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Cross-section transmission electron microscope observations of diamond-turned single-crystal Si surfaces
Phase transformations during microcutting tests on silicon

B. V. Tankikella and A. H. Somasekhar
Department of Materials Science and Engineering, North Carolina State University, Raleigh, North Carolina 27695-7907

A. T. Sowers and R. J. Nemanich
Department of Physics, North Carolina State University, Raleigh, North Carolina 27695-7907

R. O. Scattergood
Department of Materials Science and Engineering, North Carolina State University, Raleigh, North Carolina 27695-7907

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Controlled slow-speed microcutting tests were made on single crystal silicon. Micro-Raman spectroscopy confirmed the presence of amorphous silicon within the microcutting grooves as well as in the debris particles removed from the grooves. These results indicate that pressure-induced transformation to metallic silicon can occur during microcutting and the ductile metallic phase will facilitate the cutting process. Raman spectroscopy further indicated the presence of large residual tensile strains in some areas of the microcutting grooves. © 1996 American Institute of Physics.

Silicon and germanium will undergo plastic flow without fracture under hardness indenters and in other situations where the contact loading generates large hydrostatic pressures. The plastic flow mechanisms in these situations will be facilitated by the pressure-induced transformation of diamond cubic phase to a β-tin metallic phase. Gridneva et al. used in situ electrical resistivity measurements to confirm the presence of the metallic silicon phase under a loaded diamond indenter. Amorphous silicon has been observed using transmission electron microscopy (TEM) at hardness indentations and scratches after the indenter is unloaded. It was concluded that the amorphous phase exists because the pressure-induced metallic phase does not transform back to the diamond cubic structure upon unloading, but instead is “trapped” as an amorphous phase. TEM studies have also revealed the presence of the amorphous phase after precision machining using diamond cutting tools. Since the heat generated at the tool–workpiece interface during precision machining may be sufficient to locally melt silicon, there is the possibility that amorphous silicon phase produced during machining results from rapid cooling of the molten phase and not from the pressure-induced metallic phase. Hydrostatic pressure during machining would still facilitate the process because of pressure-induced reduction of the melting point. The work reported here involved controlled microcutting tests on silicon using a diamond tool. The tests are similar to precision machining tests (typically done at cutting speeds of 5–10 m/s), but they were done at very slow speed of 5 m/s to eliminate the possibility of local melting. Raman spectroscopy was used for characterizing the microcutting grooves since it can detect the presence of amorphous silicon as well as any residual stresses that may be present. The micro-Raman technique was used because the focused laser spot size has the same spatial scale as the microcutting grooves.

Device grade single crystal p-type (100) silicon was used as the test material. A system consisting of a piezoelectric transducer (PZT) actuator, motorized state, and an acoustic emission sensor was developed for microcutting tests and this has been described elsewhere. A freshly sharpened Vickers diamond was used as the cutting tool. For the results reported here, the diamond was oriented such that a [011] microcutting direction was parallel to a diagonal of the Vickers diamond. A load profile consisting of a load ramp, hold, and unload ramp was generated using the PZT actuator with feedback control. The cutting speed was 5 µm/s. Acoustic emission was used to detect the crack–initiation threshold. Below a threshold load the order of 0.05 N (depending upon test conditions), microcutting grooves in silicon were fully ductile with no fracture damage present. This was the regime of primary interest for the micro-Raman observations.

Micro-Raman spectroscopy was carried out at room temperature using an ISA U-1000 scanning monochromator equipped with an Olympus BH-2 optical microscope. The spectra were excited with the 514.5 nm line of an Argon–ion laser, which was focused to an apparent spot size of about 3–4 µm in diameter for these samples. The Raman spectra were taken in the 400–600 cm⁻¹ region, which includes the characteristic peaks normally associated with crystalline, microcrystalline, and amorphous silicon. A spectral resolution of ≈4 cm⁻¹ was employed, and the laser power at the sample was ≈5 mW. The contribution of spot size and (low) power was chosen to optimize the signal while minimizing laser-beam heating.

Figure 1(a) shows a scanning electron microscopy (SEM) micrograph of a microcutting groove at a load of 0.04 N, which is below the crack–initiation threshold of 0.05 N for the test conditions used here. Detailed SEM observations showed no evidence of fracture along the entire groove. Figure 1(b) shows the same groove at a higher magnification. The cutting direction is from left to right in Fig. 1. The grooves in Fig. 1(b) show periodic features that have a spacing in the cutting direction of 0.25 µm. These features are not a result of mechanical resonance in the test system (chatter) and they are notably similar to the features observed in grooves cut into pure copper using a round-nosed diamond cutting tool. The latter were shown to cor-
respond to intermittent shear bands formed ahead of the cutting tool. That these same types of periodic features appear in silicon is consistent with the presence of a pressure-induced metallic silicon phase that can accommodate the intense shear processes required for this kind of ductile deformation mode. Increasing the cutting speed from 5 to about 200 μm/s did not change the spatial frequency of the features in silicon, as was also observed for cutting speed changes in copper. Significant amounts of cutting debris can be observed along the flanks of the microcutting grooves in Fig. 1(a). Similar debris was observed as an “extrusion ribbon” from low-load Vickers indentation sites as can be seen in Fig. 2 for a 0.15 N Vickers indentation (above the crack-initiation threshold for static indentation). The specific volume decrease associated with the pressure transformation from diamond cubic to metallic β-tin structure is 22% and, thus, the hardness indentation is accommodated by both conventional plasticity and the extrusion of the metallic silicon. The ribbonlike morphology evident in Fig. 2 is clearly indicative of the ductile nature of the metallic silicon phase.

