal silicon is an important basic material used to manufacture electronic and photovoltaic devices. Ductile mode of diamond wire sawing is a promising method for silicon wafering in order to produce wafers with minimal surface damage. To achieve ductile mode, the correct applying of cutting parameters and careful wire design is necessary. This study investigates the scratching of monocrystalline silicon by the abrasive particles of different geometry, which simulates the material removal process in diamond wire sawing. Diamonds, crushed and spherical tungsten carbide (WC) particles served as abrasives. Experiments show that spherical abrasives enhance ductile mode cutting significantly decreasing brittle damage when compared to irregular shape particles. Spherical WC particles permit to increase the critical load and critical cut depth of ductile-to-brittle transition from 5 to 10 times. The depth of the damaged subsurface layer decreased from 5 µm to 0.2 µm due to the absence of brittle cracks. A uniform regular distribution and appropriate suitable density of abrasive particles is obligatory for cracking reduction. For that, the method of diamond particles uniform deposition with the controlled density by a polymer binder combining high modulus and adhesive capacity with good flexibility was elaborated. The method includes preliminary diamond particles fixation on a thin resin layer providing high uniformity and subsequent strong fixation by a thicker resin layer. The research on ovalization of diamond particles was performed for smoothening cutting edges. The method is based on the activation of the graphitization process at sharp edges of particles under the action of metal salts at increased temperatures.

1. Introduction
Diamond wire sawing (DWS) is the primary manufacturing process for silicon wafer production [1, 2]. The cutting parameters have a significant influence on wafers’ mechanical strength [3, 4] and the sawn surface quality [5-7] due to brittle nature of silicon resulting in increasing cracking during manufacturing. Effectiveness of the cutting process depends on speed, feed rate and tension of wire [5-7, 8-14]. Experimental results demonstrate [7, 8, 10, 13, 14] that more advantageous for obtaining minimal damage of sawn surface connected with mainly ductile material removal is a reduction of the workpiece feed speed and wire tension or increasing the saw wire movement speed. The formation of defects and value of the damage depth on the sawn surface depend on shape [7, 8, 13, 15-17] and size [5, 14] of diamonds as well as the distribution and density [7, 9, 14, 17, 18] of diamond particles on the wire. Sharper [15-17] and bigger particles [5, 14] generate more saw marks and micro-dents. The crack-free sawn surface is more likely to be obtained by increasing the abrasives density on the saw wire surface.
The condition of the cut surface of silicon depends on crystallographic orientation of the cutting direction [19] and crystalline defects such as dislocations and grain boundary [20]. Diamond grains during silicon cutting undergo micro-chipping, abrasion wear and some observed phase transformation on surface resulting in flattening (rounding) of the diamond grains leading to formation more smooth silicon surface [5, 21, 22]. However, sporadic pulled out diamond particles from cutting wire produce scratches and cavities on surfaces of the silicon wafer [6, 22]. Silicon surface damage diminution and increasing the extent of ductile mode cutting could be obtained by altering the cutting fluid properties due to lowering the friction coefficient and the chemo-mechanical effect of the cutting fluid on silicon, which lowers its hardness [15, 23, 24]. Ultrasonic vibration assisted wire sawing is more effective in reducing fracture damage and workpiece surface quality improvement compared with the conventional diamond wire sawing [18, 25]. Compared with electroplated diamond wire, resin-bonded diamond wire has the advantage in suppressing crack damage due to a more uniform protrusion distribution of abrasives and higher-elasticity of resin layer [26, 27].

The crack damage suppression is one of the important aspirations of various researches to minimize the subsurface damage [5, 14, 17, 28] and surface roughness [5-7] of the wire-cut silicon wafers which may be obtained by the realization of the ductile mode cutting [29]. The ductile cutting occurs when the local pressure at the cutting edge of the tool reaches the pressure of phase transformation (about 12 GPa) when the diamond cubic structure of silicon will transform to plastic metallic phase leading to ductile shear based material removal. Upon pressure unloading, metallic phase of silicon converts to amorphous phase. However, contact pressure more than 16 GPa will lead to brittle fracture. Thus, the processing span of ductile regime machining is rather marginal, and failure to control the process may result in catastrophic brittle fracture. To promote the ductile mode cutting is possible by using spherically tipped scribes or round particles [15, 16], which induce uniform cutting pressure and lower subsurface tensile stresses due to increased contacting cutting areas. The studies [5, 30] demonstrated that rounding and blunting of the sharp tips of the diamond particles on wire resulted in silicon surface morphology change from brittle fracture to ductile cutting. Our previous experiments [31, 32] showed that spherical abrasives enhance ductile mode cutting when compared to arbitrarily shaped particles, yielding a smoother surface with significantly fewer micro-cracks.

