Production of Cu Nanodots by Ion Sputtering Cu on Mechanically Polished (110) Si-Substrates

C. Ballesteros*, A. Rodríguez** and T. Rodríguez**

*Departamento de Física, Escuela Politécnica Superior, Universidad Carlos III de Madrid, Avda. Universidad 30, 28911 Leganés, Madrid. Spain, balleste@fis.uc3m.es
**Departamento de Tecnología Electrónica, E.T.S.I.T, Universidad Politécnica de Madrid, 28040, Madrid, Spain

ABSTRACT

We report on a new method for the production of Cu nanocrystals by 5kV Ar^+ ion sputtering of Cu metallic ribbons at glancing incidence, on top of (110) Si-substrates. The size, shape, crystal structure, in-plane distribution, and concentration of the nanocrystals have been analysed by standard, high resolution, and scanning transmission electron microscopy. The Si-substrate surface topography induced by mechanical polishing favoured the formation of the Cu nanodots. The best results were obtained in (110) Si-substrates polished down to a 0.3 µm finish. The nanocrystals distribution was partially ordered, the nanodots were aligned along the scratch line pattern induced by the mechanical polishing.

Keywords: Nanodots, Ion sputtering, TEM

1 INTRODUCTION

Many of the interesting materials properties are controlled by the size, shape, and regularity of nanometric structures. The production of ordered nanostructures on top of semiconductor substrates in a simple and reproducible process is interesting for many research groups because of its potential applications [1,2].

The formation of nanocrystals structures or quantum dots, QD, with uniform size, a high density and lateral order is essential for technological applications in quantum devices. Among the hurdles which have to be overcome, lateral ordering is one of the most challenging issues. Metallic nanostructures have a high potential for the production of nanoscale electronic, optoelectronic or magnetic devices.

Techniques such as ion beam sputtering [3,4], self-assembling due to the Stranski-Krastanow growth mode and combined with anisotropic strain or ordering in the growth plane [5-8] have been used to obtain lateral order in QDs distributions. Most of these methods are time consuming, involving very often lithographic processes to modify the surface morphology or high vacuum and low temperatures in reconstructed surfaces, making them very valuable for basic research but not of easy implementation in factory work.

Recent results on the production of self-aligned metal nano-columns embedded in amorphous Al_2O_3 matrix [9] open new possibilities for easily accessible techniques of production.

In this communication we address the utility of a glancing parallel horizontal sputtering geometry onto (110)-Si substrates, modified by mechanical polishing to obtain a homogeneous distribution of Cu nanocrystals.

2 EXPERIMENTAL DETAILS

The sputtering experiments were carried out in the system described in figure 1. Cu nanocrystals have been produced by 5kV Ar^+ ion sputtering of Cu metallic ribbons at glancing incidence, 8º, on top of (110) Si-substrates mechanically polished down to a 0.3 µm finish. The Si-substrate was placed into a rotating specimen holder and maintained at liquid nitrogen temperature in an oil-free vacuum of $2x10^{-4}$ torr.

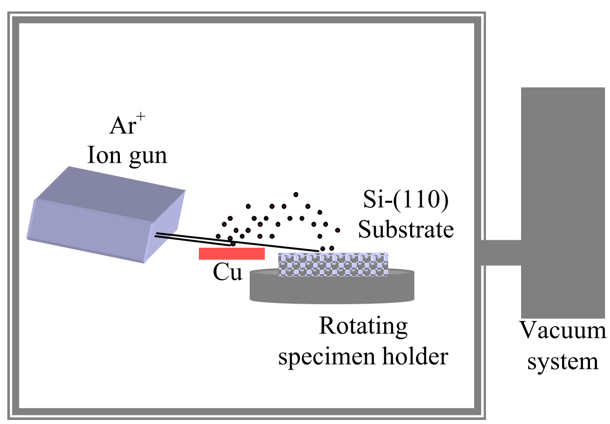

Figure 1: Scheme of the sputtering system with the geometry used in the experiments. The angle between gun and substrate was 8º.

SRIM program has been used for calculate the sputtering yield as a function of the incidence angle from normal incidence; the results are summarized in table 1.

TABLE 1

Incidence angle (º)	Yield (atoms/ion)
30	8.52
45	12.3
60	15.4
75	13.7
80	14.2
85	11.9

Plan view specimens suitable for transmission electron microscopy (TEM) were prepared by standard procedures: mechanical grinding, dimpling and ion milling in a liquid-nitrogen-cooled holder. The electron diffraction, energy-dispersive x-ray analysis and TEM-STEM images were carry out using a Philips Tecnai 20F FEG analytical microscope operating at 200 kV, equipped with STEM modulus and a dark field high angle annular detector (HAAD) for Z-contrast analysis.

3 RESULTS AND DISCUSSION

Figure 2a is a typical plan-view bright field electron micrograph of the sample, the corresponding STEM dark field, Z-contrast image is shown in figure 2b. Both images show a distribution of nanodots on the surface, the good match between dark and bright areas and the X-ray fluorescence spectra confirms the three-dimensional growth of the Cu dots. The crystal structure of the dots was analyzed by selected area electron diffraction, SAD, in very thin areas of the TEM sample. An example is shown in the inset of figure 2a. Using the substrate spot as an internal calibration the reflections are found to correspond to a Cu fcc crystal structure.

