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Nanofabrication of self-organized periodic ripples by ion beam sputtering

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ABSTRACT

Ion beam sputtering can induce the formation of regular self-assembled surface nanostructures on different materials. Several experimental variables, such as the ion energy, the ion incidence angle, the ion fluence, together with the sample surface properties and temperature, have been proven to control in a complex and not completely codified manner the formation of periodically arranged dot, hole or ripple nanopatterns. In this work, we have studied how to apply ion beam sputtering to induce periodic ripples of specific aspect ratio on silicon surfaces, namely ripple height a of about 10 nm and period $\lambda \leq 50$ nm, since these topographies can be appealing for technological applications. Silicon surfaces were irradiated with increasing O^+ and Xe^+ ion fluences at fixed ion energy and incidence angle. We have verified that some combinations of the chosen parameter values produced the desired structures. The obtained results also indicate that with a further refinement of those parameter values a better control of the aspect ratio of the obtained ripples is possible. Therefore, this work is a contribution to the final aim of exploiting ion beam sputtering as a fast, cost-effective and single-step method to fabricate well-controlled patterns over large surface areas at length scales beyond those of both standard and e-beam lithography.

1. Introduction

In 1962 Navez et al. [1] first reported the development of periodic structures by Ion Beam Sputtering (IBS) of glass surfaces and discovered their dependence on the incidence angle of the ions. In the following years, many studies have been performed in order to understand the physical mechanisms that regulate the development of periodically arranged dot or ripple nanopatterns on the surface of different materials [2–7]. This spontaneous topography formation represents an unwanted surface flatness degradation in IBS applications for surface analysis/depth profiling (e.g. secondary ion mass spectrometry [8,9], ion milling [10]). On the other side, being essentially a self-assembling phenomenon, it can be exploited to build sub-micrometer and nanometer structures on semiconductor, metal and insulator surfaces. In fact, the nanostructuring of surfaces by IBS has some attractive advantages over the top-down lithographic techniques, as it is a fast, cost-effective and single-step method to fabricate self-organized nanopatterns over large surface areas. This may lead to consider IBS as a bottom-up technique alternative or complementary to the conventional lithography for the patterning of materials at the nanoscale.

The formation and the characteristics of self-organized topographies depend on several experimental parameters related both to the incident ion beam (such as ion species, kinetic energy, incidence angle and ion fluence), and to the sample conditions (such as pristine surface and sample temperature). Considerable advances have been made to reach a comprehensive theoretical understanding of the dot and ripple formation process. In 1988, Bradley and Harper proposed a theoretical model to explain the formation of sputtering-induced ripple topographies [11]. These result from the competition of two phenomena: a curvature-induced roughening instability, i.e. an increasing amplitude instability due to the sputter yield variation with surface curvature, and a smoothening process by surface diffusion. On a short time/low fluence scale, the process can be approximated by a linear continuum equation, which predicts a constant wavelength and an exponential growth of the amplitude. By increasing the bombarding time/fluence or, alternatively, the incidence angle [12,5], the non-linear effects are no longer negligible and important surface profile changes appear. First, the coarsening of the ripple wavelength and the ripple amplitude saturation can be observed [12]. Secondly, the patterns become more and more faceted at the downstream side with respect to the ion beam direction and typical sawtooth profiles emerge [7,13]. Eventually, for grazing incidence angles or longer bombarding times, the surface smoothing is observed [14].

Despite the valuable theoretical studies, a common general framework for all the different experimental observations is still missing [6], whereas for some technological applications it is instead mandatory to understand how to effectively tune the experimental parameters to produce on large areas periodic nanostructures with the required dimensions. For this reason, the aim of this work was to see if it was

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possible to systematically order the knowledge gained with experiments described in literature and derive an empirical model allowing the prediction of the outcome when pushing parameters out of the already explored ranges. In particular, the choice of the experimental parameters controlling IBS on silicon surfaces was considered, in order to obtain periodic nano-ripples with a pre-defined aspect ratio $AR = a/\lambda \geq 1/5$, where a is the height and λ is the wavelength (period). Ripples of a period of about 50 nm and height a close as much as possible to 10 nm would be potentially useful for nanoelectronics and energy storage applications [15]. For ripples of lower height, different applications have been already envisaged, namely electrochemical devices [16], plasmonics [17], and nanostructured materials for bio-devices [18].

2. Materials and methods

Six different irradiation conditions (see Table 1) were chosen after comparing and integrating many published experimental results, allowing the definition of a “reference empirical schema”, describing how to change the experimental parameters to control the ripple sizes. Among others, references [13,19,20] provided the starting point of the six experiments, as the ripple heights and wavelengths therein discussed were already in ranges close to those expected. The same incident ion species were chosen, i.e. Xe^+ and O^+, and the empirical model was applied, with the aim to obtain ripples of period $\lambda \leq 50$ nm and height a close as much as possible to 10 nm, therefore modifying the ripple sizes reported in [13,19,20].

Ion irradiations were performed using a low energy ion implanter (Danfysik A/S, Denmark, Model1050) equipped with a gas-fed Chordis ion source. 5 keV Xe^+ and 1 keV O^+ ion beams, with fluxes of 0.6×10^{13} and 1.1×10^{13} cm^{-2} s^{-1} respectively, were electrostatically raster scanned (f_x, f_y ~ 1 kHz) full area over 1×1 cm^2 Si(100) samples, which were tilted 50° or 55° with respect to the ion beam. Ion fluences between 8.0×10^{16} and 2.0×10^{18} cm^{-2} were irradiated on six different samples. A vacuum $< 2 \times 10^{-6}$ mbar was maintained in the implantation chamber. Table 1 gives an overview of all irradiation conditions of investigated samples. All the samples were irradiated at the Institute of Ion Beam Physics and Materials Research at the Helmholtz-Zentrum Dresden-Rossendorf (HZDR), Germany.

