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Formation of self-organized nanostructures on Ge during focused ion beam sputtering

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ABSTRACT: We report the formation and self-organization of nanoscale structures during normal-incidence focused ion beam sputtering of Ge. Ridge-shaped ripples with large height-to-width ratios form. We provide evidence that the ripples form spontaneously from nanostructured networks at doses below 10^{17} ions·cm^{-2}. Highly oriented structures running in the fast scan direction evolve at higher doses. We attribute the ripple formation to spatio-temporal overlap of FIB induced micro-explosions.

1. INTRODUCTION

It is of both theoretical and practical interest to understand surface morphological evolution during ion beam bombardment. Spontaneous rippling of surfaces by keV ion irradiation has been observed on a wide variety of materials, and has been reviewed recently (Valbusa *et al.* 2002; Makeev *et al.* 2002). The orientation of the ripples is usually determined by the incidence angle of the ion beam relative to the surface normal, and sometimes by the surface crystallography (Valbusa *et al.* 2002). The Bradley-Harper mechanism (Bradley and Harper 1988) is generally accepted as the origin of ripple formation in most situations: Because of the distribution of the ion's energy over the depth below the point of impact, concave regions of the surface are sputtered more rapidly than convex regions, resulting in a surface morphological instability. This roughening effect is opposed by a smoothening effect, typically surface diffusion or viscous flow, that has a different dependence on the wavelength of the morphology than the roughening effect. The interplay of these two effects determines a characteristic wavelength for the surface pattern. For normal ion incidence, the ripples don't "know" which way to orient, and hillocks or pits are observed. However, ripples may still form when surface crystallography introduces azimuthal anisotropy in either the roughening or smoothening mechanisms (Valbusa *et al.* 2002). In this paper, we report an unusual observation of ripple formation, which cannot be explained using these or other existing models.

2. EXPERIMENT

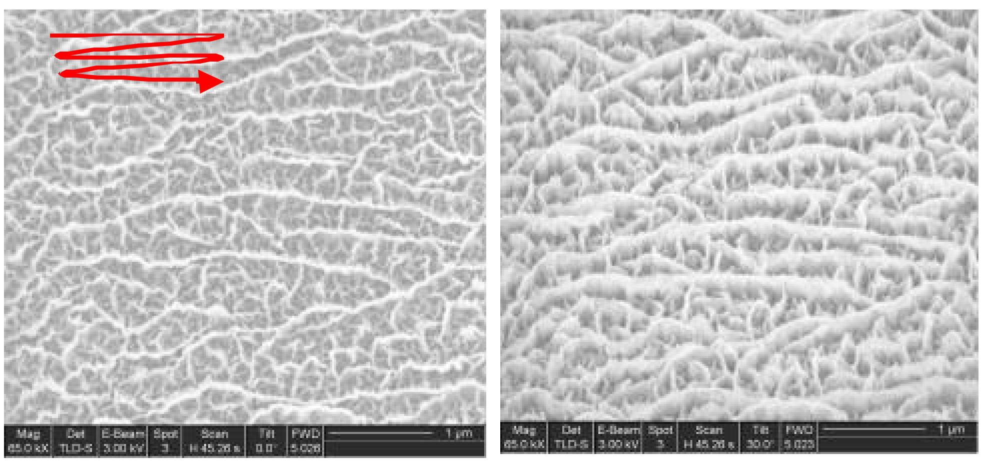
The focused ion beam (FIB) experiment was carried out using an FEI dual beam FIB-Scanning Electron Microscope (SEM) delivering 30 keV Ga^+ to a Ge(001) wafer surface in a background pressure of 1.4×10^{-6} mbar at room temperature. The incident ion beam was parallel to the surface normal and the ion beam current was 100 pA. The beam was rastered in a boustrophedonical scan across a pre-defined region of the sample surface, during which time the beam would dwell at each discrete location for 0.1 μs and then move rapidly to an adjacent location. Separation between adjacent locations was set to nominally 50% overlap, which in this case meant a 10 nm center-to-center spacing for a 20 nm diameter beam. The current profile within the beam is believed to be roughly Gaussian.

The irradiated surfaces were observed using both *in-situ* SEM and *ex-situ* contact mode AFM in air.

3. RESULTS

3.1 Self-Organized Ripple Formation

Because keV ion irradiation at room temperature is expected to amorphize the Ge surface after an exceedingly low dose ($<1\times10^{14}$ ions$\cdot$cm^{-2}) and thereby eliminate all surface anisotropy, one expects to form a surface topography of hillocks or depressions. Instead, when the ion dose was increased to above 2×10^{17} ions$\cdot$cm^{-2}, we observed ridge-shaped ripples as shown in Fig. 1(a). The ripples have irregular spacing, ranging roughly from 100 to 400 nm in most cases. Fig. 1(b) shows the same FIB sputtered area viewed at an angle. The tilted SEM image makes it possible to estimate the heights of ripples, and the heights can be measured using contact mode AFM. The ripple heights vary over a wide range from 10 to over 100 nm. The spot center-to-center distance of 10 nm in both scan directions is an order of magnitude smaller than the ripple spacing. Therefore, the ripple formation can be attributed to self-organization rather than FIB direct writing.



