Study on surface integrity and ductile cutting of PV polycrystalline silicon and wear mechanisms of electroplated diamond wire

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Abstract
This study aimed to evaluate and better understand the mechanical and crystalline responses of polycrystalline silicon sawn by diamond wire sawing. To simplify the multi-wire sawing kinematic, an endless wire saw with a single looped diamond wire welded was used. The wire cutting speed and feed rate were varied in order to evaluate the characteristics of surface morphology, surface roughness, and subsurface damage. The analysis of brittle-ductile transition and residual stress of the sawn surface and silicon chips were performed with Raman spectroscopy. The wear and failure mechanism of the diamond wire were analyzed. The results showed that sawn surface is composed of brittle and ductile regions and the predominance of one of these directly affected the surface roughness \( R_a \). The ductile cutting mode induced the predominance of microgrooves and ploughing over the sawn surface and led to formation of an amorphous layer with residual compressive stress around \( -192.3 \) MPa. Micro-cracks in subsurface were identified and it reached a minimum depth of \( 7.2 \pm 1.6 \) µm. Chip fragments and elongated chips were observed and latter is practically amorphous. The wire wear analysis indicated that during the cutting there is deformation of the Ni-layer, exposed grits, and grit pullout. The main wear mechanisms are Ni-matrix removal and abrasive wear of the diamond grit. A better surface quality of polycrystalline silicon was obtained on increasing wire cutting speed and decreasing feed rate. Thus, the looped diamond wire sawing allows to reach a high surface quality of the silicon wafer, since the material removal occurs more in ductile cutting mode, generating a smooth surface with shallow subsurface damage.

Keywords Diamond wire · Surface quality · Brittle-ductile transition · Diamond wear · PV silicon wafer

1 Introduction

In recent decades, there has been increased concern regarding environmental protection due to a reduction in reserves of conventional energy sources and a high level of \( \text{CO}_2 \) emissions. This has led to a growing demand for clean energy and has encouraged many researchers to focus effort on making solar energy a viable renewable energy option [1, 2]. Despite the advantages of solar energy, the high cost associated with solar cell manufacturing is one of the main factors that limits its growth. Several steps are involved in solar cell processing and Peguiron et al. [3] reported that wafer cutting by multi-wire sawing accounts for around 26% of the processing cost and the crystalline silicon ingot, the main substrate used to produce the wafer, represents 80% of the total cost.

The main cutting process for silicon wafer manufacturing is multi-wire sawing based on a loose abrasive slurry and a steel wire, but this is gradually being replaced by the use of a wire with fixed diamond grits [4]. This approach is associated with a higher material removal rate, lower cost per wafer, and better surface quality, besides being environmentally friendly [5, 6]. However, crystalline silicon has the typical characteristics of a brittle material and thus damage to surface integrity of the wafer, such as surface
roughness, fractures, scratches, micro-cracks, residual stress, and so on, is one of the most serious issues arising during the processing [7]. The silicon wafers must comply with stringent requirements of surface integrity to ensure the performance and lifetime of a solar cell. Thus, there is great interest in better understanding the phenomena involved in the material removal and the influence of the cutting parameters during silicon cutting by diamond wire sawing. This can be useful for optimization of the multi-wire sawing process aimed at reducing the cost per crystalline silicon wafer [8].

Theoretical and experimental approaches have been studied to improve our understanding of the silicon surface and subsurface characteristics formed during machining processes. Li et al. [9] established a mathematical model to predict the surface roughness of polycrystalline silicon sawn by diamond wire sawing. Considering a random distribution of the characteristic abrasives, their position over the wire, and brittle and ductile modes during the cutting, the model was able to predict the surface roughness value obtained on varying the cutting parameters and they have validated on experiments under $v_c \leq 1.2$ m/s and $v_f \leq 0.75$ mm/min. Chung and Le [10] studied the depth of cut per diamond grit in the diamond wire sawing process, based on polar coordinates to identify the grit position for the generation of a wire profile. Different depths of cut were observed due to the random diamond distribution, which led to both brittle and ductile cutting modes. Pu et al. [11] used a slicing process to cut polycrystalline silicon, combining the diamond wire with slurry SiC abrasive sawing under $v_c \leq 1.2$ m/s and $v_f \leq 0.9$ mm/min. The novel slicing process reduced the saw marks, the main characteristic of diamond wire sawing; however, more craters and pitting took place over the sawn surface. High surface roughness values were observed on increasing the fraction mass of free SiC abrasives.

