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Diamond Atomic Layer Epitaxy

Eighth Quarterly Report

Contractor:	SI Diamond Technology, Inc. (formerly Schmidt Instruments) 2435 North Blvd. Houston, TX 77098
Contract:	N00014-92-C-0238
Effective Date:	1 October, 1992
Expiration Date:	31 March, 1994
Reporting Period:	1 July 1994 - 1 October 1994
Principal Investigators:	Mark S. Hammond and Keith D. Jamison, (713) 529-9040
Project Manager:	Marianne McGonigal
Sponsored By:	BMDO Innovative Science and Technology Office
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Work during the current reporting period has focused on the following items:

(1) Auger and ELS of deposition with methyl bromide on diamond.

(2) Studies of methyl bromide and ethylene reaction with silicon (100) and (111).

(3) Further studies of hydrogen on diamond(100) and surface reconstructions.

(4) Set-up and testing of load-lock chamber for hot filament CVD.

Auger and ELS of depositions with methyl bromide on diamond. Auger and ELS analysis was performed on a diamond(100) sample on which carbon was deposited with cracked methyl bromide. This analysis was carried out *ex situ* with a JEOL Auger microprobe which was recently acquired by SI Diamond Technology. Mask lines from the tantalum straps which were used to mount the crystal on the sample holder are apparent in electron microscope images of the sample. The carbon KLL Auger signal from the sample at about 272 eV is observed to have the lineshape which is characteristic of carbon in diamond (as opposed to graphitic) form. In addition, the $\pi\pi^*$ plasmon which has been observed at approximately 6.5 eV energy loss for graphitic carbon is not observed in electron energy loss spectroscopy (ELS) of this sample which was performed at 350 eV beam energy. Both of these results point to deposition of carbon in diamond form and are shown in Figure 1.

Studies of methyl bromide and ethylene reaction with silicon (100) and (111). Work has continued during this reporting period attempting to deposit carbon on silicon by ALE. A sputter gun was added to the chamber during the last reporting period and is now routinely being used to generate clean, reproducible surfaces for the depositions. Experiments looking into the deposition of cracked methyl bromide on silicon have continued. Unfortunately, exposure of silicon to cracked methyl bromide does not seem to deposit significant quantities of carbon on the surface. Analysis of the exposed surfaces with Direct Recoil Spectroscopy

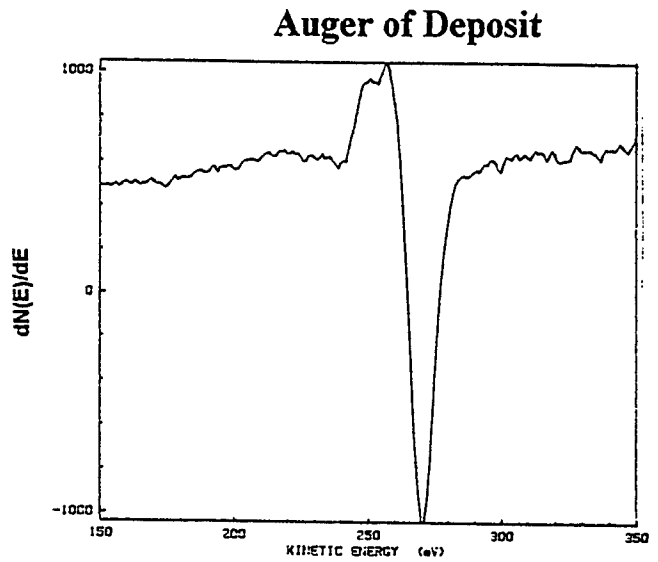
indicates that very little hydrogen and carbon are present on the surface, but a high coverage of bromine is present after exposure. This data is shown in Figure 2. Absolute bromine coverages can be estimated by a comparison of the peak areas for sodium scattered from silicon and sodium scattered from bromine in the DRS spectra. Estimated bromine coverages are approximately 45% of a monolayer on the (100) face and 70% of a monolayer on the (111) face of silicon, whereas bromine coverages observed in depositions on diamond are in the range of 15% of a monolayer. It appears that a large amount of bromine is produced from the cracking of the methyl bromide and that bromine has a very high sticking probability on the silicon. Thus the surface sites for adsorbing CH_x species are quickly blocked by bromine. In addition, bromine is known to etch silicon, so we believe that it will be necessary to initiate carbon depositions on silicon with some other carbon-containing compound.

