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Research article

Defect-free grinding of silicon nitride at high material removal rate

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ABSTRACT

Owing to the extreme hardness and toughness of sintered silicon nitride (Si_3N_4), the material is used in high stress and/or temperature applications such bearings, turbines, and combustion engines. Unfortunately, the same properties which make it ideal for use also make it particularly difficult to machine – microcracks, inclusions and spalling are all common. While prior research has shown that it is possible to grind sintered Si_3N_4 without inducing surface damage so long as material is removed entirely under ductile flow, but grind forces associated with ductile Si_3N_4 material flow are so small as to render the material removal rate (MRR) impractical. Prior researchers have attempted to solve the MRR problem through laser-assisted machining. Laser ablation, by inducing a steep thermal gradient, weakens material through surface and subsurface cracks. Grinding of fractured weakened Si_3N_4 has been done at upwards of 50 % higher MRR. There are, however, issues with laser ablation, which prevent its widespread use. Laser ablation severely disrupts the microstructure of Si_3N_4. Because cracks propagate along and through grain boundaries, the irregular morphology makes accurately predicting crack growth from ablation and during subsequent grinding highly problematic. In this proof-of-concept work, researchers determined that it is possible to irradiation weaken Si_3N_4 without cracking it, and the material can be ground defect-free at a highly productive MRR. Findings suggest present laser-assisted machining methods which fracture weaken Si_3N_4 prior to grinding may not be the best way to maximize MRR.

1. Introduction

First discovered in the mid-nineteenth century, Si_3N_4 describes a range of engineered ceramics. Each is characterized by extremely high strength, toughness, and hardness as compared to metal. Four methods are commonly used to produce Si_3N_4.

1. Reaction–bonded silicon nitride (RBSN)
2. Hot-pressed silicon nitride (HPSN).
3. Sintered reaction-bonded silicon nitride (SRBSN)
4. Sintered silicon nitride (SSN)

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None of the above-mentioned manufacturing methods produce defect-free Si_3N_4 and all suffer machinability problems. For example, because of high porosity (e.g., ∼30 %) RBSN has a high propensity for brittle fracture during grinding. To correct for porosity, HPSN is made under sufficient pressure to collapse and bond pores. The resulting material is of approximately 40 % higher density, two times higher hardness, and three times higher rupture modulus as compared to RBSN. Unfortunately, the highly dense HPSN material requires substantial grind energy input to remove modest amounts of material. To address the grindability problem, hot isostatically pressed silicon nitride (HIPSN) parts are made in nearer net shape than HPSN. For all the cost involved in making HIPSN, open pores still exist which, in turn, led to the development of sintered silicon nitrides (SSN). Densification through sintering offers SSN parts higher fracture toughness and rupture modulus as compared to HIPSN. Achieving densification entails upwards of 20 % shrinkage. Sintered parts must, therefore, be extensively ground to achieve desired final shape.

Prior researchers have attempted to solve the Si_3N_4 grindability problem through laser-assisted machining. Lasers are used to induce steep thermal gradients in Si_3N_4, resulting in surface and subsurface cracks. Grinding of fracture weakened Si_3N_4 has been done at upwards of 50 % higher MRR [1]. There are, however, several issues with laser-assisted machining preventing its widespread adoption. Laser ablation cracks the material. It also severely disrupts microstructure. Because cracks propagate along and through grain boundaries, the irregular morphology makes accurately predicting crack depths highly problematic. Moreover, during grinding laser induced cracks grow, which requires subsequent finishing and polishing operations. This research sought to reframe the Si_3N_4 grindability problems by answering three questions.

1. Is it possible to laser weaken Si_3N_4 without cracking it?
2. If so, could the irradiation weakened material be ground free of defects?
3. If so, would the defect-free MRR be productive?

2. Literature review

Finishing Si_3N_4 typically involves diamond abrasive wheel grains scratching away material. When scratch separations are large enough that abrasive grains do not interact, MRR is low. When abrasive scratch separations are small enough that they do not overlap, MRR is also low. Prior researchers have found efficient MRR for Si_3N_4 occurs over intermediate grain spacing (50–100 μm) and under moderate force (30–40 N) applied [2].

