Deformation and removal of semiconductor and laser single crystals at extremely small scales

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Abstract
Semiconductor and laser single crystals are usually brittle and hard, which need to be ground to have satisfactory surface integrity and dimensional precision prior to their applications. Improvement of the surface integrity of a ground crystal can shorten the time of a subsequent polishing process, thus reducing the manufacturing cost. The development of cost-effective grinding technologies for those crystals requires an in-depth understanding of their deformation and removal mechanisms. As a result, a great deal of research efforts were directed towards studying this topic in the past two or three decades. In this review, we aimed to summarize the deformation and removal characteristics of representative semiconductor and laser single crystals in accordance with the scale of mechanical loading, especially at extremely small scales. Their removal mechanisms were critically examined based on the evidence obtained from high-resolution TEM analyses. The relationships between machining conditions and removal behaviors were discussed to provide a guidance for further advancing of the grinding technologies for those crystals.

Keywords: deformation and removal, semiconductor, laser crystal, transmission electron microscopy (TEM), grinding

1. Introduction
Semiconductor and laser crystals are essential substrate materials for modern electronic and photonic devices that are widely used in communication, energy, medical, and military industries. The development of advanced melt-growth methods [1–5] has enabled the mass production of large-size single crystal ingots for semiconductors such as silicon (Si), gallium arsenide (GaAs) and gallium oxide (Ga₂O₃), and laser crystals such as yttrium aluminum garnet (YAG) and gadolinium gallium garnet (GGG). Usually, an as-grown single crystal ingot must be machined into thin substrates with high surface quality and dimensional precision, and more importantly, free of subsurface damage (SSD). Given the hard and brittle nature of semiconductor and laser single crystals [6], grinding is commonly used to planarize the substrates after being sliced and achieve the required surface quality prior to further polishing. An optimal balance between surface quality and processing time is the key for the success in the grinding of those substrates. Therefore, the deformation and removal behaviors of those single crystal materials were investigated extensively under multiscale loading conditions. Nevertheless, the initiation and evolution of defects in semiconductor and laser single crystals induced by mechanical loading are yet to be...
thoroughly examined in a systematic manner, especially at extremely small scales.

In this review, the initiation and evolution of crystal defects in semiconductor and laser crystals induced at extremely small scales were systematically summarized, and their roles in the deformation and removal mechanisms of the selected single crystals under variable grinding conditions were examined. The primary aim of this work is to demonstrate how the defects induced in instrumented loading at extremely small scales can be used for optimizing the grinding processes for those single crystals. Specifically, in section 2 of this review, crystal structures, possible defects and phase transformation, and mechanical properties of typical semiconductor and laser single crystals are summarized. In section 3, we analyze the initiation of the crystal defects in those single crystals induced by instrumented nanoindentation and nanoscratch, which are the two methods being widely used to mimic the grit-work material interaction in a grinding process. In section 4, the deformation and removal characteristics of semiconductor and laser single crystals involved in grinding are discussed, with an emphasis on the grinding process that has extremely small material removal, so the deformation should occur in the ductile-like removal regime [7–9]. The outline of this review is schematically shown in figure 1, where the topics being discussed in this work are highlighted in red.

2. Crystal structure and mechanical property

2.1. Crystal structure

Most of the semiconductor and laser single crystals are inorganic solid-state compounds, in which atoms are distributed in a certain crystal structure, purely or mainly through strong covalent/ionic bonding [10]. The crystals thus have stable crystal structures and high melting points, hence are ‘hard’ and ‘brittle’, which are significantly different from metals or metallic materials that exhibit more ductility under mechanical loading. Therefore, semiconductor and laser single crystals are often categorized as difficult-to-machine materials.

Table 1 shows the crystal structures, mechanical properties and applications of typical semiconductor and laser single crystal materials, including Si, GaAs, β-Ga2O3, YAG and GGG. This review focuses on these selected single crystals that have representative features, and more importantly, can be grown by melt-growth methods that have great potential to vastly reduce the cost for mass production, even though some of them are currently still highly priced. Among those selected materials, monocrystalline silicon is the mostly recognized first generation semiconductors, and has been used in electronic industries for decades and nowadays more than 95% electronics devices are fabricated on Si substrates [11]. According to [12], Si has a face centered cubic (fcc) structure with a lattice spacing of 0.543 nm, as shown in figure 2(a) and the neighboring Si atoms are held by strong covalent bonds, which leads to the stable crystal structure of Si. As a result, monocrystalline Si has a melting point of 1410 °C, a hardness of 13 GPa as measured using nanoindentation [6], and a fracture toughness of 0.62–1.69 MPam1/2, which is thus categorized as hard and brittle. Similar to Si, GaAs is a typical second generation semiconductor and has demonstrated superior electronic and optoelectronic properties for high frequency applications [13]. β-Ga2O3 attracts attention for its superior properties owing to the incredibly large bandgap of 4.7–4.9 eV [14]. Rare-earth garnet crystals, such as YAG and GGG, have been widely used in solid-state lasers and scintillation detectors due to their excellent optical homogeneity and steady chemical and physical properties [15, 16]. Note that crystal defects in those single crystals are often induced in their machining processes, even with extremely small loads and the associated deformations involve the change of crystal structures. Therefore, high resolution transmission electron microscopy (HRTEM) must be used for the deformation characterization, as
Table 1. Crystal structure, mechanical property and applications of selected single crystals.

| Selected materials | Crystal structure | Unit cell | Hardness (Gpa) | Toughness (MPam\(^{1/2}\)) | Crystal phases | Applications | References |
|--------------------|------------------|-----------|----------------|-----------------------------|----------------|-------------|------------|
| Si                 | Fcc              | \(a = b = c = 0.5430\) nm, \(\alpha = \beta = \gamma = 90^\circ\) | 13 | 0.62–1.29 | Si-I, Si-II, Si-III, Si-XII | Electronics, photonics, solar power electronics, diode/transistor | [6] |
| GaAs              | Fcc              | \(a = b = c = 0.5650\) nm, \(\alpha = \beta = \gamma = 90^\circ\) | 10.1 | 0.45 | Zinc blende, Rocksalt | Electronics, solar energy | [18] |
| \(\beta\)-Ga\(_2\)O\(_3\) | Monoclinic       | \(a = 1.2214\) nm, \(b = 0.3037\) nm, \(c = 0.5798\) nm, \(\alpha = \gamma = 90^\circ, \beta = 103.8^\circ\) | 14.5 | 0.79 | \(\alpha\), \(\beta\), \(\gamma\), \(\delta\), \(\varepsilon\) and \(\kappa\)-Ga\(_2\)O\(_3\) | Power electronics, diode/transistor | [19, 20] |
| YAG               | Cubic            | \(a = b = c = 1.199\) nm, \(\alpha = \beta = \gamma = 90\) | 20.2 | 1.82 | N.A. | Laser optics | [21] |
| GGG               | Cubic            | \(a = b = c = 1.238\) nm, \(\alpha = \beta = \gamma = 90\) | 13 | 1.2 | N.A. | Laser optics | [22, 23] |
shown in figure 2(b), where the crystalline structure of $\beta$-Ga$_2$O$_3$ are clearly presented at atomic scale along the zone axis of [010] [17].

