Micro-IBA analysis of Au/Si eutectic “crop-circles”

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When a thin gold layer is deposited onto the native oxide of a silicon wafer and is annealed at temperatures greater than 600 °C, peculiar circular features, few micrometers in diameter, with a regular polygon at the centre of each circle, reminiscent of so-called “alien” crop circles, can be observed.

A model has been recently proposed in Mathews et al. [1], where the formation of such circular structures is attributed to the interdiffusion of gold and silicon through holes in the native oxide induced by the weakening of the amorphous silica matrix occurring during the annealing process. The rupture of the liquid Au/Si eutectic disc surrounding the pinhole in the oxide causes the debris to be pulled to the edges of the disk, forming Au droplets around it and leaving an empty zone of bare silicon oxide.

In this paper, we present a morphological study and a RBS/PIXE analyses of these circular structures, carried out by scanning electron microscopy and by 4 MeV C microbeam, respectively. The results confirm the depletion of gold in the denuded circular zones, and the presence of gold droplets in the centers, which can be attributed to the Au segregation occurring during the cooling stage.

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1. Introduction

Solid-state dewetting of metallic thin films during annealing is a well-known phenomenon occurring by thermally activation of atomic diffusion and driven by interface film/surface energy minimization lowering. It consists in the agglomeration of the film during annealing, forming holes and droplets, randomly distributed through the film substrate. These metallic droplets can catalyze the growth of nanostructures to be used for numerous applications ranging from bio sensors to photonic devices [2,3].

One of the prerequisites for metallic thin film dewetting, is the presence of a diffusion barrier, which isolates the metal film from the substrate. If the substrate is silicon, CVD or thermally grown oxide effectively prevent any interdiffusion. However, if the barrier is given by a thin native oxide layer, the weakening of the amorphous matrix induced by the annealing process, causes the opening of pinholes, through which the metal can effectively diffuse into the silicon substrate, forming an eutectic layer, which spreads around the pinhole.

This is what we experienced during the deposition of thin (few tens of nanometers) Au film on silicon substrates, with nanometer thick oxide layers, annealed at temperatures greater than 600 °C. We observed peculiar circular features, surrounded by gold droplets and with a regular polygon at the centre of each circle, whose shape depends on the orientation of the substrate.

In a recent paper [1], a systematic investigation on the role of the annealing temperature and of the Au layer thickness allowed the formation mechanism of these circular nano-structures to be properly modeled: the opening of narrow channels in the native oxide induces the formation of a liquid AuSi eutectic layer with a thickness determined by the thickness of the deposited Au film. The central polygons occur in correspondence of the oxide channel openings and represent regions filled with a AuSi alloy which, during cooling, segregated into pure Au and Si. Their typical dimensions vary in the range of few micrometers and are related to the diameters of the circles, which extend up to few tens of micrometers.

In this paper, we report on a morphological investigation, which provides results in excellent agreement with those reported in [1] and extend the analysis to a differently oriented substrate. Moreover, since, to our knowledge, no elemental distribution analyses of these “crop circles” have been reported so-far, here we report