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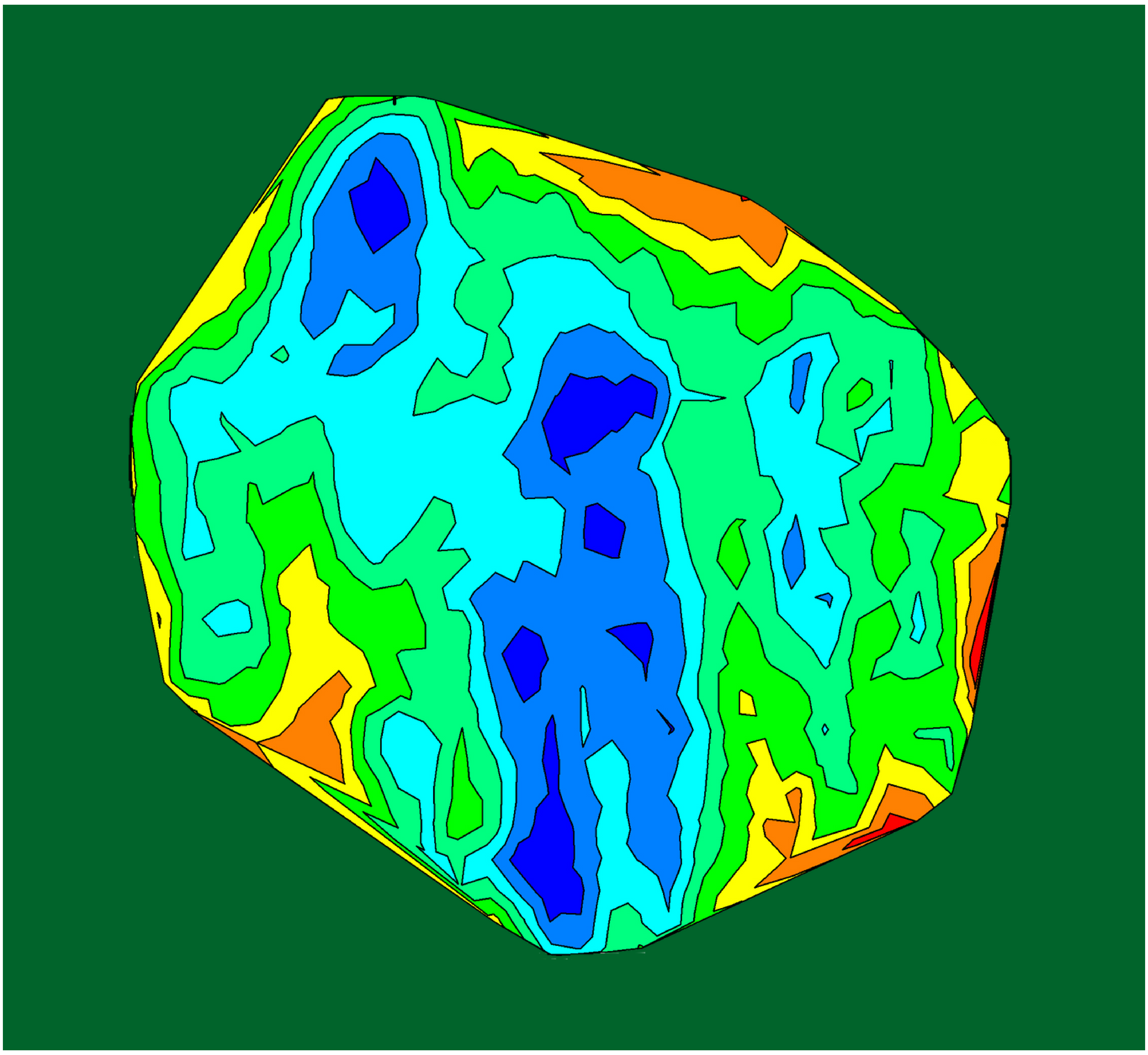

