An Investigation of the Effects of Diamond Machining on Germanium For Optical Applications

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Abstract

This investigation will examine differences in the diamond machined surface structure of various mono and polycrystalline germanium material forms. Germanium substrates were machined under controlled conditions and evaluated for surface texture and subsurface damage. Results of the substrate evaluations along with details of the sample preparations and machining are given.

Introduction

Germanium is a common material of choice for infrared optical components. It possesses excellent transmission characteristics over the important infrared spectral regions between 2 and 15 microns. Optical grade germanium is manufactured and available in several forms, both as a mono and polycrystalline material. Diamond machining is one technique for fabricating optical components from germanium. The suitability of using diamond machined germanium for infrared applications has been established.[1,2]

This investigation will examine differences in the diamond machined surface structure of various mono and polycrystalline germanium forms. Specifically, substrates of Czochralski and cast polycrystalline and near <1:1:1> oriented monocrystalline materials were used. These substrates were diamond machined under controlled conditions and evaluated. Surface texture was evaluated by the techniques of stylus (contact) profilometry, micro-interferometric (non-contact) profilometry, and Nomarski (differential interference contrast) microscopy. Subsurface damage and surface stress conditions were evaluated by Raman spectroscopy.

Germanium Substrates

The germanium used in this investigation was classified as optical grade material. Optical grade germanium is produced from material that has been zone-refined[3] and then appropriately doped to permit maximum transmittance in the 8 to 12 micron spectral region. The zone-refining process yields material with a resistivity greater than 40 ohm-cm, is N type in electrical conductivity, and is spectrographically pure as measured via an emission spectograph. The growth of the germanium boule can be accomplished either by a vertical or horizontal process, the crystal growth is similar in both. The vertical process utilizes the Czochralski technique. In the Czochralski technique molten germanium, the melt, is maintained at a temperature slightly above its melting point. The crystal is begun by bringing a seed crystal, whose temperature is slightly below the melting point, into contact with the liquid. Heat from the melt in the vicinity of the seed flows into the seed thereby cooling the adjacent liquid sufficiently to cause localized solidification onto the end of the seed. Atoms of germanium crystallize and replicate the structure of the seed as the seed is withdrawn from the melt. Thus a single nucleus will grow until the entire material of the melt is consumed to form a boule. Once the boule has been grown it is further annealed to relieve residual stresses in the material.

The determination of the crystalline nature of the material begins with the selection of the seed. Although it is possible to grow either a mono or polycrystalline boule starting from both mono and polycrystalline seeds, the seed choice is generally monocrystalline. The introduction of crystalline boundaries occurs when the germanium in the melt does not align with the orientation of the boule lattice as it makes the transition to a solid phase at the solid-liquid interface. There are a number of reasons why this misalignment can occur. When this misalignment does occur, a grain boundary can be formed resulting in the production of a polycrystalline boule. For optical applications in the infrared, polycrystalline material is indistinguishable from monocrystalline material if it is free from inhomogeneities, birefringence and localized absorptivities. Specifications for optical grade germanium are given in table 1.[4]

Standard	
Crystalline Form	polycrystalline
Conductivity Type	N-type
Resistivity Range	4-40 ohm-cm
Absorbtion Coeficient, at 25 deg C	0.035 cm^{-1} max at 10.6 μm
Oxygen Content	less than 0.03ppm
Holes and Inclusions	not larger than 0.002 in
Premium	
Crystalline Form	monocrystalline
Resistivity Range	to specifications within the 4-40 ohm-cm range
Absorption Coefficient, at 25 deg C	as low as 0.02 cm^{-1} at 10.6 μm

Table 1 Material Specifications for Optical Grade Germanium

Three optical grade germanium samples were used in this investigation. A sample of monocrystalline material oriented in the near <1:1:1> direction was used. Monocrystalline substrates can be produced in any orientation, including the <1:0:0> and the <1:1:0> orientations common in electronic applications. The <1:1:1> orientation is most commonly supplied for optical applications, when monocrystallinity is requested, because maximum size substrates can be cut from this geometry. The two other samples were polycrystalline: one Czochralski grown and the other horizontally cast. In both samples, individual grain dimensions typically exceed one millimeter. All three substrates were fabricated to a 50 mm diameter with 10 mm axial thickness. The surface to be machined was spherically generated to a 90 mm convex radius with a bonded grinding wheel impregnated with 280 grit diamonds. The 90 mm radius was used to ease the metrology considerations and to simulate a typical infrared component surface. A contoured geometry requiring turning was chosen, as opposed to a flycut surface, to insure that the effects of two-axis machining motions required to produce aspherical surfaces would be considered.

