Energy-filtered PEEM imaging of polycrystalline Cu surfaces with work function contrast and high lateral resolution *

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Using nanoESCA, a novel energy-filtered PEEM instrument equipped with an aberration corrected energy filter, we have imaged micro-sized (1 μm) polished copper grains with UV (4.9 eV) and VUV (21.1 eV) radiation. Energy-filtered UV-PEEM images allow to reach a lateral resolution of 40 nm. From series of energy-filtered images, reconstructed VUV microspectra taken over typical areas of 600 nm², directly show up variations in the local work function, on an absolute energy scale, of grains having different crystalline orientation. Work function values measured from fitting of the photoemission threshold are consistent with $\langle 111 \rangle$, $\langle 100 \rangle$ or $\langle 110 \rangle$ crystalline orientations (work function of 4.9, 4.6, and 4.5 eV, respectively). This experiment opens new possibilities in the local characterization of surfaces in the sub-micron range with high energy- and lateral resolutions.
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I. INTRODUCTION

Copper has an increasing importance in micro- and nanoelectronics, since it replaces progressively aluminium for interconnects. The necessity to use a barrier layer that can prevent diffusion into the active semiconductor and the preparation process lead to the crystallization of the copper film in microstructures which present different crystallographic orientations (damascenes lines). Electro-migration and resistivity of the interconnect depends on the microstructure and the texture of the layer [1, 2]. In addition, the increase of copper lines resistivity for line width below 100 nm still needs to be understood. This will require the implementation of spectroscopic techniques with high spatial resolution.

In the present work, we used a photoemission electron microscope (PEEM), known to achieve ultimate lateral resolutions in the 10-20 nm range, in order to image the microstructure of polycrystalline copper layers used for interconnects. Using UV photons, PEEM can produce on flat samples images with work-function contrast, which is directly related to the local grain crystallographic property. The PEEM was coupled with a novel band-pass energy filter for aberration corrections. Reconstructed nanospectra directly show up variations in the local work function of single grains.

II. EXPERIMENTAL

Copper blanket films of 1.5 μm thickness were electroplated uniformly on a TaN/Ta barrier layer seeded with a 100 nm thick layer of sputtered copper, and then annealed at 400°C, 6 Torr for 30 min. Then, a CMP step (Chemical and Mechanical Polishing) was performed [3] in order to obtain a very flat surface (rms roughness of 0.4 nm as measured by Atomic Force Microscopy); this provided ideal conditions for PEEM imaging [4]. Electron Back-Scattering Diffraction on the same samples revealed the presence of copper grains having $\langle 111 \rangle$, $\langle 110 \rangle$ and $\langle 100 \rangle$ orientations perpendicular to the surface.

For the energy-filtered PEEM imaging, we used a nanoESCA instrument. It is a new model developed in co-operation between two companies (Omicron Nanotechnology GmbH and Focus GmbH) and two universities. It consists in a standard, fully electrostatic PEEM electron optical column coupled with two hemispherical analyzers in an antisymmetric tandem configuration. This set-up corrects the leading angular aberration term (α^2 -term) of the first analyzer and gives an achromatic image at the exit of the second analyzer; it also retains the time structure of the electron signal to a much higher degree. The lateral resolution obtained in an energy filtered mode and with synchrotron radiation X-rays is presently 120 nm [5]. Using a laboratory UV source, a lateral resolution of 40 nm in the energy filtered mode has been demonstrated [6]. Besides enabling high lateral resolution, the main advantage of the energy filtering PEEM mode compared to conventional PEEM is the possibility to collect photoelectron spectra related to a single grain, and obtain a true measurement of the local work-function by analysis of the secondary emission onset, without relying on the photoemitted intensity.

In our experiments, the extractor voltage used in the

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FIG. 1: Energy filtered images of the Cu/TaN/Ta surface after 5 min sputtering. Image field of view : 15 μm . Kinetic energy of the photoelectrons : 4.45 eV (a) and 4.9 eV (b).

PEEM column was 12 kV and the sample-extractor lens distance was 1.8 mm. This generated an electric field at the sample surface of 6.67 kV/mm, thus lowering of the work function due to the Schottky effect [7]. The sample was sputtered with Ar^+ ions at 500 eV in a pressure of 10^{-7} mbar during several cycles lasting 10-20 minutes to remove the native oxide layer. The total sputtering time was 75 min. The sample was then illuminated with two lamps (Hg at $h\nu = 4.9$ eV and HeI at $h\nu = 21.2$ eV) successively, at an impact angle of 80° off-normal. Series of images were taken at increasing kinetic energies of the secondary photoelectrons with a step of 0.05 eV. Then, several plots of the photoemitted intensity versus the photoelectron kinetic energy, were performed. In addition to topographic images, micro- or even nano-spectroscopic informations were thus obtained and treated to extract the local work function without relying only on the secondary electron intensity [8]. Analysing the onset of the true secondary photoelectrons excited by higher photon energies is suitable for the determination of the work-function [9].