2.2. Crystal defects

Defects refer to the imperfect arrangement of atoms in a single crystal, which might be induced during crystal growth [24–26] or by the deformation caused by mechanical loading [17, 27–29]. The defects can be briefly categorized as point defects (vacancy, interstitial defects, and impurities), line defects (dislocations), planar defects (stacking faults, twinnings, and grain boundaries), and volume defects (voids, cracks, and amorphization) [30]. Typical defects such as edge dislocation, twinning, and amorphization are illustrated in figure 3. Defects play important roles in the fabrication of wafers and can affect the performance of final electronic and photonic devices [31]. Usually, defects in semiconductor wafers are undesirable and may cause failures or shortened lifetime of the fabricated electronic devices. However, initiation and evolution of defects are the prerequisite for the material removal in a machining process. For instance, slipping or cracking often leads to the chunk removal of materials, and dislocations or amorphization introduced during grinding may facilitate the material removal in the subsequent mechanical and/or chemical polishing process. Therefore, the knowledge of initiation and evolution of the defects in crystals is crucial for the understanding of the deformation and removal of single crystals involved in a machining process.

2.3. Phase transformation

Besides the aforementioned crystal defects, the phase of an original crystal structure may be transformed into a metastable phase that is commonly observed in semiconductor single crystals being deformed under mechanical loading. For example, pristine Si is of a fcc structure, but at least 13 other phases were reported at elevated pressures up to 250 GPa through theoretical calculation or pressure cell experiments [32]. Moreover, new phases are still emerging when unconventional approaches such as ultrafast laser-induced confined micro-explosion were successively used to handle Si crystal [33]. However, the most commonly reported polymorphs of Si under conventional mechanical loading are Si-II ($\beta$-Sn, body centered tetragonal), Si-III (BC-8, body centered cubic), and Si-XII (R-8, rhombohedral) [34–36] and their crystal structures are shown in figure 4 [37].

GaAs and Ga$_2$O$_3$ also has different high pressure phases. Besides the most stable zinc-blende phase, GaAs has a rocksalt structure [38]; while Ga$_2$O$_3$ has metastable structures, such as $\alpha$, $\gamma$, $\delta$, $\varepsilon$, and $\kappa$-Ga$_2$O$_3$ together with the most stable $\beta$-Ga$_2$O$_3$ phase [19]. The knowledge of metastable phases induced upon mechanical loading is critical in the understanding of the metastable phase-associated deformation of semiconductor materials.

Figure 2. (a) Fcc structure of monocrystalline Si [12] and (b) HRTEM image of $\beta$-Ga$_2$O$_3$ [17]. (a) This crystal structure of silicon image has been obtained by the author(s) from the Wikimedia website, where it is stated to have been released into the public domain. It is included in this article on that basis. (b) Reprinted from [17], Copyright 2017, with permission from Elsevier.

Figure 3. Typical crystal defects in semiconductor and laser crystals: (a) edge dislocation, (b) twinning and (c) amorphization. [30] 2015, reprinted by permission from the publisher (Taylor & Francis Ltd).

Figure 4. Crystal structures of metastable phases of Si. [37] 2015, reprinted by permission from the publisher (Taylor & Francis Ltd).
3. Deformation under instrumented mechanical loading

The deformation and removal mechanisms of single crystals involved in a machining process are complicated. It is very challenging, if not impossible, to analyze merely based on the machining parameters, because the early stage deformation of the machined single crystals is often concealed [43–48]. Therefore, to understand the mechanisms of deformation and removal events occurring in a grinding process, instrumented mechanical loading using a well-shaped diamond tip or single diamond grit is often employed to mimic the grit—work material interaction involved in a fine machining process, especially at its early stage. In doing so, a thorough understanding of the deformation and removal behaviors under mechanical loading can thus be obtained through studying initiation and evolution of crystal defects, and phase transformation of crystallite at nanometric scales, under controllable mechanical loads. Such instrumented mechanical loading can be achieved through indenting, scratching and single grit cutting at nanometric scales. The first two methods are the focus of this review as they are widely used and the research on single grit cutting is relatively limited.

3.1. Nanoindentation induced deformation

Nanoindentation was originally developed to measure the hardness and elastic modulus of a solid material or thin film at nanoscale in a non-destructive way. Later, it was utilized to investigate the deformation behavior of a material and its mechanical properties can be extracted by relating the induced deformation patterns to the applied loading conditions [17, 27, 49–52]. During nanoindenting, a diamond tip is used to deform a specimen with a controlled normal load that varies from a few micro-Newton (μN) to several tens of milli-Newton (mN). With the aid of TEM analysis, nanoindentation makes it possible to investigate the development of crystal defects in a single crystal with a wide range of stress levels.

3.1.1. Deformation of Si under nanoindentation. The deformation of single crystal Si induced by nanoindentation has been intensively studied [27, 34, 51, 53–59]. In the pioneering work of Wu et al [51], the deformation of single crystal Si under a Vickers indenter with a load of tens of mN was systemically investigated based on the direct evidence obtained from cross-sectional TEM examination. This study showed that the deformation pattern of single crystal Si included the formation of amorphous Si (α-Si), crystal defects, lattice distortion, and the refinement and rotation of grains, but no microcracks were observed in the subsurface zone under the Vickers indenter. More detailed knowledge on the deformation behaviors of monocristalline Si was obtained by taking the effects of tip geometry and loading conditions into account [52, 54, 57].

