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From crater eruption to surface purification of raw silicon: A treatment by pulsed electron beam

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ABSTRACT

The high-current pulsed electron beam treatment can induce crater eruption at the impurity particle sites, thus providing an efficient method for surface purification. In the present work, raw silicon materials of 2N-, 5N- and 6N-purity levels are treated by this method for the objective of testing the surface purification effect through the crater eruption mechanism on silicon. The surface morphology evolution is followed to trace the crater development in relation to treatment parameters such as energy density (1.8, 2.0 and 3.0 J/cm^2) and pulse number (1, 3, 5 pulses), and to the sample purity levels. It is confirmed that the amounts of craters reflect the purity level of the initial samples. Raman spectroscopy shows red shifts, up to 2.2 cm^{-1}, caused by the tensile stress due to impurity re-dissolution. The accompanied transient temperature field is also simulated, which supports the crater eruption mechanism.

1. Introduction

The high-current pulsed electron beam (HCPEB) technique features electron beams of low-energy ~30 KeV, high peak current ~10^3 A, and short pulse duration ~μs. The instantaneous energy density can be as high as 1–10 J/cm^2, and the heating and cooling rates in the treated material can reach 10^9 K/s [1–4]. Additionally, due to the high electron beam penetration capability of micrometers in depth, it can produce preferential sublayer melting and crater eruption, and eventually surface evaporation. Due to the differences of heat capacities, conductivities and other physical parameters, the electron beam energy is deposited preferentially on secondary phases, defects and grain boundaries. So the high-current pulsed electron beam technique is an effective method for surface purification via its unique crater eruption mechanism [4–6].

There are three kinds of impurities in raw silicon: electrically active impurities (B, P, Al), transition metal impurities (Fe, Mn, Ni, Cu, Cr, Ti, Mo), and alkalis metal impurities (Mg, Ca), which mainly exist as silicides (FeSi, Fe-Al-Si), carbides (SiC), oxides (MgO, CaO), and other types of compounds locating at crystal boundaries and in solid solutions such as B, P, Al [7]. It is then expected that irradiating a low-purity silicon by HCPEB would produce a surface purification effect via the crater eruption mechanism.

In this article, different-purity silicon materials are treated by HCPEB and possible surface purification is envisaged by carefully following their crater eruption behaviors. The accompanied thermal field is also simulated to describe the surface temperature distribution.

2. Experimental

Silicon samples with impurity levels of 2N (99%), 5N (99.999%), and 6N (99.9999%, single crystal wafer) were used. They were cut into 1 cm × 1 cm pieces and polished. The irradiations by HCPEB were carried out using the following parameters: energy densities of 1.8 J/cm^2 (accelerating voltage 23 kV, anode-target distance 20.5 cm), 2.0 J/cm^2 (accelerating voltage 23 kV, anode-target distance 14 cm), 3.0 J/cm^2 (accelerating voltage 27 kV, anode-target distance 14 cm); pulse duration of 2 μs; number of pulses of 1, 3, and 5. The beam cross-section area is about 30 cm^2, and the pulse repetition interval is about 10 s. The samples and HCPEB treatment parameters are shown in Table 1.

Surface and cross-section microstructures were observed by using optical microscopy (OM) and scanning electron microscopy (SEM). The crater profiles were observed by the NewView 5022 surface profilometer. Raman spectra were collected on an inVia Raman Microscope with an unpolarized HeNe laser (λ = 632.8 nm), a spot size of 2 μm.

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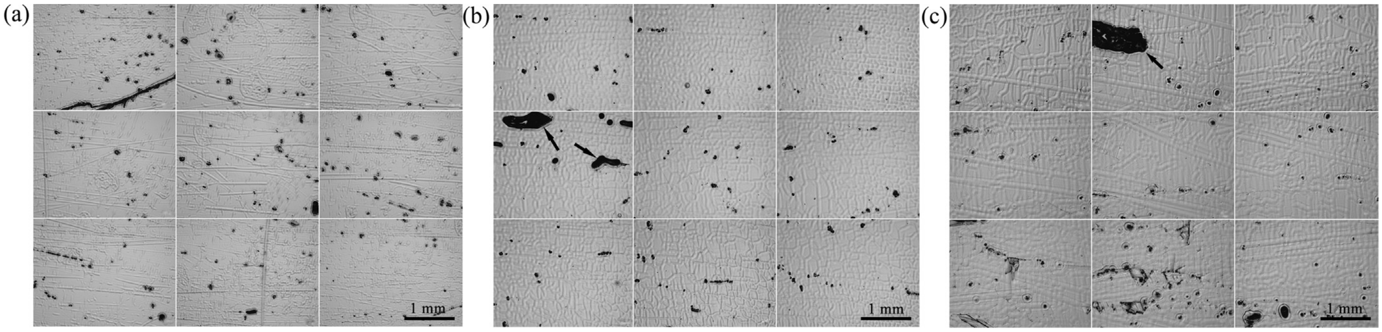

Fig. 1. Optical micrographs of the surface morphologies on the 2N-purity silicon, treated by the energy density of 1.8 J/cm^2 and 1 pulse (a), 3 pulses (b), and 5 pulses (c).