The energy required for diamond grains to remove Si_3N_4 is substantial. The specific grinding energy for sintered silicon nitride ground with a diamond abrasive wheel have been found to range from about 40 J/mm3 (at high MRR) to 130 J/mm3 (at low MRR) [3]. While it might seem paradoxical that higher grinding energies are required at lower MRRs, the reasoning has to do with chip size. At lower MRRs abrasive grains remove smaller chips. Small chips are removed by ductile flow as opposed to fracture. Ductile flow is a slow process requiring a comparatively high amount of energy. On the other hand, larger MRRs produce larger chips. Large chips are removed by fracture. Fracturing is, therefore, a comparably faster material removal process requiring less energy. The tradeoff being fracture imparts surface and subsurface cracks which propagate through and across the material's crystalline microstructure grain boundaries.

According to the *Handbook of Ceramics Grinding and Polishing,* as chip thickness falls below 0.35 μm, specific grinding energy increases sharply. Above this transition point, specific energy decreases slowly as chip size increases. Interestingly, researchers out of the School of Mechanical and Electrical Engineering at Guangdong University of Technology found not one, but three energy transition

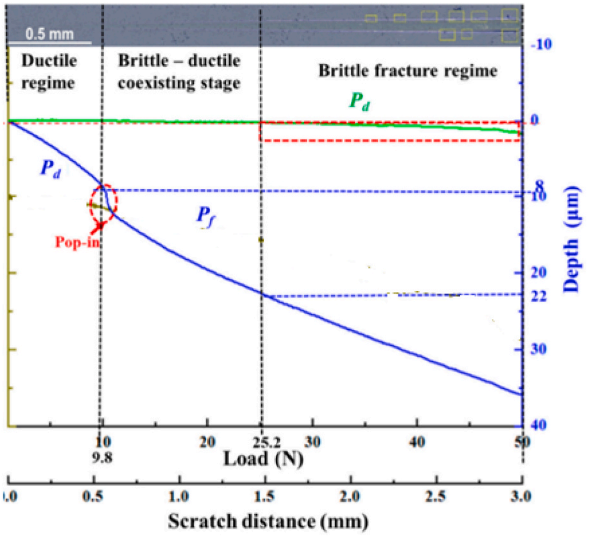

Fig. 1. Three energy transitions for grinding HIPSN.

points when grinding Si_3N_4 [4]. Transitions are shown for the grinding of HIPSN in Fig. 1.

The ductile regime in Fig. 1 is characterized by small abrasive grain scratch distances (<0.5 mm), shallow depths of cut (<0.73 μm), and low force (<10 N). Si_3N_4 material is removed in this region by ductile flow until a "pop-in" occurred. At this point, material removal is by both ductile flow and brittle fracture. The ductile–brittle stage entails 0.5–1.5 mm scratch distances, 8–22 μm depths of cut, and 10–25 N forces. It is in this region where researchers at Guangdong University of Technology first observed radial cracks and tears. The third and final stage of material removal involves 1.5–3 mm scratch distances, 22–35 μm depths of cut, and 25–50 N forces. Here researchers found surface defects magnified because all material removal is by brittle fracture.

As important as abrasive cutting depths and spacing are, energy transition models based solely on them fail to address the heat embrittlement problem. The *Handbook of Ceramics Grinding and Polishing* quantifies Si_3N_4 grinding temperature as:

$$\theta_m = \frac{1.13 R u v_w^{1/2} a^{3/4}}{(kpc)^{1/2} d_s^{1/4}} \quad 1$$

- a = depth of cut
- c = specific heat
- d_s = diameter of grinding wheel
- k = thermal conductivity
- R = fraction of the grinding energy entering the workpiece
- u = grinding energy
- v_w = linear velocity of workpiece
- ρ = density

The predominance of energy grinding Si_3N_4, per Eq. (1), has been found to be conducted as heat raising workpiece temperature by θ. For example, grind tests conducted on HPSN at low work speed (vw = 8 m/min), moderate depth of cut (a = 0.02 mm) using a diamond grinding wheel of 177 mm diameter (ds) required 50 J/mm3 of grinding energy (u). With the upper limit of energy used to form chips found to be 5 J/mm3, it was concluded that 90 % of grinding energy entered the HPSN workpieces as heat. Substituting these values into Eq. (1) yields a grinding zone temperature of approximately 9OO°C. Such a temperature practically ensures cracking considering even short time exposure of HPSN to a temperature of 800 °C has been found to impart a brittle, glassy phase [5].