Würzner et al. [12] studied the effect of $v_c \leq 15$ m/s on the sawn surface of polycrystalline silicon, which revealed the presence of both tensile and compressive residual stress as well as an inhomogeneous surface roughness when using a high $v_c$. Costa et al. [13] showed that silicon sawn by electroplated diamond wire sawing undergoes phase transformation on applying $v_c \geq 10$ m/s. It was revealed that the intensity and bandwidth of the a-Si phase increases gradually in the $v_c$ range from 10 to 20 m/s. Yin et al. [14] showed that when the ratio between $v_f$ and $v_c$ is constant (i.e., when these two velocities are varied proportionally), there is no effect on the surface morphology and surface roughness in the slicing of polycrystalline silicon. Li et al. [15] numerically simulated diamond wire sawing to study the brittle and ductile modes and average penetration depth of the grits. The authors showed that the number of grits acting in ductile mode is less than 10% under the cutting conditions simulated with $5 \text{ m/s} \leq v_c \leq 10$ m/s, but it was not evaluated on experimental tests. Yin et al. [16] proposed a prediction model for sawing of poly-Si wafer with crack-free surface. They simulated the sawing under $v_c$ from 20 to 50 m/s. However, the experiments were performed on reciprocating sawing with limited cutting condition of $v_c \leq 1.3$ m/s and the subsurface damage induced by the diamond wire sawing was not analyzed.

Another aspect that can influence the sawed surface is diamond wire wear, as reported by the Knoblauch et al. [17] and Pala et al. [18] which showed the wear mechanisms on tracking single diamonds grits during the sawing of silicon, without analyzing the surface integrity of the silicon wafer. Kumar and Melkote [19] evidenced a higher scratch force and greater wear of the diamond indenter during the scratch test of polycrystalline silicon compared with monocrystalline silicon. Moreover, Goel et al. [20] reported that the inhomogeneity of the polycrystalline silicon favors the chip formation and removal compared with monocrystalline silicon. The authors revealed a disordered local structure at cutting zone which suggested high-pressure phase transformation (from Si-I to Si-II phase) and subsequent amorphization of the machined surface of poly-Si.

With regard to the surface characteristics of silicon wafers induced by diamond wire sawing, Chen et al. [21] noted that the saw marks on the polycrystalline silicon are difficult to remove by the conventional texturing process. Niu et al. [22] added that the amorphous phase and fragile damage on the sawn surface of the polycrystalline silicon affect the texture etching efficiency and photoelectric conversion efficiency. Therefore, the cutting parameters need to be optimized in order to produce silicon wafers with a sawn surface that has smaller fractured regions, less amorphous layers and residual stress, lower surface roughness, and minimal subsurface damage depth.

In this sense, this study aims to evaluate and understand the mechanical and crystalline responses of solar-grade polycrystalline silicon sawn using electroplated diamond wire sawing process for photovoltaic application. To suppress the complex cutting kinematic of the multi-wire sawing that presents multiples cycles of cutting due to the many stages of acceleration and deceleration for reversion of the wire movement and larger length of diamond wire, the present paper has employed a continuous cutting movement using a diamond wire butt-welded into a loop. This kinematic presents only one degree of acceleration and deceleration in cutting cycle profile, which allows to reach high $v_c$ and ensures a constant $v_c$ and $v_f$ during all cutting time, without reversion of the diamond wire.

The effect of the variations in the cutting parameters of high wire cutting speed ($v_c$) and feed rate ($v_f$) of the endless diamond wire sawing process was studied. The sawn polycrystalline silicon specimen was analyzed to determine the surface morphology, brittle-ductile transition, and
residual stress using micro-Raman spectroscopy, surface roughness, subsurface damage, chip morphology, and wire wear. The results obtained in this paper revealed the main phenomena involved during diamond wire sawing of polycrystalline silicon and the cutting conditions that allows to achieve high quality of the silicon wafer, being useful for optimization approach of the wafering processing in photovoltaic industry.

2 Materials and methods

2.1 Machine tool

The experiments were carried out on an endless wire saw machine (see Fig. 1) based on aerostatic technology, which ensures low friction on bearing and sliding. The test rig allows a continuous cutting movement using a single looped wire wrapped on pulleys that are fixed on aerostatic bearings. The Detail A in Fig. 1 shows the kinematic of the cutting process used in this study and the cutting parameters of wire cutting speed ($v_c$), feed rate ($v_f$), and wire tension, which are defined by the operator. A three-phase motor (370 W) powers the pulley A-axis and a frequency inverter is used to set the $v_c$ up to 26 m/s. The poly-Si workpiece is fed against the diamond wire on perpendicular movement to the vector of $v_c$. The feed movement occurs along the $Y$-axis on an aerostatic slide and it is driven by a step motor/ball bearing system using a control interface to set the $v_f$. A pneumatic cylinder is used to set the wire tension, which is the axial force acting on the diamond wire. The deflection of a spiral spring is controlled by the air pressure, which ensures a constant wire tension in cutting ($X$-axis). The specimen thickness is set by adjusting a micrometric screw ($Z$-axis).