Department of Physics, Prithvi Narayan Campus, Pokhara
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Himalayan Physics

An Experimental Study On Irradiated Interface Of Silicon

Research Article

Min Raj Lamsal[1] *

Department of Physics, Prithvi Narayan Campus, Tribhuvan University, Pokhara

Abstract: Atomic Force Microscopic (AFM) studies of Mega electron-volt (MeV) ions irradiated silicon surface morphology has been studied to a fluence of 5×10^8 ions/cm^2. Interesting features of cracks of 50 nm in depth and 100 nm in width have been observed on the irradiated surface. The features seemed to have been caused by the irradiation-induced stress in the irradiated regions of the target surface. The observed feature of crack seems to be mainly due to the high electronic energy loss of the irradiated ions on the surface that induces the stress in it. It confirms that the coarseness of the microstructure of a material directly affect the mechanical properties.

Keywords: Irradiated silicon • AFM • Pull-off force • Silicon-on- insulator (SOI)

1. Introduction

Atomic Force Microscope (AFM) is one of the prime instruments by which atoms and molecules can move around in a controlled fashion so that atomic manipulation is possible. It can visualize and measure surfaces in 3-D with high resolution. Spectroscopy is an AFM based technique to measure, and sometime control the polarity and strength of the interaction between the AFM tip and the sample. Although the tip-sample interaction can be studied in terms of the tip-sample force and thus force spectroscopy. In force spectroscopy, the cantilever-tip assembly acts as a force sensor. Hence, improvements to AFM probe and scanning methods, applications including critical dimension measurement on small structure characterization as well as nanolithographic facility are its potentials.

Keeping all these in mind, present work is associated with nanofeatures present at the surface/interface, taking silicon crystal as the sample in a direction to reform failure of the detection or sensor instruments using functionalized AFM tip and its force spectroscopy. So, problem addressed in this work are:

- role of widespread defect in substrate-crystal, affects in device functioning.
- extend our knowledge about unusual properties behaving at nanointerface.

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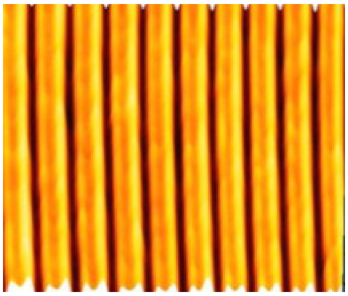

Figure 1. AFM image of generated nano-pattern of silicon structure

Measurement of materials property at an interface with structures in nanometer range is now emerging as an interesting topic both for basic and applied research. It is found that the interaction of high energy, mega electron volt (MeV) ions with a single crystalline target material such as silicon, in such a manner that following a Coulomb explosion mechanism and shockwave formation, there is localization of vibrational energy [1, 2]. The imparted energy is transferred through the single crystalline lattice such that the chains of atoms form facile pathways aiding such energy transfer. The energy is deposited in predetermined regions of the crystalline lattice to form irradiation interfaces. These strained regions are of immense interest for the formation of artificial strained structures.

Investigation of these findings as the presence of irradiated interface on silicon surface is of main concern. As the stress applied by MeV is directly proportional to strain produced and hence the mechanical deformation would take place at irradiated interface region. Such finding motivates our attention whether one can develop a quantitative technique by which one can understand presence of anisotropy and quantitative magnitude of strain that appears at irradiation interface.

Mechanical properties of materials can be studied through analysis of force-distance (F-D) curve generated with an Atomic Force Microscope (AFM). AFM force-distance curves are obtained by monitoring the cantilever deflection as a function of the vertical displacement of the piezoelectric scanner. Laser based optical detection system have been used for cantilever deflection measurement in our system. By force-spectroscopy one can systematically determine the dependence of the interaction on probe-sample distance.