Work to investigate whether ethylene might be used to begin carbon deposition on silicon is in progress. Ethylene has been reported to adsorb on silicon without activation in surface science studies carried out by others and has also been commonly used as a carbon source in the growth of silicon carbide. Therefore, we thought it might be a good candidate to start carbon depositions on silicon. Experiments we have carried out thus far seem to indicate that ethylene does indeed stick on silicon without activation even at room temperature and can be used to put a significant amount of carbon on the surface. DRS spectra showing the appearance of carbon on a silicon surface exposed to ethylene is shown in Figure 3. After exposure of the silicon to ethylene, no LEED pattern can be obtained of the surface indicating that the surface has become disordered. We have found that a LEED pattern can be restored by annealing of the surface or by exposing the ethylene covered surface to cracked methyl bromide. Annealing the surface has been shown by other workers to cause some diffusion of carbon into the bulk and we also see evidence of this in the DRS. Note the change in the carbon DRS signal after annealing in Figure 3. Exposure of the ethylene covered surface to cracked methyl bromide results in a bromine covered surface, but a fairly high surface temperature is also necessary to desorb the bromine. Thus it is not clear that all diffusion of carbon into the bulk can be prevented. We are continuing work to find ways of making ordered carbon layers on top of silicon and are also planning to try depositing on β-SiC.

Further studies of hydrogen on diamond(100) and surface reconstruction. A small amount of time during this reporting period was spent looking at surface preparation and reconstruction for the diamond(100) surface. Researchers at NRL have found that the smoothest (100) diamond surfaces they have been able to prepare were generated by treatment of the surface with a hydrogen plasma in an Astex microwave plasma system. These surfaces exhibit high quality (2x1)/(2x2) LEED patterns without annealing of the samples in vacuum. NRL prepared a diamond(100) sample for us in this manner and we have compared this sample with diamond(100) samples prepared by other methods in our lab. One diamond was prepared by exposing to a hydrogen plasma in an RF asher and another diamond was prepared by exposing to atomic hydrogen in a hot filament CVD chamber. Both the sample prepared at NRL with the microwave plasma and the sample prepared in the hot filament CVD chamber exhibited high quality (2x1)/(2x2) LEED patterns without annealing. The microwave sample had only hydrogen on the surface when examined with ion spectroscopies and the hot filament sample had hydrogen and some fluorine on the surface. The origin of the fluorine is not known. The RF asher sample was found to be covered with silicon and oxygen; it is suspected that these contaminants came from etching of the glass walls of the asher. Both the microwave sample and the hot filament sample seemed to have higher quality surfaces than the samples

prepared by our usual preparation method (polishing with diamond paste and acid cleaning), so the hot filament treatment will be used to prepare diamond(100) samples in the future. We would also like to see if the hot filament treatment is also useful for preparing good surfaces on the (111) and (110) planes of diamond.

Set-up and testing of load-lock chamber for hot filament CVD. In anticipation of growing thicker diamond layers on top of carbon layers deposited by ALE on silicon or other substrates, we have added the necessary equipment to perform hot filament CVD in our load-lock chamber. This will allow us to transfer samples from the ALE chamber into the CVD chamber for growths without breaking vacuum. The CVD chamber has been tested and the only modification which still needs to be made is that the sample heating arrangement needs to be improved so that we can grow at sample temperatures above 800°C.



