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Controlled fabrication of Si nanostructures by high vacuum electron beam annealing

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ABSTRACT

Silicon nanostructures, called Si nanowhiskers, have been successfully synthesized on Si(1 0 0) substrate by high vacuum electron beam annealing (EBA). Detailed analysis of the Si nanowhisker morphology depending on annealing temperature, duration and the temperature gradients applied in the annealing cycle is presented. A correlation was found between the variation in annealing temperature and the nanowhisker height and density. Annealing at 935 °C for 0 s, the density of nanowhiskers is about $0.2\,\mu m^{-2}$ with average height of 2.4 nm grow on a surface area of $5\times5\,\mu m$, whereas more than 500 nanowhiskers (density up to $28\,\mu m^{-2}$) with an important average height of 4.6 nm for field emission applications grow on the same surface area for a sample annealed at 970 °C for 0 s. At a cooling rate of $-50\,°C\,s^{-1}$ during the annealing cycle, 10–12 nanowhiskers grew on a surface area of $5\times5\,\mu m$, whereas close to 500 nanowhiskers grew on the same surface area for samples annealed at the cooling rate of $-5\,°C\,s^{-1}$. An exponential dependence between the density of Si nanowhiskers and the cooling rate has been found. At 950 °C, the average height of Si nanowhiskers increased from 4.0 to 6.3 nm with an increase of annealing duration from 10 to 180 s. A linear dependence exists between the average height of Si nanowhiskers and annealing duration. Selected results are presented showing the possibility of controlling the density and the height of Si nanowhiskers for improved field emission properties by applying different annealing temperatures, durations and cooling rates.

1. Introduction

The potential applications of Si nanowhiskers in photonic and optoelectronic devices [1], field effect transistors [2,3], solar cells [4] and chemical [5] or biological sensors [6,7] are the major focus of recent nanoscience research in Si nanowhiskers. This is due to the fact that Si nanowhiskers show special electronic, optical, chemical, mechanical, and thermal properties that differ from bulk Si.

The key challenge facing the practical realization of devices based on Si nanowhiskers is the identification of suitable nanofabrication techniques because it is desirable to be able to control the size, density and location of arrays of Si nanowhiskers for future application. Among different fabrication methods for Si nanowhiskers, the most widely applied techniques are chemical vapour deposition (CVD) catalyzed by Au nanoparticles with vapour–liquid–solid (VLS) mechanism [8,9], molecular beam epitaxy (MBE) with physical vapour deposition (PVD) [10], electron beam evaporation (EBE) [11], and electron beam lithography [12]. However, most of these have constraints to develop as practical and high density industrial nanofabrication tools. In previous studies, we demonstrated the growth of Si nanowhiskers on untreated Si (1 0 0) using high vacuum electron beam annealing (EBA) [13,14]. The process is rapid and straightforward, and requires no pre-treatment of the Si substrate. This novel nanofabrication technique is extremely flexible, allowing precise control over all fabrication conditions. Also good uniformity, compatibility and high density of Si nanowhiskers have been obtained, which makes it a potential industrial technique for Si nanowhiskers fabrication. Si nanowhiskers with small apex radius have been applied as the cold cathode material for field emission study and a current has been observed [15–18].

In this work, we provide a detailed analysis of the dependence of Si nanowhisker morphology on annealing temperature, cooling rate and annealing duration by high vacuum EBA which improves the understanding of the mechanism by which the nanowhiskers form. Selected results are presented showing the possibility of controlling the Si nanowhiskers by different annealing temperatures, durations and cooling rates. A two-stage mechanism is applied to explain the results kinetically and thermodynamically.

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2. Experimental

Samples were cut from n-type Si(1 0 0) maintaining the native oxide layer. The electrical resistivity of the wafer is 10 Ω cm. No surface cleaning and treatments were performed on the substrate. The samples were loaded into the electron beam annealer [19] in which a raster scanned 20 keV electron beam with current up to 3.5 mA was used. The operating gas pressure was 2×10^{-7} mbar. Annealings were carried out by a computer controlled feedback system based on a thermopile detector. True temperature measurements were performed using a 2-color-pyrometer which also faced the front side of the samples. The as-grown samples were investigated by a Nanosurf atomic force microscope (AFM) using a Si probe with diamond coating (tip radius $\leq$10 nm, half cone angle 10° at apex). The annealing temperature range was selected from 935 to 970 °C because previous results suggest that this is an idea temperature range for forming Si nanowhiskers on Si substrate for field emission applications [20]. The annealing duration was varied between 0 and 180 s. The heating rate was kept constant at +5 °C s^{-1} and the

