In-Situ Infrared (IR) Detection of the High Pressure Phase Transformation of Silicon during Scratching Test

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In this paper a new method of in-situ detection of the high pressure phase transformation of semiconductor materials during dead-load scratching is described. This method is based on the simple fact that single crystal silicon is transparent to IR light while metallic materials are not transparent to IR radiation. The sample material used here is silicon, but the same approach can be applied to germanium and other materials, such as ceramics (SiC), which have appropriate optical properties.

It has been established that silicon goes through a Si-I to Si-II (covalent diamond cubic to metallic) phase transformation under a pressure around 12Gpa, i.e. approximately the hardness of silicon. This Si-II phase is metallic, which is presumed to result in reduced transmission of radiation at IR wavelengths, i.e. metals are not transparent to IR radiation. In this paper experiments are described in which an IR laser-detector system is used in conjunction with a pre-loaded (dead weight) scratching device to detect the metallic high pressure phase of Si in-situ. Loads used are 10, 20, 30 and 40 mN. The apparatus is designed such that IR light passes through a diamond tip (diamond is transparent to the IR laser used), which is a 90 degree conical tip with nominal radius of 5μm, onto the interface/junction between the diamond tip and the silicon as the diamond tip is translated on the silicon wafer. The IR diode laser used in these experiments is an 8mw Infrared laser with a wavelength of 1330 nm. An Infrared detector is placed below the silicon wafer to sense the transmitted IR light. Results show a decreasing trend of IR transmittance after each scratch (multiple scratches are made at each load condition) and an average of ~10% decrease after 4 scratching cycles in the same groove. This indicates, in conjunction with previously reported results (Patten, 2003), that a metallic phase has been generated during scratching and the metallic phase has preferentially blocked the IR laser light. AFM and SEM have been used to measure the groove shape and to study the material generated in the pile-up (displaced) area. These images show that ductile deformation, for the loads investigated, correlate with the generation of the metallic silicon during simulated machining.

Key words: Metallic high pressure phase transformation Infrared Silicon Semiconductor

1. Introduction

High pressure phase transformations during machining of brittle materials have recently attracted the attention of a number of researchers [1]. Significant amounts of work and papers in this area and related fields (diamond anvil cell and nanoindentation) have been published especially in the last twenty years [8]. Much of this previous effort has been focused on investigating the series of phase transformations that occur during the processing of ceramic materials and semiconductors. In the case of silicon, the sample material in this paper, over 12 different phases generated upon loading and unloading in indentation tests of silicon have been identified [2]. Nanoindentation tests are by far the most widely used experiments in this field [3]. Papers on nanoindentation mainly evaluate the effect of different loading and unloading situations (load and depth, and rate effects), indenter geometric shape, stress-strain effects, and thermal effects under pressure. Direct detection of these high pressure phases is generally not available except for diamond anvil cell experiments [4] .Most published work, to our knowledge, suggesting a high pressure metallic phase transformation rely on post process evaluation techniques such as Raman spectroscopy [5]. Direct evidence of a metallic phase transformation, the authors of this paper believe, has much more practical value from an industrial manufacturing point of view. Due to the nature of high pressure phase transformation in brittle materials, in-situ study of this phenomenon has been very difficult, primarily due to the small size scale (micrometers) and high pressures (GPa). Developing a new instrument or modifying a current instrument to make such an in-situ measurement would take considerable time and effort. In this paper a new in-situ approach to detect the metallic phase in silicon during scratching is described. The apparatus (a modified stylus profilometer) is an extension of previous work on in-situ electrical resistance heating of Si during scratching [6]. Results give direct evidence of the existence of the high pressure metallic phase of silicon during scratching and this correlates well with previous reported and related work utilizing electrical detection [7.]

2. Infrared in-situ detection of metallic high pressure phase transformation in Silicon

The previously developed nanoscratching setup is augmented with a laser source and detection system[2]. The laser beam is coupled to a fiber optic cable and then passes through a diamond tip, which is attached to the diode laser's fiber ferrule. The whole system can be easily built but the phenomenon behind this simple experiment might inspire some new thoughts and novel applications.

