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Contributed paper

CLEAVAGE OF SILICON SINGLE CRYSTAL SURFACE PRODUCED BY COMPRESSION PLASMA FLOW ACTION

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Abstract. Modification of silicon single crystal surface by the compression plasma flow (CPF) action is studied. It has been found that during single pulse surface treatment regular fracture features are obtained on the Si (111) and Si (100) surface in the target central part. Some of these regular structures can become free from the underlying bulk, formed as blocks ejected from the surface. These surface phenomena are results of specific conditions during CPF interaction with silicon surface.

1. INTRODUCTION

High-power pulsed energy streams interaction with material surfaces results in surface modification, as well as the material removing from surface in the form of vapor, liquid droplets, or solid flakes due to evaporation, sputtering, ablation, exfoliation etc. High temperatures and consequent thermal stresses, as well as mechanically strained surface during treatment, result in significant deformation and fracture of the layer, induced defects, cracking and exfoliation of the coating. Also, cracks are occurng being characteristic of a molten material which is resolidified very quickly. Surface and interface properties are very important for semiconductor devices and their engineering applications. In this experiment supersonic compression plasma flow (CPF) is used for silicon single crystal surface modification. In central part of treated silicon surface regular fracture features are obtained. It was found that some of these structures as blocks can be ejected from the surface. In the periphery part of silicon samples surface highly oriented periodic cylindrical shaped structures are obtained. Surface cleavage and exfoliation phenomena, as well as ripple structures formation, as the results of specific conditions during CPF interaction on silicon surface are, also, observed and studied.

2. EXPERIMENTAL SETUP

Si (100) and Si (111) surfaces of single crystal were treated with quasistationary CPF produced by magnetoplasma compressor (MPC). This quasistationary plasma accelerator (plasma gun) is described elsewhere [1-3], therefore only a few details are given here for the sake of completeness. The MPC consists of the specially designed electrode system [1]. Conically shaped cathode of MPC defines the profile of acceleration channel. Using nitrogen as working gas at 500 Pa pressures and 800 μ F, 4 kV capacitor bank, the obtained current maximum was up to 100 kA and time duration up to 150 μ s with current half period ~70 μ s. The continual ionization processes are taking part in working gas introduced in interelectrode region. The ionized gas (plasma) is steadily accelerated and permanently compressed.

The advantages of MPC, as compared to other types of plasma accelerators, are high stability of generated CPF and high plasma parameters (electron density up to $4 \cdot 10^{17}$ cm⁻³ and temperature up to 4 eV), as well as the CPF time duration (quasistationary stable phase is 40-50 µs) and large flow velocity (~40 km/s in nitrogen) sufficient for material surface modification. Beside that, the operation in the ion current transfer mode with the minimization of the electrodes erosion represents an additional and very important advantage of the quasistationary plasma accelerators in comparison with the classical ones. Magnetic flux conservation is a particular characteristic of CPF. During the action of CPF on a sample surface, due to CPF deceleration and frozen-in magnetic field, current loops (vortices) are formed.

For the studies of CPF interaction with silicon surfaces, commercial one-side polished n-type silicon wafers (100 and 111 orientation) 300 µm thick and 10 mm in diameter were used. The sample is mounted on the cylindrical brass holder of the same diameter, and placed in front of the MPC cathode at the distance of 5 cm. Silicon samples are exposed to a single plasma pulse. To investigate the morphology of treated silicon surface, optical microscopy (OM), scanning electron microscopy (SEM) and atomic force microscopy (AFM) were used.