Figure 2 shows the profiles of $v_c$ and $v_f$ when performing on unidirectional and continuous cutting movement with diamond wire into a loop. By employing the endless wire sawing, it can be ensured that $v_c$ remains constant during the cutting of the silicon, since the cutting cycle time ($t_{cy}$) have only one degree of acceleration ($t_a$) and deceleration ($t_d$). In this sense, it can reach a high $v_c$ and a more stable wire tension, increasing the stability of the cut and leading to predominance of material removal by a ductile cutting mode, that is an important factor to ensure high surface quality of silicon wafer.
2.2 Cutting tool and workpiece

A commercial Ni-electroplated diamond wire, manufactured by Norton Saint-Gobain Abrasives, was used as the cutting tool. The surface of the diamond wire is shown in Fig. 3a. The wire has an outer diameter of 350 µm and the diamonds of 30–45 µm are fixed on surface of a steel wire by electroplating using Ni as the layer coating. For the experiments, a diamond wire with a length of 1 m was used. To perform the continuous cutting movement, the diamond wires were butt-welded employing a diamond wire welding device and a welding procedure developed and validated for this purpose [23], which is shown schematically in Fig. 3b.

The procedures of diamond wire preparation and butt-welding process involve the following steps: (i) the cut-off of a short length of a commercial diamond wire; (ii) the sanding of the wire ends to provide flatness, parallelism, and smoothness of abutting surfaces; (iii) fixation of the wire on clamping die to ensure alignment of the abutting surfaces; (iv) an electrical source is used to apply a current (A) with time controlled, and then butt-welding and subsequent heat treatment of the welded joint is provided by Joule effect and force; and (v) the welded joint is sanded in order to remove the burr formation in the welding region.

The workpiece used in the sawing experiments was a polycrystalline silicon block with a parallelepiped geometry and dimensions of 25 mm × 50 mm × 7 mm. The polycrystalline silicon blocks, with a purity of 99.9999999%, typically used for photovoltaic application, were manufactured following a new metallurgical route of refining, purification, and solidification of solar-grade polycrystalline silicon. The workpieces were provided by a partner laboratory at the Institute of Technological Research—São Paulo (IPT-SP).

2.3 Experimental setup

The cutting parameters varied with the wire cutting speed \( v_c = 10, 15, \) and 20 m/s) and feed rate \( v_f = 0.3, 0.5, \) and 0.7 mm/s). The tension of the diamond wire was maintained constant at 20 N. All polycrystalline silicon specimens were sawn with a thickness of 500 µm. Following a \( 3^2 \) full factorial design, each experiment was performed three times under same cutting condition, which resulted in a total of 27 cutting tests. For each cutting experiment, a new (unworn) diamond wire was used to avoid the effect of cutting tool wear on surface integrity.

2.4 Characterization method of the surface integrity and performance of diamond wire

The sawn surface morphology was characterized by scanning electron microscope (SEM) (TESCAN, model VEGA3) to investigate the mechanism of material removal from the sawn surface of the polycrystalline silicon specimens. A micro-Raman microscope (Renishaw®, model 2000), coupled with an Ar-laser (wavelength of 514.2 nm) and optical lens (500×), was employed to identify the phase transformation and to confirm the hypothesis regarding the mechanism of material removal. The residual stress was calculated based on the shift of the Si-I phase peak on the Raman spectra, following the method proposed by Weinstein and Piermarini [24]. The reference value of 521 cm\(^{-1}\) for the Si-I phase was used and Eq. (1) was applied.

\[
\omega = \omega_o + 0.52 \times P
\]

where \( \omega \) (cm\(^{-1}\)) is the spectral value for the Si-I phase, \( \omega_o \) (cm\(^{-1}\)) is the reference spectral value for the Si-I phase, and \( P \) is the calculated residual stress (kilobar).

The surface roughness was measured using a contact profilometer (Taylor Hobson, model FTS i-120) with a diamond stylus tip (radius of 2 µm and cone angle of 90°). According to the ISO 4288/1998 standard, a sampling length of \( l_s = 4 \) mm and cut-off length of \( \lambda_c = 0.8 \) mm were adopted. The surface roughness was measured along the feed direction. The data processing was carried out in the software MoutainsMap Universal 7.1® and the parameter \( R_a \)

![Fig. 3](image) a Ni-electroplated diamond wire. b Wire welding process
was obtained according to guidelines in the ISO 4287/1997 standard.

The subsurface damage was analyzed using a bevel-polishing method, as recommended in the ASTM F 950–02 standard [25]. The procedure (see Fig. 4) consisted of placing the polycrystalline silicon specimen at an inclination (slope, β) using a support and embedding it in an acrylic resin. A metallographic preparation was performed with sanding (220 to 2000 mesh) and polishing (diamond slurry of 0.25 µm). Chemical etching was carried out using 1 ml HF + 3 ml H₂O + 0.25 g CrO₃, with a time of 20 s. The micro-crack depth in subsurface region of the poly-Si specimen was determined applying an image-processing method of SEM micrographs as described in reference [4] and the slope (β) was determined using an optical microscope (Leica, model DM400M).