The purposes of the present studies are more deep figuring out the effect of different shape particles on ductile-to-brittle transition [15, 20, 33] at scratching [11, 15, 19, 34] which simulates the abrasive interaction in diamond wire sawing of silicon wafers (1); the elaboration of the method to deposit diamond particles on steel wire surface with uniform distribution and controlled required density (2); to study the ovalization (rounding) the diamond particles for blunting the sharp edges and corners of the typical arbitrarily shaped industrial diamond particles (3).

2. Abrasiveness of different diamond and tungsten carbide particles against single crystal silicon

The scribing experiments were performed by using two types of scratching machines; the first permits increasing scribing depth controlling vertical displacement and the second permits gradually increasing a normal load while both recording the depth and the friction force as a function of the displacement of a scribe. The abrasive particles were deposited using epoxy resin on a hardened steel ball (Ø 4 mm) made of bearing steel (AISI 52100, HRC 60) serving as a pin. Three types of abrasive particles were used in the experiments; diamond particles (1), crushed tungsten carbide (WC) particles (2) and spherical WC particles (3) ranging in the size from 7 to 50 µm (Figure 1). The particles were separated to five sized groups to observe a size effect (7-18 µm, 14-25 µm, 25-35 µm, 28-40 µm, 35-50 µm). The scribing was performed along the [110] crystallographic direction on the (100) crystallographic surface orientation of monocrystalline mirror-polished silicon wafer with a constant speed of 100 µm/s along an entire scribing length of 5 mm. Figure 1 shows representative images of the diamonds (a), spherical (b) and crushed (c) WC particles. After scribing, the resulting surface morphology was analyzed by optical, scanning electron microscopy (SEM) and micro-Raman spectroscopy.

The scratching experiments revealed that with the increase the normal load and scribing path, the fastest transition from ductile (without any cracks, only plastic silicon removal) to brittle mode scribing
was observed at scratching by diamond particles. The longest scribing distance and the biggest normal load at ductile-to-brittle transition are inherent to the spherical WC particles. Figure 2a demonstrates the typical single brittle scratch after passing a transitive point of ductile cutting, which is characterized by absolute absence of brittle damage and divergent cracks around all scratches formed in ductile mode (Figure 2b). The moment of the ductile-to-brittle transition were easily recognizable due to typical cracking noise appearance and the location of that transitive point was discovered by the presence of the first appearing cracks around the scratch as it is shown in Figure 2c. Such difference in the abrasive action of different kind of particles is explained that the sharp edges and corners of diamond and, in slightly less extend, crushed WC particles (Figures 1a and 1c) cause the big contact pressing (above 20 GPa) resulting in brittle damage initiation at lower load. In contrast, WC spherical particles (Figure 1b) smoothly touch silicon in extended contact areas providing appropriate range of contact pressure favorable for phase transformation of silicon from the crystalline to metallic and amorphous phase (around 12 GPa). According to Raman spectroscopy, the inner area of scratches is amorphous.

**Figure 1.** SEM images of diamonds (a), spherical (b) and crushed (c) WC particles (28-40 µm).

The decrease of the ductile-to-brittle transition depth with the increase of the particles size is observed for all type of particles. The smaller particles contacted silicon in multiple points, and hence reduced contact pressure when normal load increased with increase in scribing depth. The results demonstrate the feasibility of the spherical abrasive particles application for providing ductile mode of wire sawing of silicon.

**Figure 2.** SEM image of the typical brittle scratch (a), optical images of the wholly ductile scratches (b) and SEM image of the ductile scratch in the point of ductile-to-brittle transition (c).