The samples were evaluated prior to diamond machining to determine how much stock removal would be required to "clean" the surfaces of damage from the generating process. The samples had an average profile roughness (PR_a) of 1.0 micron and a peak-to-valley profile (PR_t) of 10.0 microns as measured across the full 50 mm surfaces. The disrupted layer caused by the spherical generation extends below the measured profile peak-to-valley roughness. An amount equal to three times the profile peak-to-valley roughness (ie. 30 microns) was removed to insure the erasure of any damage due to the generation. In the first machining pass of 12.5 microns (0.0005 in) each of the surfaces was rendered specular.

Diamond Machining Conditions

The samples were diamond machined on a Rank Penumo MSG-325, two axis, CNC, laser interferometric feedback contouring lathe. The diamond tool used for the machining was a Rank Pneumo catalog tool, designation CO25WG. The diamond in this tool is a natural gem-quality single crystal stone. It is fabricated to have an edge radius with controlled waviness of less than 0.5 microns (20 microinches) over a sweep angle (i.e. cutting window) of 100 degrees. In addition, the tool has a negative 25 degree top rake and 10 degree front clearance angle with a nominal cutting edge radius of 0.63 mm (0.025 in). This diamond tool's geometry is considered typical for the contouring of germanium. The samples were machined at a work spindle speed of 1500 rpm and a surface feed rate of 2.54 mm/min (0.10 in/min) using sprayed cycloparaffin mineral spirits (OMS) for chip removal and as a lubricant. The final depth of cut on each sample was 0.75 microns (30 microinches).

Parameters for the diamond machining of these samples were chosen based upon general experience for the machining of germanium. The material removal rate has been found to yield good results in germanium when the quotient of feed to rotational speed does not exceed 2.5 microns/rev (100 microinches/rev) and maximum depths of cut do not exceed 25 microns (0.001 in). Using these parameters the machining appears to remove material in a ductile fashion with no evidence of brittle fracturing on the surface. In addition, the diamond tool cutting edge is not excessively worn at these cutting parameters. This is demonstrated by the full surface interferogram of the final surface turned shown in figure 1. Other less conservative parameters for the machining of germanium have been published.[5]

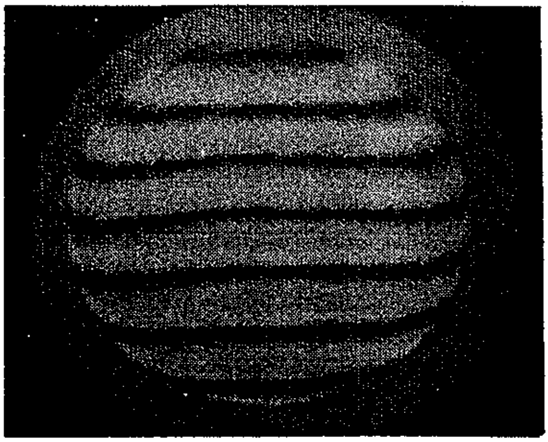

Figure 1 Full Surface Interferogram

Surface Texture Comparisons

Three techniques were used to investigate differences in the surface texture of these samples., These techniques were: stylus (contact) profilometry, micro-interferometric (non-contact) profilometry, and Nomarski (differential intereference contrast) microscopy. The stylus profilometry was performed on a Rank Taylor Hobson Form Talysurf, measuring in the roughness mode using a 0.8 mm cut-off figure and an ISO filter. The micro-interferometric profilometry was performed on a Wyco Digital Optical Profiler NCP-1000M at a magnification that produced a 665 micron scan length. The Nomarski microscopy was performed on an Olympus Vanot T microscope.

Four stylus measurements were taken on each sample: one near the edge, the second across the center, another at half the radial distance on the surface, and the last with the sample rotated 90 degrees again at half the radial distance. All traces were taken orthogonal to the direction of machined surface lay. The values of the measurements given in table 2 are the means of the arithmetic average (R_a) across five cut-off lengths, a total scan length of 4.0 mm (0.16 in) per trace.

	edge	center	half	half-90	average
monocrystal near <1:1:1>	61 (0.24)	64 (0.25)	53 (0.21)	58 (0.23)	59 (0.23)
polycrystal Czochralski	58 (0.23)	64 (0.25)	58 (0.23)	61 (0.24)	60 (0.24)
polycrystal cast	66 (0.26)	66 (0.26)	61 (0.24)	56 (0.22)	62 (0.25)

Table 2 Arithmetic averages (R_a) in angstroms (microinches)

These results are just above the 40 angstrom limit of the instrument's accuracy for surface texture measurements. By using a micro-interferometric profilometry technique better accuracy was obtained but at the expense of a shorter scan length due to the curved surface. Using the micro-interferometer it was also possible to selectively search for grain structure effects on the polycrystalline surfaces. Micro-interferometric rms surface texture measurements of the three samples ranged from 30 angstroms (0.12 microinches) to 66 angstroms (0.26 microinches) as measured orthogonal to the machine lay. In the interference microscope it is possible to observe the grain boundaries in the polycrystalline samples. Their effects on these rms surface texture measurements were estimated to be at most 15 angstroms (0.06 microinches). It should be noted that these surface grain boundary effects on texture and hence on scatter (TIS) would be significantly less than one percent at the germanium "cut-on" frequency and negligible in the 8 to 12 micron spectral band.