From surface integrity analysis, the optimum cutting condition (v_c and v_f) was determined. The main factors considered to define the optimum condition were the presence of ductile regions on the sawn surface, smooth surface roughness (small Rₐ value) and shallow subsurface damage. On applying the optimized cutting condition, the analysis of silicon chips and the wear of the diamond wire were analyzed. Firstly, the silicon chips were collected in situ using a conductive carbon adhesive tape and the chip morphology and its residual phases were analyzed by SEM and Raman spectroscopy, respectively.

To analyze the wear and failure mechanisms of diamond wire, it was performed experiments under the same optimum cutting condition until the diamond wire breaks. This approach was used in order to induce a more intense wear on Ni-matrix bonding and diamond grits, as well as to evaluate the failure characteristic of the welding joint. SEM images were acquired in 5 main stages to perform the analysis of the wire wear. To understand the influence of welding process on the failure mechanism of welded diamond wire, the welding joint of the diamond wire was analyzed by SEM/EDS and optical microscopy, identifying the microstructure and elementary composition.

3 Results and discussion

3.1 Morphology of the sawn surface

Analysis of the sawn surface morphology provided qualitative information on the sawn surface damage and chip formation during the cut by diamond wire sawing. Figure 5 shows the SEM micrographs of the sawn surface of the polycrystalline silicon specimen applying variations in the cutting parameters. Increasing v_f resulted in the formation of deeper and wider craters, which were predominant on the sawn surface. An increase in v_f led to high normal force per grit, resulting in greater penetration depth of the grit, as noted by Marinescu et al. [26]. According to the considerations of Bifano et al. [27], excessively increasing the penetration depth of the grits causes crack nucleation as a response of the polycrystalline silicon. Therefore, brittle fracture increases, which leads to the formation of chip fragments and the appearance of larger and wider craters. As consequence, when the v_f increases in the diamond wire sawing of poly-Si, the quantity of plastic grooves decreases over the sawed surface morphology.

Moreover, the anisotropy of the poly-Si contributes to the formation of a surface with more craters, since the diamond grit acts along the crystallographic orientation, with different slip and cleavage planes. This agrees with the findings of Kumar et al. [28] who showed that silicon grain randomly oriented as well as the presence of discontinuity like twin boundary and grain boundary along to multiples scratches induce to a variation in the cracking frequency and have influence on the cutting behavior. Based on the cutting condition evaluated, it can be stated that increasing v_c from 0.3 to 0.7 mm/s led to the formation of deeper and wider craters on the sawn surface of the poly-Si specimen.

The opposite effect on the sawn surface morphology was observed on increasing v_c. As shown in Fig. 5, maintaining v_f constant and increasing v_c from 10 to 20 m/s resulted in more plastic grooves and scratches over the sawn surface of the polycrystalline silicon. This difference in the mechanical behavior of the poly-Si specimen is attributed to fact that a high v_c will reduce the penetration depth of the grits and favor the material removal in the ductile regime, such as a ductile material. Since increasing v_c leads to rise the engagement frequency of the grit on the poly-Si, the normal and cutting forces per grit decreases and the penetration depth per grit will also reduce. Consequently, the brittle fractures, like craters and pits, over the sawn surface will be shallower and narrower, increasing the probability to have kinematic edges acting mainly in the ductile mode.

![Fig. 4 Bevel-polishing method applied to evaluate the subsurface damage](image-url)
acting on the silicon grains with different crystallographic planes. The SEM micrographs show this behavior through the presence of periodic plastic grooves and scratches over the sawn surface.

Although there are differences in the morphological aspects, all SEM micrographs of sawn surfaces of the polycrystalline silicon specimens have regions with craters and grooves/scratches. On varying the cutting parameters, the predominance of these regions was affected and this can be attributed to the mechanism of material removal. The material removal in brittle mode becomes dominant with higher $v_f$, whereas on increasing $v_c$, the ductile mode becomes dominant. Both brittle and ductile cutting modes were observed in the sawn surfaces analyzed, and this can be a result of anisotropy of the poly-Si as well as of variations in the protrusion and microgeometry of the diamond grits [13]. Pala and Wegener [29] noted that the diamond grits on the wire surface present different microgeometries and hence the levels of protrusion lie within a range. This different microgeometries/shape of the diamond grits changes the local stress state in grit-silicon contact, leading to a variation in critical depth of cut ($h_{cu}$), resulting in the formation of the sawn surface with ductile and brittle regions [28, 30].