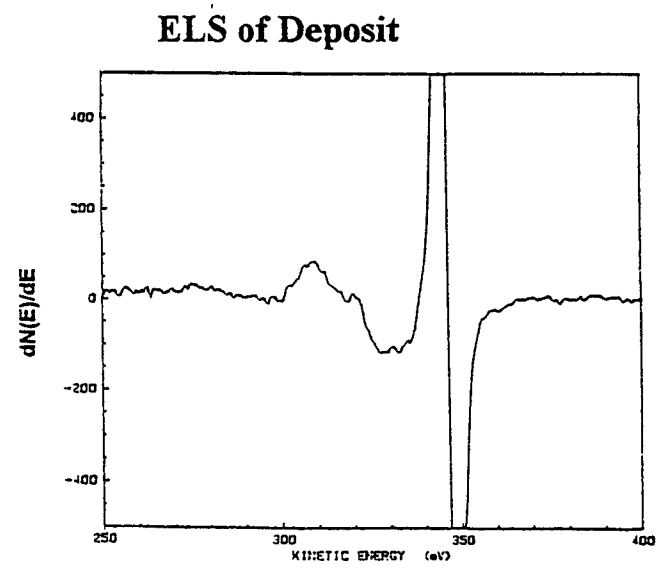


Figure 1. Auger and ELS spectra of carbon deposit on diamond(100) produced with cracked methyl bromide.

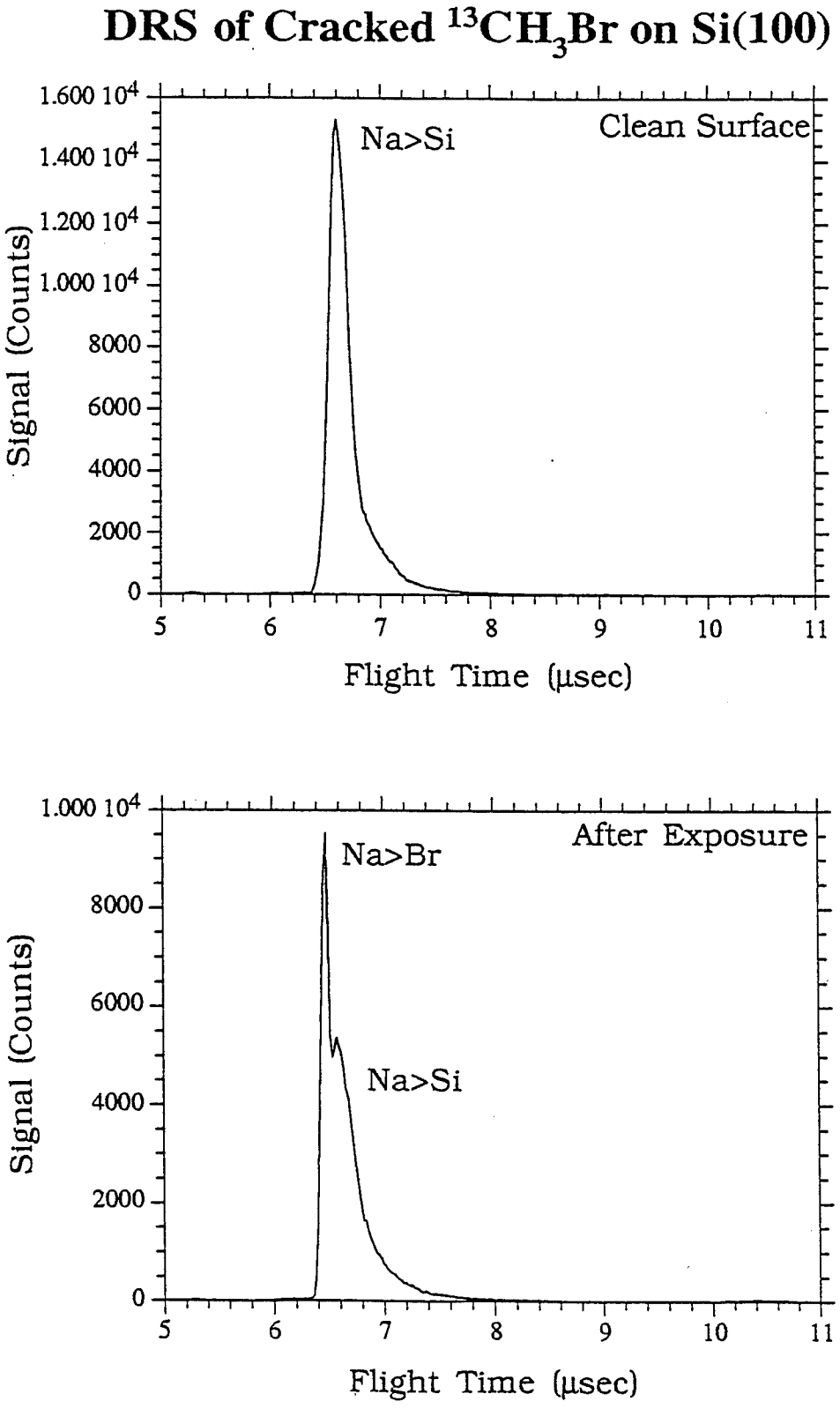


Figure 2. DRS spectra of silicon exposed to cracked methyl bromide.