The variation of the F-D spectrum in AFM is such that the far right side of the curve is defined to be where the scanner is fully retracted at which the force experienced by probe is null. As the probe approaches the sample surface, the force is increased in contact region [3]. In addition to this, far below the null position there is a point in F-D curve where probe retracts from the sample surface is snap-off point. We address these two cases as minimum loaded and heavily loaded conditions of the cantilever respectively. The difference between the minimum and maximum loading condition of the cantilever is pull-off or adhesion force [4]. In other words, at some critical distance the tip feels an attractive force from the surface and falls quickly towards the surface until contact [5] and sample motion bends the tip backwards. When the sample is retracted, the tip continues to touch the sample, even in post equilibrium position. The tip will adhere until the force extracted by the cantilever is enough to break the adhesion. The cantilever then springs back to the resting position. The difference in the minimum of retracting curve and the tip resting position is known as adhesion force. This situation reserves the potentiality to assess the surface energy of the region of the sample surface under consideration.

In this work, variation of pull-off force as measured by AFM has been presented in 2D plot summarized the features that are present at interface. Present work aims to provide better understanding of unusual variation of pull-off or adhesion and hence the nature of binding interaction that exists in irradiated interface. The pull-off force distribution in 2D plot has been analyzed across the length and width of the irradiated interface. Moreover, surface condition of the irradiated interface has been analyzed with the help of AFM images as well as corresponding plot of pull-off force. In brief, in this work, effort has been made to use force spectroscopy as a tool for detection and characterization of material inhomogenities present at interface.

2. Methodology

This is a laboratory based experimental work which starts with etching technology. Etching was done at nano-lab PN Campus, Pokhara. Further, formation of interface on silicon sample was performed and finally characterization of the sample had been done. Single crystal silicon was taken as the sample. To make contamination free and defect free sample surface, etching technology was proceeded over it. Designing an interface on it, a nichrome wire of nano-dimension was stretched while passing radiation coming from paletron. Then sample was ready for AFM examination.

Further, our region of interest was on the silicon surface where lack of irradiation through MeV ion energy loss would produce an irradiation interface, simply by creating a hot region (or region under irradiation) on one side and a cold region next to it. Location of the lattice site at cold region was spatially separated from the region of ion impact such that even induced MeV could not affect the protected cold region on the sample surface. A spatial filtering made by square grid composed of Ni wire having square cross section with 40 μm edge length, at a spacing of 850 μm placed between 5 mm to 20 cm in front of the sample silicon. Choosing a wire here is quite important. Wire was chosen to be of thickness of approximately double the range of the MeV employed. This prevents any possibility of forward sputtering from the nickel grid into the silicon (100) crystal would remove. Time of flight (TOF) secondary mass spectroscopic (SMS) analysis was carried out on the sample before and after irradiation. After irradiation the surface of the crystal was treated with dash etchant to a depth of about 50nm to remove sample contamination or damage.

Sample mounted on a piezoelectric tube, is driven towards the tip under computer control to take images. The motion of the tip is monitored as a function of sample displacement. AFM images in contact mode were recorded with Thermo Microscopes Autoprobe Electronics Module Model No. APEM 1000, CA USA. Before taking data on spectroscopy mode, cantilever probe was fully calibrated as per its force constant. Using spectroscopy mode of Proscan Data Acquisition software, 20 different locations were chosen at the interface of the AFM images on irradiation interface. And finally 16 data points belonging to F − D response were recorded for each location.

Employing an anisotropic KOH etching, in p-type mirror polished silicon chip with resistivity 4 $\Omega - \text{cm}$, produce a 27 μm thick silicon sample prepared and mounted on a specially designed pressure cell. Argon gas was used to pressurize the cell, which retains the gas pressure with the help of a self-activating valve. For the experiment reported here, the cell was pressurized to 3 bars. The pressurized cell is mounted on the AFM scanner to perform scans. The pull-off force reported here was recorded by symmetrically moving the sample on the X-Y stage of the head of the AFM scanner. The force constant of the cantilever employed is 0.26 N/m. The instrument was automatically programmed to collect force distance curves (from which the pull-off force data was retrieved) from 16 points along a line.

