Mapping ductile-to-fragile transition and the effect of tool nose radius in diamond turning of single-crystal silicon

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Abstract
Although it has long been known that tools with more negative rake angles allow the ductile regime when machining monocrystalline silicon, little has been discussed about the tool-material interaction. The microgeometric contact of the tool tip at this interface plays an essential role in the material remotion (ductile or brittle). In this paper, the tool rake angle was varied in order to change the value of the undeformed chip thickness once the tool cutting radius, formed in front of the tool rake face, changes when the tool rake angle becomes more negative. Based on the statistical design of the experiment applied to cutting tests, a map is built to relate the values of transition pressure in different crystallographic directions. This map assisted in determining the machining conditions with a ductile response into a broader spectrum based on the variation of the tool rake angle. The results obtained allowed to answer questions under which machining conditions and tool geometry account for better surface finishes, lower machining forces, and lower residual stresses. The response surfaces, from statistical design, provided answers capable of establishing under which cutting radii yielded more ductile material removal and avoided a brittle response related to the anisotropic behavior of the material. Finally, the brittle-to-ductile transition mapping determined a more suitable machining condition to diamond turn Fresnel lenses in single crystal silicon.

Keywords Monocrystalline · Silicon · Diamond tool · Transition pressure · Rake angle · Surface finish

Nomenclature

| Symbol | Description |
|--------|-------------|
| Rc     | Cutting radius (µm) |
| α      | Tool rake angle (°) |
| Re     | Tool nose radius (µm) |
| dc     | Depth of cut (µm) |
| dc∗    | Critical depth of cut (µm) |
| dce    | Effective depth of cut (µm) |
| dce∗   | Critical-effective depth of cut (µm) |
| dc110  | Critical depth of cut in the direction [110] (µm) |
| re     | Cutting edge radius (nm) |
| f      | Feedrate (µm/revolution) |
|Fc     | Cutting force (N) |
| Ft     | Thrust force (N) |
| Ff     | Feed force (N) |
| kN     | Transition pressure (GPa) |
| h      | Thickness of cutting (nm) |
| h∗     | Critical-effective thickness of cutting (nm) |
| hemax  | Maximum-effective thickness of cutting (nm) |
| h∗110  | Critical-effective thickness of cutting in the direction [110] (nm) |
| As     | Cutting section area (µm²) |
| Asε    | Effective area of cutting section (µm²) |
| Ra     | Arithmetic average of surface roughness (nm) |
| Rq     | Root-mean-square surface roughness (nm) |
| Rt     | Maximum height of the surface roughness (nm) |
| C      | Safety factor |

Highlights
- Cutting forces used to show anisotropy effects with changes in crystallographic direction.
- Inverse relation between residual stress and cutting forces.
- Establish optimal surface finish condition as a function of the rake angle value.
- Negative rake angle increases cutting radius enhancing the critical thickness of cutting.

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1 Introduction

Monocrystalline silicon is an essential structural and optical material [1] and is broadly used in micro/nanoelectromechanical systems [2]. It has a typically fragile behavior at room temperature and atmospheric pressure [3]. This brittle response may be partially reverted to ductile when the deformation process reaches a value of transition pressure related to phase transformation hindering the manifestation of harmful microcrack propagation into the surface and subsurface of the semiconductors crystal [4, 5].

The brittle-to-ductile transition applies to elemental crystals such as silicon and germanium and is studied using microindentation and machining experiments [6, 7]. The studies applied to explain the ductile response of semiconductor crystals from the 1990s onwards [8–12] refer to concepts of pressure/stress-induced phase transformation initially demonstrated in the 1960s [13–17]. Silicon experiences 12 different phases under compression ranging from 0 to 230 GPa [18]. Some of these phases are thermodynamically stable, while others are metastable. Among these phases, the metallic characteristic is the most relevant in mechanical processes, making silicon susceptible to plastic deformation [19, 20]. This phase occurs under hydrostatic stress between 10 and 13 GPa [21] by changing the natural structure of silicon (diamond cubic) to a more dense, metastable structure, known as beta-tin (β-Sn). The β-Sn metastable phase is of great interest since when the decompression speed is rapid, this phase becomes amorphous [19, 22], and, in turn, maintains the desired physical characteristics in the induction of the β-Sn phase by mechanical loading [20].

In semiconductor crystals machining with a single point diamond cutting tool, hydrostatic pressure with the presence of a shear component tends to lower the values of transition pressure in different crystallographic directions [23–25]. The material removal mechanism tends to occur predominantly in a ductile mode providing a crack-free surface with optical quality [24, 26]. This ductile mode material removal is intrinsically related to the critical thickness of cutting [9, 27]. The critical thickness of cutting when machining with round nose diamond tools is determined using the uncut shoulder idealized model [24], as shown by Fig. 1a, b. The cracks will reach the cut surface and harm the finish when the critical thickness of cutting is below a critical height (dc*).

The tool geometry (e.g., tool nose radius and rake angle) affects the dominant mode of material removal [28]. The value of the transition pressure, depth of cut, and critical thickness determine this removal mode [29]. The critical thickness of cutting is directly proportional to the rake angle (α) and the tool nose radius (Re) [28]. However, the rake angle directly relates to the tool nose radius interaction in the tool material interface: due to the machining process dynamics, the rake angle variation changes the rake face area in contact with the material, i.e., the chip cross-section area. The contact area involved by tool radius acting is defined here as cutting radius (Rc). For the same tool nose radius (Re), the cutting radius (Rc) changes when the tool rake angle varies (Fig. 1c).

