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Assessment of Subsurface Damage in Ultraprecision Machined Semiconductors

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Abstract—The subsurface damaged layer in ultraprecisison machined single crystal Ge was examined by ion channeling. Single crystal Ge surfaces were prepared by chemo-mechanical polishing, mechanical polishing with 1/4 μm diamond abrasive, single point diamond turning and ultraprecision orthogonal flycutting. The extent of subsurface lattice disorder was compared to the crystal's orginal surface quality. Ion channeling is seen to be useful for quantitative measure of lattice disorder in finely finished surfaces.

1. Introduction

The destruction or alteration of subsurface lattice order when semiconductor materials are processed into final form has long been of interest. Most recent focus is on assessing damage depths which approach the limits of capability of all but a few techniques. Study of the subsurface damage which results when Ge is finely polished or single point diamond machined is of both practical and fundamental interest. Germanium's use as substrate material for solar cells and in the manufacture of x-ray monochromators, as examples, points to the need for assessing the damaged layer which effects both the conditions for epitaxial growth and device performance. In addition, basic questions regarding the physics which governs material removal at dimensions approaching the atomic scale persist. Significant interest in the so-called "ductile-regime" machining and grinding of brittle materials has resulted in several investigations aimed at identifying the mechanisms of material removal at depths of cut where there is the appearance of ductile cutting of an otherwise brittle material [1-3]. These studies are based on a three dimensional diamond turning operation, and examine the transition from a damaged to undamaged surface along the shoulder of a cut resulting from interrupted machining. The transition point, or critical depth of cut, is seen to vary with feed rate, workpiece crystallographic orientation, tool rake angle and lubricant, and has been observed to be in the range of 50-300 nm for Ge [2]. Whereas the change from pitted to not-pitted region is apparent, there is still debate on the actual mechanism responsible for material removal [4]. In addition, the appearance of a non-pitted surface may not necessarily indicate the absence of a damaged subsurface. Quantitative information in the literature regarding the depths of subsurface damage is limited. In the present study, we report results on the assessment of lattice disorder resulting from Ge which has been chemo-mechanically polished, mechanically polished and single point diamond machined.

2. Preparation of Surfaces

Single crystal n-type Ge supplied by Eagle-Picher Research Laboratory, Miami, OK was used in this study. The material was sawn from a bulk crystal, and then etched with a proprietary etch to remove all saw damage. The prepared surfaces included chemo-mechanically polished, mechanically polished, and single point diamond machined with both three dimensional facing cut and orthogonal flycutting geometries. For the polishing and face cutting experiments, $5\times5\times3$ mm rectangular specimens were oriented such that a 5×5 mm face was parallel to a (111) plane, and one of the 3×5 mm sides was parallel to a $(\bar{1}10)$ plane. For orthogonal flycutting, the specimens were $12\times15\times1$ mm with the 1×15 mm face parallel to the (001) plane and a 12×15 face parallel to a (010) plane. For Ge, the

(111) is a preferred cleavage plane. The specimens were oriented using Laue back-reflection x-ray diffraction to within ± 0.5 degrees.