In both TEM and STEM Z-contrast images the nanodots distribution and the surface topography are visible. A partial alignment of the dots is also observed indicating that the distribution of the nanodot across the surface is affected by the line pattern induced by the previous mechanical polishing. The geometry of the sputtering system and the sample rotation favoured a partial milling of the surface by impacts of Ar^+ ions coming directly from the gun; as a result, the decrease of the substrate roughness and the homogenization of the surface during the deposition occur.

Figure 3 is a HREM micrograph showing a detail of one of the polycrystalline Cu nanocrystals where the polycrystalline structure of the dot is imaged. Moiré and lattice fringes are visible. The grain size varies from dot to dot and the average value is tens of nanometers.

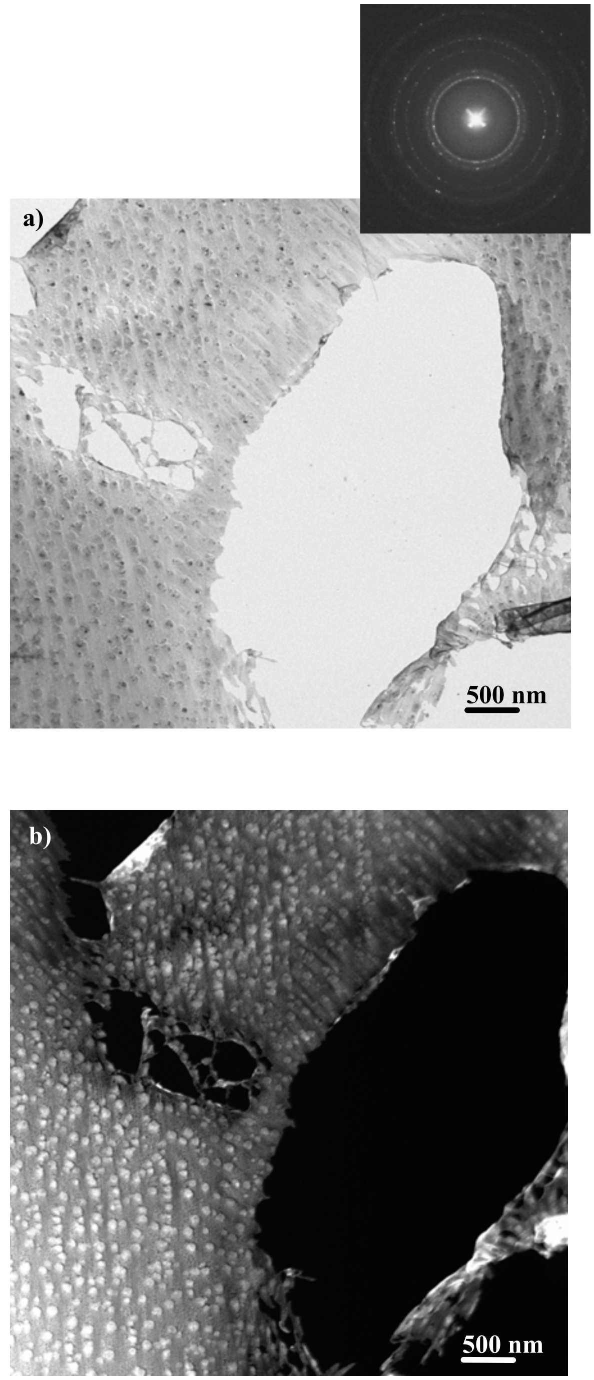

Figure 2: a) bright field electron micrograph from a region with nanodots. Inset Cu polycrystalline SAD pattern b) corresponding STEM dark field, Z-contrast image. The nanodots distribution on the surface is affected by the line pattern induced by mechanical polishing.

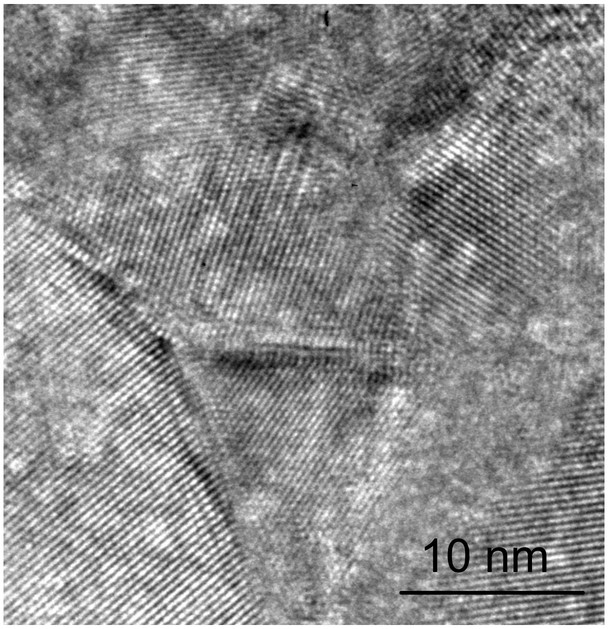

Figure 3: HREM micrograph of one of the polycrystalline Cu nanodots. The grain size varies from dot to dot. Moiré and lattice fringes are visible.

