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Cutting mechanics and subsurface integrity in diamond machining of chalcogenide glass

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Abstract

Infrared-transparent chalcogenide glasses are important for the manufacturing of optics for thermal imaging. These brittle materials can be diamond machined, but material removal rates are often limited by the occurrence of surface/subsurface damage. In this paper, the cutting mechanics of orthogonal cutting, orthogonal flycutting and ball-milling of a common chalcogenide glass (As₄₀Se₆₀) are measured and analyzed. The nature of the resulting surface/subsurface was characterized with atomic force microscopy, Raman spectroscopy, and nanoindentation. Results of this study contribute to both the fundamental understanding of material behavior and the cost-effective production of novel freeform infrared optics.

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1. Introduction

For centuries, optical devices have consisted of axi-symmetric elements - often spherical - due to the relative ease of manufacturing and metrology. Recent advances in optical simulation software, manufacturing equipment and control have broadened the design space by allowing nearly arbitrary optical surfaces - freeform optics - to be manufactured. Current manufacturing technologies, such as CNC diamond machining, are most suited to the tolerance and surface finishes required for longer wavelength applications (e.g., infrared (IR)) [1]. However, many challenges associated with the diamond machining of IR freeform optics remain. Here, we address the connection between the cutting mechanics of brittle IR materials and surface and subsurface integrity. While this work targets IR optics, techniques are applicable to optics functioning in the visible spectrum as well.

It is well known that some brittle materials can be successfully diamond turned to produce an optical quality surface [2-8]. Lucca et al. [6] showed that under certain conditions brittle materials can be machined in a manner that leaves a surface free of significant brittle fracture, while under other conditions, tensile stresses in the cutting zone result in significant surface fracture. IR optics have been diamond turned in brittle semiconductor materials including Si and Ge. Chalcogenide glasses, which contain S, Se, or Te, have numerous advantages for use in IR imaging systems. Owen et al. [1] showed that chalcogenide glass can be milled to produce optical quality freeform surfaces with little visible surface fracture. Owen et al. [1] and Troutman et al. [9] demonstrated that significant changes in specific cutting energy in these glasses, for both continuous and interrupted cutting, coincide with the production of fracture-dominated surfaces. In this paper, we correlate these changes in cutting mechanics with quantitative measurements of surface and subsurface integrity. This is of importance for maximizing the lifetime of optics designed to function at high power or in harsh environments.

We focus on As₄₀Se₆₀, a brittle chalcogenide glass. This material has a low glass transition temperature and is suitable
for molded IR optics. Despite being brittle, the material can also be ultra-precision machined with relatively high material removal rates [1]. To investigate the machining mechanics, orthogonal turning and interrupted flycutting experiments were performed and the cutting mechanics were correlated to uncut chip thickness \( t_c \). Using this knowledge, ball-milled patches were generated for measurement of surface and subsurface integrity. Atomic force microscopy (AFM), Raman spectroscopy, and nanoindentation were used to characterize surface and subsurface integrity.

2. Cutting Mechanics Characterization

2.1. Orthogonal turning

Machining experiments were conducted on a Moore Nanotechnology 350FG ultraprecision diamond machining center. Cutting forces \( F_c \) and thrust forces \( F_t \) were measured while machining the outer diameter of a 30 mm diameter, 5 mm thick cylindrical workpiece. A “dead sharp” single crystal diamond (SCD) tool with 60° included angle, 0° rake angle, and 10° clearance angle was mounted on a Kistler 9256C1 miniature cutting force dynamometer. The chip had a width, \( w_c \), determined by the depth of cut, and uncut chip thickness, \( t_c \), determined by the feed per revolution.

With the surface speed held constant at 2 m/s, and a fixed chip width of 100 μm, \( t_c \) was varied from 200 nm to 2.0 μm. For each set of parameters, 80 revolutions of cutting data were used to generate average forces. To minimize initial subsurface damage, the surface was pre-machined with a \( t_c \) of 100 nm for 250 revolutions prior to each experiment. Experiments performed in ascending and descending order agreed, indicating that the initial surface condition was the same for each experiment. Figure 1 shows average \( F_c \) and \( F_t \) as a function of \( t_c \) and representative SEM images of the chips.