The chemo-mechanically polished (111) surface-oriented specimens were prepared by first removing about 75 μm of material by lapping with 9 μm Al_2O_3 abrasive, subsequently removing about 15 μm by mechanically polishing using a 1/4 μm diamond abrasive, and finally removing about 10 μm by chemo-mechanically polishing by using a proprietary solution. The mechanically polished (111) surface were prepared by removing about 75 μm of material by lapping with 9 μm Al_2O_3 abrasive, and than removing about 25 μm by mechanically polishing using a 1/4 μm diamond abrasive. For the single point diamond face cutting surface preparation, a (111) oriented crystal was first chemo-mechanically polished using the sequence described above to minimize the subsurface damage which would be required to be removed by diamond turning. The specimen was than mounted with glycol phthalate to a lapped aluminum substrate which was in turn held on a vacuum chuck of a commercial diamond turning machine. The crystal was positioned with its centerline approximately 28.6 mm off the spindle axis, with the $(\bar{1}10)$ aligned with the cutting direction. The (111) face of the crystal was machined at a nominal depth of cut of 1.25 μm. The feedrate used was 10.21 μm/rev, and the cutting speed at the crystal centerline was 0.72 m/sec. No lubricant was used. Multiple passes at the final depth of cut were taken to assure that the surface was representative of the depth of cut of interest. A single crystal diamond tool having a nose radius of 5 mm, a 0 degree rake and a 5 degree clearance angle was used. The edge profile, as measured with an atomic force microscopy technique previously reported [5], was found to be a true radius of $50\ nm \pm 10$ nm. An additional (111) oriented specimen was prepared with an etched surface such that ion channeling could be performed on it, providing a measure of the orignal bulk crystal quality.

As discussed above, two additional specimens were prepared by single point diamond flycutting. Orthgonal cutting was performed on the (001) plane in the [010] direction by rotating a flat nose, single crystal diamond tool (0 degree rake, 5 degree clearance ange) 76.2 mm off the spindle axis of the diamond turning machine, and infeeding a 15×1 mm rectangular specimen. The crystal was positioned with its length tangent to the tool motion, also 76.2 mm off axis. The magnitude of the off-axis dimension compared to the length and width of the specimen allowed for orthogonal "planing" to be achieved. This configuration is the same as that reported elsewhere [6]. The rotational speed (cutting velocity) was held constant at 0.78 m/sec, and the infeed rate corresponded to the depth of cut. No lubricant was used. Surfaces were produced with depths of cut of 20 nm and 400 nm.

3. Channeling in Crystals

Channeling is the influence of a crystal lattice on the trajectories of energetic particles. Since its discovery in the mid-1960's channeling has been extensively used to study the crystalline structure of solids at the near surface (< 1 μm depths). Rutherford Backscattering Spectrometry (RBS) is a well established technique for probing surface and near surface atomic composition. When used under channeling conditions, RBS can provide information about both atomic composition and crystal structure. Applications include study of the lattice position of impurities of crystalline solids (e.g., ion implanted surfaces) and study of the nature of crystalline or amorphous thin films. A thorough treatment of channeling is presented in the text by Feldman *et al.* [7]. The type of interaction of an ion beam with a single crystal solid depends upon the energy of the incident beam. For example, energies on the order the oder of 10 eV are typically used film deposition and epitaxial growth, sputtering is performed with heavy ions in the 1 keV range, and implantaion requires energies on the order of 100 keV. Channeling is achieved with ions having MeV energies. Whereas sputtering and ion implantion leave the surface structure substantially disturbed, the high energy, light particles used for channeling can penetrate deeply into the crystal without significant surface damage. Techniques used to study the crystal sructure of solids such as x-ray and neutron diffraction rely on radiation with wavelengths comparable to the lattice spacing ($\sim 10^{-7}$ mm) such that the crystal is seen as a diffracion grating to the incident radiation. An incident beam of MeV He ions with wavelength on the order of 10^{-11} mm sees the crystal not as a diffraction grating but as columns and rows of atoms

which direct its motion through the crystal. Channeling relies on more than simply the geometric transparency of a crystal lattice; the ions are steered during their travel through the crystal by the interatomic potential fields throughout the lattice. As the ions pass by an atom, most make small energy transfers (~10 eV) with outer electrons. A few experience close encounters with the nucleus resulting in large energy loss (~100 keV).