Previous works [34, 60] showed that amorphization was the most commonly observed deformation phenomenon during nanoindenting of monocrystalline Si and the α-Si region was located directly beneath the indenter, i.e. a V-shaped α-Si region was observed under the Vickers indenter [51]. The crystal structures of the nanoindentation induced α-Si were later studied by use of HRTEM and nanodiffraction to reveal the transition path for the formation of α-Si [61, 62]. The unloading rate was found to be the controlling factor to affect amorphous structures during indenting of silicon [61]. The average atomic distance of α-Si varied from 0.25 to 0.28 nm when using a relatively fast unloading rate of 3 mN s⁻¹ and ranged from 0.23 to 0.24 nm at a slower loading rate of 0.6 mN s⁻¹ [61, 62]. This is because at the faster unloading rate α-Si phase was transformed from Si-II phase; whereas at the lower unloading rate, α-Si phase was more likely transformed from Si-XII and Si-III.

Figure 4. Crystal structures for metastable phases of (a) Si-II, (b) Si-III and (c) Si-XII. Reproduced from [37]. © IOP Publishing Ltd. All rights reserved.
\(\alpha\)-Si was firstly induced in the plastic deformation of monocrystalline Si during nanoindentation at relatively small loads, or small applied stress, other crystal defects can also be generated with increased applied stress. Zardui et al. [63] used a spherical diamond tip, aimed to minimize the effect of tip shape on stress concentration, to indent a Si substrate and found that amorphization and defects lying in \(111\) lattice planes were induced and their penetration depth increased with the increased load. As shown in figure 5(a), similar phenomena were observed by Bradby et al. [27], as stacking faults, nanotwins and dislocation were induced in the indented monocrystalline Si at a maximum load of 80 mN [27]. Specifically, nanotwins [64] and stacking faults [51] along \(111\) planes were detected on the shoulder of the indent, as seen in figures 5(b) and (c), and dislocations along \(111\) planes were gradually penetrated into the deformed region, as shown in figure 5(a). A single dislocation along \(111\) plane was generated at a relative low load of 30 mN using a spherical tip with radius of 5 \(\mu\)m, and multiple dislocations along both \(111\) and \(111\) planes were observed after nanoindentation at a higher applied load [63]. It is thus reasonable to derive that for the same indenter shape being used a higher applied load (or stress) would lead to a greater dislocation density in monocrystalline Si.

To mimic the penetration event of a diamond grit involved in an ultraprecision grinding process, nanoindentation was carried out at very low loads on Si surfaces. In situ nanoindenting in TEM showed that at the initial stage no phase transformation occurred, but dislocations were observed, as shown in figure 6 [65]. It was the first time that dislocation formation and propagation was observed in Si in situ. It is worth noting that the difference between the in situ and ex situ nanoindentation tests should be attributed to the different confinement conditions in an ultra-thin TEM specimen and a bulk material.

The formation of high pressure Si phases was observed when the indenting load was sufficiently high [27, 49, 54, 56, 63, 66, 67]. Bradby et al. [27] found a thin layer of polycrystalline material identified as rhombohedral Si-XII phase under a sharp and spherical tip, after the release of the load at a relatively slow unloading rate. In contrast, \(\alpha\)-Si was formed when unloaded at more rapid rates. As indicated by the cross-sectional TEM image in figure 7(a), a high pressure phase of Si-XII was formed directly on the top surface of the indented Si and the selected area electron diffraction (SAED) pattern in figure 7(b) was indexed as Si-XII. Zardui et al. [63] and Lloyd et al. [67] both reported the presence of Si-III phase on the indented Si surfaces using cross-sectional TEM analysis. Phase transformation was also reflected on the load–displacement curves, as crystalline nucleation at relatively low unloading rates would lead to pop-outs on the curves, while amorphization at a relatively high unloading rate resulted in no pop-out [50].

HRTEM images enabled the direct observation of the crystalline structures of high pressure phases [58, 68]. The high resolution TEM images shown in figures 8(a)–(c) refer to Si-I, Si-III and Si-XII phases, respectively, which are consistent with the simulated high resolution images as shown in figure 8(d) for Si-I, (e) for Si-III and (f) for Si-XII [58]. Apparently, HRTEM results provide the direct evidences of the existence of both Si-III and Si-XII phases. Very recently, a new phase transformation pathway was reported, in which a transition from Si-I to Si-VI was demonstrated by in situ TEM with atomic scale resolution [64].

The phase transformation route for single crystal Si under mechanical stress can be summarized in figure 9. In summary, the pristine Si-I phase transforms into Si-II upon applied stress, but the formation of either \(\alpha\)-Si or high pressure phases is dependent on the loading and unloading conditions: when the applied stress is sufficiently low, only \(\alpha\)-Si is formed after unloading; when the applied stress is sufficiently high, \(\alpha\)-Si is formed at high unloading rates, while high pressure phases will be formed at relatively low unloading rates. Moreover, nanocrystals with the same crystal structure as Si-I might recrystallize from \(\alpha\)-Si.

Molecular dynamics (MD) simulation was often used to understand the mechanism for phase transformations induced by nanoindentation [53, 69–73]. MD simulation enables the monitoring of positioning of Si atoms under mechanical loading, so that the crystal structures after transformation would be identified convincingly, such as the transformations from Si-I to Si-II during loading and to Si-III, Si-XII and \(\alpha\)-Si during unloading. In general, the MD simulation results agree well with the experimental results [58, 68] presented earlier. Apparently, MD can be a useful complement to TEM, which...
Figure 6. TEM images taken from a video of an in situ nanoindentation into silicon showing: (a) defect free Si sample prior to indentation, (b) and (c) the elastic strain contours at the initial indenting stages, (d) and (e) dislocations started to nucleate, (f) the residual dislocations and strain contours after releasing the applied pressure. [65] 2005, reprinted by permission of the publisher (Taylor & Francis Ltd).

Figure 7. (a) Dark field cross-sectional TEM image of a spherical indent on Si made at a maximum load of 20 mN and (b) the SAED pattern of polycrystalline region. Reprinted from [27], with the permission of AIP Publishing.
help understand the deformation mechanism of crystal materials.