Engineers at Sumitomo Electric Industries Ltd. attempted to use the grinding heat problem to solve the embrittlement cracking problem [6]. The idea was to grind Si_3N_4 shallow enough with sufficient heat to *deposit* a layer of material on top of existing surface defects in the purchased material. Engineers determined that a diamond abrasive grinding wheel of organic binder spinning at 26 m/s with an average abrasive grain size of 5–50 μm was ideal for deposition grinding. Moreover, abrasive concentration needed to be no less than 75 and no more than 150. Infeed was controlled within 0.005–0.1 μm per rotation of the grinding wheel. Material was fed under the grinding wheel at 25–75 m/s.

Clearly, there are several practical issues with Sumitomo's additive approach to defect-free grinding of Si_3N_4. Commercial surface grinders, typically capable of no more than 0.5 m/s table speed, are nowhere near the 25–75 m/s needed. Even if such a machine could be built, the grinding system would need to maintain <0.1 μm rectilinear control. Existing ultra-precision grinders (UPG) control total displacement on the order of microns not submicron. Assuming machining speed and displacement constraints could be overcome, there is still the problem of form. Commercially purchased Si_3N_4 requires stock removal on the order of 1–2 mm not because of surface defects, but to correct for geometric inaccuracies. Sumitomo's additive grinding approach could never correct for sintering shrinkage or hot press forming errors. By 2014 Sumitomo Electric Industries Ltd. had allowed its Si3N4 grind patent to expire.

Researchers at the Institute for Precision Machining and High Frequency Technology in Deggendorf Germany also attempted to solve the Si_3N_4 grinding crack problem using heat. Albeit their approach was completely different than Sumitomo's. Laser heat was used to induce steep thermal gradients in Si_3N_4 samples. Cracks weakening of Si_3N_4 has allowed practitioners of laser-assisted machining to grind away the fractured surface layer at approximately 50 % lower force [7]. Present research in the field of laser-assisted machining centers around selecting laser power, pulse duration, scan speed, and repetition rate to maximize MRR [8].

There are, however, several issues with the present approach to laser-assisted machining of Si_3N_4 which stand in the way of widespread use. Attempts to quantify how much material needs to be removed to eliminate cracks have been unsuccessful. Part of the problem is Si_3N_4 cracks are not stationary. They grow under laser induced thermal stress [9] as well as subsequent grinding. Owing to high retained stresses, cracks have also been observed to grow even after lasers are turned off [10] and grinding has stopped [11]. The other issue is predicting how cracks will grow. Cracks propagate along inter- and *trans*-granular boundaries [12]. Unfortunately, present methods of laser-assisted machining impart cracks, phase transformation and chemical reactions, all of which highly disrupt microstructure. Lacking a regular crystalline lattice makes predicting crack magnitude, direction, and growth highly problematic [13].

3. Methodology

This research sought to determine if it was possible to overcome laser-assisted machining limitations. The goal was to determine if it was possible to irradiation weaken Si_3N_4 without cracking the material, then grind it defect-free at a productive MRR. This proof-of-concept work involved two SSN samples each measuring 25 mm wide x 25 mm thick x 50 mm long. The test sample was laser irradiated. The control sample was not. On each sample, the irradiated test space area measured 242 mm^2 (e.g., 6.35 mm wide x 38.1 mm long). Across both samples, researchers.

- Assessed surface defects and microstructure using a Keyence VHX 7000 Digital Microscope at 500x and 1000x magnification
- Measured surface roughness before and after grinding with a Zygo NewView 7000 using a150 μm bipolar scan
- Determined surface hardness with a Wilson Tukon 1202 micro hardness gauge set to 50 g
- Evaluated heat affected zone depth via oxygen content measured in cross section using a field emission scanning electron microscope with EDAX (LYRA3 XMU FIB)

Irradiation was done with a Nd:YAG solid state laser. Laser type, pulse (<10 ps), wavelength (1032 nm) and operating parameters (as listed in Table 1) were selected to be just within what prior researchers had determined was possible to ablate Si_3N_4 [14].

During irradiation (Fig. 2) and subsequent surface grinding, a FLIR thermal camera was used to ensured surface temperature did not exceed a maximum of 800 °C. The 800 °C maximum temperate was selected as prior researchers established the maximum temperature range use for Si_3N_4 in an oxidizing atmosphere was from 1000 °C to 1400 °C [15].