Atomic Force Microscopy (AFM) images were acquired with a scanning probe microscope (Unisolver P47H, NT MDT Co.). The analyses were performed in semi-contact mode with a silicon tip having a nominal radius of less than 10 nm. Scan areas of 2×2 μm^2, 5×5 μm^2 and 10×10 μm^2 were acquired with a resolution up to 1024×1024 pixels. The 10×10 μm^2 images (not shown) were used to confirm on a larger spatial scale the hereinafter discussed results. The root mean square (RMS) surface roughness, representing the standard deviation of surface heights, was calculated for each scanned area and used to compare the roughness of the irradiated surfaces. The periodicity of the patterns was evaluated by the discrete two-dimensional Fast Fourier transformation (2D-FFT), as well as by inspecting the cross sections of the AFM topographies, which were taken along the projected irradiation beam direction.

The same irradiated samples were also analyzed by field-emission Scanning Electron Microscopy (SEM, Jeol JMS 7401F). Analyses were carried out in plan-view, setting an acceleration voltage of 5 kV.

Table 1
The irradiating sample conditions.

Sample name	Implanted ion species	energy (keV)	Fluence 10^{18} (ions/cm^{-2})	Incidence angle (°)
Xe_1	Xe^+	5	0.080	55
Xe_2	Xe^+	5	1.5	55
Xe_3	Xe^+	5	2.0	55
O_1	O^+	1	1.0	50
O_2	O^+	1	2.0	50
O_3	O^+	1	2.0	55

3. Results

Fig. 1 shows the AFM topography images acquired on the three Xe^+ irradiated samples. For each image, the corresponding 2D-FFT image and a vertical linear profile are shown. Fig. 1A shows the presence of a pattern with an irregular short range correlation and structure heights of about 1–2 nm. On the contrary, the surfaces of Fig. 1B and C show a regime of surface smoothing, since the resulting RMS roughness is comparable with the initial RMS roughness The SEM images are not shown, since the modified surfaces have morphological features below the instrument detection limit.

The presence of a regular topography is instead recognizable in all the samples irradiated with O^+ ions. In general, following the ion fluence increase from Fig. 2A to C, the analysis shows the development of periodic ripples (with a distribution of wavelengths as described by the 2D-FFT images shown in the insets of Fig. 2), with a nearly constant wavelength of ~40 nm, 50 nm and 60 nm respectively, and corresponding heights of ~2.5 nm, 3 nm, and 15 nm. These patterns are superimposed on a less regular topography of longer wavelength and higher amplitude, which can be associated to the long time-scale and non-linear regime proper of the sputtering process [5]. In fact, in Fig. 2C, which corresponds to the highest fluence, the larger wavelength structure definitely dominates on the high-frequency rippled structure, and the cross section clearly shows a sawtooth profile, which is typical of long bombarding times [5,7].

In Fig. 3, the SEM images of the same samples of Fig. 2(A–C) show, on a larger field of view, the regularity of the obtained nano-ripples. In Fig. 3C, which corresponds to the highest fluence, shingle-like faceted surfaces are clearly visible in place of the regular ripples of Fig. 3(A,B), which are indeed typical of lower ion fluences.

4. Discussion

Starting from the experimental settings given in [13,19,20], in order to modify the ripple features to values of $\lambda \leq 50$ nm and of a of about 10 nm, the empirical model derived from the literature prescribes values of fluence and incidence angle that move towards a quasi-linear experimental regime, so that a weak dependence of λ on irradiation time is expected while the height a increases [5,12]. In addition, the ion energy has to be chosen to have λ values in the required nanometer range, as it is known that at room temperature the ripple wavelength increases with the ion energy [5]. It is generally acknowledged that the surface remains flat up to a critical incidence angle. Some authors also gave evidence that this critical angle shifts to lower values with decreasing ion energy [5], and that the linear instability develops on a faster time scale for increasing incidence angles, leading to an earlier onset of nonlinear effects [5,12]. Therefore, by choosing low values of the ion energy and incidence angle, we aimed at increasing the aspect ratio and at delaying as much as possible the onset of nonlinear features, such as the coarsening of the wavelength, the appearance of large triangular and faceted hillocks or of shingle-like faceted surfaces, and, on longer times, the surface smoothing regime.

A different outcome of the Xe^+ experiments with respect to the O^+ experiments is clearly outlined in Figs. 1 and 2. Because of a large mass difference between Xe and O, the absolute value of the (physical) sputtering rate and its incident angle dependence are different. The oxygen reactivity might also give additional differences in the fabrication process of ripple structures [21].

In Fig. 1 (B–C), the silicon surfaces after Xe^+ irradiation show the effects of a long-term non-linear regime. In fact, only for the first ion fluence (Fig. 1A) a pattern was produced having an irregular short range correlation and structure heights of about 1–2 nm. Therefore, in this case the quasi-linear regime probably starts and ends at definitely lower fluence values.

In Fig. 2(A–C), showing the results of O^+ irradiation, a sufficiently stabilized non-linear regime is present as well, but, for samples of