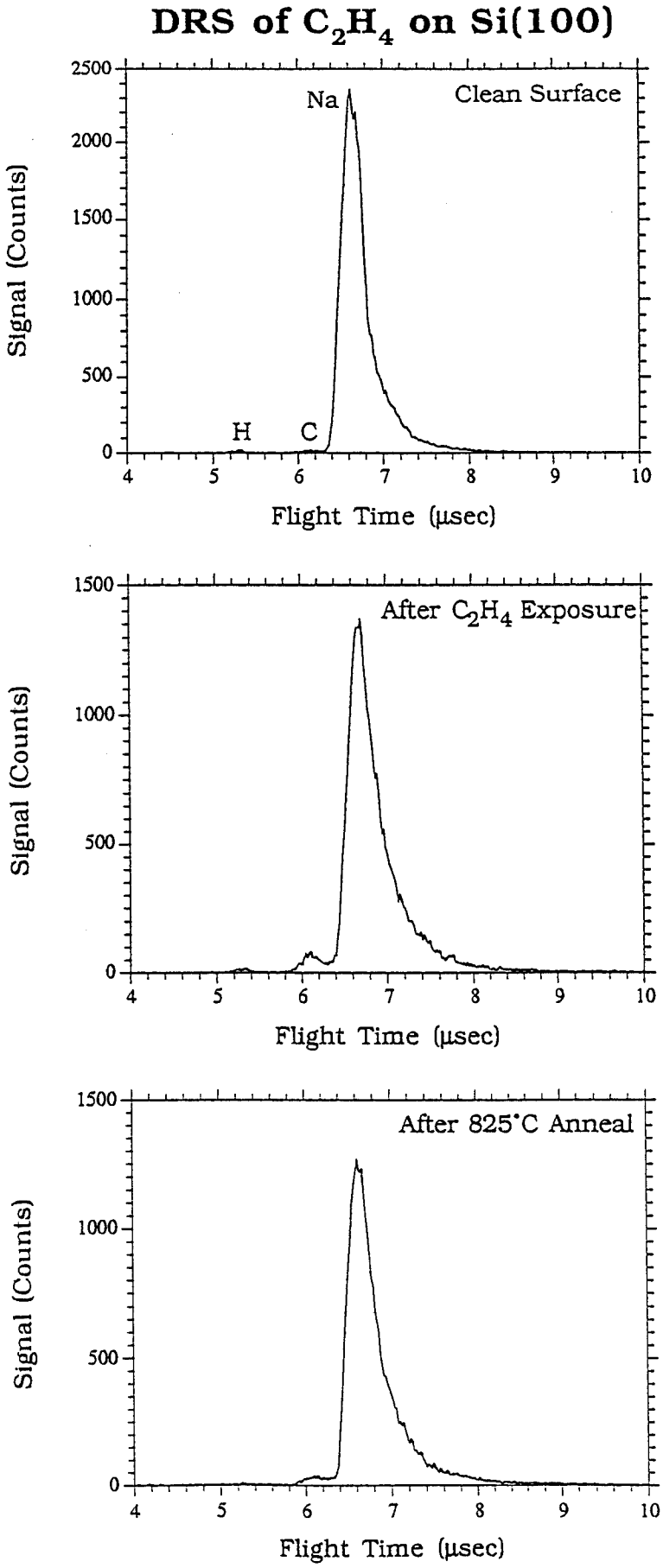


Figure 3. DRS of silicon exposed to ethylene. Top: before exposure. Middle: after exposure. Bottom: after annealing ethylene-exposed surface.