X-ray Diffractometers (XRD) measurements were performed by the Philips X'pert, Holland with Cu-Ka radiation for 45 KV of anode voltage and 40 mA current at each stage of the processing. X-rays of wavelength 15.4 nm were chosen for all XRD measurements. Pressure cell with extended silicon membrane (i.e., pressurized) was placed on sample holder with some special arrangements. Keeping in mind that sample plane is horizontally adjusted such that there is no any disturbance for incoming X-rays and detector during the measurements.

3. Results and Discussion

Microstructural investigations are useful as they are capable of direct resolution of the defect structure. AFM reveals about the structure of the defected solids. Except in ultrahigh vacuum, the surfaces of the AFM tip and the sample are always covered, either partially or completely, with an adsorbed layer of molecules from the environment. Therefore, the interaction between the tip and the sample surface may be mediated through these adsorbed species. On the other hand, often the tip and the sample surface are intentionally exposed to chemical by the design of the experiment. In these cases, the tip (or the sample) is said to be functionalized. It is generally believed that the displacement spike of MeV irradiation may involve a region of the crystal containing as many as 10^6 atoms sites, at the centre of which are lattice vacancies with the interstitials being closer to the periphery of the spike [6]. Owing to the thermal motion of the atoms many vacancy interstitials pairs spontaneously recombine, thereby reducing the amount of damage in the region of the displacement spike.

Microscopic studies have shown the increase of defects such as dislocation of loops and their tangling appear. These dislocations are displaced in specific regions, due to the process of vacancies and interstitials, which increases their cumulation. Thus, different kinds of defects (dislocations and interstitials) and their complexes appeared during irradiation. The strongly damaged regions were imaged in the microscope as black (B) islands and the less damaged regions were imaged as white (W) islands. For all fluencies the regions W with micro-hardness smaller than in non-irradiated silicon were observed. Micro-hardness is larger in the regions where the concentration of dislocation loops is high. The W, regions have a small number of the dislocation loops, and single punctual defects were seen using AFM. The dislocation loops are placed in specific B regions, which increase in size with the increase of fluence due to a process of vacancies and interstitials accumulation. Using an AFM it is found that the W regions contain smaller number of dislocation loops and a large amount of single point defects with their cumulations.

In Figure 3, it is clearly seen that the surface has cracked in three directions, while in Figure 4 the same feature have been clearly viewed (for the smaller scan size). The depth and height of the feature have been measured using the software available with the AFM and they were found to be of 47 nm and 103 nm respectively. The observed feature seems due to the irradiation-induced stress in the target material. The electronic flux of MeV Si ions in silicon was found to be of 2.43 MeV/micron, while the nuclear flux is 1.94 KeV/micron. Since the electronic energy loss is maximum at the surface and the observed feature has the depth of 50 nm only, the observed features seemed mainly due to electronic energy loss of the incident ions. The energy loss in the near-surface region seems to be high enough to lead the various effects in the irradiated region. It has been shown that [7] once amorphization takes place; the in-plane stress and strain begins to decrease. The decrease of the in-plane strain may results in the flow of silicon atoms out of the target plane in the irradiated region. When amorphous materials are subjected to swift heavy ion beam irradiation, they behave like a viscoelastic fluid. Moreover, in swift heavy ion irradiation, there is an occurrence of thermal spike [8] . This thermal spike leads to high local heating, which also leads to the observable effect. It has also been shown that the final state of material within a thermal spike is more disordered and thus strained with respect to the initial state. This strain gives rise to a stress even if the sample was originally stress-free, because of the constraints imposed by the un-irradiated surrounding matrix.

