Nuclear Inst. and Methods in Physics Research, B 413 (2017) 6–12

Contents lists available at ScienceDirect

Nuclear Inst. and Methods in Physics Research B

journal homepage: www.elsevier.com/locate/nimb

Fracture analysis of surface exfoliation on single crystal silicon irradiated by intense pulsed ion beam

Jie Shen[a,b,c], Ijaz Shahid[a,b,c], Xiao Yu[a,b,c,e], Jie Zhang[a,b,c], Haowen Zhong[a,b,c], Xiaojun Cui[a,b,c], Guoying Liang[a,b,c], Xiang Yu[a,b,c], Wanying Huang[a,b,c], Sha Yan[d], Gaolong Zhang[a,b,c], Xiaofu Zhang[a,b,c], Xiaoyun Le[a,b,c,*]

[a] School of Physics and Nuclear Energy Engineering, Beihang University, Beijing 100191, PR China
[b] International Research Center for Nuclei and Particles in Cosmos, Beihang University, Beijing 100191, PR China
[c] Beijing Key Laboratory of Advanced Nuclear Energy Materials and Physics, Beihang University, Beijing 100191, PR China
[d] Institute of Heavy Ion Physics, Peking University, Beijing 100871, PR China
[e] School of Space and Environment, Beihang University, Beijing 100191, PR China

ARTICLE INFO

Keywords:
Intense pulsed ion beam
Single crystal silicon
Finite element method
Exfoliation
J integral

ABSTRACT

Surface exfoliation was observed on single crystal silicon surface irradiated by Intense Pulsed Ion Beam (IPIB). As the strong transient thermal stress impact induced by IPIB was mainly attributed to the exfoliation, a micro scale model combined with thermal conduction and linear elastic fracture mechanics was built to analyze the thermal stress distribution along the energy deposition process. After computation with finite element method, J integral parameter was applied as the criterion for crack development. It was demonstrated that the exfoliation initiation calls for specific material, crack depth and IPIB parameter. The results are potentially valuable for beam/target selection and IPIB parameter optimization.

1. Introduction

During the past several decades, material surfaces and thin layers modification with intense pulsed ion beams (IPIB) has been a novel technique [1–3]. IPIB are energetic charged ion beams with high intensity (1–10^4 A/cm^2), short pulse duration (≤1 μs) and high instantaneous power density (10^6–10^{10} W/cm^2). Due to the fast energy deposition on the material surface, it results in fast temperature rise (up to 10^{10} K/s [4]), melt, vaporization, sublimation, ablation, re-solidification, recrystallization and shock wave. Therefore, IPIB has been proved promising in material surface enhancement [5], polishing [6], mixture [7], film deposition [8] and nanopowder synthesis [9].

However, after IPIB irradiation some microstructures including micro scale cracks [10], craters [11] and exfoliation [12,13] may be introduced. Among them, the exfoliation, as the result of interlaminar fracture initiation and propagation will reveal the material substrate and facilitate the erosion and secondary fracture because of the stress concentration. Even though sometimes interlaminar fractures may not cause exfoliation, their existence may significantly reduce the adhesive strength between the surface layer and substrate, thus affect the material performance. Therefore, it becomes important to understand the generation mechanism of interlaminar fracture.

To explain the crack generation, shock wave theory was applied [14] after it was theoretically predicted [15,16] and experimentally observed [17]. After IPIB energy is deposited into the surface layer rapidly, high-gradient temperature and stress field can be formed. Shock wave with pressure at several GPa is thus generated and plays a dominant role in sub-layer modification. An accurate theory later was proposed as a criterion of shock wave generation [18].

Nevertheless, shock wave will not necessarily lead to interlaminar fracture or exfoliation. Instead, shock wave sometimes facilitates the enhancement of microhardness [14,19] within the modified region. In that case, the interaction between the thermal shock wave and target material should be taken into consideration. It is of great necessity to develop an effective method, which can enable one to judge the possibility of the interlaminar fracture initiation in a specific combination of IPIB and target material before practical application.