Thus, the nominal value of the chip cross-section area corresponding to the zero-degree rake angle formed under the nominal values feedrate and depth of cut will change as the rake angle becomes more negative. Thus, the critical thickness of cutting will vary with the rake angle due to the cutting radius change. The enhancement of machinability of semiconductor crystals by increasing the ductile response of using a more negative rake angle [30, 31] may be related to the change in the value of the cutting radius. A consensus has not been reached on the excellent value of this angle to correspond to the best ductile response in material removal.

The crystallographic orientation and direction of a single crystal influence the material removal mode [7, 32]. Since single crystal silicon is an anisotropic material, the direction in different orientations presents different ductility responses. The orientation (100) is the most ductile among them. When cutting single crystal silicon on the plane (100), the material removal mode changes with the change in the directions [33]: direction [110] depicts a more brittle response [34, 35], and thus the critical thickness of cutting presents lower values when compared to the direction [100] [36].

It is recognized that studies concerning the influence of tool rake angle, nose radius, and machining conditions have long been investigated. However, these studies do not mention or even consider this issue related to the tool cutting radius, which changes with the rake angle, consequently changing the volume of deformed material in front of the tool. Thus, this study intends to evaluate the ductile–brittle response in the tool-material interface for rake angle more negative. The aim is to respond with continuity characteristics for the ductile response trend when the rake angle becomes more negative, along with variation in machining conditions. Furthermore, the anisotropy effect, resulting from the change in direction, still needs to be quantified to avoid this harmful effect in manufacturing optical components.

2 Material and methods

2.1 Object of the study

The specimens (20 × 20 mm) from single crystal silicon wafers with (100) surface orientation, applied in
ultra-precision machining with a round nose diamond tool, has 1–10 Ω cm resistivity, P-type (Boron concentrations: 10^{15}–10^{16} atoms cm^{-3}), 55 mm diameter and 500 μm thick. Table 1 shows some physical and mechanical properties of silicon [37]. The main outputs assessed in this study are cutting forces, surface finish, and the onset of uncut shoulder cracks propagation in machining.

A commercially available diamond turning machine, the Aspheric Surface Generator Rank Pneumo ASG 2500, carried out the experiments. This machine is composed of a very rigid system with a T-base carriage configuration: hydrostatic bearing, driven with pulse-width-modulated dc servomotors, rotary-to-linear motion through 5 mm pitch balls crews, and position feedback using a laser interferometer. The positioning resolution of the X–Z axis is 10 nm, according to the machine manufacturer.

The commercial monocrystalline diamond tools used for the experiment have a nose radius of 762 μm (Fig. 2a) and 100 μm (Fig. 2b). The clearance angles, rake angles, and cutting edge radii are 10°, 0°, and 40 nm, respectively. Figure 2c

| Material property | Value |
|-------------------|-------|
| Density (g/cm³)   | 2.328 |
| Melting point (°C)| 1,420 |
| Young modulus (GPa) (100) | 131 |
| Shear modulus (GPa) | 79.9 |
| Fracture toughness (MPa.m^{1/2}) | 0.95 |
| Poisson’s ratio | 0.266 |
| Hardness load 10 g Hvicker (GPa) | 11.3 |
Fig. 2 Monocrystalline diamond tools with cutting edge radius of the order of 40 nm. a) Nose radius $R_{ε} = 762 \, \mu m$ and $α = 0^\circ$. b) Nose half-radius $R_{ε} = 100 \, \mu m$ and $α = 0^\circ$. c) Edge radius $r_{e} = 40 \, nm$. SEM scanning electron microscope, AFM atomic force microscopy.

shows the measurement of the cutting edge radii of the diamond tool done in an atomic force microscope (AFM). This microscope has operated with a standard 50–60° conical silicon nitride stylus of 15–20 nm radius tip, with a cantilever spring constant of $\sim 0.06 \, N/m$. Conventional contact mode was employed where the stylus is scanned raster style over, with contact forces of typically 10–100 nN. The tool’s cutting edge measurement was carried out similarly to what has been described elsewhere [38].

### 2.2 Machining experiment and data analysis

The statistical technique applied to the machining experiment was the central composite design (CCD). This planning is an increased first order factorial by additional points to estimate second-order surface parameters. The method adopted here was that of rotation proposed by Box et al. [39]. Two factors were analyzed: the tool feed ($f$) and the tool rake angle ($α$).

Table 2 presents the CCD’s statistical matrix for the execution of the machining experiment. The statistical matrix’s lowest feedrate and tool rake angle values were established at 2.5 $μm/rev$ and $0^\circ$, respectively. In order to achieve the minimum point ($−1.41$) of the matrix, given by $f = 2.5 \, μm/rev$ and $α = 0^\circ$, was adopted a central point (0) so that the points $+1$ and $−1$ varied by 1 $μm/rev$ for feedrate $f$ and $15^\circ$ for rake angle $α$. Thus, Eq. 1, which determines the additional points ($±1.41$), provides the desired minimum point ($f, 2.5 \, μm/α, 0^\circ$).

$$e_{(±1.41)} = e_{(0)} ± 1.41 \left( \frac{e_{(+1)} - e_{(-1)}}{2} \right)$$

where $e$ is the value of $f$ or $α$ corresponding to the coding.