As a result, an inclusion produced in the bulk is compressed to a size between its original volume and a stress-free transformation volume. The shape of the inclusion determines the anisotropy in the components of the constrained strain and that of elastic strain thus produced. In the case of a spherical inclusion, the stress-free and the constrained strain are hydrostatic in nature. Thus the deformation caused by the resulting elastic strain and stress, which are hydrostatic as well, are identical in all directions. However, for the ellipsoidal or cylindrical shape of the inclusion, the constrained strain and the elastic strain and stress produced in the inclusions have shear components too. If the length of an inclusion is much larger than its diameter, i.e., $l >> d$, these shear components lead to the growth in the transversal directions but almost negligible change in the longitudinal directions. The complete process thus leads to a microscopically observed anisotropic growth effect. During the irradiation there are four different effects in which each manifest itself by characteristic changes in the mechanical stress state of the materials i.e., densification, stress relaxation by radiation-enhanced plastic flow, anisotropic expansion and stress generation and transient stress relaxation. These phenomena can cause the stress-induced cracks on the surface due to swift heavy ion irradiation. Moreover, the maximum momentum transfer, due to same atomic size of the incident ions and host material, leads to knockout (sputtering) of the silicon atoms towards the surface that may result in more stress on the surface. Stress induced cracks with a depth of 50nm and width of 100nm are found, however, the mechanism of the formation of cracks is not yet clearly understood.

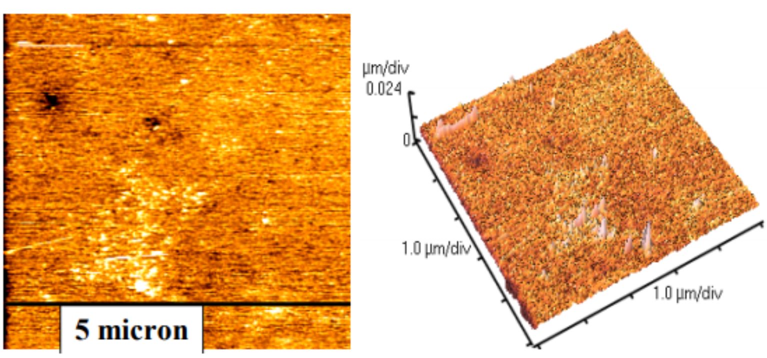

Figure 2. AFM micrographs of an un-irradiated p-silicon surface, which seems to be smooth and featureless.

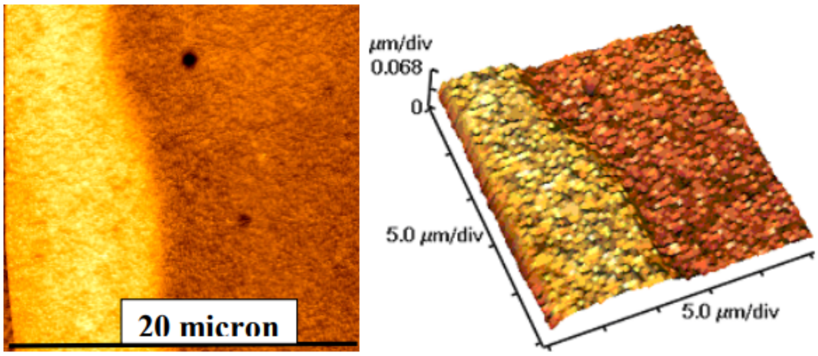

Figure 3. AFM micrographs of p- Silicon surface irradiated with MeV ions to a fluence of 10^{11} ions cm^{-2}.