3.1.2 Deformation of GaAs under nanoindentation. The fact that Ga–As atomic bonding strength in single crystal GaAs is weaker than that of Si–Si bond in Si [74, 75], together with the anisotropic properties of GaAs, makes the deformation pattern of GaAs significantly different from that of Si. Thus, it is expected that the deformation mechanism of GaAs is different from that of Si. The nanoindentation studies on the deformation mechanism of GaAs were mainly focused on the (001) crystal plane [29, 38, 76–80]. Those studies used different indenters including Berkovich [81, 82], Vickers [18, 76, 83], and spherical diamond tips [84–86]. Pop-ins were commonly observed on their load–displacement curves, which should be attributed to the sudden nucleation of slips in the GaAs being indented. The critical loads for the occurrence of pop-ins were considerably affected by tip geometries [84, 87].

Prior to the nucleation of slips, dislocation was firstly induced in GaAs at extremely low loads (typically smaller than 0.2 mN when using a sharp cube corner indenter), verified by TEM examination [88]. The dislocation activity in GaAs induced by nanoindentation was also investigated by means of MD analysis [89]. TEM was employed to examine the deformation from both plane and cross-sectional views, and an example is shown in figure 10 [90], where TEM imaging revealed complicated dislocation arrangements in the indented GaAs including both stacking faults and perfect dislocations. The dislocations in the rosette arms were critically investigated [81, 90–93], showing that the asymmetrical rosette was attributed to the effects of tip geometry. Moreover, the rosette arm along the [110] direction was constituted of perfect dislocations, and the length of rosette arm depended on the mobility of dislocations. Twin structures occurred in GaAs when further increased the indentation load [84, 85], which is shown in figure 11(a) with its HRTEM image of twins in figure 11(b). The crystallographic structure of the induced twins was demonstrated to be strongly related to the apex angle of the indenter [94, 95]: diverging twins were formed when the indenter apex angle is smaller than 70.5° that is the angle between two {111} lattice planes of GaAs, as shown in figure 11(a); otherwise, convergent twins would be induced. Further increase in indentation load would induce median cracks [94–96]. The formation of cracks was attributed to the highly concentrated stress field induced by the sharp diamond tips [97].

The phase transformation frequently observed in the deformation of Si was not experimentally observed during nanoindentation of GaAs, but the powderization or even amorphization was reported, which was under the circumstances of ultra-high pressure or extremely severe deformation conditions [18]. Although MD simulation suggested that the phase transformation [38] from original zinc-blende to rock-salt like structure would occur during nanoindentation of...
GaAs, however, such transformation has never been experimentally verified. One of the possible explanations is because a reverse transformation might occur during unloading of GaAs.

The effect of n-type (Si) or p-type (Zn) doping on the deformation of GaAs was investigated using nanoindentation by Le Bourhis et al [29]. They also studied the dislocation activity by indenting along two different orientations using a Berkovich indenter. It was found that the indentation rosette structure appeared to be affected by doping and indenter orientation, but the hardness, shear stress, and critical load of pop-in event were not obviously influenced [29].

In summary, the deformation patterns of single crystal GaAs induced by nanoindentation include dislocation, twining, stacking faults, and amorphization when the deformation is insufficient to induce micro-crack. According to our literature study, no high-pressure phase was observed experimentally.

3.1.3. Deformation of $\beta$-Ga$_2$O$_3$ under nanoindentation. Monocrystalline $\beta$-Ga$_2$O$_3$ is a newly developed and a representative for the third-generation semiconductor materials. Till now, the studies of the mechanical properties and deformation behaviors of $\beta$-Ga$_2$O$_3$ are lacking [17, 98]. Wu et al investigated the nanoindentation-induced deformation in $\beta$-Ga$_2$O$_3$ single crystal using TEM [17] and found that stacking faults along the (200) lattice planes and twinning structures with (201) lattice plane as twin boundary were firstly induced, as shown in figure 12(a). HRTEM images together with the corresponding electron patterns in figures 13(a) and (b) illustrated the characteristics of stacking faults and twinning structures, respectively. Subsequent to stacking faults and twinning, dislocations started to initiate at relatively high loads on (101) lattices planes. With the further increase in indentation load, densities of stacking faults, twins and dislocations became higher, forming a deeper defect layer. When the indentation load was sufficiently high (such as the case of 10 mN when using a conical diamond tip with a radius of 1 $\mu$m...
crack was initiated and propagated along (200) lattice planes (figure 12(b)) and (201) lattice planes were bent (figure 12(c)).

### 3.1.4. Deformation of laser crystals under nanoindentation

Nanoindentation induced surface/subsurface deformation of single crystal GGG was investigated by use of electron microscopic analyses [99]. Plastic flow lines were found to be distributed hierarchically on the indented surface from SEM examination. A typical cross-sectional TEM image of the indented GGG using a Berkovich tip with a tip radius of 40 nm is presented in figure 14(a), where no subsurface...
cracks or full dislocations can be observed. HRTEM images in figures 14(b) and (c) indicated that ductile deformation in the subsurface of GGG includes both polycrystalline nanocrystallites and amorphous phase. The so-called ‘elastic to plastic transition’ was investigated by analyzing the load–displacement curves using a spherical indenter: the occurrence of pop-in events was found to be related to this transition.

YAG single crystals is the other typical laser crystal for solid-state lasers at multi-kW scale. Elastic modulus, hardness, and fracture toughness of single crystal YAG were obtained from the nanoindentation made using a Berkovich tip with a tip radius of 40 nm [100]. Only plastic deformation was found on the indentation surface when the applied load was sufficiently low. Radial cracks were generated on the indentation surface when the load was sufficiently high. The lengths of cracks increased with the increased applied load. However, the detailed indentation-induced subsurface deformation of YAG is yet to be investigated using cross-sectional HRTEM observation.

### 3.2. Nanoscratch induced deformation

Nanoscratch is a similar process to nanoindentation that involves the interaction of a diamond tip and a workpiece material. Somehow different from nanoindentation, both lateral and normal forces are applied during scratching, which is thus more analogically mimic the removal event involved in grinding. Also, the deformed area induced by nanoindentation is extremely difficult to characterize by use of TEM, because very small impression is left on the surface. In contrast, a nanoscratch can be characterized with ease, even though the deformation is at extremely small scale, making observation of the initiation of single dislocation/defect possible. As a result, nanoscratch is widely used for characterizing the deformation of semiconductor and laser single crystals induced by mechanical loading in a much more controllable manner, especially at extreme small scales.