The purpose of this work is to build such an accurate analytical model, which will be useful to better understand the mechanism of the surface crack and exfoliation. Eventually, optimization of IPIB parameter and rationalization of target material will enable one to avoid these undesirable microstructures. To specifically investigate the exfoliation mechanism, single crystal silicon was purposely chosen as the sample in both of experiment and simulation for the following two

* Corresponding author at: School of Physics and Nuclear Energy Engineering, Beihang University, Beijing 100191, PR China.
E-mail address: xyle@buaa.edu.cn (X. Le).

http://dx.doi.org/10.1016/j.nimb.2017.09.031
Received 1 August 2017; Received in revised form 26 September 2017; Accepted 26 September 2017

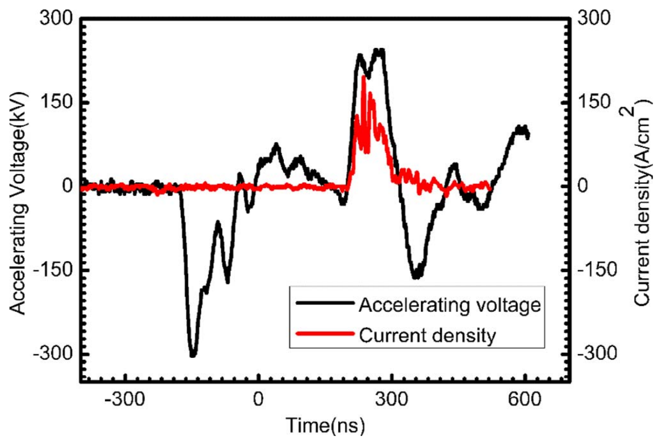

Fig. 1. Accelerating voltage and current density waveforms of IPIB from TIA-450.

reasons. On one hand, single crystal materials exclude various uncertainties, such as impurity, grain structure, grain boundary, surface roughness, etc., which may bring other possibilities to the beam-target interaction and make the stress release process complicated. On the other hand, silicon is a typical brittle material, which absorbs relatively little energy prior to fracture without plastic deformation. In this way, it can be beneficial to theoretical analysis if the energy can be efficiently used and the fracture occurrence can be maximized under IPIB irradiation.

2. Experiment

Specimens of high purity single crystal silicon (1 0 0) were mechanically polished and cut into pieces manually at dimension of $10 \times 10 \times 1\ mm^3$. Then they were cleaned ultrasonically with acetone. The irradiation experiment was carried out with TIA-450 accelerator at College of Materials Science, Shenyang Ligong University. Typical waveforms of IPIB parameters are demonstrated in Fig. 1. The beams from the accelerator consist of 70% C^{n+} and 30% H^+, and the ion energy, pulse duration (full width at half maximum) current density and maximum cross-sectional energy density are 250 keV, 100 ns, 180 A/cm^2, 1.0 J/cm^2, respectively. After multiple shots of IPIB irradiation, sample surface morphology was characterized with optic microscope, scanning electron microscope (SEM) on Hitachi S-4800 and atomic force microscope (AFM) on Agilent 5100 in different scales.

3. Theoretical modeling

3.1. Finite element method simulation

For finite element method, it is of great importance in fracture mechanics analysis to define the load applied onto the study object first. Afterwards, the main idea is to get the stress distribution around the crack tip within a specific area and use an appropriate criterion to assess the possibility of crack growth. Some simulation details are referred in Fig. 2. Worth to mention, considering the thermal loading mode in this case, simple mode I loading is taken into consideration.