In summary, the mechanisms involved in the material removal from the sawn surface can be attributed to anisotropy of the polycrystalline silicon and the different protrusion of the diamond grits, which results in different penetration depths of the diamond grits acting on silicon grains with different slip and cleavage planes. Therefore, a high $v_f$ increases $h_{cu}$ above the critical value, which produces a surface with a lot of craters. On the other hand, on increasing $v_c$, the $h_{cu}$ value is reduced considerably and this leads to a predominance of ductile material removal. However, as reported by Chung and Le [10], there is still a number of grits with high $h_{cu}$, which leads to cutting in the brittle mode, resulting in a sawn surface with mixed brittle and ductile cutting modes.

### 3.2 Brittle-ductile transition and residual stress due to phase transformation

The mechanisms of material removal were confirmed by detection of the residual phases on the sawn surface of the polycrystalline silicon specimen. The Raman spectroscopy results are shown in Fig. 6. The Raman spectra in Fig. 6a show the residual phase in a region with craters (shown in the inset). The spectra exhibit a single peak at 521.2 cm$^{-1}$ that represents the Si-I phase. According to the hypothesis for brittle mode described above, excess penetration depth of the diamond grits can cause micro-crack propagation and the formation of chip fragments. Although the silicon-diamond grit contact produces phase transformation, a volumetric overlap of the chip occurs and thus no residual secondary phase remains on sawn surface. Therefore, the craters expose only pure crystalline silicon of the Si-I phase.
The Raman spectra for the region with microgrooves show that, in addition to the Si-I phase, there was an amorphous phase (a-Si) with a bandwidth in the region of 425–505 cm$^{-1}$ (see Fig. 6b). The presence of a-Si as a residual phase is indicative that the silicon has a disordered local structure due to the occurrence of phase transformation. Kovalchenko and Milman [31] noted that the a-Si phase is found on the machined surface of silicon when the ductile mode occurs during the cut. Würzner et al. [12] also reported the amorphization of the sawn surface of polycrystalline silicon under conditions of high $v_c$. Costa et al. [13] showed that the phase transformation of silicon sliced by diamond wire sawing can occur with $v_c \geq 10$ m/s, however with greater intensity compared to monocrystalline silicon. This difference in the a-Si intensity when compared with monocrystalline silicon is due to the microstructure of the polycrystalline silicon that present grains with varied crystallographic plane, which results in different critical value of $h_{cu}$ during the chip formation and removal. As a result, the sawn surface presents a lower residual stress and few dislocations in the subsurface region [20].

Based on a study of Knoblauch et al. [17], who determined the protrusion of the grit from the Ni-matrix for the same Ni-electroplated diamond wire, machine tool, and similar cutting conditions, it can be assumed that the penetration depth of the diamond grit is around 2 μm. Although many grits effectively assume the cut, it is possible that only some are responsible for producing high-pressure phase transformation on the machined surface, which can cause small elastoplastic deformations as well as ploughing and microcutting [13]. Moreover, a high cutting speed increases the temperature in the cutting zone, which can also induce ductile shearing. Based on the Raman spectra shown in Fig. 6b, which exhibited a bandwidth associated with the a-Si phase, it can be considered that there was ductile cutting of the polycrystalline silicon specimen during the diamond wire sawing.

The calculated residual stress results are shown in Fig. 7 (values were converted from kilobar to MPa). It was observed that the residual compressive stress is higher in the ductile region (microgrooves) than in the brittle region (craters). As seen in the Raman spectra, the microgrooves region presented a bandwidth associated with the a-Si phase, indicating a more intense compressive state on the sawn surface. This higher compressive stress can be attributed to the surface formation by ductile cutting mode, which leads to the formation of smooth grooves on the sawn silicon surface [7]. Based on this finding, it can be stated that the stress state of the sawn polycrystalline silicon surface results from material removal in ductile regime.

### 3.3 Behavior of the surface roughness

Figure 8 shows the average values for the surface roughness parameter $R_a$ obtained on varying the cutting parameters. The $R_a$ values ranged between 0.520 and 0.904 μm. It was observed that with an increase in $v_f$, the surface roughness is increased. This behavior is in accordance with the characteristics of the sawn surface morphology, since with high $v_f$ there was mainly fragile cutting, which led to the predominance of craters on the sawn surface. According to Costa et al. [32] and Zhang [33], the surface roughness increases proportionally with the depth and width of the craters formed on the sawn surface of polycrystalline silicon. Yin et al. [14] reported similar behavior for the surface roughness $R_a$ of polycrystalline silicon specimens with an
increase in \( v_f \). However, the minimum \( R_a \) value reached was 1.103 \( \mu m \), since a \( v_c \) of 0.4 \( m/s \) was employed by the authors, which is considerably lower (factor of 25 times) than the minimum \( v_c \) value of 10 \( m/s \) used in this study. All the mean values for surface roughness were below \( R_a = 0.801 \, \mu m \) in the study reported herein.