#### 3.2.1. Deformation of Si under nanoscratch

The scratching-induced deformation of single crystal Si was firstly investigated by Minowa et al in early 1990 [101]. Since then, scratching was often used to reveal the deformation and removal mechanisms of monocrystalline Si [102–114]. Gassilloud et al [103] investigated the effects of peak load and scratching speed using a Berkovich tip and found that residual scratch morphologies were strongly influenced by scratching velocity and applied load. An elastic-plastic regime was formed at relatively low loads, while a fully plastic regime was formed at relatively high loads. TEM results [103] showed that Si nanocrystals were embedded in the amorphous matrix at lower scratching speeds, while at higher speeds the transformation area was completely amorphous. Raman spectra were also acquired on the micro-scratch scar and a high pressure phase of Si-XII was detected when the scratching load was beyond a threshold value. A thorough Raman spectral analysis was made on the scratches made at variable scratching rates, revealed that a high pressure phase Si-XII existed when scratching rate was sufficiently low, and only α-Si was formed at relatively high scratching rates. An elastic contact mechanics model based on Hertz’s theory [103] was used to calculate the stress field under scratching in order to interpret the observed phenomena.

Wu et al [28, 60] utilized nanoscratch to systematically investigate the deformation pattern of single crystal Si at extremely low stress levels with the aid of HRTEM. As shown in figure 15(a) [28], only stacking faults were generated underneath the α-Si layer when the applied load was extremely low. The SAED pattern as seen in the inset in figure 15(c) shows that the maximum intensity of the diffuse diffraction ring (caused by α-Si) intersects with the \{111\}α diffraction spots of the Si-I phase, suggesting that α-Si could be transformed directly from Si-I phase. This is significantly different from the well-documented transformation route of monocrystalline Si induced by nanoindentation [36, 115, 116]. As mentioned above, lateral loading was found to play a key role in the amorphization of Si during nanoscratching, stacking faults and twins were nucleated at a threshold load lower than that for the nucleation of dislocations [60]. Dislocations started to be generated along Si \{111\} planes and penetrated into the Si substrate when the load was further increased as evidenced in figures 15(b) and (c) [28]. Further, HRTEM observations revealed that the dislocation induced by nano-scratching was a total dislocation with a Burgers vector of ±\{110\} [110]. The threshold load for the emission of dislocations during scratching was predicted by Fang et al using an analytical model [117]. It was proposed that the threshold load for dislocation nucleation during nanoscratching was always lower than that during nanoindentation, confirming the important role of lateral force in the generation of crystallite defects. When the scratching-induced stress was sufficiently great, α-Si was reversely recrystallized into Si-I nanocrystals, and high pressure Si-III and Si-XII phases were also formed inside the α-Si matrix, as presented in figure 15(d) [28, 60]. Ductile mode scribing in Si was found to be crystallographic orientation dependent [118]: (i) ductile removal preferentially occurred in \{111\} plane to \{110\} plane, (ii) the \{001\} plane exhibited to be more brittle during nanoscratching, and (iii) in the same crystal plane scratching along the \{111\} direction exhibited more ductile removal than that along the \{100\} direction.

Stress distribution is closely related to the tip geometry and its effect on the ductile-to-brittle transition was investigated using spherical tips with variable radii [119]. It was found that a larger tip radius tended to induce surface cracks, while sharp tips were prone to create median and/or lateral cracks. Also, friction would affect the critical depth of cut (DOC), as lowering the friction coefficient resulted in a higher critical DOC [119].

To more truly reveal the deformation pattern of Si in a grinding process, scratching was conducted at the speeds varying from several to tens of meters per second [120–123], which is significantly higher than that achievable in nanoscratch (at the scale of micrometers per second). When the scratching depth was sufficiently small, an amorphous layer was formed directly on the top of pristine Si-I prior to...
the formation of chips. When scratching depth was sufficiently high, high pressure phases and $\alpha$-Si coexisted in the scratch grooves and the ratio of $\alpha$-Si phase to high pressure phases increased with the increase of scratching speed [123]. The results are consistent with the conventional nanoscratch experiments [28, 103], indicating that nanoscratching at a relatively low speed does mimic the Si deformation occurred in the grinding process. In other words, the scratching speeds might only affect the value of the critical DOC, but not change the deformation pattern of Si [124].

Local temperature in the deformation zone will unavoidably rise in the grinding process, even though coolant was usually used. Thus, knowledge of the scratch-induced deformation in Si at elevated temperatures is important in gaining a complete understanding of the deformation mechanism in grinding. It was found that scratch hardness of Si decreased up to 46% as temperature increased from room temperature to 500 °C [125]. It was also reported that scratch depth and width increased at elevated temperatures due to the thermal softening effect [125]. As a consequence, the surface roughness of Si wafers being processed by micro-laser assisted machining could be improved [126]. It is interesting to note that high-pressure Si phase was absent in the scratched tracks at 500 °C as reported by Chavoshi et al [125]. While using similar high temperature scratching method, Alreja et al reported the presence of metastable silicon phases under various furnace-controlled temperature conditions, i.e. RT to 500 °C [127]. Nanoscratching at elevated temperatures is still at the infancy stage and it is expected that the complication at relatively high temperatures may cause some controversial results.

Molecular dynamics analysis would help understand the deformation of Si under diamond asperities to simulate nanosliding [128, 129] and nano-scratching [130]. Through MD simulation, it was found that a thin amorphous layer always existed on the top of the deformed Si, but there was absence of dislocations when the penetration depth of asperity was sufficiently small [129]. Four-coordinated diamond cubic silicon can be transformed into the six-coordinated $\beta$-silicon form when the penetration depth or the stress being induced was sufficiently high [129]. Importantly, the transformation of $\beta$-phase to the amorphous phase of Si is reversible. It should be noted that MD models could only simulate very limited
penetration depth, at a scale of several nanometers, which was much smaller than the depths used in nanoscratch, usually several tens or hundreds of nanometers. Therefore, MD simulation cannot fully represent a pragmatic nanoscratching process.

Overall, the deformation pattern of single crystal Si induced by nanoscratching includes amorphization, twinning, formation of stacking faults, dislocation and high pressure phases, and recrystallization when the deformation is at extremely small scales.