![Fig. 1. Measured forces from orthogonal turning experiments. Linear fits of the low \( t_c \) regime are shown by solid lines. Insets show chips from the two regimes (400 nm, top) and (1.2 μm, bottom).](image)

At \( t_c \) values above 600 nm, the chips were fragmented indicating a material removal mechanism dominated by brittle fracture. With decreasing \( t_c \) down to roughly 600 nm, cutting forces were found to decrease slowly. At \( t_c \) values near 600 nm, both \( F_c \) and \( F_t \) increase by approximately a factor of five. The increase in \( F_c \) is accompanied by a significant jump in the energy per unit volume of material removed, from 0.2 J/mm³ to 1.3 J/mm³, as reported previously by Owen et al. [1]. Further decrease in \( t_c \) results in a rapid, nearly linear decrease in the cutting forces. The mechanics of this force behavior remain unknown, but could potentially indicate a visco-plastic response, strain rate sensitivity, thermal sensitivity, or other material behavior. Chips created when \( t_c \) was less than 600 nm were not consistent with the fragmented chips observed for higher \( t_c \), but were instead curled.

2.2 Orthogonal flycutting

To study the effect of an interrupted cut under orthogonal cutting conditions, an orthogonal flycutting arrangement was developed. In this configuration, the workpiece was pre-machined to leave only thin ribs. Each rib was machined using a flycutter that was 100 mm in diameter, with the same tool used for the orthogonal turning. A more detailed description of both the turning and flycutting experiments is given in Troutman et al. [9]. At a constant surface speed of 2 m/s, cutting experiments were performed on workpieces with rib widths of 1 mm, 2 mm, and 4 mm, corresponding to tool-workpiece contact times of 0.5 ms, 1 ms, and 2 ms.

For each rib, six total experiments were performed at each \( t_c \) and the progression from low to high \( t_c \) was repeated three times. Figure 2 shows the mean values of \( F_c \) and \( F_t \) for each of the three progressions. Force magnitudes are in agreement with Fig. 1 and relatively little variation due to rib size is evident. Further, the increase in \( F_c \) and \( F_t \) occurred at a higher \( t_c \) between 800 nm and 1.0 μm. The uncertainties in the mean values of \( F_c \) and \( F_t \) were greatest near the transition region with standard deviations of 19 mN and 15 mN at \( t_c \) equal to 1.0 μm and 1.2 μm respectively. At all other values of \( t_c \), the standard deviation was less than 4 mN. The possibility that the transition in cutting mechanics is altered by an interrupted cut is also supported by observations in ultraprecision diamond ball-milling where brittle materials such as Ge can be successfully milled with zero rake angle tools [10], whereas the turning literature indicates a negative rake angle is required [5].

![Fig. 2. Measured forces during orthogonal flycutting.](image)

2.3 Ball-milling experiments

To investigate the changes in the governing mechanics of the milling process and their effect on the nature of the generated surface and subsurface generated, chalcogenide glass was raster ball milled. A SCD, single flute, 1 mm diameter, zero rake angle ball end-mill was used to machine 2 mm x 2 mm patches with the parameters summarized in

[1] J.R. Troutman et al., 2016, Procedia CIRP 45, 135–138.
Table 1. Ball-milling parameters for the patches.

| Patch | Feedrate (μm/rev) | Stepover distance (μm) |
|-------|-------------------|------------------------|
| A     | 0.3               | 12                     |
| B     | 0.3               | 60                     |
| C     | 10                | 12                     |
| D     | 10                | 60                     |

3. Surface and Subsurface Characterization

A commercial Bruker AFM operating in tapping mode was used to examine the surface topography and measure the average surface roughness (Ra) of the milled patches. Confocal Raman spectroscopy in a backscattering configuration was performed with a frequency doubled Nd:YAG laser (λ = 532 nm) and a 10x/0.2NA microscope objective. The laser power was minimized to avoid heating and modification of the specimen. The near surface hardness and elastic modulus of the patches were quantified by nanoindentation experiments performed with a force-controlled commercial nanoindenter using a Berkovich diamond indenter. The instrument compliance and indenter area function were obtained by performing indentations in fused silica and tungsten using the procedure of Oliver and Pharr [11] and consistent with ISO 14577-4. The loading/unloading sequence was as follows: (1) a 30 s increase to a maximum force of 2,500 μN; (2) a 60 s hold at the maximum force to allow any time dependent plastic effects to diminish; (3) a 10 s unloading to 10% of the maximum force, (4) a 60 s hold at 10% maximum force to measure thermal drift; and (5) a 2 s final unloading.