2.1 Experimental design

The experimental setup is shown in Fig-1. The main part of the system is a profilometer with precision tracing driver (Surfcom110B). The scribing length and speed are controlled by the tracing driver. A dead weight-load is added onto the stylus that holds the ferrule. The load direction is perpendicular to the wafer's surface. The silicon wafer is a p type (100) 4 inch diameter wafer with a thickness of 475~575 micrometer. A 90 degree cone diamond tip (5µm nominal radius) mounted on the ferrule end is used as the scratching tool, see Fig-2. A low power laser (1310 nm wavelength, 8mW power) and corresponding detector (INGAAS DETECTOR 3000MICRON DIA, 900-1700nm) are used as the illumination and detection devices respectively. The diamond tip is gold coated to minimize stray radiation from being emitted from the ferrule end and diamond stylus. Fig-3 and 4 are the SEM images of the gold coated diamond tip before and after scratching test. The area which has the gold removed is about 4 um in diameter. This small area represents the aperture of the illumination system; i.e. the IR radiation mostly emerges from this small area. The small hole ~ 4 micrometer in diameter is visible at the tip of the diamond; i.e. on the radius of the diamond tip. This small hole also represents the area of contact between the diamond tip and the silicon wafer, i.e. the gold coating is removed due to the contact between the diamond tip and the silicon wafer.

2.2 Test of laser signal

The laser output power was calibrated using an IQ-1200 power meter. The diamond tip is attached to the fiber ferrule using epoxy. A thin layer of gold, 2000 Angstrom thick, is vacuum coated on the diamond tip, which is used to converge or concentrate (guide) the laser beam to the contact area between the tip and the wafer. The coating, and small aperture size, brings the laser power down to several micro watts (from the peak of 8mW). The epoxy may also attenuate some of the power of the laser beam. The beam size of the laser, from the fiber, is about 10 µm. The detector reading versus laser power measured on the power meter has been calibrated and shown in Chart-1. The laser runs at 18mA driving current during scratching.