(a) (b)

Fig. 1 SEM images after ion dose of 1.04×10^{18} ions$\cdot$cm^{-2}. (a) View normal to Ge surface showing ripples oriented in the fast scan direction. The arrow indicates the raster direction. (b) The same area viewed at 30° tilt.

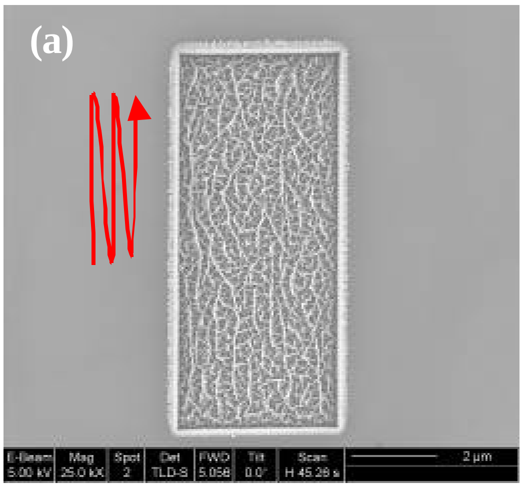
3.2 Ripple Orientation vs. Fast Scan Direction

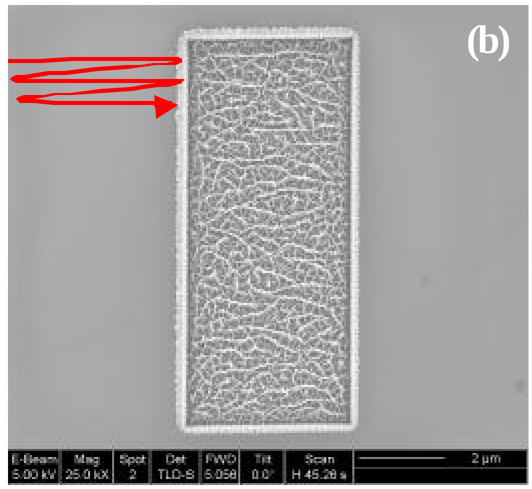
In Fig. 1(a) the ripples are oriented in the fast scan direction. This always occurs, as shown in Figs. 2(a), (b) and (c). It should be especially noted, by comparing Fig. 2(a) with Fig. 2(b), that the orientation of the ripples is not determined by the boundary of the FIB sputtered area under the conditions studied here.

3.3 Surface Morphological Evolution with Increasing Dose

We used the FIB to raster a 6 μm square under these conditions and continuously monitored the surface morphological evolution with increasing dose from 10^{15} ions$\cdot$cm^{-2} up to over 10^{18} ions$\cdot$cm^{-2}. At a low dose level of 3.47×10^{15} ions$\cdot$cm^{-2}, the rastered area was found to be lower than the original surface, as shown in Fig. 3(a). This result is expected due to the effect of ion beam sputtering.

However, as the dose level increased to 8.67×10^{15} ions·cm^{-2}, nanostructured networks formed (Fig. 3(b)) and part of the rastered surface "swelled" above the original Ge surface level by a few nanometers (Fig. 3(c)). The surface swelling continued with increasing dose, with the peaks reaching a maximum of 120 nm above the original surface at a dose level of 5.2×10^{16} ions·cm^{-2}. The process was accompanied by coarsening of the nanostructured networks (Fig. 3(d)). We were unable to measure the profile of the portions of the "swelled" surface that lay in between the peaks, or to assess what fraction of the surface lay above the original surface location. As ion dose increased further to above 2×10^{17} ions·cm^{-2}, ripples oriented in the fast scan direction (see Figs. 1 and 2) developed from the gradually coarsening nanostructured networks and all parts of the rastered once again descended below the location of the original surface.





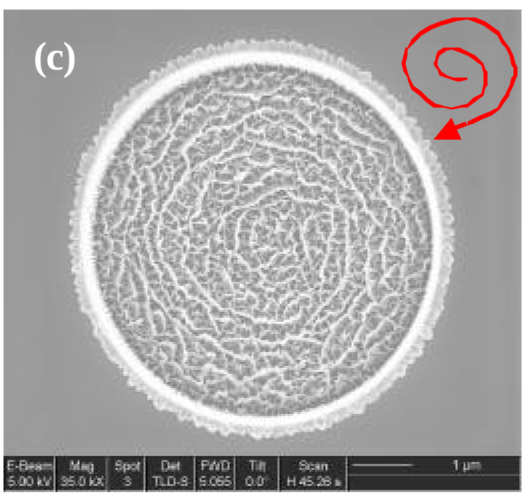


Fig. 2 SEM micrographs showing that ripples are largely orientated in the fast scan direction. The arrow in (a), (b) or (c) indicates the scan direction.