Table 1
Sample purity levels and HCPEB experiment parameters.

	Energy density 1.8 J/cm^2	Energy density 2.0 J/cm^2	Energy density 3.0 J/cm^2
1 pulse	2N, 5N, 6N	5N, 6N	5N, 6N
3 pulses	2N, 5N, 6N	6N	6N
5 pulses	2N, 5N, 6N	6N	6N

3. Results and discussion

3.1. Surface morphologies and crater densities

The OM micrographs of the complete surface area of 2N-purity silicon, treated at the energy density of 1.8 J/cm^2 for 1, 3 and 5 pulses, are shown in Fig. 1. The crater density is counted, which is equal to the numbers of craters divided by the surface area, which is 51.7 mm^2. The crater densities are about 2.32, 2.41 and 2.79 mm^{-2} for the samples irradiated by 1, 3, and 5 pulses, respectively. The counting error of viewing area is about 0.02 mm^{-2}. The dark zones, as arrowed in Fig. 1(b) and (c), are initial holes on the surface.

Fig. 2 compares the surface OM micrographs of all the three 2N, 5N and 6N-purity silicon pieces, which are irradiated by HCPEB at the same energy density of 1.8 J/cm^2 for the same pulses of 1, 3 and 5, as in Fig. 1. As expected, the electron beam treatment induced a large amount of crater eruptions on the surfaces of the low-purity silicon, and the crater densities decrease along with increasing purities. With one pulse, the crater densities are 2.32 mm^{-2} for the 2N sample, 0.32 mm^{-2} for 5N, and 0.09 mm^{-2} for 6N. On the 2N-purity polycrystalline silicon surface, the crater eruptions are abundant. The size and the shape of the craters can be very different. The crater arrowed by a_1 represents a typical morphology, with a round hole encircled with multiple fringes. The crater arrowed with a_2 has an irregular shape, possibly caused by simultaneous eruptions due to the uneven impurity distributions. On the 5N-purity polycrystalline silicon surface, the crater density is reduced obviously as compared to the 2N sample. There is nearly no crater

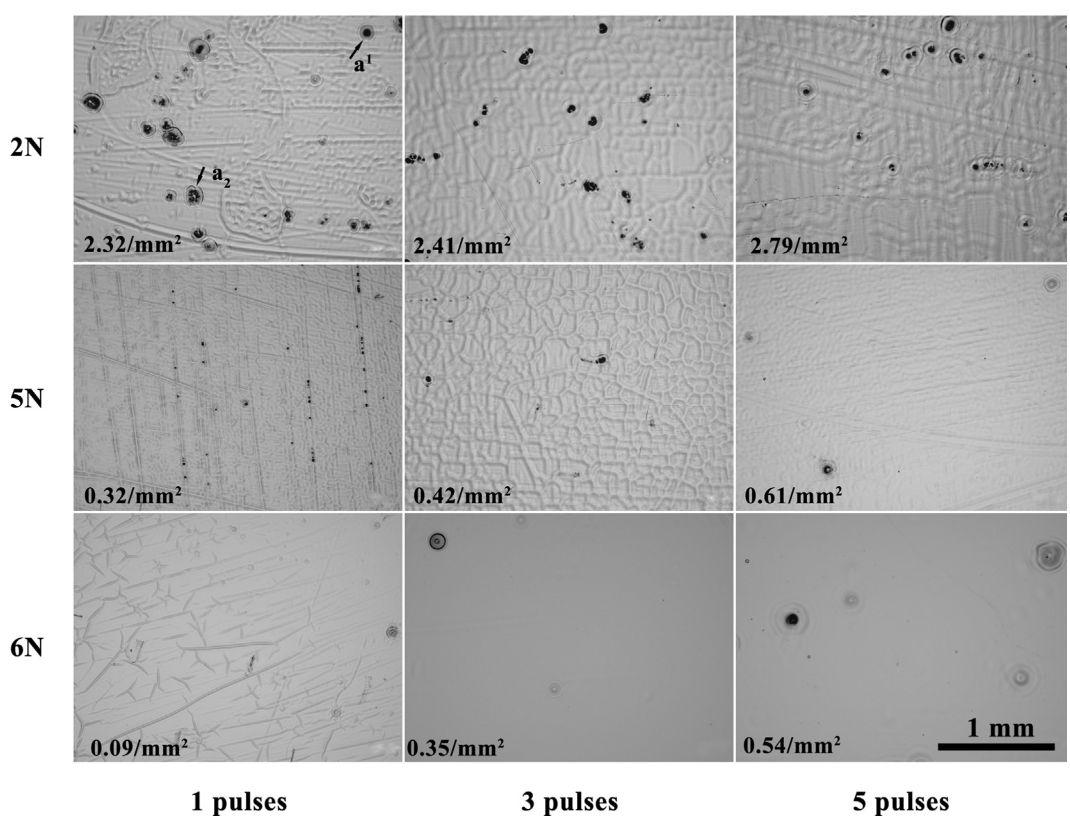

Fig. 2. Optical micrographs of the surface morphologies on the 2N, 5N and 6N-purity silicon materials treated by 1.8 J/cm^2, 1 pulse, 3 pulses, and 5 pulses.