Fig. 1 shows schematic representations of the interaction of an in beam with a single crystal. Fig. 1a shows a perfect crystal which has one of its principal planes of symmetry aligned with the incident beam. As the beam encounters the crystal, some ions make close impact with the atoms at the surface, and experience large angle scattering events. The majority are able to penetrate the crystal and are steered by the rows and sheets of atoms. The backscattered energy spectra for this aligned case show a small surface peak and a subsequent reduction in yield of backscattered particles which results from the atoms at the surface shielding those directly below from close encounter with the beam. If the surface is randomly positioned with respect to the incident beam, channeling will not occur, a surface peak will not be observed, and the energy spectra will be characteristic of Rutherford backscattering from an amorphous solid. The alignment of the crystal with the beam accounts for a reduction in backscattered yield of about 98%, and is the basis on which channeling is used as a probe for surface structure. If the crystal contains a thin, disordered or amorphous layer (Fig. 1b), the scattering yield at the surface increases over that for the aligned case. If the layer is thin enough, some ions can make their way through the disordered layer, and will encounter ordered structure below. In this case, the backscattered yield will dercrease, and a measure of the thickness of the disordered layer based on energy loss can be made. A channeling experiment requires a source of collimated monochromatic ions, a detector of scattered particles, and precise crystal manipulator to align the crystal with the incident beam. Channeling is achieved when the crystal is aligned to within 1 degree of the beam -i.e., the critical scattering angle is on the order of 1 degree.

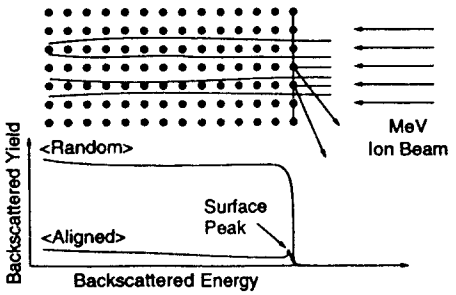

Fig. 1a). <Aligned> and <random> energy spectra for a perfect crystal.

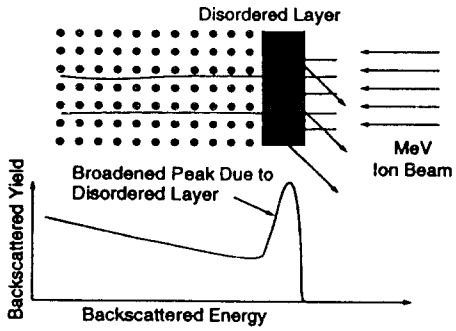

Fig. 1b). Energy spectra for a single crystal containing a disordered surface layer.

4. Results and Discussion

After the crystal surfaces were prepared, they were characterized with RBS under channeling conditions. A schematic of the experimental set-up is shown in Fig. 2. For the (111) oriented specimens, a collimated beam of 2 MeV $^4He^+$ ions with a spot size of approximately 2×2 mm was directed at the surface. The crystals were mounted on a goniometer which enabled precise alignment of the [111] direction with the beam. The backscattered particles were

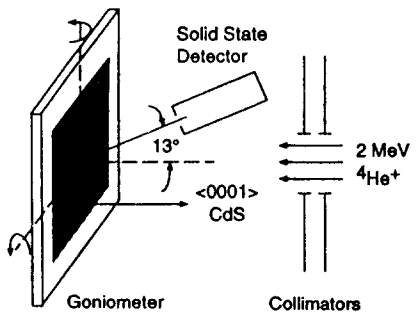

Fig. 2. Schematic of channeling set-up.

collected with a nuclear detector positioned 13 degrees off the beam axis. The solid angle for collection was 2.4 msr. The chamber operated at 4-5 $\times$ 10^{-7} Torr. Although the goniometer had to position within 0.01 degree, alignment to within 0.1 degree was sufficient to achieve channeling. (Recall that the critical sattering angle is about 1 degree). The crystal was tilted 4 degrees off its initially positioned axis in both planar directions (x and y), and then stepped through a series of 0.2 degree increments in one axis while holding the other axis fixed. This was done in all four directions resulting in an 8 degree $\times$8 degree "box" around the beam axis. A window of 1 MeV was set up in the region of expected backscattered energies, and energies measured by the detector were than integrated at each 0.2 degree step until the crystal was exposed to a fixed incident charge (in this case, 2×10^{-8}C). The channeling yield versus tilt angle can than be plotted. Yield is measured in counts where 1 count = 1 particle. If the surface channels, 8 planar dips associated with the 4 major and minor axes result. Plotting the measured polar location of the dips, and recognizing that if the [111] direction was perfectly aligned with the beam axis they should occur equispaced at 45 degrees apart, allows for the minor correction to the crystal orientation. Although minimum surface damage is expected when aligned for channeling with high energy, light particles, the beam location used for the final data collection was slightly translated from the location used for alignment.