### 3. Diamond particles deposition on the steel wire surface

The uniform distribution of particles on the surface of the wire is required to minimize the defectness and roughness of the silicon cut surface. At present, two methods are used for applying diamond particles to a wire surface. These are the galvanic method and the extrusion of a mixture of diamond powder with polymer melt. In the case of the galvanic fixing, the particles suspended in an electrolyte medium move
to the surface both under the action of gravitational forces and under the action of an ion flux stimulated by an electric field. After the initial acts of fixing some particles at random points on the surface of the wire (or another tool body of arbitrary shape), a local increase in the field strength occurs at these points. The density of ions flux increases correspondingly. Due to ions flux transports particles toward the steel surface, the probability of the next particle fixation is increasing in the vicinity of the previously fixed particle. The repulsive forces between the particles are due to the presence of adsorbed ions of the same name on their surface cannot resist forces that promote aggregation of abrasive particles on the surface. The action of the mentioned mechanism is further enhanced by the large curvature of the surface on which the particles are deposited. Additionally, with galvanic fixing is extremely difficult to exceed the maximum surface density of the fixed particles, dictated by their size and geometry. In the case of the extrusion a polymer melt containing abrasive particles onto a wire surface, the formation of aggregates of particles on the surface can be prevented by uniform mixing of the particles with the binder melt and the optimal selection of technological parameters of extrusion. It is possible, in some extent, increasing the density of particles on the surface of the wire by increasing their concentration in the melt. However, many particles located in the polymer layer do not have direct contact with the surface of the wire. As far as the rigidity of polymer bond is much less than the rigidity of a steel wire or the cut silicon, the contribution of such particles to the cutting process will be significantly less than the contribution of grains having hard contact with the surface of the steel wire. In both cases, the placement and fixing of particles with the formation of a cutting tool occurs simultaneously.

The third approach providing a significant decrease in the tendency of particles to dense aggregation involves the separation of the stage of primary fixing the particles on a thin (about 0.1 of diamond particle diameter) layer of a viscous binder providing a uniformity and controlled density and the stage of final fixing. The primary step should be applying of abrasive particles from the suspension. The obvious choice of disperse medium for this operation is water. It was found that to balance the forces acting on a thin viscous layer of the binder from the side of the wire and from the side of the particle it is not enough to provide the same angle of wetting by the binder of the surfaces of steel and diamond. Even in the case of perfectly balanced wetting, the difference in curvature between the wire and the particle leads to a local redistribution of the ligament, contributing to the aggregation of particles on the surface of the wire.

The best conditions for reducing aggregation of abrasive particles on a substrate arise when the surface of the steel substrate and the surface of the diamond particles have an equal lyophilicity coefficient. The lyophilicity coefficient $\beta$ determines as the ratio between the heat of wetting a given surface with water to the heat of wetting the same surface with benzene ($\beta = Q_{\text{H2O}}/Q_{\text{C6H6}}$). The equality of the lyophilicity coefficients of the surface of diamond particles and steel wires can be ensured by modifying their surfaces. In this case, a pair of capillary forces acting on a viscous binder from the side of the wire surface, on the one hand, and from the side of the diamond surface, on the other, is additionally balanced by the third force, which tends to hold the diamond particles in an aqueous dispersion medium. This allows us to level the effect of the difference in the curvature of the wire and particles on their interaction with a viscous bindle. The second condition for creating a negative feedback between the local surface particles density and the probability of adding the next particle to an adjacent group is the creation of a weak repulsion between the particles. This problem was solved by treating the modified diamond powder with a dilute solution of ammonium or phosphonium base. At the same time, an adsorbed layer of likely charged ions is created on the surface of each particle, which provides weak repulsive forces between them. The visual information about the difference in the structure of ensembles of particles formed at different ratios of the lyophilicity coefficients of diamond and steel with modified surfaces is presented in Figure 3.
Figure 3. Schematic diagram of diamond particles distribution on steel wire surface (a-c) with different ratios of the lyophilicity coefficients of diamond and steel, and corresponding SEM images (d-f) of the steel surfaces with deposited diamond particles.

The method for manufacturing a cutting wire with uniform diamonds distribution and their desired density includes the following stages:

1. Preparation of water-based suspension of diamond powder. The diamond particles surface should be preliminary modified to obtain an equality of lyophilicity coefficients of diamond and steel ($\beta_{\text{diamond}} = \beta_{\text{steel}}$). The surface of the steel wire was treated by a solution of silicon organic compound which permits to create of a certain predetermined relation of wetting angles by water and benzene. In its turn, the surface of diamond particles was treated by another silicon organic compound with different lengths of attached organic chains. The choice of silicon organic compounds is determined by the necessity of the unification of surface properties of the steel and diamonds. The concentration of diamonds in suspension will be determinative for the required diamond density.

2. A deposition a thin layer of resin having a notably smaller thickness than diamonds mean size (not more than 10 %) on the steel wire core enabling positioning of diamond particles.

3. Dipping the steel wire core covered by the first layer of resin in the diamonds based suspension.

4. A polymerization of the first binder layer for initial fixing the diamond particles.

5. A deposition a second layer of resin enabling strong retention of the diamond particles onto the surface of the steel wire core in process of abrasive cutting.