Surface hardness was evaluated by averaging hardness values before and after laser irradiation on the test sample at the same four locations. Any possible cracking and/or change in microstructure were evaluated on the test sample at the same locations before and after irradiation. The 500–1000x magnification level was selected because industrial users of Si_3N_4 typically use dye penetrant for nondestructive evaluation of cracks. Prior researchers established that dye penetrants provide reliable detection of crack from 0.8 mm to 1.3 mm in length [16]. A 500 - 1000x magnification was sufficient to see cracks at a fraction of this magnitude. To determine heat affected zone depth, measurements were taken in cross section on the test sample before and after irradiation using a digital microscope and field emission scanning electron microscope with EDAX.

Defect-free grindability after irradiation was assessed. The researchers did this by recording MRR for both the irradiated and control samples. All samples were ground with normal force under 10 N. The 10 N upper limit was selected because prior researchers established 10 N as the maximum normal force by which Si_3N_4 material could be removal entirely under ductile flow [3]. Grinding tests were conducted using a Grind-X model Okamoto horizontal spindle surface grinder. Normal grinding force (per Fig. 3) was measured with a Kistler 9251a force transducer sensor.

A Labview program was created to record normal force data using an NI DAQ system. Grinding temperature was recorded using a FLIR thermal camera. Grinding was done per parameters listed in Table 2.

4. Results

Using a 100 W, 125 J pulse laser of <10 ps with scan speed of 1 mm/s the silicon nitride sample heated to 740 °C. Sample surface hardness reduced 12 % (Fig. 4).

Prior to laser irradiation, the sintered silicon nitride samples, per Fig. 5, exhibited an α-β crystalline surface structure.

After laser irradiation, per Fig. 6, the α-β crystalline surface structure was gone. The only regular lattices seen were shades of the pre-sinter, hexagonal α starter phase.

After laser treatment, the surface measured 25 μm Rz. Digital microscope measurements in cross-section, per Fig. 7, indicated that the heat affected zone extended approximately 90 μm deep into the sample.

Field emission scanning electron microscope with EDAX confirmed that the heat affected zone depth was approximately 90 μm.

Per grinding conditions listed in Table 2, researchers were able to grind the irradiated sample at a traverse rate of 10.9 mm/s. This equated to a specific material removal rate of 0.027 mm3/mm sec. At this MRR, normal grinding force was ~10 N (Fig. 8) and temperature was ~100C.

Digital microscope measurement of the irradiated sample after grind showed no image (Fig. 9) or scan (Fig. 10) evidence of surface damage. Rz measured 2.9 μm.

By comparison, the nonirradiated sample could not be ground defect-free under 10 N normal force using Table 2 parameter values. With all other parameters unchanged, depth of cut was reduced to 0.5 μm. This minimum depth of cut was used because it was the minimum value which the machine was capable. The resulting MRR was 0.0054 mm3/mm sec. Nominal normal grinding force, per Fig. 11, was ~50 N. Grinding temperature was ~100 °C.

The nonirradiated grind sample after digital microscope measurement showed image (Fig. 12) and scan (Fig. 13) evidence of surface damage. Rz was 231 μm.

5. Conclusions

For a sintered Si_3N_4 sample at 0.5 μm depth of cut and a mere 0.0054 mm3/mm sec. MRR, researchers were not capable of

Table 1
Laser parameters.

Parameters	Values
Power (W)	100
Repetition rate (kHz)	400
Scan speed (mm/sec)	1

Fig. 2. Laser & thermal camera set up.

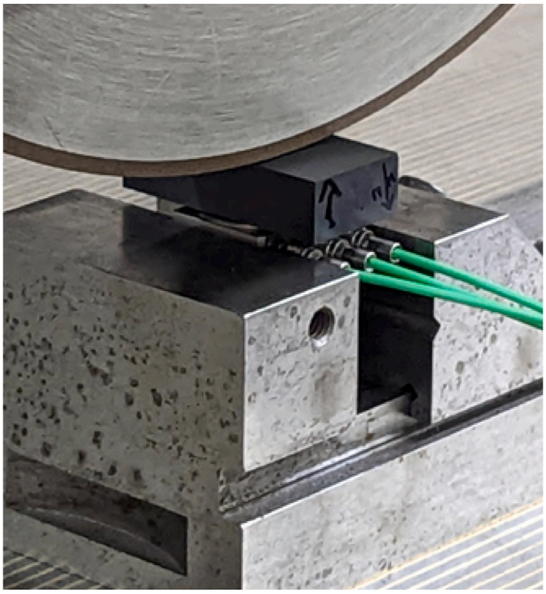

Fig. 3. Grinding force measurement.