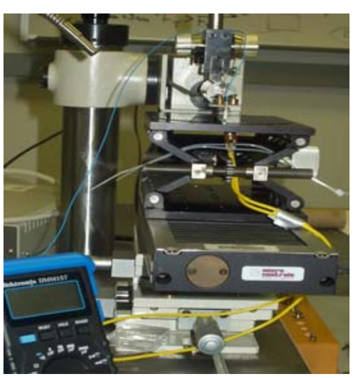

Fig-1 Experimental setup

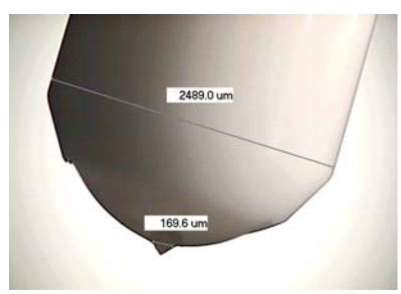

Fig-2 Diamond tip attached on the ferrule

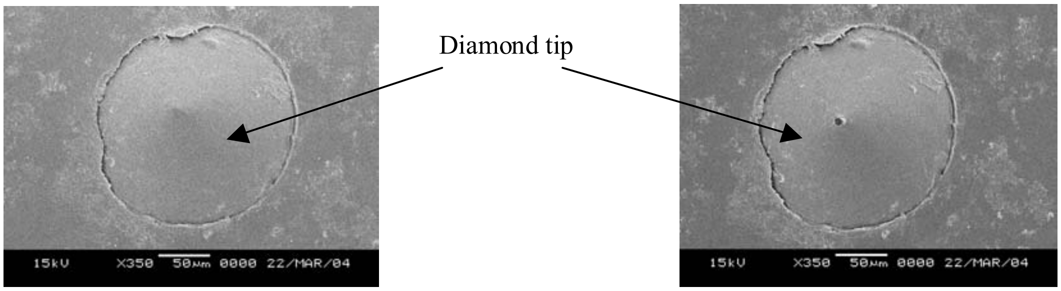

Fig-3 Gold coated tip before scratching test

Fig-4 Gold coated tip after scratching test

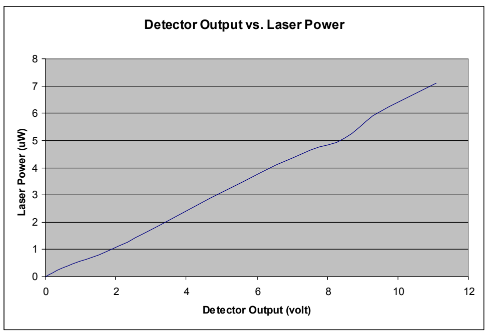

Chart-1 Detector Output vs. Laser Power

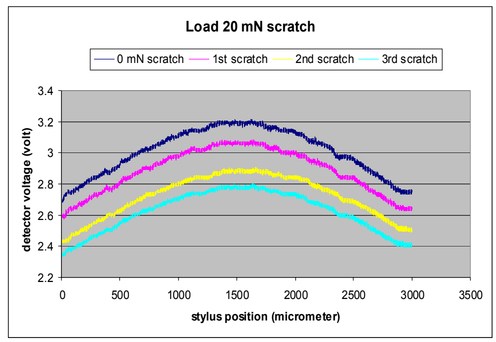

Chart-2 Load 20mN Scratch

2.3 Experimental procedures and results

The Infrared detection experiment is carried out as shown in Fig-5. A pre-determined length of scratch (~4mm) is made across the silicon wafer. This scratch coincides with the diameter of the IR detector beneath the wafer. The sensing area of the detector is 3000 Micrometers in diameter. The diamond tip starts scratching from one side of the detector, going across the full range of the detector and stops on the other end of the detector. The detector, which is fixed below the wafer, measures the Infrared laser signal coming through (transmitted) the silicon wafer. This signal is collected using Labview software. Different weights from 20 mN to 50mN have been tried. Scratching direction is [110].

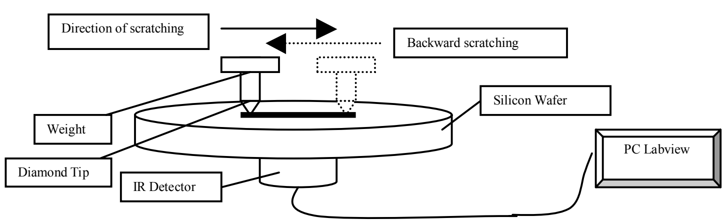

Fig-5 Schematic of scratching procedure

Chart-2 shows a series of 4 scratches at 20mN load; the top curve represents zero weight scratching. This scratch is made when the tip touches the silicon wafer and transverses across, just as one would do in a surface roughness test. The zero weight represents the nominal preload of the surface profilometer or about 0.7 mN. The following three curves represent scratches that are done in the same track on top of the zero weight scratch. For the various loads of 20mN, 30mN, 40mN, 50mN (only the results for the 20mN scratch are presented), the voltage (IR transmitted signal) decreases after each scratch and overall there is about 10% to 15% signal decrease after the 3rd scratch, compared to the zero weight scratch.

3 Discussion and conclusion

The experimental results agree with our premise that a layer of metallic silicon phase, which is generated under high pressure (12~16GPa), blocks some of the infrared light. If there is no metallic phase, then the infrared light would pass through the crystal structure of silicon (covalent or semiconducting silicon) and there should be no signal change detected. This (detector) voltage decrease is direct evidence of the existence of high pressure phase transition (semiconductor to metal) in silicon. This also agrees with earlier work on electrical heating of the transformed metallic phase [4] .

As the loaded diamond tip scratches the silicon wafer, a layer of the high pressure metallic phase, about the thickness of the groove depth, is generated underneath the diamond tip. This metallic phase only exists during the contact, i.e. as long as the high pressure is present. As soon as the tip moves on, the material behind the diamond tip back transforms to an amorphous structure. When the next scratch occurs, the high pressure phase is reformed under the diamond tip. As the groove gets deeper and wider, the metallic zone also grows in extent (scaling with the groove size) resulting in greater attenuation of the IR laser beam. The decreasing detector signal voltage, with subsequent scratches, matches this theory. If this were not the case, the IR laser transmitted signal would actually increase with subsequent scratches as more of the gold coating would be removed (corresponding to the larger groove dimensions). This larger aperture would allow corresponding more of the IR laser to emerge from the diamond tip. The fact that the IR signal decreases, in light of the above, clearly demonstrates the existence of the increasing size of the metallic high pressure phase of silicon under the diamond stylus tip. One of the potential applications of this experimental result is to develop a new concept of semiconductor material machining, which involves preferentially heating and softening (with an IR laser) the high pressure metallic zone to achieve tool wear reduction as well as enhanced ductile machining. It has been shown experimentally that at temperature above 0.3 to 0.4 T_{ml}, the microhardness drops abruptly with increasing temperature[5]. Temperatures in the range of 400 to 600 degrees C would substantially soften silicon and promote augmented ductile machining with potentially drastically reduced cutting forces and less tool wear.

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