3.2.2. Deformation of GaAs under nanoscratch. Little was done to study the deformation of GaAs using nanoscratching [85, 131–135]. Micro-scratches made using diamond tips of variable geometries (cube corner, conical, and Berkovich) were used to investigate the mechanics of GaAs deformation for industrial applications [132, 133]. Wasmer et al creatively cut the longitudinal TEM lamellae along a scratch, which makes it possible to directly compare the crystal defects induced from indenting and scratching [85]. Figure 16 shows that twinning was the main deformation mechanism under indentation, whereas only slip bands and perfect dislocations were induced during scratching.

The effect of peak normal load on the deformation pattern of GaAs under nanoscratching was investigated by Wu et al [136]. As shown in figure 17(a), the impression was very shallow when the load was small (i.e. 1 mN in this case) and only slight pile-up appeared on the shoulder of the nanoscratch, which was also observed in previous studies [85, 95]. When the normal load was sufficiently high, a median crack was initiated [95, 96, 137], as shown in figure 17(b). The crack was initiated and propagated in a perpendicular direction into GaAs bulk. Previous studies suggested that median cracks could be initiated due to high stress concentration underneath spherical [84] and Berkovich tips [67]. It was verified by the HRTEM results as shown in figure 17(c) that parallel platelets, which were determined to be a twinning structure, can be clearly seen in the area near the scratch impression. Note the white lines in figure 17(c) indicated the reflective atomic planes which were characteristics of a twinning structure. The density of twins increased with the increased applied normal load [136]. Moreover, the twinning structure was also verified by an SAED analysis as shown in figure 17(d). Diffraction spots, indicated by the white arrows in figure 17(d), were superimposed on a <110> diffraction pattern of GaAs, which represents the diffraction from the reflective twinning planes. Parlinska-Wojtan et al [95] proposed that during nanoindentation, if the tip apex angle was larger than 70.5°, namely the angle between two {111} planes in GaAs, there would be convergent twinning patterns formed during nanoindentation; otherwise, divergent twinning patterns were expected. Both convergent and divergent twinning patterns were seen in figure 17, this might be due to the difference in tip geometry and the effect of lateral force. Direct evidence of lattice bending induced by nanoscratching at atomic level was demonstrated by Wu et al and it appeared to be a new deformation pattern in GaAs, in addition to previously reported dislocation, twinning and stacking faults [135].

3.2.3. Deformation of laser crystals under nanoscratch. The knowledge of nanoscratch-induced deformation of laser crystals was rarely reported. Li et al conducted some scratching studies on GGG crystals using a Berkovich tip [16, 138], with which the brittle to ductile transition for this crystal was investigated. Amorphization and formation of nano-crystals were found to be the dominated deformation patterns in the plastic flow zone [16]. Micro cracks would be generated when the normal force was increased beyond a threshold value and median cracks would be initiated when the normal force was sufficiently high. The effect of strain rate on the scratch-induced deformation of GGG was also reported [138] and increasing the scratching velocity could effectively improve plasticity and reduce the critical depth for brittle-to-ductile transition. As shown in figure 18, nano-sized polycrystals and amorphous phases were found to be the dominated crystal defects in the scratched GGG, irrelevant of scratching velocity [138]. The corresponding TEM results indicated that the increased scratching velocity led to slipping planes appearing in multiple directions as shown in figure 19(b), which subsequently prevented the generation of long slipping planes and hence reduced the depth of SSD. In other words, the depth of damaged layer decreased with the increasing of scratching velocity, as indicated by the yellow arrows in figure 19.

Surface morphologies of the YAG single crystal scratched by a Berkovich tip with a tip radius of 2 μm was examined by Li et al with the aid of SEM [100]. Their work enabled the identification of the critical load for the ductile-to-brittle transition of this crystal, as the first radial crack was observed on the scratched surface when the applied load was approximately 18 mN in this case. It should be noted that the ductile-to-brittle transition was judged based on the surface
cracks, rather than a complete understanding of subsurface damage, which could underestimate the critical load.

4. Deformation and removal of single crystals involved in machining

Grinding, cutting, lapping, and polishing are the main machining approaches currently used for the fabrication of semiconductor wafers and laser crystal substrates. Grinding is often used for stock removal to achieve the required thickness, flatness and parallelism, while lapping and mechanical polishing processes are employed for the removal of the residual damage layer after grinding to obtain the required surface integrity. Thus if the grinding process can minimize its residual damaged layer, the processing time for the final finishing process can be reduced, thus lowering the overall manufacturing cost. In the applications, the quality requirement of semiconductor and laser crystal wafers is so stringent that an atomically smooth surface and a damage-free

Figure 17. High magnification TEM images of the nanoscratches made using a conical tip at different loads of (a) 1 and (b) 6 mN, (c) HRTEM image taken at the shoulder of a scratch, showing twinning structure and (d) its corresponding SAED pattern [136].

Figure 18. (a) Bright field TEM image of scratched GGG single crystal, HRTEM images showing (b) nanocrystals and (c) amorphous [16].
subsurface must be guaranteed. To achieve this, knowledge of the deformation and removal of those crystal materials involved in machining need to be explored. It should also be noted that as grinding, lapping and polishing are usually employed in the process, the three procedures should be optimized in order to reduce the overall manufacturing cost. This suggests that different processing combinations may be adopted for different crystals. Nevertheless, subsurface damage, or SSD, cannot be fully removed after grinding/lapping/mechanical polishing [139]. Therefore, a final finishing process usually employs chemical mechanical polishing (CMP) that can generate atomically smooth and damage free surfaces [140–142]. As SSD must be removed in the final finish process, the smallest possible SSD layer generated in grinding/lapping/polishing is very beneficial to its subsequent process, in terms of machining efficiency and cost [143]. It should be noted that this review does not cover the deformation and removal involved in CMP process because it concerns a great deal of chemically enhanced material removal. Also, grinding at nanoscale, lapping and mechanical polishing are all abrasive machining processes involving similar grit-work material interactions, so that in this work only the literatures concerning grinding induced deformation and removal will be reviewed.