The characteristic Raman peak for diamond cubic crystalline silicon is displayed in the micro-Raman spectrum shown in Fig. 3(a), which was made on virgin silicon crystal. Figure 3(b) shows a micro-Raman spectrum made with the beam focused within a microcutting groove like that shown in Fig. 1. These grooves are about 3 μm in width, so that a large fraction of the laser beam spot is within the groove. The spectra were taken from regions of the groove that did not contain cutting debris and, therefore, these results are representative of the structure of the cut surface immediately below the groove. The asymmetric background peak centered around 475 cm\(^{-1}\) indicates the presence of a substantial amount of amorphous silicon within the groove (quantitative analysis was not attempted because of unknown differences in optical absorption and other factors noted in Ref. 9). The sharp peak at approximately 520 cm\(^{-1}\) in Fig. 3(b) can be attributed to the crystalline silicon that is present along the flanks and underneath the groove. The maximum peak shift is less than two wave numbers below the standard (room temperature) silicon peak position at 521 cm\(^{-1}\). This peak shift can be used to estimate the maximum sample heating during the Raman scattering measurements. Previous results have shown that a 1 cm\(^{-1}\) peak shift corresponds to a 50 K temperature rise. Thus, the results indicate a maximum temperature of 100 K. Previous work has shown that temperatures of this order will not cause changes in the amorphous or crystalline phases.

An interesting “double-peak” feature was observed in the micro-Raman spectra taken from certain areas of the groove and also from some of the debris along the groove flanks. Figure 4(a) shows such a double-peak feature taken within a microcutting groove while Fig. 4(b) shows a double-peak feature obtained from a debris particle. The sharp peak at 520 cm\(^{-1}\) [Fig. 3(b)] is present in Figs. 4(a) and 4(b). The background asymmetry of the spectra further indicates the presence of the amorphous silicon phase [this is less evident in Fig. 4(b) because the higher count rate for this particular spectrum obscures the broad peak due to the amorphous silicon]. The second peak in the double-peak spectra in Fig. 4 is shifted downward to 510 cm\(^{-1}\), and it is broadened relative to the peak at 520 cm\(^{-1}\). This feature suggests
the presence of small domains of crystalline silicon formed within the parent amorphous phase by a recrystallization process. Assuming a purely hydrostatic strain state, a peak shift downward of ten wave numbers corresponds to a tensile transformation strain $\Delta V/V$ of 2.1% within the crystalline domains.\textsuperscript{18} Assuming further that the domains are spherical and that the crystalline and amorphous silicon phases are elastically isotropic with identical elastic constants, Eshelby’s solution for the transformation strains within embedded inclusions\textsuperscript{19} shows that $\Delta V/V$ should be one-half of the initial density difference between the crystalline and amorphous phases (stress-free strain). Since amorphous silicon is typically 5% less dense than the crystalline phase,\textsuperscript{20} the tensile strain value $\Delta V/V=2.1\%$ is in good agreement with the prediction.

It is important to note that the maximum temperature rise due to laser-beam heating is insufficient to recrystallize the amorphous phase. This implies that recrystallization must occur during the slow speed microcutting. Although the details of the recrystallization process cannot be established from the results presented here, adiabatic heating within the shear bands discussed in context with Fig. 1(b) could provide the required (thermal) driving force.

It is pertinent to compare the results obtained here with results obtained for precision machining of silicon and germanium. Puttick et al.\textsuperscript{8} recently reported the occurrence of amorphous silicon on silicon surfaces machined at a speed of about 5 m/s. Amorphous layers up to 100 nm in thickness on the machined surfaces were found using TEM and Rutherford backscattering. These authors remained uncertain as to the origin of the amorphous silicon. They suggested that it could be a result of the pressure-induced phase transformation to the $\beta$-tin structure or to localized melting of silicon under the frictional heating generated by machining. A rough estimate suggested that the combination of pressure-induced reduction of the melting point and frictional heating at the 5 m/s machining speed used could be sufficient to cause localized melting of silicon. The TEM results reported by Morris et al.\textsuperscript{3} showed mixed regions of crystalline domains and amorphous phase in the chips formed during precision diamond machining of germanium at 5 m/s machining speeds. The chips also possessed a lamellar structure indicative of an intermittent shear process.

In summary, the Raman spectroscopy results reported here show the presence of amorphous silicon within microcutting grooves machined on single crystal silicon surfaces and also in the cutting debris outside of the grooves. Since the machining speeds were very low, localized melting due to intrinsic heat generated at the tool–workpiece interface during machining is clearly not required for the production of amorphous silicon. The latter can be attributed to the presence of the pressure-induced metallic silicon phase that then reverts to an amorphous phase. Raman peak shifts also indicate the occurrence of a diamond cubic silicon phase under large residual tensile strains. This was attributed to the presence of recrystallized domains of the crystalline phase within the less dense amorphous phase. Although the mechanisms for the recrystallization process have not been verified, adiabatic heating within shear bands formed during machining could provide the necessary driving force.

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