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Novel microstructures on the surfaces of single crystal silicon irradiated by intense pulsed ion beams

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ABSTRACT

An ion beam treatment of high purity single crystal silicon specimens was performed with different shots by the irradiation of intense pulsed ion beams (IPIB), which were generated by an accelerating voltage of 350 kV and with the current density of 130 A/cm^2. As the result of irradiation, the formation of various microstructures caused by the irradiation effect, especially the thermal effect is confirmed by SEM and XRD analysis, and the corresponding processes are described and related explanations are given.

1. Introduction

IPIB have been developed over decades for various applications, including surface treatment, films deposition and nanophase powder synthesis. The beams' (with ion currents 1–5 kA, and ion energies E ~350 keV) short pulse duration ($\leqslant$1 μm) and high deposited energy density (~J/cm^2) without reflection make them promising for wide commercial use. The key process of the IPIB surface treatment is the induced rapid melt and re-solidification at up to 10^{11} K/s, which may at the same time, introduce microstructures or defects, such as craters, cracks and ripples, thus affecting the performance of modified materials [1–4].

Many related researches have been conducted to study the mechanism of the formation of these microstructures or defects, at the same time to study the interaction between the beams and targets [5,6]. As demonstrated and widely recognized, the whole process during the very limited interaction time is basically driven by the thermal effects.

In this work, to investigate the thermal effects and obtain a visible understanding, high purity single crystal silicon and copper were chosen as the target materials to perform the irradiation experiment. Different from common materials, high purity 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 process and make the physical scene more complicated.

2. Experiment

Specimens of high purity single crystal silicon and copper were mechanically polished and cut into pieces manually at dimension of $10 \times 10 \times 2$ mm^3. Then they were cleaned ultrasonically with acetone.

The irradiation experiment was carried out with the TIA-450 accelerator at College of Materials Science, Shenyang Ligong University. The beams from the accelerator consist of 70% C^{n+} and 30% H^+, which was controlled by the accelerator's anode material. The key beam parameters for the experiment are listed below: average ion energy, current fluence J, power density Pp, energy density Ep, pulse duration τ corresponding with 350 keV, 130 A/cm^2, 2.4×10^7 W/cm^2, 1.44 J/cm^2 and 60 ns respectively; and the number of pulses varying within 1, 5, 10, 30. As you can see in [7,8], the uneven cross section distribution of the ion beam, along with the instability of the beam parameters determined that different samples irradiated by the same number of shots may have slightly different behaviors. After IPIB irradiation, SEM analysis was conducted on Hitachi TM-1000 or S-4800, and XRD analysis was conducted on X-Pert PRO.

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3. Results and discussion

3.1. Heat conduction

As is well known, heat conduction plays an important role after the energy deposition onto the material surface. But due to different material properties, the surface behavior differs. An example was present in Figs. 1 and 2, where different shaped craters were observed. Worth to mention, the thermal conductivity of single crystal Cu and Si at room temperature are approximately 400 W/(m * K) and 150 W/(m * K), respectively.

In rapid IPIB-target interaction, fast warming at up to 10^{11} K/s must be followed by fast heat conduction. Metals can always get round shaped craters while low-heat-conductivity materials like silicon sometimes only get quadricorn craters. The XRD analysis in Fig. 3 revealed that new orientation silicon (201) was formed during the melt and recrystallization and the formation of the quadricorn craters is greatly related to the anisotropy of the single crystal materials, especially the thermal conductivity difference.

Imagine that in the rapid heat conduction, various materials' crystal symmetry properties are usually too weak to influence the pattern, because when the conductivity symmetry just starts to make differences to the thermal field distribution, the whole process of fast melt and resolidification have already come to the end. But if the heat conduction is slowed down slightly in the case of silicon, the conductivity symmetry is enabled to win in the competition against the phase transition during the time revolution, thus its internal symmetry characteristic can be incarnated after the pulse.