To enable an accurate measure of lattice disorder at the surface of the ultraprecision machined crystals, the bulk surface quality was measured. An oriented specimen, taken from the same wafer as the prepared specimens, was etched to provide a damage free (111) surface. The ratio of the integrated minimum signal from the aligned spectra to that of the spectra obtained at the same energy for the randomly oriented crystal is known as the minimum yield, χ_{min}, and is used as a measure of crystal quality. χ_{min} can be calculated from first principles with knowledge of the lattice structure and the thermal vibrational amplitudes of the atoms. Fig. 3 shows a typical result obtained for the [111] aligned and random spectra of the Ge used in the study. The random spectra were obtained by measuring the backscattered energies while the crystal was continuously positioned with random orientations to the incident beam. The spectra were obtained by integrating the energies until the crystal had been exposed to a fixed incident charge (between 2-6 μC). Whereas the aligned spectra will vary for specimens of different crystal quality, the random spectra will be the same for materials with the same atomic composition. The crystal quality, as determined from the spectra in Fig. 3 resulted in a χ_{min} of 0.039. This is indicative of high crystalline quality, and is consistent with the theroretical value of χ_{min} of 0.036 for the [100] direction of Ge calculated by Feldman [7].

Fig. 4 shows the spectra obtained for the (111) oriented crystals. Shown are spectra for surfaces which have been (1) etched, (2) chemo-mechanically point diamond, (3) mechanically polished with

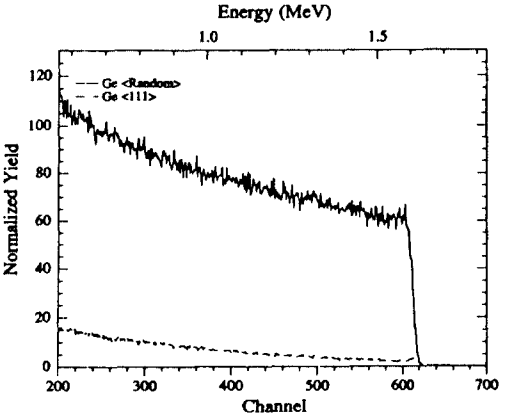

Fig. 3. <Random> and <111> aligned spectra obtained for an etched Ge crystal.

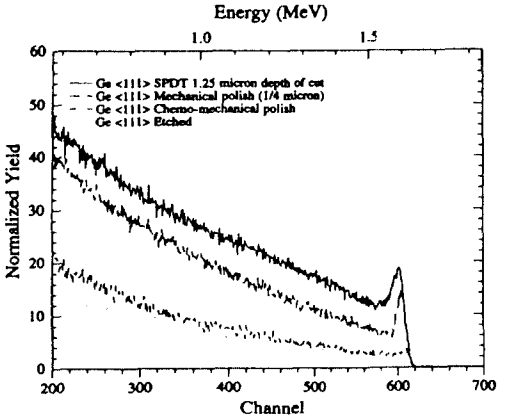

Fig. 4. Spectra obtained for various processing of <111> oriented Ge.