In contrast, it was verified that on increasing \( v_c \) from 10 to 20 \( m/s \), the surface roughness \( R_a \) reduced. This behavior is associated with the ductile mode being the dominant mechanism of material removal during cutting. The sawn surface of the poly-Si exhibited a decrease in the presence of craters but there were more microgrooves, with less fragile damage, as observed in the SEM micrographs in Fig. 5. Pala et al. [34] complement that when ductile cutting is dominant, there is a decrease in the surface roughness. The ductile mode may favor a reduction of the surface roughness because a higher \( v_c \) reduces significantly the normal and tangential forces per single grit, decreasing the penetration depth, leading to the ductile cutting mode predominating. Pu et al. [11] and Li et al. [9] cite that a higher quality of the polycrystalline silicon wafer, in terms of surface roughness, is obtained on increasing \( v_c \).

### 3.4 Analysis of the subsurface damage

The subsurface characteristics of polycrystalline silicon specimens sawn by electroplated diamond wire sawing were determined by the bevel-polishing method. In general, median micro-cracks were the main type of residual damage found at the subsurface, as shown in the Fig. 9. Klocke [35] and Marinescu et al. [26] affirm that brittle machining is commonly characterized by the appearance of micro-cracks due to the low fracture toughness of these materials (for crystalline silicon \( K_{IC} = 0.93 \, MPa \, \sqrt{m} \) [36]). Considering the model of micro-crack initiation and propagation based on indentation employing a single indenter, it is known that the median micro-cracks appear beneath the indentation region and have the same normal load direction [37]. However, machining presents both normal and tangential loads, which affects significantly the elastic/plastic stress field induced by the action of multiple diamond
grits, leading to a change of the fracture initiation angle and resulting in inclination of the median micro-cracks [32].

Since continuous cutting movement of the looped diamond wire runs in one direction, it suggests that the action direction of normal and tangential forces remains unchanged. In this sense, it is expected that the direction and inclination of the median micro-crack should also remain the same. However, Fig. 9 shows that the inclination direction of the median micro-crack is irregular since the poly-Si has a complex crack system in which the presence of grains with varied crystallographic plane and discontinuity can serve as crack nucleation sites [28]. It can also be observed a larger density of median micro-cracks in subsurface of the poly-Si sawed. As shown in Fig. 9, the median micro-cracks are slightly inclined (δ), with irregular inclinations, and arise mainly from bottom of periodical grooves produced along of the wire cutting movement.

Based on the SEM micrographs and micro-crack depth (MD), it is possible to evaluate the influence of the cutting parameters on subsurface damage of the poly-Si specimen. On comparing Fig. 9a, b, it can be observed that the average MD in the subsurface increased from 8.1 ± 2.4 to 9.4 ± 2.0 µm. This is attributed to increased $v_f$ while $v_c$ remains constant, since this will increase the penetration depth of the grit, leading to the formation of deeper micro-cracks. With an increase in $v_c$ from 10 to 15 m/s, for the same $v_f$, there was a slight reduction in the average MD (8.6 ± 2.7 µm), as shown in Fig. 9c. This reduction is the results of the smaller penetration depth of the diamond grit, which reduces the micro-crack propagation in the subsurface.

Figure 9d shows the subsurface of the poly-Si sawn applying the cutting parameters of $v_c = 20$ m/s and $v_f = 0.3$ mm/s. Under these conditions, there was a lower average subsurface damage depth (average MD = 7.2 ± 1.6 µm). Considering the load of a single diamond grit, the normal and tangential loads are reduced significantly with the highest $v_c$ and lowest $v_f$ values, resulting in shallower micro-crack depth. Although the a-Si phase was detected on the sawn surface under these cutting conditions, the presence of subsurface damage suggests that there was not a purely ductile cutting. No dislocations in the silicon were observed, as this rarely occurs. This is attributed mainly due to the anisotropy of the poly-Si that present silicon grains with different slip and cleavage planes, resulting in mixed brittle and ductile modes occurring at the same time.

### 3.5 Silicon chip formation and morphology

It was observed that optimum cutting condition is under $v_c = 20$ m/s and $v_f = 0.3$ mm/s and the silicon chips were collected during the experiments. Two different morphological types of silicon chips were observed.
chip fragments (Fig. 10a) and elongated chips (Fig. 10c). Polycrystalline silicon has the characteristics typical of a brittle material, where material removal occurs through chip formation due to the propagation of micro-cracks. As shown in Fig. 10b, undesirable fracture damage is produced when there is excessive $h_{cu}$ of the diamond grit, which introduces damage at the subsurface and chip fragments are formed. Figure 10a clearly shows chip fragments as a result of the brittle cutting mode. Raman spectra indicates a predominance of the pristine crystalline phase (peak at 521.5 cm$^{-1}$) and lower intensity of the secondary phases Si-XII and Si-III with peaks at 351 and 445 cm$^{-1}$, respectively.