The measured average size of the nanodots was 50 nm and their density 10^{10} cm^{-2}. Selected area electron diffraction and convergent beam electron diffraction experiments indicate that the polycrystalline nanodots do not show any orientation related to the one of the Si-substrate.

The mechanical polishing of the substrate prior to ion sputtering forms scratches and favours likewise the oxidation of the surface and the formation of an amorphous Si oxide layer on top of the substrate. When depositing a metal on an oxide surface, the Volmer-Weber three dimensional growth is expected due to the lower surface free energy and interface energy of the metal with respect to those of the oxide, in good agreement with the distribution of polycrystalline Cu-dots described before.

To determine more accurately the Cu distribution on the surface, EDX mappings using the K_a and $K_ß$ lines of Cu have been made. An example is presented in figure 4. The EDX mapping for the K_a line of Cu and the STEM-HAAD Z-contrast image are shown. From the image, the distribution of the Cu on the surface can be associated with the presence of nanodots.

A very similar pattern distribution can be imaged for treatment times ranging from 60 min to several hours. This incomplete recovery of the surface is an indication of the interplay of several competitive mechanisms during the sputtering deposit.

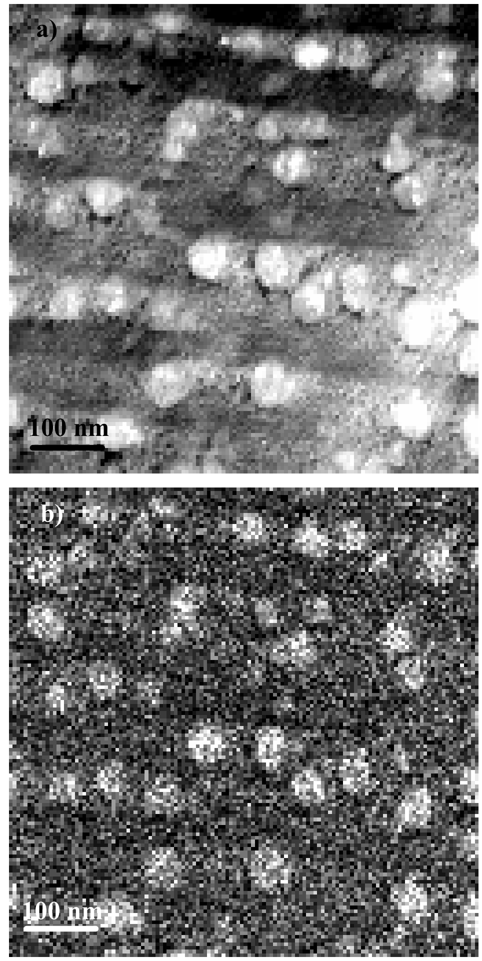

Figure 4: a) STEM Z-contrast image b) The EDX mapping for the Ka line of Cu of the same area.

Among those mechanisms we can suggest two: the direct impact of Ar^+ ions from the gun on the substrate and/or substrate + Cu deposited atoms, giving rise to a partial re-sputtering and the collision of the Ar+ ions with the Cu sputtered atoms during its flight, providing a driving force pushing the Cu atoms to the substrate. The compromise between both mechanisms is the responsible of the incomplete recovery of the substrate.

On the other hand, the line pattern induced by mechanical polishing in the substrate, changes its topography, favouring the milling along the scratches and modifying too the diffusion of the atoms. It is a common fact that in laterally ordered QD's distributions there is an anisotropic diffusion in the surface induced by surface reconstruction, anisotropic strain or ordering in the growing plane [5,8].

The line pattern and the geometry of the sputtering process can be adapted to other materials with interesting technological applications, like metal magnetic materials or semiconductors like Ge or Si.

4 SUMMARY

In summary we have shown that a distribution of polycrystalline metal dots can be deposited at the surface of Si substrates by a single sputtering process.

These results are promising and suggest that this simple method can be used in other materials. The glanzing incidence of the Ar^+ ions magnified the sputtering yield for most part of materials. The competitive mechanisms responsible for the formation of the dots distribution, based in the interplay between the roughening induced by ion sputtering and mechanical polishing and the smoothening due to partial milling of the surface and surface diffusion, can be controlled by the sputtering geometry and the ion beam energy, and open new possibilities for controlled deposition of laterally ordered distributions of nanocrystals.

5 ACKNOWLEDGMENTS

This work was funded by Spanish Government through CICYT Project MAT2004-04580-C02-01. TEM work has been made at the LABMET associated to Red de Laboratorios de la Comunidad Autónoma de Madrid. The help of M.I. Ortíz with TEM sample preparation is acknowledged.

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