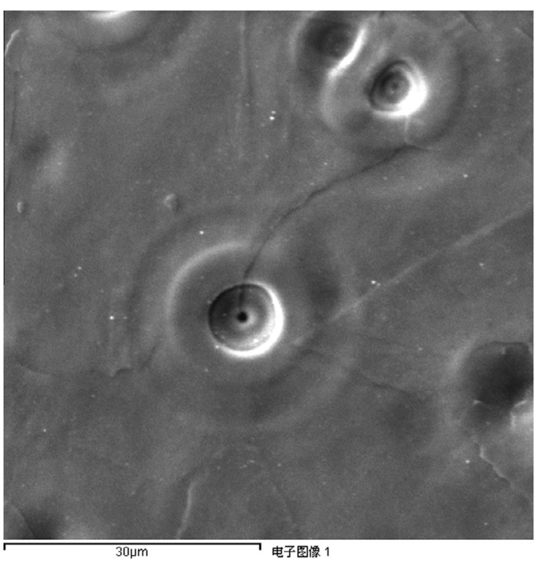

Fig. 1. Surface of Cu (100) irradiated by 10 shots of IPIB.

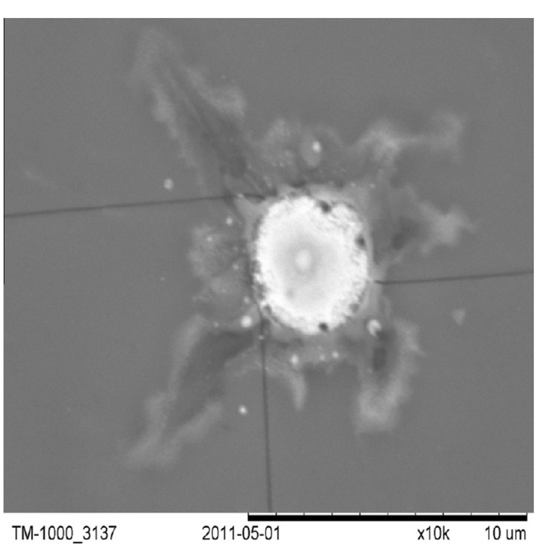

Fig. 2. Surface of Si (100) irradiated by 10 shots of IPIB.

What's more, when compared to copper, the much smaller viscosity of silicon at melt state [9,10] can make the necessary time for the flow of liquid state material and pattern formation much shorter, which will significantly increase the possibility of conductivity symmetry's victory.

3.2. Heat convection

It was presented in Fig. 4 that the melt and resolidification process did occur during the short IPIB-target interaction. In molten state materials, if the temperature is gradually increased by a heat source, heat conduction between different layers with different height will take place at the primary stage and at the next stage heat convection will follow up when the temperature gradient become large enough.

In [11] we can see that under the IPIB irradiation, the silicon surface temperature increase from room temperature to up to 1700 K within 50 ns, which will inevitably introduce large temperature gradient, thus heat convection and even turbulence will take place. The obvious undulations on the surface of Fig. 4 remind us of the occurrence of rapid heat conduction process along with the convection process at molten state.

3.3. Bénard–Marangoni Convection

Since 1916, when Rayleigh firstly gave out his explanation to the novel convection phenomenon discovered by Bénard [12], Bénard Convection has become a hot topic and a research subject for physicists, mathematicians and many other scientists. Its pattern formation in nonequilibrium systems has received considerable attention as well [13]. As illustrated in [14], when Bénard Convection is driven by surface tension gradient instead of buoyancy, it is named Bénard–Marangoni Convection. Previous work [15] has tried to use Bénard–Marangoni Convection to describe the physics in cladding process with laser beams, which share a lot of similarities with IPIB.

In the IPIB irradiation and energy deposition process, an approximately planar hot source and extremely large temperature gradient was introduced. These similar conditions necessary for Bénard–Marangoni Convection are provided, thus under some specific circumstances, Bénard–Marangoni Convection patterns are possible to be discovered on the surface of materials, just as showed in Fig. 5. It is a recording of the instantaneous Bénard–Marangoni Convection during the fast interaction process of IPIB and the target. It experimentally testified the occurrence of convection under the IPIB thermal effect.

Another proof of this conclusion is the evaluation of the process period. As demonstrated in [15] where the thermal–hydrodynamic process under IPIB irradiation was simulated and a molten period of around 500 ns was obtained, while the required time for the formation of a 20-μm crater was only about 150 ns. Considering the lower melting point of silicon, the molten time and the hydrodynamic process for the pattern formation are at the same order, which enables the occurrence of the Bénard–Marangoni Convection in this case.

Usually Rayleigh number Ra is used as an important parameter in the description of a flow field and the function of buoyancy:

$$\mathrm{Ra} = \alpha g \Delta T d^3 / \gamma K \quad (1)$$