4.1. Fundamentals of brittle-to-ductile transition

Grinding is an abrasive machining process that has been extensively used to machine semiconductor substrates and laser crystals to achieve the desired surface roughness and dimensional precision [44, 46, 143–149]. To achieve a high precision finish surface, ultrafine grain of diamond is employed to generate ductile-like deformation and removal. Grinding processes are, however, very complicated, owing to their complex stress status and the elevated temperatures in the grinding zone. The work material experiences repeated rubbing, plowing, scratching, and shearing by multiple diamond grits, thus the aforementioned deformation mechanisms of single crystals under instrumented mechanical loading may be used as a guide for optimizing the grinding process.

To achieve the best possible surface integrity in the grinding of semiconductor and laser crystal materials that are very brittle, their material deformation and removal should not involve brittle fracture or cracking. In other words, the grinding must be carried out in the so called ‘ductile’ removal regime. The effect of DOC on the brittle-to-ductile transition in the grinding of brittle materials may be understood with the aid of Bifano’s model, in which the critical grinding depth can be estimated as [9]:

$$d_c = \Psi \frac{E}{H} \left( \frac{K_c}{H} \right)^2,$$

where $E$ is the material’s Young’s modulus, $H$ is hardness, $K_c$ is fracture toughness, and $\Psi$ is material’s brittle-ductile transition factor, which varies with different brittle materials. Bifano’s model provides the criteria for the occurrence of ductile removal in the grinding process when the DOC of individual abrasive is smaller than $d_c$. The DOC of an individual abrasive grit can be estimated by calculating the maximum undeformed chip thickness ($h_m$). $h_m$ for face grinding can be calculated as [150]:

$$h_m = \left( \frac{2}{Cr} \right)^{0.5} \left( \frac{r}{v_f} \right)^{0.5},$$

where $C$ is the density of active abrasives, $r$ is the width to depth ratio for an undeformed chip, $f$ is the feed rate and $v_f$ is the wheel velocity. The value of $h_m$ for surface grinding can be calculated using the following equation [151]:

$$h_m = \left( \frac{3}{C \tan \alpha} \right)^{0.5} \left( \frac{v_w}{v_f} \right)^{0.5} \left( \frac{d_c}{d_w} \right)^{0.25},$$

where $C$ is the density of active abrasives, $\alpha$ is the semi-included angle for the undeformed chip cross section, $d_c$ is the wheel diameter, $v_w$ is the workpiece feed rate, $v_f$ is the wheel speed and $a_c$ is the wheel DOC. Based on Bifano’s model, to enable the ductile-like removal in the grinding of a brittle solid, the grinding parameters must be designed so that $h_m$ is smaller than $d_c$. It should be mentioned that this model works well for some brittle materials, but could considerably underestimate or overestimate the critical depth for ductile removal due to the complex nature of abrasive machining and microstructure of brittle materials.
4.2. Grinding induced deformation and removal of Si

The characteristics and performance in the grinding of single crystal Si were intensively investigated, including the effects of grinding wheels [46, 140, 143, 152], grinding induced surface/subsurface damages [46, 153–155], and optimization of grinding parameters for better surface quality [44, 144, 148, 153, 156–164]. As the material removal involved in silicon grinding is often controlled at nanometric scales to ensure the ductile-like removal [153, 155, 160–163], such a grinding process was also termed as nanogrinding. In this work, only the deformation and removal of single crystal Si carried out in the ductile region will be reviewed.

The grit size of a grinding wheel plays a critical role in the deformation and ductile-like removal of Si. AFM imaging of the ground Si surface demonstrated that grinding grooves were apparent and became finer as the grit size decreased [153]. The grit size effect on subsurface damage was extensively examined by use of cross-sectional TEM [153, 155, 165], and the amorphous, elastically stressed, and plastically deformed layers were found to be the main deformation structures, as shown in figure 20 [155]. When a grinding wheel with a fine grit size of 3000 in mesh was used, only pure amorphous phase and elastically stressed layer were observed in ground Si monocrystal, as evidenced in the TEM image in figure 20(a) and its corresponding SAED pattern in figure 20(b). No evidence of nanocrystals was found in the amorphous layer. In contrast, when a diamond wheel with a coarser grit size was used, severely deformed layer was observed in the ground Si as shown in figure 20(c) and nanocrystals with the same crystal structure as the pristine Si were formed based on the SAED pattern shown in figure 20(d). The subsurface with a more severely damaged layer was obtained when a very coarse grit wheel was used, as shown in figure 20(e) and nanocrystals other than the Si-I phase were also confirmed by the SAED pattern in figure 20(f). HRTEM images of the nanocrystals embedded in the amorphous in figure 20(e) are shown in figures 20(g) and (h), demonstrating the existence of high pressure phase of Si-III. The residual stress, after rough and fine grinding, was found to be in the range of a few hundreds of MPa and tens of MPa, respectively [165]. To sum up, less severely damaged subsurface layer can be obtained using grinding wheels with finer grit sizes, but sacrificing the material removal rate. Thus, to reduce the overall processing cost associated with grinding, an optimal combination of grinding procedures with different grit sizes is necessary, such as a coarser wheel for removal while a finer wheel for finishing.

The conventionally used mesh sizes of grinding wheels for silicon machining were below 5000, corresponding to grit size of above ~2 μm. As a consequence, the surface roughness (Ra) of a ground Si wafer was above 8 nm [153, 158] and the depth of subsurface damage layer was in the scale of 100–200 nm [153, 155, 166]. To further improve the surface finishing by grinding, an ultrafine diamond wheel of a grit size of 0.25 μm could be adopted, which would produce an extremely smooth surface of an average roughness of 0.6 nm [46]. The TEM examination indicated that the total thickness of subsurface damage layer was less than 60 nm, as shown in figure 21 [46]. The grinding results indicate that the damaged layer needs to be removed in the following polishing process is less than 100 nm, which is promising and exaggerates the possibility to be adopted in the product line. However, it should be noted that the grinding conditions became more sophisticated when an ultrafine grinding wheel was employed, i.e. inappropriate grinding parameters would cause burning as the wheel could be easily overloaded and thus more grinding heat would be generated [46]. The phase transformation of Si wafer under various grinding conditions were investigated using HRTEM [154] and the formation of nanocrystals appeared to be strongly influenced by the thermal status of a grinding process when using a diamond wheel with ultrafine grits. Specifically, the higher grinding heat being generated would enhance the transformation of Si-I to high-pressure Si phases [154]. Note that the wheel speed would also affect the ground surface in term of the depth of a damaged layer, but its effect is less significant [46].