4. Discussion

Top view surface topography images of the milled patches obtained by AFM are shown in Fig. 3. The raster direction for machining was vertical so that at low feed rates (Patches A and B) cusps with 1 mm radius of curvature are produced. The height scale for patches A and C (created with the lowest stepover distance) was 50 nm and for patches B and D (created with the highest stepover distance) was 500 nm. Cross sectional analysis of the patches (not shown here) showed a dominant cusp structure in patches A and B coming from the tool stepover. For patches C and D, circular features separated by 10 μm (feedrate) are superimposed on the cusp structure. As expected this is most evident in patch C where the stepover and federate are nearly equal. Local surface roughness was measured in 10 μm × 10 μm AFM scans (not shown) to determine the acceptable minimum penetration depth during nanoindentation. The local Ra value was less than 6 nm for all patches.

Raman spectroscopy was performed on the patches to attempt to measure differences in the quality of the near-surface region. The spectra, shown in Fig. 4, consisted of two relatively broad peaks, the first with higher intensity centered at approximately 230 cm⁻¹ and the second lower intensity peak centered at approximately 110 cm⁻¹. The position and shape of both peaks are consistent with previous reports of the Raman response of As₄₀Se₆₀ and other AsSe alloys [12,13]. No differences in spectra were observed between patches. This indicates that unlike Ge [3], Raman spectroscopy was not able to measure differences in the resulting residual stress or nature of the chalcogenide glass.

The near surface mechanical response of the patches was investigated by nanoindentation experiments. Figure 5 shows typical force vs. penetration depth curves obtained for the patches at a maximum force of 2,500 μN. The curves show loading, the hold at maximum force (P_max) and unloading. The curves can be used for the evaluation of hardness (H) and reduced elastic modulus (E_r). The initial portion of the unloading curve was fit to a power law, which then allowed for the determination of the stiffness during unloading (S = dP/dh) and the contact area (A) at P_max. H and E_r were then obtained by H = P_max/A and E_r = μ²S(2A)/E, (where 1/E_r = (1 - μ²)/E + (1 - μ²)/E_i, and E and μ are the elastic modulus and Poisson’s ratio for the specimen and E_i and μ_i are the same values for the diamond indenter. The obtained H and E_r values for the patches are shown in Fig. 5. The results shown represent the average values of 10 indentations along with their standard deviation. An average E_r value of 26.4 GPa was obtained for patch A which is equivalent to an elastic modulus of 24.7 GPa using a Poisson’s ratio of 0.29 [14] and indenter modulus of 114 GPa and Poisson’s ratio of 0.07 [15]. This value can be compared to the value of 22.2 GPa reported in the literature [16]. An increase in the feedrate or stepover distance (for patches B and C) is seen to have no measurable influence on H and E_r. However, when both the feedrate and stepover distance were increased to their...
highest values (for patch D), higher values of \( H \) and \( E_r \) were obtained. Further studies are required to determine the effect of more aggressive machining on the material, and the underlying cause of this increase.

To better understand the deformation mechanism during nanoindentation, examination of the residual impressions on patches A and D (created with the lowest or highest geometric parameters) were performed by AFM. Figure 6 shows these residual impressions with a scan area of 5 \( \mu m \) × 5 \( \mu m \) and height scale of 100 nm. No obvious fracture was observed around the corners on either of the impressions. On the other hand, while the material appears to sink-in along the edges of the impression on patch A, no similar behavior can be seen on patch D and the edges of the impression are distinctly visible. Although the origin of this altered behavior is not clear at this point, it indicates a difference between the elastic-plastic response of the indented material on patches A and D [17].