In the ultra-precision diamond turning of semiconductor crystals, rotation is not a significant factor for ductile or brittle response effect. In addition, the finish generated by the process fundamentally depends on the feedrate and the sharpening state of the tool’s cutting edge: any imperfection along the tool’s cutting edge length in contact with the machined surface will reproduce on the surface finish. The maximum workpiece diameter (20 mm) corresponds to 60 m/min (1000 rpm). This cutting speed is not high enough to cause wear as the linear cutting distances are
very short in these tests. The significant effect of the cutting speed when machining with single point diamond tools is related to tool wear over long distances (over 2.5 km) [40]. In this work, the machining was only a few meters. To further minimize the effects of cutting speed, a cutting fluid (Alkalisol 2050) was used. Besides, the maximum rotation offered by the machine is only 2000 rpm.

The order of execution of the points (samples) in the experiment was carried out at random, as shown in Table 2.

Thus, machining tests were carried out on monocrystalline silicon samples randomly, with a total of 10 machining conditions established based on the central composite design (Table 2). Feedrates ranged from 2.5 to 5.5 µm/rev at a depth of cut of 10 µm.

As shown in Fig. 3a, the first condition (track 1) is at the center of the experiment, and the last (track 10) is at the ultimate track. The schematic diagram of the step structure, generated by subsequent machining conditions from track 1 to 3, is illustrated by Fig. 3b: when machining the first track,

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**Table 2** Experimental matrix of the central composite planning. Input factors: machining feedrate $f$ (µm/revolution) and tool rake angle $\alpha$ (°)

| Coding | $f$  | $\alpha$ | $f$  | $\alpha$ | Number* |
|--------|------|----------|------|----------|---------|
| +1.41  | 5.5  | -45°     | -1   | -1       | 3.0     |
| +1     | 5.0  | -37.5°   | -1   | +1       | 3.0     |
| 0      | 4.0  | -22.5°   | +1   | -1       | 5.0     |
| -1     | 3.0  | -7.5°    | +1   | -1       | 5.0     |
| -1.41  | 2.5  | 0°       | -1.41| 0        | 5.0     |
| 0      | -1.41| 0°       | -1.41| 0        | 4.0     |
| 0      | +1.41| 0°       | +1.41| 0        | 4.0     |
| 0      | 0    | 0°       | 0    | 0        | 4.0     |

* The samples execution random order (tracks)

** Machining environment: wet cutting (Alkalisol 2050)

** Depth of cut (dc): 10 µm

** Spindle rotation rate: 1000 rpm

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![Fig. 3](samples_tracks.png) Samples (tracks) of the machining experiment. a Experiment finished (workpiece). b Machining protocol for samples
the tool traveled across the entire surface; in the second and third track, it stopped beforehand so as not to eliminate track 1 and track 3; thus, successively up until the ultimate track of the experiment. The width of the zone generated by each condition is about 1 mm. Cutting fluid (Alkalisol 2050—produced by Alkalis Brazil) was applied to machining in all zones.

Figure 4 shows the experimental setup. This system consisted of a tool holder, an angled base, and a dynamometer. The angular base was attached to the dynamometer and the tool holder to the angular base in the position corresponding to the desired rake angles for the experiment (Fig. 4). The silicon sample was fixed with heat-softening glue on a fixing bracket, which was then fixed onto the vacuum chuck of the machine spindle (Fig. 4). The room temperature was 20 °C during machining.

2.2.1 Cutting forces

The acquisition system to measure the machining forces consists of an acquisition board (400 kHz), a multi-channel load amplifier, and a piezoelectric dynamometer Kistler, model 9652C2 (0 to 250 N; natural frequency of 2 kHz), all commercial. The dynamometer was positioned in such a way that the x-axis provided the thrust force (Ft), the y-axis provided the cutting force (Fc), and the z-axis provided the feed force (Ff). A sampling frequency of 130 kHz was used to record the forces at each direction (x, y, and z).

Figure 5 shows the thickness of cutting (h), the depth of cut (dc), and the cutting section area (As), corresponding to the layer to be removed, which is in front of the tool. The effective thickness of cutting (he), the effective depth of cut (de), and the effective area of cutting section (As e) are those correlated with the tool inclination and will be influenced by the rake angle (α).

The value of the transition pressure (kN) was determined by the decomposition of thrust and cutting forces, distributed in the effective area of cutting section (As e), as expressed by Eq. (2).

\[ k_N = \frac{N}{A_{se}} = \frac{F_c \cos \alpha - F_t \sin \alpha}{A_{se}} \]  

(2)

\[ A_{se} = \frac{f \cdot dc}{\cos |\alpha|} \]  

(3)

f: tool feedrate per revolution; dc: depth of cut; α: tool rake angle

2.2.2 Micro Raman spectroscopy

In this study, the Raman spectra were taken with an HR800 Evolution micro-Raman spectrometer from Horiba-Jobin–Yvon using the line with wavelength 532 nm of the Nd-YAG laser. The laser power was lower than 0.5 mW to avoid undesired heating effects. The backscattered light was collected, and its spectrum was analyzed by a double
monochromator and detected by a liquid-nitrogen cooled charge-coupled device. The adjustment curves applied to the data were Lorentzian curve fitting.

2.2.3 Surface quality

The cut surface finish was measured using a VEECO non-contact high-resolution profiler Wyko NT 1100. The parameter of the areal surface roughness used was root-mean-square roughness \( R_q \). Six measurements were carried out for each machining track: three measurements in the direction [100] and three measurements in the direction [110] and then estimated an average and standard deviation \( R_q \) for each cutting condition.

2.2.4 Uncut shoulders and critical thickness of cutting

The Olympus LEXT OLS4100 laser scanning digital microscope non-contact 3D was used to analyze the uncut shoulders of each cut track (Fig. 6), formed by the depth of cut (dc) and the cutting radius \( (R_c) \) from the machining experiment (Fig. 3). The cutting radius \( (R_c) \) was estimated focusing the microscope on each uncut shoulder, where the tool stopped and was withdrawn.