With a smaller depth of cut ($h_{cu} \leq h_{cu, crit}$), brittle materials undergo phase transformation and shearing. The formation of microgrooves on the machined surface and elongated chips are considered responses of the ductile cutting mode. Figure 10c shows elongated chips with a lamellar morphology, suggesting that there was a shear deformation mechanism acting during the silicon-diamond grit contact, resulting in plastic flow that led to the formation of elongated chips. This characteristic is consistent with free-damage regions (microgrooves) present on sawn surface. The Raman spectra revealed that the silicon chip is practically amorphous, since there was a lower peak intensity related to the Si-I phase and a larger bandwidth of amorphous phase (a-Si) in the region of 407–512 cm$^{-1}$.

As shown in Fig. 10d, the change in the metallic state (to Si-I from Si-II) under high pressure (10–13 GPa) is also the main mechanism of ductile chip formation in the case of poly-Si. Although the conditions favor cutting in the ductile mode, the surface integrity presents phase transformation.

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**Fig. 10** Silicon chips obtained applying $v_c = 20$ m/s and $v_f = 0.3$ mm/s: a chip fragments; b formation of chip fragments; c elongated chip; d formation of elongated chip.
transformation (amorphous or secondary phases), residual defects (dislocations), and elastic recovery due to shear deformation of the silicon. In the cutting of poly-Si, the multiple scratching along different crystallographic planes produce shearing which can form chips with nanocrystals [20]. This is consistent with the Raman spectra results, which show the presence of the a-Si phase, due to the partial amorphization of silicon grains. Different slip systems can be observed in the grains that form the poly-Si, which leads to ductile shearing of some silicon grains and splitting via brittleness of others. Since the chips are formed of amorphous and crystalline silicon, peaks of the Si-I and a-Si phases are present on the spectra.

Considering the findings of Li et al. [15] and Liu et al. [38], around of 10% of the kinematics edges take the ductile mode cutting, affecting the sawn surface, and they are restricted to the position angle on the wire cross-section of $\theta \leq 25^\circ$. Chung and Le [10] complement that the diamond grits on upper side of the wire cross-Sect. ($50^\circ \leq \theta \leq 90^\circ$) assume mainly the brittle cutting mode. Theoretically, it is possible that the collected silicon chips have been formed in the following regions of the wire cross-section: at upper region of the wire, in which the diamond grits assume predominantly the brittle cutting, there is the formation of fragmented chips (Fig. 10c); at side region of the wire ($\theta \leq 25^\circ$), where grits present smaller $h_{cu}$, occurs ductile cutting (Fig. 10d). As the diamond wire sawing is a stochastic machining process, it is very difficult, if possible, to determine the exact moment and identify the single diamond grit that produces the respective chips collected. Thus, the regions of the diamond wire in contact with workpiece in which the silicon chips were formed can be just theorized.

### 3.6 Performance of the diamond wire: wear and failure mechanisms

The diamond wire was analyzed by SEM before (Fig. 11a) and after of the sawing experiments (Fig. 11b–e) under $v_c = 20$ m/s and $v_f = 0.3$ mm/s. As shown in Fig. 11a, the diamond wire is composed of a steel wire core coated with diamond grits embedded on the wire surface using Ni-layer as bonding and this characteristic can be observed when it is in the unworn stage. The initial contact between the diamond wire and the workpiece leads to the removal of the Ni-layer. This first wear mechanism is due to the lower hardness of Ni (200–400 HV) compared with silicon (1000 HV) and diamond (6000–10,000 HV), resulting in plastic deformation of the Ni-layer to the diamond grit sides, which increases their base area, and generate the partial removal of the Ni-layer, as can be seen in the Fig. 11b. The Ni removal exposes the cutting edges of the diamond grits and the Ni-layer around the diamond grit produces a stronger bonding over the wire [17]. In this stage, the sawing process...
presents higher cutting ability since sharp cutting edges of
the diamond grits are in contact with the surface of the
poly-Si specimen.

After the diamond grit is exposed, the silicon-diamond
grit contact leads to tribologically induced micro-fracture,
micro-chipping, rounding, and flattening. As the volume
of material removed increases, the diamond grits acting
in cutting are worn more intensely, generating larger edge
radius and gradual reduction of their protrusion [18, 39].
Furthermore, the presence of impurities in poly-Si like
carbide and nitride increases the local hardness, which
results in greater wear of the diamond grits [19]. These
observations suggest that the wear mechanisms of the
diamond wire also involve abrasive wear of the diamond
grits.