The evaluation of the surfaces by Nomarski microscopy gave qualitative information about the surfaces. In the Nomarski evaluation of the polycrystalline samples, the grain structure could not be readily distinguished. The surfaces for all three samples were free of the pitting usually observed when germanium is machined at excessive feed rates. On the monocrystalline sample, the three-lobed crystallographic structure was also not present. None of the samples appeared to have evidence of brittle fracture material removal. As viewed in the microscope at 300x, the predominant surface texture trait was a long-term undulation superimposed on the feedline cusps. This is evident in the photomicrograph in figure 2. This waviness is most probably due to the use of a negative top rake tool. For such a tool the chip morphology should result in higher than normal cutting forces and a surface that is compressively stressed.

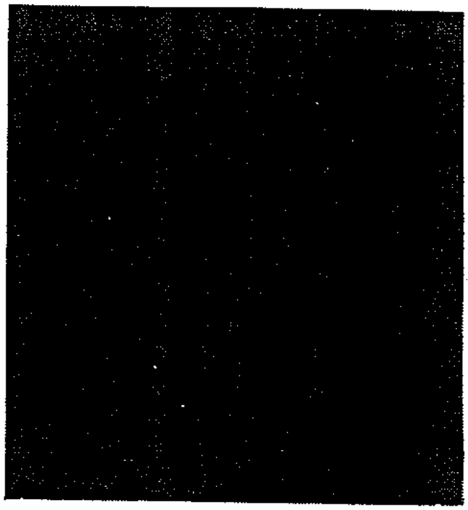

Figure 2 Czochralski Polycrystalline Surface at 300X

Subsurface Damage and Stress Conditions

The samples were evaluated for subsurface damage and surface stress conditions by a "macro" Raman spectroscopy technique.[6] In Raman spectroscopy, an incident photon interacts with phonons in the germanium crystal lattice and is inelastically scattered. From the shift in frequency of the incident to scattered light, the frequency of lattice vibrations can be determined. This lattice vibration is influenced by the presence of stress in the crystal. For germanium the unstressed Raman peak occurs at 299 cm^{-1} away from the excitation frequency. Shifts in that peak are converted to stress by a linear relationship between the two.

The 514.5 nm line of an argon laser was used to excite the samples. The elliptical footprint of the laser spot on the samples was approximately 10 mm by 1 mm and was projected on the sample about 20 mm from the center of the sample. At this wavelength the laser line has a penetration depth of approximately 0.6 microns. Table 3 contains the results for the three samples measured. Figure 3 displays the intensity of the Raman scattered light as measured by the photomultiplier after spectral analysis by the Czerny-Turner grating spectrophotometer:

Sample 1: mono near <1:1:1>; compressive stress
$1.5 \pm 0.3 \times 10^8$ Pa

Sample 2: poly Czochralski; compressive stress
$1.3 \pm 0.3 \times 10^8$ Pa

Sample 3: poly cast; compressive stress
$2.1 \pm 0.3 \times 10^8$ Pa

Table 3 Stress Conditions

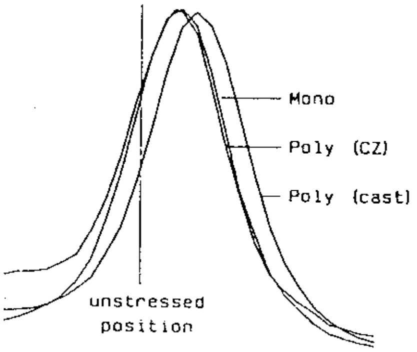

Figure 3 Raman Spectrum

With these results the following conclusions can be made. Material removal from all three surfaces has been ductile. No evidence of the tensile stresses that result from the brittle fracturing of a surface were found. This is consistent with the croscopic observations of the surfaces. Because of the relative size of the laser spot on the samples as compared with the typical polycrystalline grain size, it is expected that several grain boundaries occured within the measurement footprints on the polycrystalline samples. No evidence of the tensile stresses that would accompany crack propagation at the grain boundaries were observed. The compressive stresses at the surfaces is consistent with the chip morphology associated with machining germanium with a highly negative raked tool.

Conclusions

This investigation has examined differences in the diamond machined surface structure of various mono and polycrystalline germanium material forms. For infrared optical applications, the differences in surface structure between polycrystalline and monocrystalline germanium, as affected by diamond machining, are insignificant. Surface texture differences as a result of the presence of polycrystalline grain boundaries are estimated to be below 15 angstroms rms. The presence of grain boundaries does not appear to cause brittle fracture damage to surfaces as a result of diamond machining.

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