Nevertheless, when the rake angle becomes more negative, the chip cross-sectional area generated in front of the tool rake face forms a different curvature arc corresponding to its nose radius \( (R_e) \). For example, when a 0° tool rake angle is used (Fig. 7), the curvature arc formed by the cutting radius \( (R_c) \) corresponds to the circumference arc on the tool nose radius as schematically shown in Fig. 7 \( (R_c = R_e) \). However, when the tool rake angle turns negative (Fig. 7), the curvature are formed by the cutting radius acting on the rake face becomes larger than the tool nose radius \( (R_c > R_e) \). This cutting radius \( (R_c) \) is proportional to the rake angle \( (\alpha) \).

The increase in the cutting radius is not entirely a circumference; its actual shape resembles a parabola. However, when machining with a diamond tool, the tool nose radius is much larger than the depth of cut, and a circumference can estimate the cutting radius.

The onset of fractures is usually verified along the uncut shoulders [24]: the point where the effective thickness of...
cutting \( h_e \) reaches its critical value (Fig. 8: Fracture), and it is called the critical-effective thickness of cutting (\( h^*_e \)). At this point, the material does not support more deformations, and the phase transition ceases. Since the critical depth of cut (\( d_{c*} \)) of this fracture has been measured, the critical-effective thickness of cutting (\( h^*_e \)) can then be determined by the tool feedrate (\( f \)), the tool rake angle (\( \alpha \)), and the cutting radius (\( R_c \)), as expressed by the equations in Fig. 8.

3 Results and discussion

The statistical matrix provided the response surfaces of the cutting transition pressure and surface finish. The level curves of the transition pressure response surfaces can produce an accurate mapping of the ductile-to-brittle transition region. Based on this mapping, parameters for roughing operations were selected. The level curves
of the surface finish allowed to determine the tool rake angle to obtain an optimum value of the surface finish. In addition, the application of the critical thickness of cutting was generalized to other tool nose radius sizes and different rake angles, taking into account the influence of this angle on the nose radius contact area during chip formation. Thus, based upon these results, the study was ended with the manufacture of two Fresnel microlenses.

3.1 Machining experiment

The machining experiment determined the ductile machining region for a specific tool nose radius (Re 762 µm) and the best tool rake angle for finishing. Furthermore, the uncut shoulders were analyzed through this same experiment to generalize the optimal material removal for any tool nose radius.

Figure 9 presents a general view of the machined monocrystalline silicon sample after cutting tests, with ten tracks corresponding to the machining conditions, as shown in Table 2. The crystallographic direction [110] has darker shades than the direction [100] in the same track. This darker shade represents damage caused by microcracks on the machined surface.

Figure 9 shows that the material removal occurred predominantly in brittle mode within tracks 6 and 10; ductile and brittle within tracks 1, 3, 4, and 5; and predominantly in ductile mode within tracks 2, 7, and 8. Considering the direction where the brittle mode is predominant, direction [110], it is important to mention that the direction [110] is the softest direction on the crystallographic plane (100) [7]. The brittle fracture occurring preferentially in the direction [110] supports the results reported by Mukaida and Yan [35], who performed turning on silicon (100). Jasinevicius et al. [36] explained this converse material response: being the direction [110] the softest direction, whose transition pressure will be smaller as well (8–9 GPa) [41], and therefore, the difference between hardness and transition pressure (11–12.5 GPa) will always be broader in the direction [110] in comparison to [100] (12 GPa). The second factor to be considered is that, in machining, the shear stress component is present and lowers the transition pressure of the direction [100] by up to 40% [23, 42]. Thus, it is more difficult for the direction [110] to reach the necessary pressure for the phase transformation, and instead of having a ductile response, the response is fragile.

3.1.1 Cutting forces and material removal

The cutting forces, in an overview plot, behaved as shown in Fig. 10. Before machining, there is signal noise (time from 0 to 2 s). At the time of the tool-piece contact (starting cut), the machining forces increased and stabilized at a maximum value.

Figure 11 shows cutting force behavior on track 9 during one revolution of the workpiece, where it is possible to observe the machining forces changing as the crystallographic direction. This track was chosen because the tool exits and re-enters the part, generating a discontinuity of machining in the direction [100], facilitating the observation of the force signal interruption and its
Fig. 9 Single crystal silicon (100) workpiece from machining experiment with a diamond tool (Re 762 µm). a Track 1 in the center to track 10 on the outmost region of the experiment. b Surface damage state in <100>. c Surface damage state in <110>.
Fig. 10 Behavior of the machining forces of track 7 \((f = 3.0 \mu m/\alpha = 37.5^\circ)\) with diamond tool \((R_e = 762 \mu m)\) on monocrystalline silicon \((100)\). \(F_f\) feed force, \(F_t\) thrust force, \(F_c\) cutting force

Fig. 11 Machining forces of track 9 \((f = 3.0 \mu m/\alpha = 7.5^\circ)\) in the period of one revolution when machining silicon \((100)\) with a diamond tool \((R_e = 762 \mu m)\). Force decayed in one of the directions \([100]\) due to the cut discontinuity
resumption immediately after, at the specific direction, as marked with a dashed line in Fig. 9a.

Table 3 presents the values of the machining forces for each machining track of the experiment.

The statistical treatment of the central composite planning (CCD) of the Ft and Fc forces (Table 3) distributed over the effective area of the cutting section of the tool, Eq. (2), provided the respective brittle-ductile transition pressures as shown in Fig. 12.