5. Conclusions

With decreasing uncut chip thickness during orthogonal turning and flycutting, \( \text{As}_{20}\text{Se}_{80} \) was shown to exhibit an abrupt increase in forces at a critical value of uncut chip thickness. This critical value was higher during an interrupted flycutting process than during continuous turning. Changes in the feedrate and stepover distance during the ball end-milling were seen to alter the surface topography of the milled patches. However, it was found that average local surface roughness was not significantly affected by the milling parameters. Increasing both the feedrate and stepover distance to their highest values resulted in an increase in the hardness and reduced elastic modulus of the patches and also a reduction of the tendency of the material to sink-in during nanoindentation experiments. Understanding the differences in the mechanical response of the machined material may also provide insights into the general material behavior of chalcogenide glass under machining conditions (high stress, high strain rate, etc.). The end goal is to identify the physical mechanisms responsible for this previously unobserved machining behavior, and to link this with the integrity of the machined surface and subsurface.

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References

[1] Owen J, Davies M, Schmidt D, Urruti E. On the ultra-precision diamond machining of chalcogenide glass. CIRP Annals - Manufacturing Technology. 2015; 64(1): p. 113-116.
[2] Giovanola, JH, Finnie, I (1980) On the machining of glass. Journal of Materials Science 15:2508-2514.
[3] Puttick KE, Rudman MR, Smith KJ, Franks A, Lindsey K (1989) Single-point diamond machining of glass. Proceedings of the Royal Society of London A, Mathematical, Physical & Engineering Sciences 426(1870):19-30.
[4] Nakasui T, Kodera S, Hara S, Matsunage H, Iwaka N, Shimada S (1990) Diamond turning of brittle materials for optical components. CIRP Annals - Manufacturing Technology 39(1):89-92.
[5] Blake PN, Sattergood RO (1990) Ductile-regime machining of germanium and silicon. Journal of the American Ceramic Society 73(4):949-957.
[6] Lucca DA, Chou P, and Hocken RJ (1998) Effect of tool edge geometry on the nanometric cutting of Ge, CIRP Annals - Manufacturing Technology 47(1):475-478.
[7] Chiu W-C, Endres WJ, Thoules MD (1999) An experimental study of orthogonal machining of glass. Machining Science and Technology 4(2):253-275.
[8] Bae D-S, Yeo J-B, Lee H-Y (2013) A study on a production and processing technique for a GeSiBGe aspheric lens with a mid-infrared wavelength band. Journal of the Korean Physical Society 62(1): 1610-1615.
[9] Troutman, J, Owen J, Davies M, Schmitz T (2015) Cutting forces during diamond flycutting of chalcogenide glass. 30th ASPE Annual Meeting, Austin TX.
[10] Dutterer BS, Lineberger JL, Smilie PJ, Hildebrand DS, Hartman TA, Davies MA, Suleski TJ, Lucca DA (2014) Diamond milling of an Alvarez lens in germanium. Precision Engineering 38:398-408.
[11] Oliver WC, Pharr GM (1992) An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments. Journal of Materials Research 7:1564-1583.
[12] Iovu MS, Kamitsos EI, Varsamis CPE, Boolchand P, Popescu M (2005) Raman spectra of \( \text{As}_{20}\text{Se}_{80} \) and \( \text{As}_{20}\text{Se}_{80} \) glasses doped with metals. Chalcogenide Letters 2(3):21-25.
[13] Golovchak R, Oelgoetz J, Vlcek M, Esposito A, Saiter A, Saiter J-M, Jain H (2014) Complex structural rearrangements in \( \text{As-Se} \) glasses. The Journal of Chemical Physics 140: 054505.
[14] Mešnichenko TN, Katenko YP, Fedeshki VL, Yurkin IM, Mešnichenko TD (2001) Interrelation between the glass transition temperature, thermal expansion coefficient, and Poisson ratio for some glasses in the AV–BVI–TD (2001) system. Glass physics and chemistry 27(4): 298-305.
[15] Lucca DA, Herrmann K, Klopstein MJ (2010) Nanoindentation: Measuring methods and applications CIRP Annals-Manufacturing Technology 59:803-819.
[16] Tronov ML, Bilanich VS, Duh SN (2007) The non-Hookian behavior of chalcogenide glasses under irradiation: A nanoindentation study. Journal of non-crystalline solids 353(18): 1904-1909.
[17] McElhaney KW, Vlassak JJ, Nix WD (1998) Determination of indenter tip geometry and indentation contact area for depth-sensing indentation experiments. Journal of Materials Research 13(05): 1300-1306.