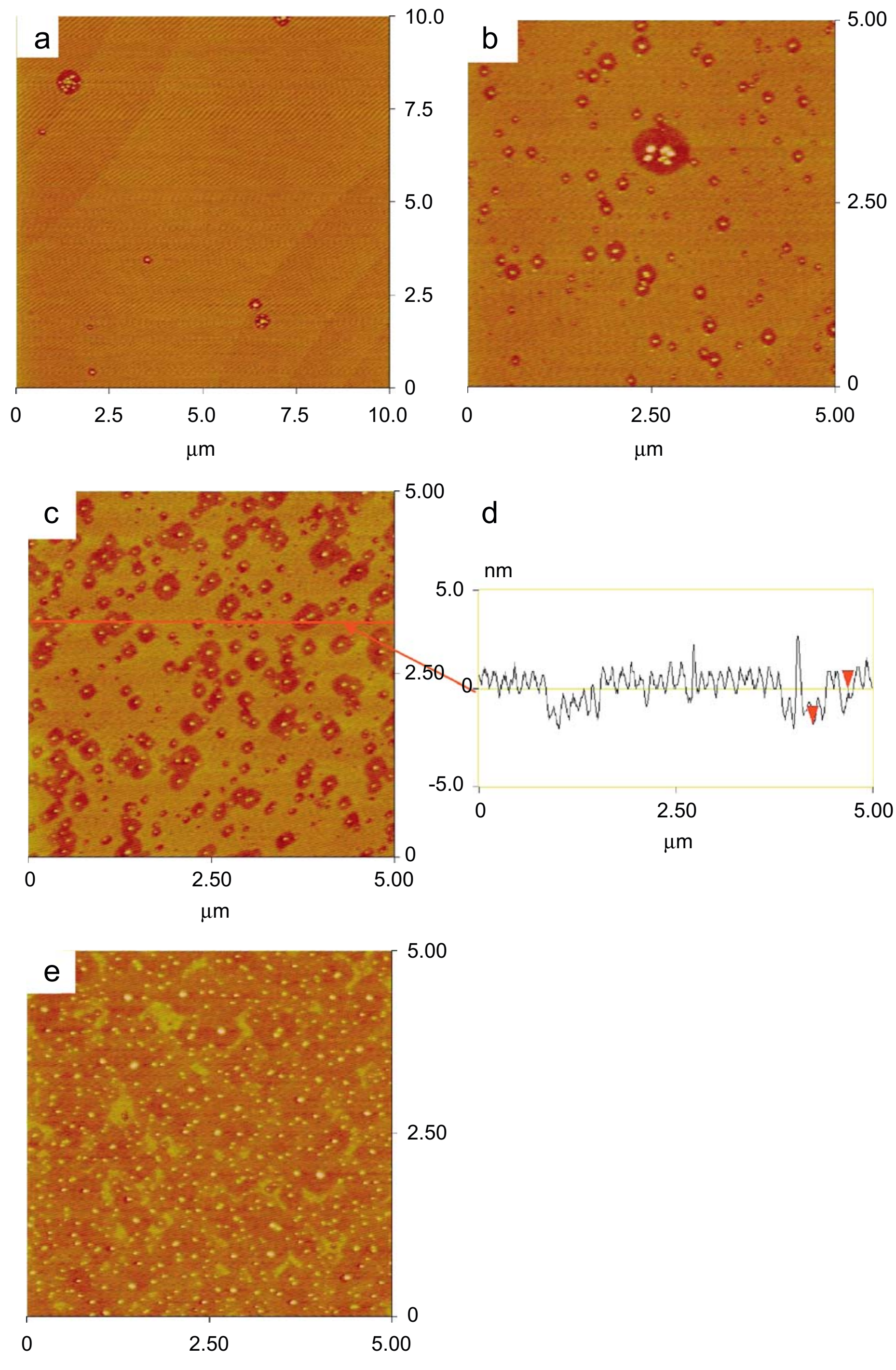

Fig. 1. AFM images of Si nanowhiskers grown on Si substrate after electron beam annealing at: (a) 935 °C, (b) 945 °C, (c) 960 °C, (e) 970 °C for 0 s with heating rate of 5 °C s^{-1} and cooling rate of −100 °C s^{-1}. (d) The line scan along the straight red line in (c).