3. RESULTS

OM micrographs of central part of the treated Si (111) and Si (100) surfaces are given in Fig. 1a and Fig. 1b, respectively. Rhombic and triangular regular fracture features are obtained in the case of Si (111) (Fig. 1a), as expected for threefold symmetry. On Si (100) surface treated by CPF, two sets of fracture lines intersecting at 90° form a grid that divides the surface into rectangular blocks (Fig. 1b). Length of a cleavage along crystal planes is up to 1 mm. It is worth to emphasize that, in the same conditions, the length of cleavage lines at Si (100)

surface is much larger than one at Si (111). SEM micrographs of the treated Si (100) surfaces are given in Fig. 2a. On the central part of the sample, some of the blocks are ejected from the surface, and large holes at the surface emerged. In this case development of subsurface fracture, parallel to the surface, is occured. Thickness of these blocks is about 10 μ m. A typical hole is shown in Fig. 2a. In order to determine cleavage plane, treated silicon surface is observed by atomic force microscopy. AFM micrographs of treated Si (100) surfaces are given in Fig. 2b. Surface profile indicates cleavage along crystal plane. Cleavage height in this figure is (1.3 ± 0.1) μ m. Estimated angle between Si (100) surfaces and cleavage plane is (54 ± 3)°. This indicates that cleavage plane is Si (111).



Fig 1. OM micrograph of silicon single crystal surface after CPF treatment: a) Si (111), image size $100 \times 100 \ \mu\text{m}^2$, b) Si (100), image size $50 \times 50 \ \mu\text{m}^2$.

The formation of observed surface features may be explained by energetic action of CPF on the surface (absorbed energy 10-15 J per pulse, flow power density $\sim 1 \cdot 10^5$ W/cm² [4]). Interaction of CPF with silicon sample surface causes the evaporation of a thin surface layer and formation of a shock-compressed plasma layer (plasma plume) [5]. Formation of this cloud of dense target plasma results in the shielding of a processed surface from a direct action of a CPF and surface plasma layer is about 1 cm. Using the high speed camera, time of interaction was estimated to be ~40 µs [5].

Energetic action of CPF causes the fast heating and melting of the surface layer and the presence of high dynamic pressure of CPF of the order of several atmospheres [4]. Namely, CPF kinetic energy thermalization causes the heating of target surface and high gradient of thermodynamic parameters occurred.

Formation of regular fracture features (Figs. 1,2) can be explained by considerable fraction of the absorbed plasma flow energy trapped into fractures rather than converted to heat energy [6]. Single crystal silicon is well known as a

typical anisotropic material and it is very brittle at room temperature [7]. Similar to silicon surface cleavage, obtained in this experiment, laser interactions with MgO single crystal caused regular fracture features [6]. Plastic deformation in MgO typically involves the growth of a relatively few dislocations that form extremely long, convoluted structures known as dislocation "multiplication". During laser interaction with MgO (111) surface triangular features are typical fracture patterns.



Fig. 2. a) SEM micrograph of a typical fracture features and holes on the treated Si (100) surface, image size $800 \times 800 \ \mu\text{m}^2$; b) AFM three-dimensional view of the Si (100) surface morphologies after CPF treatment.

The anisotropy of crack propagation direction from energetic point of view is explained with Griffith criterion [8]: in equilibrium, the mechanical energy released upon crack advance must be in balance with the energy required to create the two new surfaces. This is a necessary condition for fracture and leads to conclusion that crystal lattice planes with low surface energies are energetically favored as cleavage planes. The lowest energy cleavage planes in silicon are the {111} planes.

When a silicon single crystal is loaded to fracture, the cracks tend to initiate along a family of favoured crystallographic planes. In the case of plasma flow treatment of Si (100) surface, favoured cleavage planes are Si {111}, as are given in Fig. 2b. Angle between Si (100) surface and cleavage Si (111) plane is 54.74°.

Low adhesion between blocks and silicon bulk, and eventual ejection of blocks from the CPF treated surface (Fig. 2), can be explained by development of subsurface fracture, parallel to the surface. Cracking between the block and the bulk is growing due to local energy absorption.

4. CONCLUSION

Regular fracture features and exfoliations are observed on silicon single crystal surface treated by CPF. Surface modification is performed by fast heating of the surface in the presence of high dynamic pressure, thermodynamic parameters gradients and induced magnetic field from the CPF. During surface treatment and fast cooling phase, differential stresses in surface layer occurred. As the results of all of these processes rhombic and rectangular regular fracture features are obtained on the Si (111) and Si (100) surface, respectively. Some of these blocks are ejected from the surface.

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