The brittle-to-ductile transition was determined by comparing the transition pressure results generated in each machined track and the response surface level curves. In the crystallographic direction [100], tracks 6 and 10 underwent brittle fractures on their surfaces (Fig. 9). The band that passed above these tracks is the dash-point curve (Fig. 12c). The inner portion, delimited by the dash-point curve, defined the material removal mode as predominantly brittle, and the outer part: prominently ductile. The value of the transition pressure for direction [100] is around 12 GPa in this transition line.

The same criterion for determining the brittle-to-ductile transition was applied for direction [110]. Tracks 2 and 7 were predominantly ductile in this crystallographic direction. However, track 8 presented itself as an intermediate point as two of the four directions [110] have fractured (Fig. 9). As Fig. 12d, the band that passed below point 7 (track 7) and above point 8 (track 8), bounded by the dash-point ellipse, determined the transition region in that direction. All other bands that have suffered a fracture were found in the inner part of this trace-point ellipse. The value of the transition pressure for this band, corresponding to the direction [110], was approximately 13 GPa.

The direction [110] generated a smaller cutting and thrust force in comparison to the direction [100] (Fig. 11), supporting the results presented by O’Connor et al. [7]. In addition, the direction [110] demanded a higher compression pressure (13 GPa) for phase transformation to take place and to shift from brittle to ductile material removal mode (Fig. 12b, d). These two factors confirm what was discussed earlier.

The contour lines of these two crystallographic directions were combined for mapping the shift in transition pressure in both directions simultaneously (Fig. 13). Thus, for a given value of feedrate, there will be a critical angle corresponding to a ductile machining condition. This mapping allowed the selection of machining parameters for roughing operations. As shown in Fig. 13, for a feedrate greater than 4.5 µm/rev, more negative rake angles (−37.5°) would be indicated to achieve material removal predominantly in ductile mode. This performance improvement in ductile mode machining using a more negative rake angle was also observed for other types of crystals [43]. Although, for a very long cutting distance (30–60 km), high negative rake angles can generate a higher rate of tool wear [44].

### 3.1.2 Residual stress vs. cutting force under ductile mode

Residual stresses were estimated based on the equation proposed by Weinstein and Piermarini [45] on the shift in the characteristic crystalline peak of silicon under high compression, which revealed that the greater the compression in silicon, the more the crystalline peak shifts to the right of the crystalline peak centered in 520.3 cm⁻¹. From this experiment, the authors proposed an equation for determining the value of material stress for the peak shift, which can be expressed with a good approximation by:

\[
\sigma = 192.3(\omega - \omega_0) \quad \text{MPa}
\]  

(4)

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**Table 3** Average forces and standard deviation of the silicon machining experiment (100) with a diamond tool (Rε 762 µm) at a machining depth of 10 µm

| Experiment f (µm/rev) | α (º) | Track | Fc [100] Mean | Fc [100] Dev | Ft [100] Mean | Ft [100] Dev | Fc [110] Mean | Fc [110] Dev | Ft [110] Mean | Ft [110] Dev |
|----------------------|------|------|---------------|-------------|---------------|-------------|---------------|-------------|---------------|-------------|
| 4.0                  | 0º   | 1    | 0.715         | 0.026       | 0.585         | 0.012       | 0.828         | 0.083       | 0.470         | 0.036       |
| 4.0                  | −45.0º | 2   | 0.705         | 0.008       | 0.455         | 0.019       | 1.036         | 0.022       | 0.552         | 0.041       |
| 4.0                  | −22.5º | 3   | 0.565         | 0.097       | 0.138         | 0.008       | 1.057         | 0.208       | 0.132         | 0.008       |
| 4.0                  | −22.5º | 4   | 0.324         | 0.018       | 0.113         | 0.006       | 0.745         | 0.046       | 0.128         | 0.017       |
| 2.5                  | −22.5º | 5   | 0.512         | 0.002       | 0.191         | 0.003       | 1.239         | 0.012       | 0.403         | 0.001       |
| 5.5                  | −22.5º | 6   | 0.320         | 0.012       | 0.207         | 0.000       | 0.633         | 0.023       | 0.313         | 0.001       |
| 3.0                  | −37.5º | 7   | 0.512         | 0.028       | 0.420         | 0.013       | 0.688         | 0.046       | 0.482         | 0.038       |
| 5.0                  | −37.5º | 8   | 0.706         | 0.012       | 0.642         | 0.011       | 1.006         | 0.014       | 0.899         | 0.020       |
| 3.0                  | −7.5º  | 9    | 0.536         | 0.032       | 0.271         | 0.013       | 1.220         | 0.105       | 0.426         | 0.048       |
| 5.0                  | −7.5º  | 10   | 0.403         | 0.015       | 0.286         | 0.010       | 0.865         | 0.043       | 0.517         | 0.012       |

*Dev: standard deviation
where $\alpha_0$ is the value of the characteristic crystalline peak of silicon (from the measurements of the specimen: 520.283 cm$^{-1}$), and $\alpha_1$ is the value of the crystalline peak measured on the cut surface after machining.

Figure 14 presents Raman spectra referring to cutting conditions 7 and 8 for directions [100] and [110]. In both directions, the generation of amorphous and crystalline phases (a-Si + c-Si) is due to the Si-I in the diamond cubic structure. The broad band at 470 cm$^{-1}$ denounces the phase a-Si, and the peak around 522 cm$^{-1}$ is the phase c-Si. The stress state probed by micro Raman spectroscopy is due to this strained subsurface layer. The difference in residual stresses observed for different directions may be attributed to the depth of the damaged subsurface, which is deeper in the hardest direction [41]. The thin a-Si layer is formed on the top and by nanocrystalline grains immersed in amorphous silicon in a strained layer with defects extending downwards [7, 10, 12, 24, 27, 41, 46, 47].