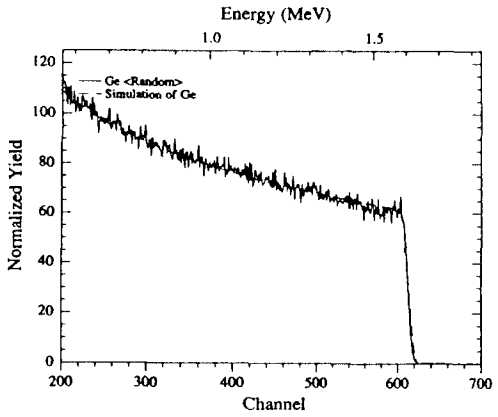

Fig. 5. Comparison of measured and calculated <random> spectra used for calibration check.

1/4 μm diamond abrasive, and (4) single polint diamond turned at a depth of cut of 1.25 μm. To characterize the depth of the disorder, energy loss in the near surface region is used. The energy loss of the backscattered particles is the sum of the energy lost on their inward and outward paths. The rate of energy loss dE/dx, or stopping power, is a function of the mass of the crystal atoms, and the mass and energy of the incident particles. Data for stopping power for a wide range of incident and target atoms has been compiled from extensive experimental studies [8]. Using this data, and known scattering cross sections and scattering geometries, one can accurately predict the results of RBS. The complexity of the calculations, however, has lead to the development of computer algorithms to perform the calulations. We have an algorithm developed by Doolittle [9] to calculate the thickness of an amorphous layer which would lead to the energry loss measured by the channeling experiments. This same algorithm was used to calculate the random spectra measured. Fig. 5 shows the calculated random spectra compared with that measured. To calibrate for the solid angle of the detector, incident beam energy, and charge collection efficiency, the calculated RBS spectra were fit to the measured curves. To arrive at the depth of the disordered layer, the simulation was to convert the measured backscattered Ge peaks into integrated equivalent amorphous layers (in units of Ge atoms/cm^2). The density of Ge implies that there are 4.41444×10^{22} Ge atoms/cm^3. This allows for a conversion of the equivalent amorphous layer data

Table 1. Summary of Ge channeling results

Process	Integrated Surface Peak 10^{17} Ge Atoms/cm^2	Effective Depth (nm)
Chemo-mechanical polishing of (111) crystal	~0.14	~2-3
Mechanical polishing with 0.25 μm diamond abrasive of (111) crystal	0.463	11
Ultraprecision diamond face cutting of (111) crystal	1.265	28
Orthogonal flycutting at depth of cut=20 nm (001) crystal in [010] direction	2.697	61

to a subsurface depth. Table 1 is a summary of the results obtained for the crystals. The depths listed in Table 1 are those for an amporphous layer which would result in the energy loss measured. If the actual surface layer was not entirely amorphous or contained some gradient of strain, the deepest location of any machining effect on the lattice would be larger. Whereas the depths listed in Table 1 do not necessarily correspond to the furthest location from the surface for which an atom was displaced from its original lattice site, the use of this approach, presents an unambiguous method of quantifying the level of lattice disorder. Channeling performed on the chemo-mechanically polished surface indicated a depth of lattice of 2-3 nm. Whereas it is clear that this surface possessed significantly less damage than the mechanically polished or diamond turned specimens, the present experiments are limited in accurate measure of damage depths at this scale. Grazing angle detector positioning coupled with surface chemical analysis (for detection of oxide layers) may be needed to further characterize the chemo-mechanically polished surfaces.

5. Conclusion

The subsurface damaged layer in ultraprecision machined single crystal Ge was examined by ion channeling. Single crystal Ge surfaces were pre-

pared by chemo-mechanical polishing, mechanical polishing with 1/4 μm diamond abrasive, single point diamond turning and ultraprecision orthogonal flycutting, and the extent of subsurface lattice disorder was measured. Minimum backscattered yield was as a measure of original crystal quality. It is clear that the chemo-mechanically polished surface possessed significantly less damage than the mechanically polished or diamond turned specimens, however the present experiments are limited in accurate measure of damage depths at this scale, and grazing angle detector positioning coupled with surface chemical analysis may be required.

Acknowledgments

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