Basing on the result of ref. [20,21], the defect density in single crystal silicon was estimated to be lower than $10^9/cm^3$. As shown in Fig. 3, a $10 \times 10\ \mu m^2$ silicon square on the sample surface was studied. A micro scale notch with 2 μm length, 0.2 μm height and 3 μm depth on the upper left corner was preset in the square. Calculated from the above defect density value, the initial number of original defects within this area should be no more than one. The notch stands for the potential crack source which probably evolved from the original defect through defect generation, migration and nucleation [22,23]. Crack source produced in the above-mentioned process will be at least 200 nm, and probably get larger size after multiple shots of IPIB irradiation.

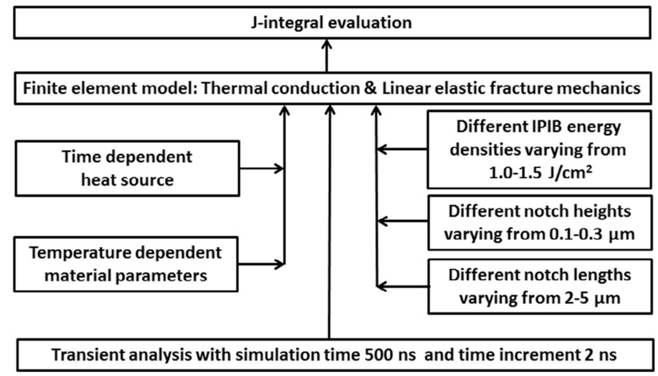

Fig. 2. Schematic diagram of the simulation procedures.

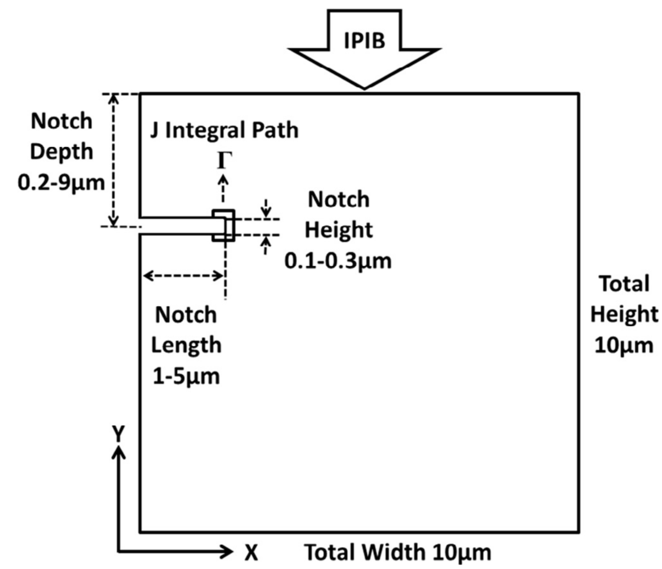

Fig. 3. Geometry schematic of single crystal silicon irradiated by IPIB with a notch surrounded by a J integral path.

Therefore, crack sources with length varying from 2 to 5 μm were utilized in this model for analysis. According to [24], the crack opening angle can significantly affect the value of the critical stress intensity factor. That's why a U-type notch was used rather than a V-type one. To make sure the path-independence and validity of J integral, the height of the crack source is restricted to be smaller than 1 μm.

In this work, as thermal stress induced by IPIB is the applied load, the thermal conduction process should be taken into consideration. Thus, Fourier heat conduction equation was adopted as the governing equation:

$$\rho C_v \frac{\partial T}{\partial t} = \lambda \nabla^2 T + P \tag{1}$$

where ρ, C_v, λ, T, t and P are the mass density, specific heat, thermal conductivity of the target, temperature, time and input energy to be defined below, respectively. The first three of them are temperature dependent parameters. ∇^2 is Laplace Operator in the two-dimensional space. Worth to mention, as the maximum temperature in this case was below the melting point of silicon, surface phase change was not taken into account.

According to our previous work [25], the external heat source term P(x, y, t) induced by IPIB was defined as:

$$P(x,y,t) = U(x) \cdot d(y) \cdot g(t) \tag{2}$$

Here $P(x,y,t)$ is the power density distribution on the target induced by IPIB. U(x) is the cross-sectional energy distribution function, which