Figure 15 presents the results of the main cutting force and residual stresses for conditions 7 and 8 (Fig. 9b, c), which showed a predominantly ductile response in both crystallographic directions. Even as the crystallographic direction changes, the main cutting force increases by almost the same proportion as feedrate. Moreover, the graph shows
that the main cutting force decreased when the cutting shifted from the hardest direction [100] to the softest direction [110]. However, it is interesting to note that the residual stresses behaved contrary to the main cutting force. This increase in residual stresses proved to be higher for lower feedrate (3 µm/rev).

3.1.3 Surface finish

According to the machined surfaces state (Table 4), these surfaces alternated between fractured (brittle mode) and crack-free (ductile mode). Tracks 6 and 10 are predominantly brittle over the machined surface within [100] and [110] crystallographic directions. Conversely, only track 7 presented a predominantly ductile response over both crystallographic directions. The roughness of these surfaces was measured using a profile. An analysis of the effect of the average showed the significant difference in the finish (Rq) of the surfaces obtained in the ductile mode (3–8 nm) and the brittle mode (100–250 nm).

The differences presented between tracks 7 (fully ductile), 6, and 10 (fully brittles) can be explained by the differences in feedrate and values of the rake angle. The former was machined with a feedrate of 3.0 µm/rev and a rake angle of −22.5°. In the case of tracks 6 and 10, they were machined with the highest values of tested feedrate and different negative rake angles. However, the response of track 10 was better in both crystallographic directions in terms of surface roughness. Although it is not possible to identify a brittle-to-ductile transition point along the uncut shoulder for both brittle cutting conditions, the portion closer to the surface of the uncut shoulder of track 10 presents with fewer and smaller microcracks when compared to the formed uncut shoulder on track 6. Track 10 used a feedrate of 5 µm/rev and a rake angle of −7.5°; meanwhile, for track 6, the feedrate was 5.5 µm/rev and a rake angle of −22.5°. Under feedrate of 5 µm/rev, the material

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**Fig. 13** Mapping the ductile region of silicon (100) from level curves shown in Fig. 12c and d for roughing operations in machining with circular diamond tool Re 762 µm
removal becomes predominantly ductile only when the rake angle reaches $-37.5^\circ$, as observed for machined track 8 (Table 4).

Regarding the other cutting conditions tested, the ductile regime was observed for remaining tracks (1, 2, 3, 4, 5, 8, and 9) in the crystallographic direction [100], but the brittle response was dominant for the direction [110].

The measurements of root-mean-square surface roughness (Rq) of the machined surfaces (Table 4) generated the response surfaces of the CCD’s statistical treatment. Figure 16 shows the level curves of these response surfaces. The regions of optical finish (Rq $\leq$ 15 nm) are restricted within the trace-point band. In the crystallographic direction [100], it is possible to obtain an optical finish with a high feedrate of 5 µm/rev coupled with a very negative rake angle ($-45^\circ$). This behavior also occurred for all combinations of feedrates and rake angles that were within the limit band (dash-point line). However, these limit bands became narrow in the crystallographic direction [110].

To achieve the same order of magnitude in the optical finish in the direction [110], feedrates from 2.5 to 5.5 µm/rev needed to work in conjunction with rake angles around $-37.5^\circ$. Furthermore, based on this, it would be an appropriate choice for a feedrate and rake angle condition that best meets the direction [110], which would therefore fully meet direction [100]. In this case, the recommended rake angle is around $-37.5^\circ$ for the best surface finish.

3.1.4 Uncut shoulders

The analysis of the brittle-to-ductile transition on the uncut shoulder enabled the determination of the critical depth of cut ($d_c^*$), corresponding to where the cracks stopped propagating (Fig. 17).

Figure 18 shows the cutting radii measured in each of the machined tracks. The equation that governs the characteristic curve of the variation of the cutting radius as a function of the rake angle was developed based on three-dimensional drawings (Eq. 5) to match the rake angles’ values exactly. The values adjusted by Eq. 5 were close to the experimental values (Fig. 18). On average, there was a 5% difference between the calculated cutting radii and the cutting radii of the machining experiment. This difference can be related to fractured surfaces.

$$R_c = R_e \left(1.0078 - 3.065 .. |\alpha|^1.804 \cdot 10^{-4}\right)^{-1} \mu m \quad dc \leq 0.1R_e$$  

(5)
Table 4  Surface state of the machined tracks cut with a diamond tool ($R_0.762\text{nm}$)

|  | 1 ($f(4.0\mu m/\alpha^0)$) | 2 ($f(4.0\mu m/\alpha-45^\circ)$) | 3 ($f(4.0\mu m/\alpha-22.5^\circ)$) |
|---|---|---|---|
| Ra | 6.43 nm | 3.33 nm | 3.33 nm |
| Rq | 7.95 nm | 4.09 nm | 4.09 nm |
| Rt | 49.35 nm | 22.33 nm | 22.33 nm |

