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Names and affiliations of applicants (* indicates experimentalists):

Prof. Corrado BORAGNO * , INFM-UdR Genova and Dip. di Fisica, Genova, Italy

Dr. Francesco BUATIER * , INFM-UdR Genova and Dip. di Fisica, Genova, Italy

Dr. Roberto FELICI * , INFM-OGG , c/o ESRF, Grenoble, France

Prof. Ugo VALBUSA * , INFM-UdR Genova and Dip. di Fisica, Genova, Italy

Dr. Alessandro MOLLE * , INFM-UdR Genova and Dip. di Fisica, Genova, Italy

Prof. Salvador FERRER * , ESRF, Grenoble, France

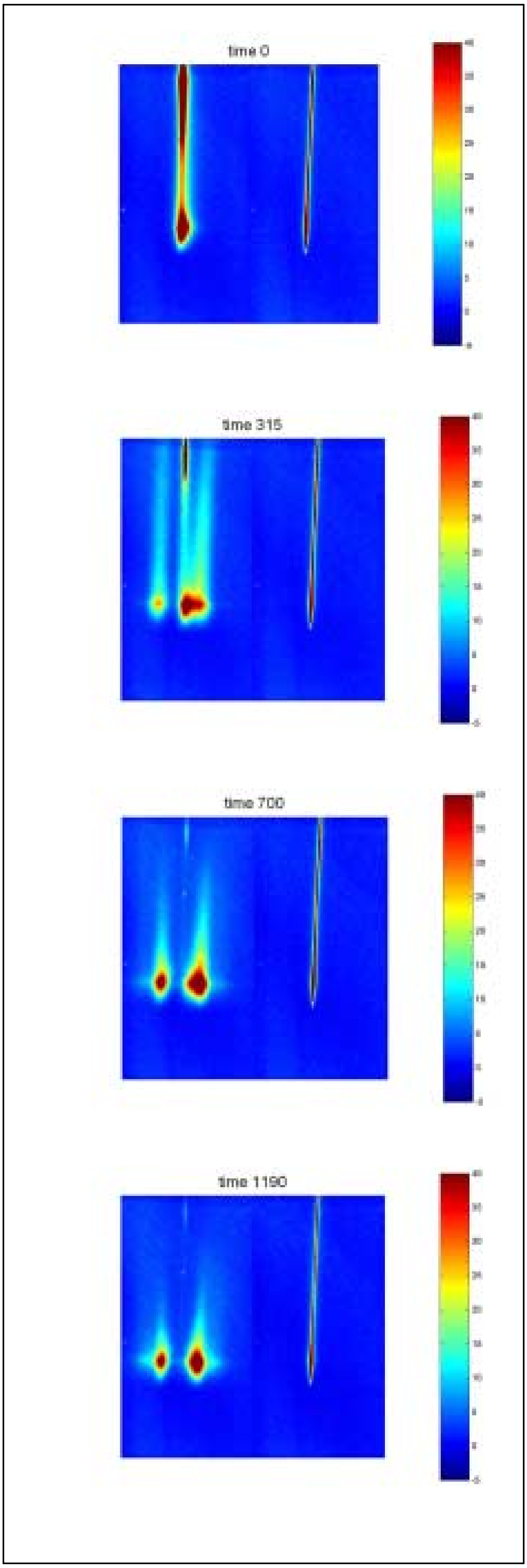
Report:

The purpose of the experiment, a prosecution of two other runs previously performed, was to follow in real time the evolution of a surface when bombarded with ions.
The main differences respect to the previous experiments which studied Ag and Cu respectively were:

1. The use of a crystal, Rh, with lower surface diffusion. This condition moves the formation of ripples/mounds to temperatures well above room temperature thus allowing to produce nanopatterns very stable when the crystal is cooled down at room temperature. This goal will open the possibility of using these surfaces as templates for film deposition or nanostamping.
2. The use of a CCD camera instead of a point detector. In this way we are able to collect more information at the same time and to compare the 2-D intensity maps with those calculated from an analysis of the scattered intensity when a nanopattern is present on a surface.

The crystal was Rh(110), aligned better than 0.2° . The surface was sputtered with Ar^+ ions, at an energy of 1 keV; an ion current in the range 3 – 8 μA was measured on the sample, corresponding to an ion flux in the range $3\ 10^{13} – 8\ 10^{13}$ ions/(sec cm^2). The ion gun was placed few degrees off the surface normal, but in the plane scattering, i.e. the plane containing the surface normal, the incident and the reflected X-ray beam.

As expected, we observed formation of regular patterns on the surface, with different symmetries depending on the sputtering temperature: at T = 130 °C , ripples are present, elongated on <001>; in the range T = 180 – 250 °C , the surface is organized in mounds with the sides aligned with the principal surface directions; at T = 280 °C , ripples are again formed, but now they form along <1-10> showing the well known effect of ripple rotation. The temperature evolution is similar to the one observed for Ag(110) and Cu (110) , but the temperature range where this effect occurs is shifted 200 K towards higher temperatures. During the experiment, we also followed the time decay of a ripple structure formed at T = 280 °C : the structure is stable till about 320 °C and at this temperature it vanishes in about 30 min. This observation confirm our initial hypothesis: the formation of nanopatterns on metal surfaces is a quite general phenomenon, and by choosing the appropriate crystal it is possible to obtain nanostructures which are stable at room temperature and above for long time.



In order to illustrate the information we got by CCD camera, we report a series of images corresponding at different sputtering time at T = 280 °C . In Figure 1, on the left are presented the images taken when the incoming X-ray beam is aligned along <1-10> direction in the real space, while on the right the images taken when the crystal is rotated by 90° (X-ray beam along <001>).

Grazing incidence geometry (GISAXS) was used; ion current was set at 3 μA ; the acquisition time was 35 sec / image. We followed the time evolution for about 1 hour. Here we present only few snapshots. For each images, the x axis represent a variation in the out-of-plane scattering angle, while the y axis represents a variation in the exit angle.

At the beginning, only the trace of the incoming X-ray beam is present. Two maxima of intensity (marked by arrows in the first panel) are present: the first corresponds to an exit angle equal to the reflectivity angle, while the second corresponds to the specular angle.

After 315 sec, two shoulders appear in the left panel: they corresponds to the formation of ripples along the <1-10> direction in real space. The intensity at the critical and specular angles decreases strongly, indicating that the total reflectivity of the surface changes due to the surface disorder. The intensity in the opposite direction remains practically the same.

After 700 sec, only the shoulders are present, while after 1190 sec it seems that also second order peaks start. We remark that in the other direction nothing changes.

We acquired images in GISAXS geometry at 4 different temperatures.

Also scattered intensity at the (2 0 0.15) and (0 2 0.15) points in the reciprocal space have been acquired in order to get information on the time evolution of the facets and of the local slope of the nanostrucutres.

In conclusion, in this experiment we demonstrated that the nanostructurization of metal surfaces is a general phenomenon and that CCD camera technique can be applied to follow in real time the evolution of the nanopatterns created by ion sputtering.