(a) Dose = 5.2×10^{17} ions·cm^{-2}.
(b) The dose is kept the same as in (a), but the fast scan direction is changed by a 90 degree rotation. The ripples also change the orientation by 90 degree to align themselves in the fast scan direction.
(c) Dose = 1.32×10^{18} ions·cm^{-2}. When FIB rasters the circular area spirally from the center outwardly, curved ripples form roughly along the spiral path of FIB spot.

4. DISCUSSION

It is possible to propose a tentative explanation of our observations. Wilson (1982) and Bellon *et al.* (1995) studied very low dose unfocused ion irradiation of Ge(001) and observed that a small fraction of the incident ions appears to produce large "micro-explosions". The surface swelling that we observe suggests micro-explosions are occurring here as well. The observation that ripples are oriented in the fast scan direction leads us to tentatively attribute the ripple formation to the spatio-temporal overlap of the FIB induced micro-explosions. Further experiments are planned.

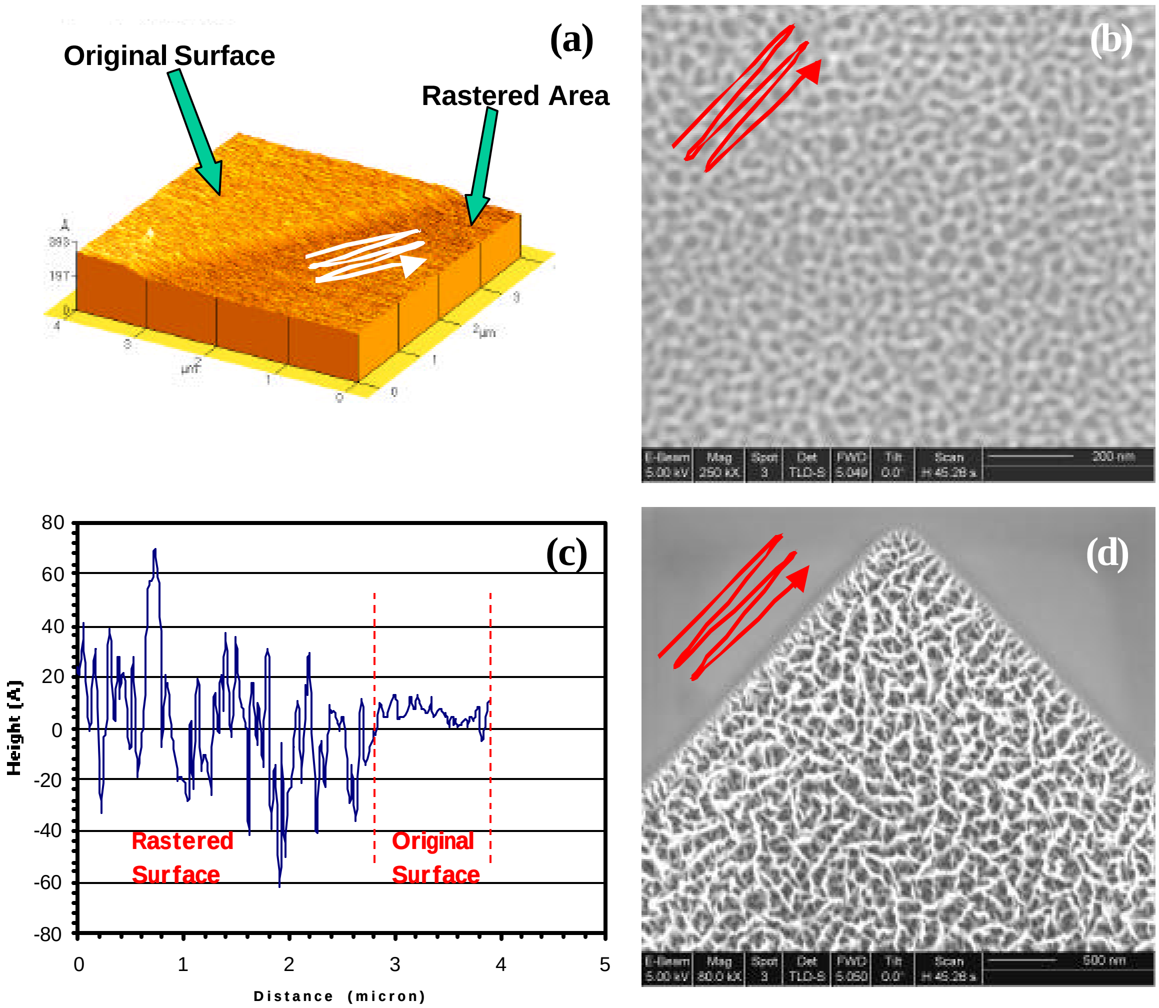


Fig. 3 (a) AFM image at dose of 3.47×10^{15} ions·cm^{-2}. (b) Nanostructured networks at dose of 8.67×10^{15} ions·cm^{-2}. (c) AFM line profile showing that part of the rastered surface shown in (b) swelled above the original Ge surface. (d) Coarsening of the nanostructured networks at a higher dose of 5.2×10^{16} ions·cm^{-2}. Arrow in (a), (b) or (d) indicates FIB scan direction.

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