Table 2
Test space grind parameter.

Parameters	Values
Depth of cut (μm)	2.5
Normal Force (N)	<10
Grinding Temp. (C)	<800
GW material	diamond
GW grit	120
GW concentration	60
GW width (mm)	6.35
GW OD (mm)	355.6
GW speed (m/s)	28
Coolant	none

achieving the <10 N normal grinding force which prior researchers determined was required for defect-free, ductile grinding. Indeed, at the nominal 50 N normal grinding force and 100 °C grinding temperature, the sample exhibited surface damage measuring approximately 100 μm deep.

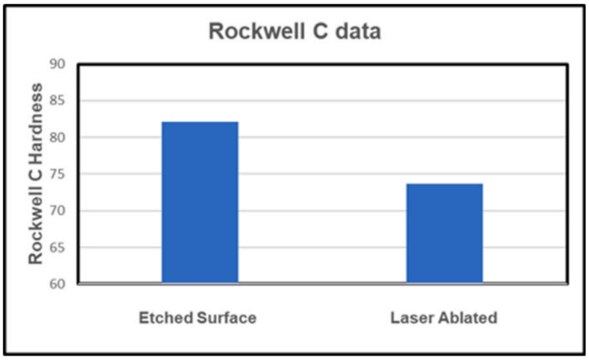

Fig. 4. Surface hardness after irradiation.

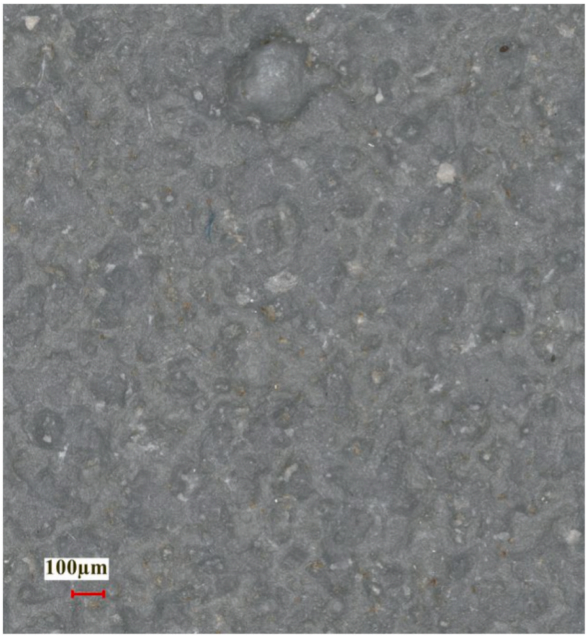

Fig. 5. α-β crystalline surface structure.

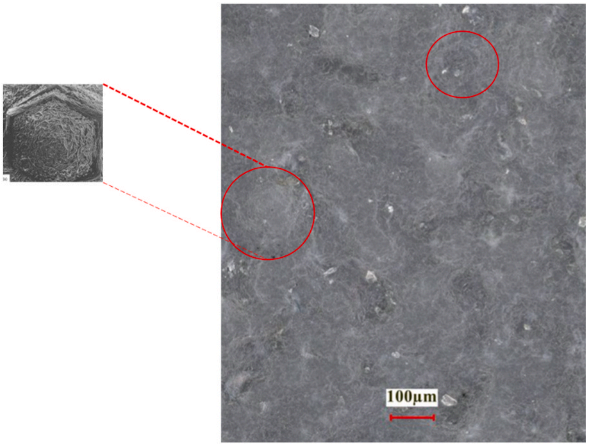

Fig. 6. Laser ablated surface.

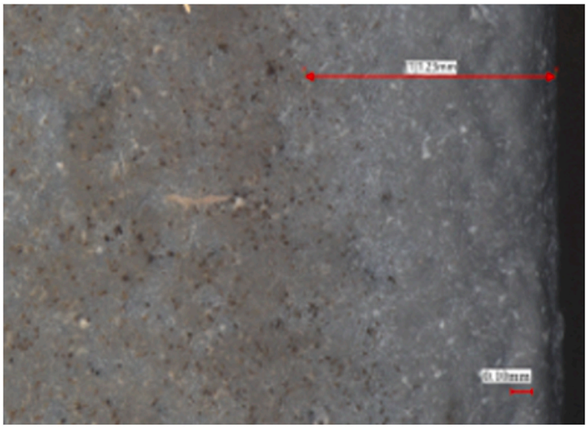

Fig. 7. Heat affected zone.