Another wear mechanism of diamond wire is grits
pullout, as shown in Fig. 11c, d. Zheng et al. [40] reported
that the excessive wear of the Ni-layer increases the
protrusion height of the diamond grit and decreases the grit
depth inside the Ni-layer, which leads to lower retention
capacity over the wire. Since the Ni-layer is removed
more excessively due to the endless cutting cycle applied
in this study, the diamond grits undergo pullout. Based on
Fig. 11b–d, it was identified only 3 pullouts for a total of
106 diamond grits analyzed, resulting in a pullout rate of
2.83%, which is a small number of cases. Figure 11d shows a
closed-up view of a region where a diamond grit underwent
pullout phenomena.

The manufacturing procedure applied to obtain a
diamond wire in the form of a loop for this purpose is
aimed at ensuring the internal rotational of the wire during
the cutting, which leads to a longer lifespan of the diamond
wire. Nevertheless, excessive and non-uniform wear was
still observed on one side of the diamond wire, as shown
in Fig. 11e. Since the diamond wire have an abrasives
monolayer, the cutting ability is reduced significantly.
Moreover, Fig. 11f shows that undesirable rupture of the
wire occurred during the cutting experiments. This is
mainly due to the welding process applied to obtain the
diamond wire into a loop, which induces the formation
of a heat-affected zone (HAZ) in a small region (around
0.5 mm).

Fig. 12 Cross-section of the wire core: a welded region; b HAZ and drawn wire regions; c EDS spectrum and color maps; d EDS line
Figure 12 shows the welded region of the wire core and recrystallized grains can be observed in the HAZ. According to the SEM images shown in the Fig. 12a, it is observed a burr rich in Ni. The Ni is present in the wire surface as bonding and diffused to the welding joint. The burr is formed as a consequence of the expulsion of the material by the action of the temperature and pressure held on the wire ends. The presence of Ni can be observed in the boundary of the recrystallized grains. In Fig. 12b, it is possible to observe the deformation lines structure of the material due to the cold drawing process, following the ASTM A228/A228M standard [41]. A transition zone is also observed, where begins the HAZ. In this region, it was not observed influence of the Ni. Moreover, due to the high temperature produced during the welding process of steel containing 0.8% C (wire core), it is possible that the deformation lines formed by cold drawing process be martensitic grains elongated in which it has been transformed into austenite grains, as shown in detail of Fig. 12a.

Energy-dispersive X-ray spectroscopy (EDS) was performed to identify the elements in the welded joint. As shown in Fig. 12c, the elemental spectrum show peaks of Fe, C, Mn, and Si as well as a small amount of P and S that are present in steel wire core. It was also revealed the presence of Ni by the elemental color maps, which confirms its presence in the welded burr region (see detail of the Fig. 12c). According to Fig. 12d, the EDS line analysis shown that the Ni is concentrated in the surrounding regions of the recrystallized grains, since its presence was identified mainly in the elements of the base material in the grains. As a result of these effects, in the welding process, a fragile microstructure is formed, which decreases considerably the rupture strength of the wire core (from 2250 to 740 MPa) reducing the lifespan of the welded diamond wire during the slicing process.

4 Conclusions

The surface integrity and ductile cutting of photovoltaic poly-Si and the wear and failure mechanisms of Ni-electroplated diamond wire were investigated during the endless wire sawing process on different cutting parameters. The results showed that both brittle and ductile cutting regimes occur during the sawing of poly-Si as consequence of its anisotropy. It was revealed that ductile regions have a-Si layer with residual compressive stress around of –192.3 MPa. The presence of brittle fractures regions increases the surface roughness, whereas the predominance of regions with ductile cutting modes decreases the surface roughness, reaching the better surface quality with $R_a = 0.52 \mu m$. Subsurface damage was mainly median micro-cracks with a minimum damage depth on 7.2 ± 1.6 μm. The main types of silicon chips were fragments and elongated chips and latter is practically amorphous. Meanwhile, the wire wear analysis showed that during the cutting there is Ni-layer deformation and removal, abrasive wear, exposed grits, and grits pullout. The wire welding process applied to manufacture the single-looped diamond wire induces to HAZ formation, which decreases their rupture strength and generated the failure of the diamond wire at welded region.

The simplest cutting kinematic provided by the endless diamond wire sawing allows to study the slicing of poly-Si under cutting conditions ($v_c$, $v_f$, and wire tension) similar to the industry. Many researchers that employed the reciprocating wire cutting movement used $v_c$ values much lower than the common values used in industry. On unidirectional and continuous cutting movement provided by the diamond wire into a loop is possible reaches a high $v_c$ and increases the stability of the wire tension, which lead to the predominance of ductile cutting material removal mode. This is a very important condition to obtain silicon wafers with high surface quality, resulting in smooth sawn surface and shallow micro-crack depth.

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Declarations

Ethics approval The authors declare that this manuscript was not submitted to more than one journal for simultaneous consideration. Also, the submitted work was original and has not been published elsewhere in any form or language.

Consent to participate and publish The authors declare that they participated in this paper willingly and the authors declare to consent to the publication of this paper.

Competing interests The authors declare no competing interests.

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