6. A polymerization of the second binder layer for the final strong fixation of the diamond particles.

4. Ovalization of diamond particles

The predominant approach to an ovalization of diamond micro-powders is to use self-abrasion of the powders when they move in rotating devices or in gas stream, however this method is applicable only for the diamonds with dimensions more than 60 µm. Our alternative approach, just for particles less than 10 µm, is a heat treatment of diamond powders in media of metal salts that increase chemical reactivity and initiate the graphitization process. Diamond micro-powders are obtained by crushing larger fractions. The sharp edges of the micro-powder particles correspond to the intersection of cracks formed in the
larger particles during crushing. Such an intersection usually occurs in the area of accumulation of dislocations or other types of defects in diamond crystals. As a consequence, in the powder particles obtained by crushing, the vicinity of the sharp edges is more reactive than the volume of the particles on average. The processing of diamond micro-powder with melts of certain combinations of metal salts containing components of variable valence was studied. It was found that, at a certain temperature, selective graphitization occurs, localized at the edges of the diamond particles. Subsequent processing of diamond micro-powder with oxidizing agents makes it possible to obtain an ensemble of grains with blunt cutting edges. The removal kinetics of the graphitized micro-volumes of diamond particles localized at sharp edges was controlled by high temperature gas analysis. That permits to avoid the excessive graphitization and to prevent undesirable further oxidation. Figure 4 demonstrates a bluntness of the cutting edges of diamond particles after the removal of graphitization products.

![Figure 4. SEM images of some diamond particles (a-c) after ovalization.](image)

5. Conclusions
(1) Spherical particles promote the ductile mode of silicon cutting at higher cutting depth and normal load permitting to avoid brittle fracture in comparison with arbitrary particles. Spherical WC particles permit to increase the critical load and critical cut depth of ductile-to-brittle transition from 5 to 10 times. The depth of the damaged subsurface layer decreased from 5 µm at brittle mode cutting to 0.2 µm at ductile mode cutting due to the absence of brittle cracks.
(2) The method of diamond particles deposition on steel surface with uniform distribution and controlled required density was elaborated. The method includes preliminary diamond particles fixation on a thin resin layer providing high uniformity and subsequent strong fixation by a thicker resin layer.
(3) The method of ovalization of diamond particles basing on the graphitization process was elaborated. The method is based on the activation of the graphitization process under the action of metal salts at increased temperature.

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References
[1] Wu H 2016 Wire sawing technology: a state-of-the-art review Precis. Eng. 43 1-9
[2] Kumar A and Melkote S N 2018 Diamond wire sawing of solar silicon wafers: a sustainable manufacturing alternative to loose abrasive slurry sawing Procedia Manuf. 21 549-66
[3] Carton L, Riva R, Nelias D, Fourmeau M, Coustier F and Chabli A 2019 Comparative analysis of mechanical strength of diamond-sawn silicon wafers depending on saw mark orientation, crystalline nature and thickness Sol. Energy Mater. Sol. Cells 201 110068
[4] Sekhar H, Fukuda T, Tanahashi K, Takato H, Ono H, Sampei Y and Kobayashi T 2020 Mechanical strength problem of thin silicon wafers (120 and 140 µm) cut with thinner diamond wires (Si kerf 120→ 100 µm) for photovoltaic use Mater. Sci. Semicond. Process. 119 105209
[5] Suzuki T, Nishino Y and Yan J 2017 Mechanisms of material removal and subsurface damage in fixed-abrasive diamond wire slicing of single-crystalline silicon Precis. Eng. 50 32-43
[6] Ozturk S, Aydin L and Celik E 2018 A comprehensive study on slicing processes optimization of silicon ingot for photovoltaic applications Sol. Energy 161 109-24

[7] Pala U, Kuster F and Wegener K 2020 Characterization of electroplated diamond wires and the resulting workpiece quality in silicon sawing J. Mater. Process. Technol. 276 116390

[8] Xiao H, Wang H, Yu N, Liang R, Tong Z, Chen Z and Wang J 2019 Evaluation of fixed abrasive diamond wire sawing induced subsurface damage of solar silicon wafers J. Mater. Process. Technol. 273 116267

[9] Li X, Gao Y, Ge P, Zhang L and Bi W 2019 The effect of cut depth and distribution for abrasives on wafer surface morphology in diamond wire sawing of PV polycrystalline silicon Mater. Sci. Semicond. Process. 91 316-26

[10] Costa E C, Xavier F A, Knoblauch R, Binder C and Weingaertner W L 2020 Effect of cutting parameters on surface integrity of monocrystalline silicon sawn with an endless diamond wire saw Sol. Energy 207 640-50

[11] Wang B, Melkote S N, Saraogi S and Wang P 2020 Effect of scratching speed on phase transformations in high-speed scratching of monocrystalline silicon Mater. Sci. Eng. A. 772 138836