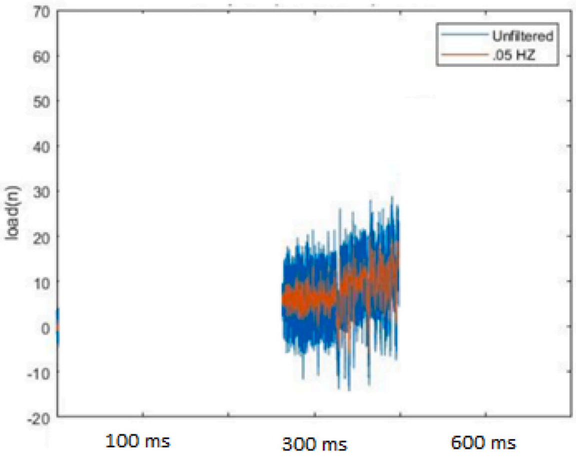

Fig. 8. Normal grinding force of laser irradiated sample.

Fig. 9. Image of laser ablated sample post grind.

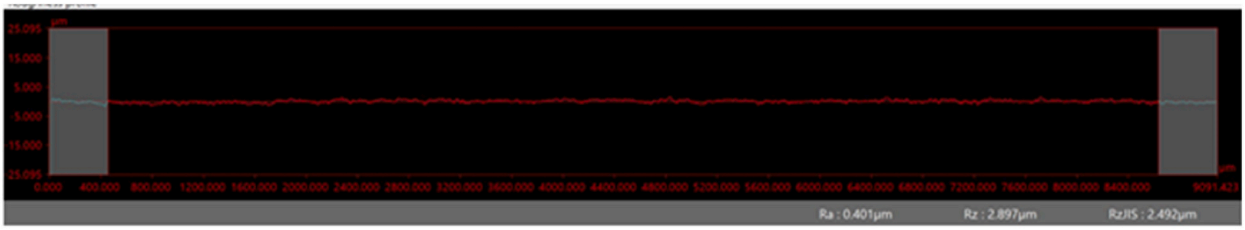

Fig. 10. Trace of laser ablated sample post grind.

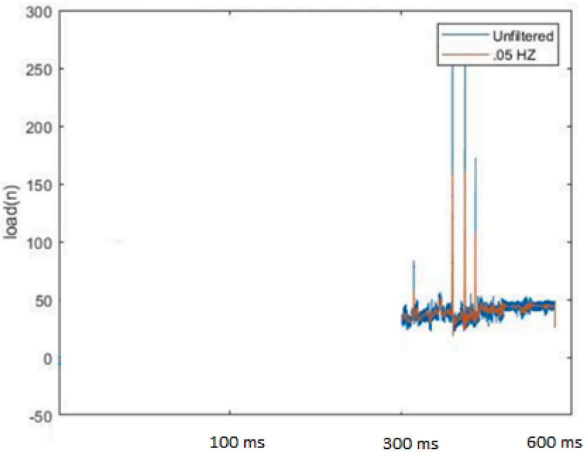

Fig. 11. Normal grinding force of nonirradiated sample.

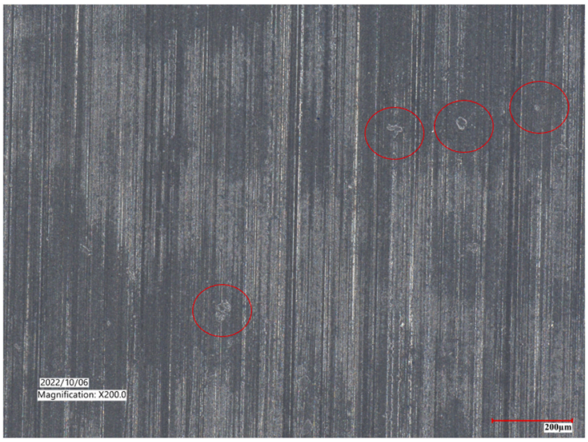

Fig. 12. Image of grind damage on nonirradiated sample.