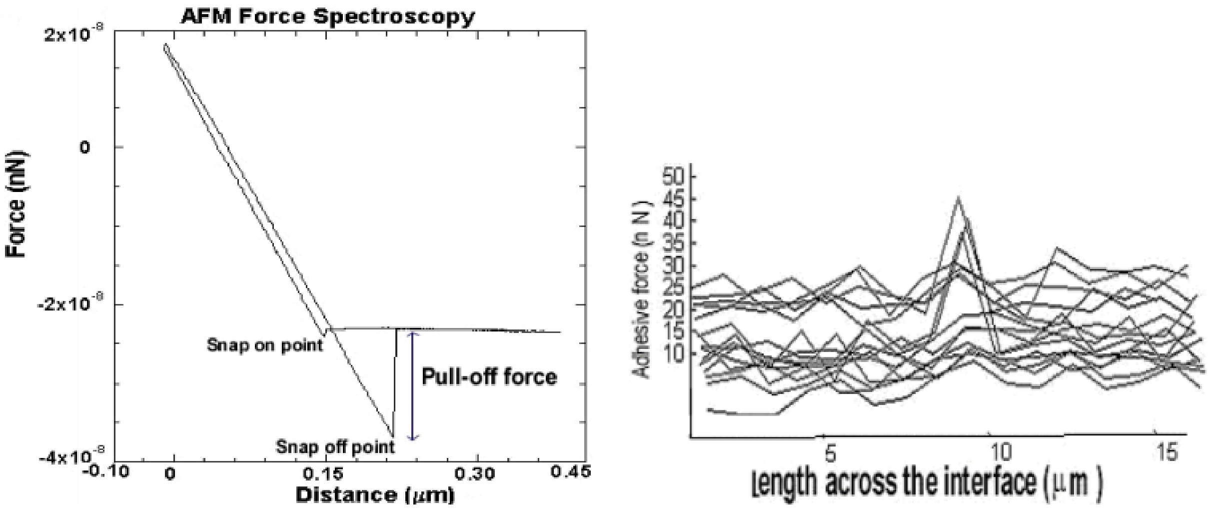

Figure 4. A typical loading curve is shown. The adhesion of the tip is extracted by the loading curve.

AFM has made its mark on wide varieties of applications, not only as a topographic mapping tool but also as a tool for studying surface interactions by means of force-distance curves, which has been emerged since 1989. Force curves (force versus distance curve) typically relate to the deflection of the free end of the AFM cantilever, as its fixed end is brought vertically towards (approach) and then away from the sample (retraction) surface. This causes the scanner to expand and contract in the vertical direction generating relative motion between the cantilever and the sample as per atomic interaction between them. In this way, AFM generates and records the force felt by the cantilever, as the probe tip is brought close to and even indent into the sample surface and then pulled away. The force required to separate the two surfaces (the tip and the sample) is adhesive or pull-off force. Force curve [9] can be used to measure the pull-off or adhesion force. The adhesion force [10] depends on the nature of the binding interaction between the tip and surface during the contact. Adhesion maximum [11] occurs where the tip has a larger contact area with the surface, while the adhesion minima are formed where the tip has the smaller contact area with surface. While moving a line across the interface from un-irradiated to irradiated part of the sample, we found that at the surface there is sharp increase in the adhesion force or there is adhesion maxima at the interface. We believe that this increase in adhesion force at the interface is due to compressive forces. These compressive forces [12] have increased the force constant. The increased force constant brought atoms closer at the interface i.e. intra-atomic spacing has decreased at interface due to increase in contact area between the tip and surface. So, adhesion force increases at the interface.

4. Conclusions

As we know properties response to a gradient is sensitive to the geometry of the microstructure and is the choice of path for energy transport. Generally, the behavior of phase depends on the characteristics of the adjacent grains and phase. The weighing procedures necessarily vary with the shape and distribution of the planes. Here we are having two phase microstructures. Results of AFM force spectroscopic study of radiation defects in silicon layers, Si/SiO2 interface trap, along with oxide formation under irradiation with MeV in silicon-on-insulator (SOI) structures are presented and discussed. SOI was fabricated by the wafer bonding. It is found that electron irradiation leads to transformation of energy-spectrum of the interface traps (relaxation of the bonded interface) in SOI. The main effect of high-energy Kr ion irradiation consists in formation of radiation defects in the top silicon layer as well as in the substrate.

5. Acknowledgements

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