[100] [110]
Table 4 (continued)

| 4 (f.4μm, α=22.5°) | 5 (f.5μm, α=22.5°) | 6 (f.5μm, α=22.5°) | 7 (f.3μm, α=37.5°) |
|---------------------|---------------------|---------------------|---------------------|
| Ra 3.82 nm          | Rq 4.68 nm          | Rt 26.37 nm         | Ra 123.16 nm        |
| Ra 2.52 nm          | Rq 3.14 nm          | Rt 20.44 nm         | Ra 139.46 nm        |
| Ra 3.04 nm          | Rq 3.75 nm          | Rt 19.59 nm         | Ra 70.43 nm         |
| Ra 150.70 nm        | Rq 202.45 nm        | Rt 1440 nm          | Ra 178.53 nm        |
| Ra 3.46 nm          | Rq 4.21 nm          | Rt 21.79 nm         | Ra 2.61 nm          |
| Ra 2.61 nm          | Rq 3.20 nm          | Rt 17.40 nm         | Ra 2.61 nm          |

The International Journal of Advanced Manufacturing Technology (2022) 120:843–867
Table 4 (continued)

| 8 (5.0 μm/α1.75°) | 9 (3.0 μm/α1.75°) | 10 (5.0 μm/α1.75°) |
|---------------------|-------------------|---------------------|
| Ra 3.71 nm | **Rq 4.62 nm** | Ra 3.60 nm | **Rq 4.57 nm** | Ra 67.51 nm | **Rq 94.78 nm** |
| Rt 26.04 nm | | Ra 91.57 nm | **Rq 140.11 nm** | Ra 128.60 nm | **Rq 187.63 nm** |
| | | Rt 1590 nm | | Ra 1490 nm | |
Since determining cutting radius (Rc), the critical-effective depth of cut (dc*) was calculated so that, according to the equations in Fig. 8, it was possible to estimate the critical-effective thickness of cutting (he*) of silicon as a function of the tool’s rake angle (Fig. 19). Thus, a tool’s rake angle variation from 0° to −45°, the critical-effective thicknesses of cutting values for the crystallographic direction [100] varied from 250 to 550 nm.

Yan et al. [46] measured the thickness of cutting versus the tool rake angle variation between −15° and −45° using nanoprecision plunge cut tests on silicon (100). The authors found that in direction [100], there was a variation in the

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**Fig. 15** Residual stress and cutting force from the track 7 (f 3.0 µm/α−37.5°) and track 8 (f 5.0 µm/α−37.5°) with a diamond tool (Re 762 µm)

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**Fig. 16** Level curves of the Rq (nm) surface roughness of the machining experiment of silicon (100) with single point diamond tool (Re 762 µm)
The increase in critical thickness was directly proportional to the negative rake angle [31].

Regarding the direction [110], it was possible to estimate the critical-effective thickness of cutting only on track 7. All other tracks showed microcrack along all the uncut shoulder width and propagated to the finished surface. Comparing the critical-effective thickness of cutting \( h_e^\ast \) from track 7 in both directions [110] and [100], \( h_e^\ast [110] \) corresponds to 35% of \( h_e^\ast [100] \). This reduction in the critical thickness of cutting in the direction [110] was observed by Yan et al. [46]. Similarly, O’Connor, Marsh, and Couey [7] concluded that the ratio between the critical thickness of cutting for [110] and [100] directions was 33%.

In addition to the critical thickness of cutting, the value of the critical depth of cut \( (d_c^\ast) \) was estimated. At the critical depth of cut, cracks propagated but did not reach the cut surface line [24]. Such depth must be greater than 3 µm for direction [100]. As shown in Fig. 9, this value was found from tracks 3 and 4: three of the four directions [100] of these tracks showed slight fractures on the surface (Fig. 9b), \( h_e^\ast \) is likely to be at the limit of its critical depth. On the other hand, for direction [110], the critical depth of \( h_e^\ast [110] \) must be greater than 10 µm. This assumption is based on track 8, which presented superficial fractures in two of the four directions [110] (Fig. 9c). Thus, it was found that \( d_c^\ast [110] \) corresponds to 3.35 times \( d_c^\ast [100] \). In machining, the cracks that propagate in the direction [110] reach greater lengths.

Therefore, as mentioned above and according to Fig. 19, in relation to the direction [110], the critical-effective thickness of cutting (nm) and its critical depth of cut (µm) can be determined by:

\[
h_e^\ast [110] = 2.6\alpha + 78 \text{ nm}
\]
Fig. 18 Cutting radius $R_c$ as a function of the rake angle

Fig. 19 Critical-effective thickness of cutting of monocrystalline silicon (100)
After determining the limits of the critical depth of cut and the critical-effective thickness of cutting, the results were applied to manufacture a diffractive optical element (DOE’s). For this, two Fresnel microlenses were machined in Si (100) with concave and convex profiles, diameter of 3 mm, 11 zones with a height of 7 µm (Fig. 20). A rake angle of −37.5° was chosen based on the analyses, seeking a better finishing response in the direction [110] (Fig. 16).

First, it was necessary to carry out a roughing step on the surface. A device adjusted the rake angle at −37.5° for the roughing operation. For this rake angle, the maximum possible tool feedrate is 3 µm/rev, as the mapping provided by Fig. 13 for ductile material removal. The depth of cut (dc = 10 µm) and diamond tool nose radius (Re = 762 µm) were the same as the experiment.
3.2.1 Fresnel lenses

After the roughing process, a half-radius diamond tool $R_e = 100 \mu m$ (Fig. 2) manufactured the Fresnel lenses. With the rake angle positioned at $-37.5^\circ$, the critical-effective thickness of cutting is around 175 nm in the direction $[110]$ (Eq. 6). Thus, two decisions can be made: case 1—would be to work within this limit of the critical-effective thickness of cutting, $h_e^*[110]$, and to set up $h_e^*[110]$ to a height corresponding to its critical depth $24$. In case 2, would be to work with a maximum-effective thickness of cutting ($h_{e\text{max}}$) always smaller than the critical-effective thickness of cutting ($h_e^*$). It was chosen to work with the second option.