[12] Wang B, Melkote S N, Wang P and Saraogi S 2020 Effect of speed on material removal behavior in scratching of monocrystalline silicon Precis. Eng. 66 315-323

[13] Huang W and Yan J 2021 Fundamental investigation of diamond cutting of micro V-shaped grooves on a polycrystalline soft-brittle material J. Manuf. Mater. Process. 5(1) 17

[14] Yin Y, Gao Y, Wang L, Zhang L and Pu T 2021 Analysis of crack-free surface generation of photovoltaic polysilicon wafer cut by diamond wire saw J. Mater. Process. Technol. 221 109674

[15] Wu H and Melkote S N 2012 Study of ductile-to-brittle transition in single grit diamond scribing of silicon: application to wire sawing of silicon wafers J. Eng. Mater. Technol. 134(4) 041011

[16] Kumar A, Melkote S N, Kaminski S and Arcona C 2017 Effect of grit shape and crystal structure on damage in diamond wire scribing of silicon J. Am. Ceram. Soc. 100(4) 1350-9

[17] Wallburg F, Kuna M, Budnitzki M and Schoenfelder S 2020 Experimental and numerical analysis of scratching induced damage during diamond wire sawing of silicon Wear 454 203328

[18] Wang Y, Li D L, Ding Z J, Liu J G and Wang R 2019 Modeling and verifying of sawing force in ultrasonic vibration assisted diamond wire sawing (UAWS) based on impact load Int. J. Mech. Sci. 164 105161

[19] Wu H and Melkote S 2012 Effect of crystallographic orientation on ductile scribing of crystalline silicon: role of phase transformation and slip Mater. Sci. Eng. A. 549 200-5

[20] Wu H and Melkote S N 2013 Effect of crystal defects on mechanical properties relevant to cutting of multicrystalline solar silicon Mater. Sci. Semicond. Process. 16(6) 1416-21

[21] Kumar A and Melkote S N 2018 Wear of diamond in scribing of multi-crystalline silicon J. Appl. Phys. 124(6) 065101

[22] Knoblauch R, Boing D, Weingaertner W L, Wegener K, Kuster F and Xavier F A 2018 Investigation of the progressive wear of individual diamond grains in wire used to cut monocrystalline silicon Wear 414 50-8

[23] Kumar A and Melkote S N 2017 The chemo-mechanical effect of cutting fluid on material removal in diamond scribing of silicon Appl. Phys. Lett. 111(1) 011901

[24] Yao C, Chen D, Xu K, Zheng Z, Wang Q and Liu Y 2021 Study on nano silicon carbide water-based cutting fluid in polysilicon cutting Mater. Sci. Semicond. Process. 123 105512

[25] Yan L, Wang Q, Li H and Zhan Q 2021 Surface generation mechanism of ceramic matrix composite in ultrasonic assisted wire sawing Ceram. Int. 47(2) 1740-9

[26] Ge M, Wang P, Bi W and Ge P 2021 Fabrication of thin resin-bonded diamond wire and its application to ductile-mode wire sawing of mono-crystalline silicon Mater. Sci. Semicond. Process. 126 105665

[27] Liu T, Ge Pand Bi W 2021 The influence of wire speed on phase transitions and residual stress in single crystal silicon wafers sawn by resin bonded diamond wire saw Micromach. 12(4) 429
[28] Li H, Yu X, Zhu X, Jin C, Zhou S and Yang D 2021 A microscopic TEM study of the defect layers in cast-mono crystalline silicon wafers induced by diamond-wire sawing AIP Adv. 11(4) 045103
[29] Kovalchenko A 2013 Studies of the ductile mode of cutting brittle materials (A review) J. Superhard Mater. 35(5) 259-76
[30] Kumar A, Kaminski S, Melkote S and Arcona C 2016 Effect of wear of diamond wire on surface morphology, roughness and subsurface damage of silicon wafers Wear 364 163–8
[31] Kumar A, Kovalchenko A, Pogue V, Pashchenko E and Melkote S N 2016 Ductile mode behavior of silicon during scribing by spherical abrasive particles Procedia CIRP 45 147-50
[32] Kovalchenko A M, Goel S, Zakiev I M, Pashchenko E A and Al-Sayegh R 2019 Suppressing scratch-induced brittle fracture in silicon by geometric design modification of the abrasive grits J. Mater. Res. Technol. 8(1) 703-12
[33] Veerabagu A, Alreja C and Subbiah S 2020 Ductile-brittle transition detection in scratching of single crystal silicon using charged particle emissions Procedia CIRP 87 378-84
[34] Budnitzki M and Kuna M 2019 Scratching of silicon surfaces Int. J. Solids Struct. 162 211-6