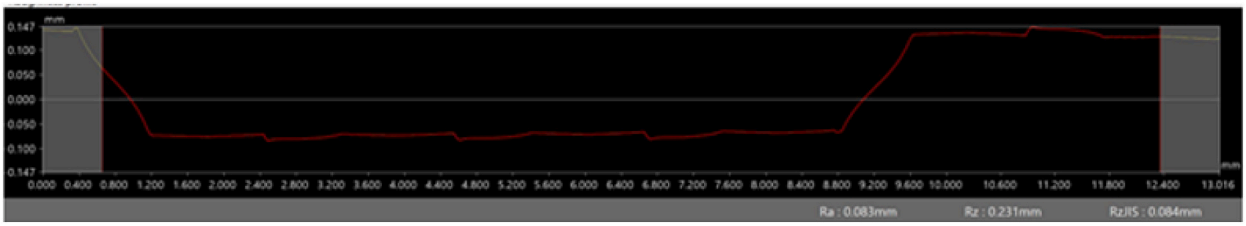

Fig. 13. Trace of grind damage on nonirradiated sample.

An identical sintered Si_3N_4 sample was laser irradiated. Researchers found that it was possible to select laser parameters (e.g.,100W, 400 kHz, 1 mm/s scam speed) which did not induce cracks or any other identified surface damage. Hardness testing indicated laser irradiation softened the material surface 12 %. The microscope showed that the sintered α-β crystalline surface structure was lost. The only regular surface structures observed after irradiation were shades of the pre-sinter, hexagonal α starter phase. The heat affected zone measured approximately 90 μm deep.

When grinding the irradiated sample at a 2.5 μm depth of cut and 0.027 mm3/mm sec MRR, researchers achieved nominal 10 N normal grinding force and ~100 °C grinding temperature. No cracks or surface defects were observed after grinding.

Discussion

Researchers found that it was possible to select laser irradiation parameters such that sintered Si_3N_4 could be both softened on the surface and structurally changed to a depth of approximately 90 μm without inducing cracks or any other defects. Localized melting from the laser is consistent with observed softening. Observed disruption of the sintered α-β crystalline Si_3N_4 structure is consistent with laser energy being sufficient to move basal planes in the heat affected zone out of stacking sequence. Absence of cracking and surface defects suggests that laser energies imparted were below that necessary for material yield.

Researchers, likewise, found that it was possible to defect-free grind the irradiation softened and strained Si_3N_4. Strain weakening of Si_3N_4 is well established [17] as is defect-free grinding under ductile material flow [4]. Test results suggest material weakening from irradiation was sufficient for fully ductile material flow during grinding.

Present methods of laser-assisted machining, which fracture weaken Si_3N_4 prior to grinding and remain cracked after grinding, have shown MRR improvements on the order of 20–50 % [1] as compared to nonablated material. In this work, the MRR was 500 % higher compared to the nonablated sample (e.g., 0.027 mm3/mm sec vs 0.0054 mm3/mm sec). Additionally, at the 500 % higher MRR the sample was ground defect-free and at a normal grinding force 500 % lower than the nonablated sample (e.g., 10 N vs 50 N). Findings suggest, per Equation (2), irradiation weakening improved defect-free MRR because the specific grinding energy required to remove material fell to a level whereby all material was removed by ductile flow.

$$\mathrm{SGE} = (Fn * Vs)/MRR \tag{2}$$

Crack propagation explains why the test part's MRR improvement was orders of magnitude over what is typical for laser-assisted machining. When grinding crack weakened Si_3N_4, MRR is limited by crack growth which finishing and polishing operations must later remove. The irradiated test part in this work had no cracks. Because more energy is needed to start a crack in an engineered ceramic than to propagate an existing one [18], grinding energy was focused on material removal.

Further work

This proof-of-concept work supports further investigation. Irradiation weakening of Si_3N_4 without cracking could be the key to unlocking much higher MRRs than are presently being achieved in laser-assisted machining. Tests with larger sample sizes are needed to determine optimum irradiation parameters as well as statistically define maximum defect-free MRR throughout the heat affected zone.

Data availability

Data associated with study has not been deposited into a publicly available repository, but data is available upon request.

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CRediT authorship contribution statement

Craig Seidelson: Writing – original draft, Methodology, Investigation, Formal analysis, Conceptualization. **Manigandan Kannan:** Writing – review & editing, Supervision, Investigation, Funding acquisition, Formal analysis, Data curation.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

Dr. Craig Seidelson reports financial support was provided by Timken Foundation Center for Precision Manufacturing

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