The tool feedrate ($\mu m/rev$) was determined by the common mathematical relationships of the maximum-effective thickness of cutting for tools with nose radius. It took the critical-effective thickness of cutting, from direction $[110]$, as a basis (Eq. 6). Thus, for ductile machining of Si (100) with a round tip diamond tool, the tool feedrate can be determined by the expression of Eq. 8.

$$f = \frac{(2.6\alpha + 78).10^{-3}}{\sin \left( \arccos \left[ 1 - d_c \left( R_c \cos \alpha \right)^{-1} \right] \right)} \frac{1}{C} \mu m/rev$$

C: safety factor $\geq 1 \alpha$ in absolute values $dc$ can be substituted for $dc^*$ for case 1

Using Eq. 8 for a tool with a tip radius $R_e = 100 \mu m$ (cutting radius $R_c = 125 \mu m$ according to Eq. 5) depth of cut $dc = 7 \mu m$ (height of the Fresnel lens), rake angle $\alpha = -37.5^\circ$ (optimal finish), and considering a safety factor $C = 2$; the tool feedrate $(f)$ for ductile machining is in the order of 0.25 $\mu m/rev$. The maximum thickness of cutting for this feedrate is 100 nm, which is less than the critical-effective thickness of cutting (175 nm) of the direction $[110]$.

The tool feedrate set up at 0.25 $\mu m/rev$ reached surface finish in the curved areas of the Fresnel lenses (Fig. 20). All curved surfaces in the lens areas were free of brittle damages, demonstrating the ability to apply the value of the critical-effective thickness of cutting for different rake angles and tool nose radii. This ductile removal was possible since these thicknesses of cutting are related to the cutting radius $(R_c)$ during machining; thus, providing the critical-effective thickness of cutting.

Nevertheless, there was a particular fracture in the vertical walls of Fresnel lenses in the tool’s plunge movement (Fig. 20). This fracture could be due to small wear at the tool tip, as shown in Fig. 21. In this case, since the tip is very narrow, the contact pressure during silicon cutting may have promoted small wear due to phenomena involving high pressures on the diamond [48]. For this particular case, the tool first performs a plunging motion into the workpiece, during which the lens edges break, and then the tool performs the cutting of the lens curvatures. The cutting becomes stable as the tool path follows the lens zone profile. If cracks develop during the plunging motion, the tool removes such cracks when cutting the curvatures in the ductile material removal mode.

4 Conclusions

In this study, transition pressure, surface finish, the influence of the rake angle on the cutting radius, and critical thickness of cutting were all characterized by the experimental-statistical analysis of ultra-precision machining of single-crystal silicon (100) with a rounded-nose diamond tool, varying tool rake angle, and feedrates. Through this approach, it was possible to suit the value of the transition pressure of silicon, during machining, with the respective critical thickness of cutting at the equivalent cutting radius formed by the correspondent rake angle, thus improving the surface finish. The main conclusions considering the mentioned factors are:

- The mapping of the response surfaces of brittle-to-ductile transition pressure enables the determination of the roughing parameters of the machining in ductile mode: high feedrate with the appropriate rake angle.
- The mapping of the surface finish response surfaces enables the determination of the tool’s rake angle for the optimal machining finish. This angle is around $-37.5^\circ$ for silicon (100).
- The residual stress value estimated on the ductile machined surfaces is inversely proportional to machining forces. The main cutting force decreased when the cutting shifted from the hardest direction $[100]$ to the softest direction $[110]$, and the residual stresses behaved contrary to that of the main cutting force. This increase in residual stresses proved to be higher for lower feedrate (3 $\mu m/rev$).
- The rake angle significantly influences the cutting radius during machining, which affects the critical thickness of cutting. The larger the rake angle, the larger the cutting radius and, consequently, the critical thickness of cutting shifts upward along the uncut shoulder as the cutting radius increases (Fig. 1c). This increase in cutting radius probably prevents the cracks from replicating into the final cut surface with the rise of the feedrate.
- Besides the differences reported in the literature on the critical thickness of cutting between directions $[110]$ and $[100]$, there are differences in the lengths that cracks travel in each direction, which reached greater lengths (around 3.35 times) in direction $[110]$. 
The results showed a correlation between the critical thickness of cutting, the tool rake angle, and the cutting radius during machining. These three factors should be considered in conjunction to achieve machining in a ductile regime and a crack-free surface finish to allow the deterministic generalization of the optimal machining parameters.

Finally, the results enabled answering questions under which machining conditions and tool geometry account for better surface finishes, lower machining forces, and lower residual stresses. The central composite design (CCD) provided responses to establishing under which cutting radii yielded more ductile material removal, avoiding brittle response, and mitigating anisotropic response. This approach will be applied to other semiconductor crystals to find a general response for determining the cutting parameters in the ductile mode for machining diffraction optical elements made of crystals such as GaAs, InSb, among others.

Author contribution Marcel Henrique Militão Din: conceptualization, investigation, methodology, data curation, formal analysis, software for cutting forces, writing—original draft, visualization, validation. José Antonio Otoboni: methodology, validation, writing—review and editing. Renato Goulart Jasinevicius: supervision, conceptualization, methodology, resources, validation, investigation, data curation, writing—review and editing.

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Availability of data and material The data sets generated and analyzed from the current study are available upon request from the corresponding author.

Code availability The software application developed for the analysis of cutting forces is available on request from the corresponding author.

Declarations

Ethics approval and consent to participate Not applicable.

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Conflict of interest The authors declare no conflict of interest.

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