For face grinding of a Si substrate or wafer, the wheel diameter is generally sufficiently large to cover the entire workpiece in one grinding revolution [46, 157]. Certainly, a smaller diamond wheel would help reduce the processing cost [152]. By carefully planning the cutting paths of the smaller wheel, it is possible to achieve a surface roughness similar to or slightly worse than that obtained using the conventional large wheel [152]. In this case, a mathematical model was developed to plan the cutting path, which helped to optimize the grinding conditions too. Nevertheless, a grinding wheel with diameter slightly larger than the wafer radius should be beneficial to improve the grinding performance.

Three dimensional MD model was established for the high speed grinding of Si by Li et al [167] to understand the speed effect on the integrity of ground surface and SSD. The equivalent stress distribution and corresponding atomic displacement in the ground Si material are illustrated in figures 22(a) and (b), respectively. It was found that a higher grinding velocity would produce larger chips and higher local temperature in grinding zone, which might help reduce SSD. However, at extremely high grinding speed, the subsurface damage layer was slightly thicker because elevated temperature might promote the nucleation and motion of dislocations [167].

Deionized water is usually employed as the coolant in the grinding of Si substrates [46, 159]. It is suggested that both inner and outer coolant nozzles should be used simultaneously to achieve the best possible results and a high flow rate was beneficial for the cooling performance [159]. Li et al proposed that water-based coolants containing graphene oxide could significantly reduce the tangential force during grinding and thus improve surface/subsurface quality [168].

4.3. Grinding induced deformation and removal of β-Ga2O3

The grinding characteristics of single crystal β-Ga2O3 were seldom reported, most likely because it is a newly developed material. Nanogrinding induced surface/subsurface deformation characteristics of single crystal β-Ga2O3 were
Figure 20. Bright-field TEM images and corresponding SAED patterns of ground Si using grinding wheels with mesh size of (a) and (b) 3000, (c) and (d) 800, (e) and (f) 400, respectively. (g), (h) HRTEM images of the nanocrystals embedded in the amorphous in (e). Reproduced from [155]. © IOP Publishing Ltd. All rights reserved.
investigated by Gao et al [20]. The severity of SSD was reportedly reduced when grinding wheels with finer grit sizes were employed. A typical TEM image and its corresponding HRTEM image of the ground β-Ga2O3 single crystal using an ultrafine grinding wheel of mesh size of 12 000 are shown in figures 23(a) and (b), respectively. Formation of nanocrystals, stacking faults, twins, and dislocations were the main mechanisms responsible for the deformation induced by
4.4. Grinding induced deformation and removal of laser crystals

The literature study showed that YAG single crystals are mainly machined through lapping and polishing that are less aggressive processes with lower dimensional accuracy than grinding [21, 169]. The studies regarding the grinding performance of this crystal material was rarely reported. Li et al investigated the precision grinding performance of YAG single crystals using different grit size wheels [21]. Two types of ground surfaces, namely ductile-like and brittle-ductile surfaces, were obtained. The grinding in ductile regime was realized using the diamond wheels with mesh sizes of 4000 and 6000. Slippages of (001) crystal planes were evidenced in the subsurface, as presented in figure 24 [21], along with the formation of stacking faults, dislocations, and distortion of atomic planes, which are shown in the HRTEM images in figures 26(a)–(e). Generation of brittle-ductile surfaces was attributed to the plastic deformation with formation of nanocrystals and nanovoids, as well as brittle fracture, when the grinding wheel with a coarser grit size was employed. As shown in figure 27, a model that depicts the effect of grit distribution on the chip formation, combines with other materials deformation models, was developed to estimate the grinding force in the ductile-like removal of the grinding process by considering the combined effect of strain rate, random distribution of abrasive radii, and elastic-to-plastic transition depth [6]. It is believed that accuracy of the predicted grinding force was substantially improved by taking the effect of strain rate into account. The thickness of damaged layer is about 200 nm with slippages along particular lattice planes. To further improve the surface quality in terms of reducing damaged layer thickness and eliminating slippage, a grinding wheel with finer diamond grits should be adopted and grinding parameters need further optimization in terms of grinding force and surface finish.

5. Summary and perspectives

In this work, the deformation and removal characteristics and behaviors of representative semiconductor and laser crystals, including Si, GaAs, β-Ga2O3, YAG and GGG single crystals, were systematically reviewed based on the available literature. The deformation and removal patterns induced by instrumented mechanical loads and grinding were summarized in table 2 for comparison purpose. This review demonstrates that the knowledge of deformation and removal mechanisms obtained from nanoindentation and nanoscratch studies can be utilized to optimize the grinding processes for those important single crystals, which are thus beneficial to improving their surface finish and manufacturing cost.

From this review it is clear that knowledge of the deformation and removal characteristics and behaviors of a specific single crystal gained from the previous studies should be taken into consideration for developing abrasive technologies for the crystal. It is thus important to use instrumented
nanoindentation and nanoscratch to gain its deformation and removal characteristics, as those approaches can produce similar deformation patterns and mechanisms to a grinding process. The first-hand knowledge being obtained should save a lot of testing time for developing optimal grinding, lapping and polishing processes.

In general, the grit DOC involved in the grinding of those brittle single crystals plays a critical role in determining their...
deformation and removal behaviors. More specifically, when the grit DOC in a grinding process is smaller than the critical DOC for brittle-to-ductile transition for a specific crystal material, ductile-like deformation and removal should occur, which enables the achievement of appropriate surface finish and subsurface integrity. Usually, a grinding wheel with finer diamond grits produces a better surface finish with less surface/subsurface damage. However, special care is needed to avoid wheel burnishing when dealing with ultrafine grinding wheels. Certainly, grinding wheels with relatively coarse diamond grits are more suitable for stock removal, but more significant subsurface damage would be generated, which thus increases difficulty of the subsequent finishing process, prolongs machining time, and increases the overall manufacturing cost. It is thus of great value to have a complete understanding of the deformation and removal mechanisms of the crystal material involved in all the finishing procedures, so that an optimal protocol can be established from both quality and efficiency perspectives. Optimization of grinding conditions to minimize subsurface damage should be the ultimate goal in an abrasive finishing process, though it is impossible to eliminate crystalline defects. Nevertheless, optimization should also be made with taking care of appropriate machining efficiency.

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