III. RESULTS AND DISCUSSION

Figure 1 shows two energy-filtered PEEM images of the sample after uncomplete sputtering process (5 min. duration), recorded with the Hg lamp at two different kinetic

FIG. 2: Energy filtered images of the Cu/TaN/Ta surface after 75 min sputtering. Image field of view : 35 μm . Kinetic energy of the photoelectrons : 4.45 eV (a) and 4.9 eV (b).

energies of the photoelectrons above the Fermi level : 4.45 eV (Fig. 1(a)) and 4.9 eV (Fig.1(b)). Copper grains exhibiting different intensities can be clearly distinguished on both image. The first image taken with photoelectrons of 4.45 eV kinetic energy looks essentially the same as the second image taken with photoelectrons of 4.9 eV kinetic energy : the contrast between different grain does not vary in image (b) with respect to image (a). As copper work function ranges from 4.4 to 4.9 eV depending on grain orientation [10], one would expect contrast variations between image (a) and (b) arising from differences in grain work function due to the different crystalline orientations. More precisely, the $\langle 111 \rangle$ grains of highest work function should appear dark at 4.45 eV and bright at 4.9 eV, and the opposite for the $\langle 110 \rangle$ grains (work function : 4.50 eV).

This is obviously not the case here. The limited surface cleaning due to the uncomplete sputtering process, with most probably still an important amount of copper oxide remaining at the surface, is responsible for this result.

Figures 2(a) and (b) present the same energy filtered images than in Fig. 1 but after an extended sputtering treatment of 75 min. The photo-emitted intensity of the grains varies significantly on these two images. The high photon flux intensity of the Hg lamp permits to discriminate by contrast different groups of grains. At such low-

FIG. 3: Nanospectra corresponding to the different grains marked with an arrow on the images of Fig. 2, and obtained with the Hg (a) and He (b) lamps. The arrows a and b indicate on the energy scale where the images a and b were taken.

TABLE I: Measured work function of the copper grains and comparison with literature data.

Grain number	Measured Φ (± 0.02 eV)	Literature Φ (eV)	Orientation
1	4.8	4.9	(111)
3	4.5	4.6	(100)
2	4.4	4.5	(110)
all	4.55	4.65	polycrystal

energy excitation, and considering (i) the very low surface roughness, (ii) the homogeneity of the intensity of the incident beam on the illuminated area, and (iii) the absence of polarization, the contrast arises principally from the local work function [4], due to the differences in crystallographic orientations. Particularly, we focus on three grains, as indicated with arrows number 1, 2 and 3 in Figs. 2(a) and (b), that are representative of the trends observed in the variations of the intensity between 4.45 and 4.9 eV : the intensity of Grain 1 and 2 is reversed but the opposite way, while intensity of Grain 3 shows up less variations. The corresponding nanospectra, taken over a

typical area of 600 nm² are shown in Fig. 3(a), and reflect the different behaviour observed for grain 1, 2 and 3. The photoemission threshold appears clearly shifted depending on the considered grain number, as a result of different intrinsic work function. Thus, the microspectra allow a first, *qualitative* assignment of the crystalline orientation of grain 1, 2 and 3 from the evolution of the threshold energy and the data from literature [10] given in Table I : Grain 1 and 2, with respectively the highest and the lowest work function, should then be (111) and (110) oriented. However, the low photon energy emission of the Hg source does not excite secondary electrons well separated in energy from characteristic direct transitions from the copper *d*-bands.

In a second step, complementary micro-spectra of the same grains presented in Fig. 3(b), were taken using the HeI radiation at 21.2 eV in the photoemission threshold region; this time, the higher excitation energy allows a clear separation of the secondary electrons signal from direct transitions. The energy position of the different thresholds appears unchanged compared to the mercury microspectra. The work function can be estimated by fitting the threshold with a Heaviside function convoluted with a Gaussian function [11]:

$$I(E - E_F) = 1 - \operatorname{erf}\left(\frac{\Phi - (E - E_F)}{\sigma\sqrt{2}}\right), \quad (1)$$

where Φ is the work function, σ is the standard deviation of the Gaussian due to the energy broadening of the analyzer at the pass energy used, and $E - E_F$ is the photoelectron kinetic energy above the Fermi level, measured at the He I excited-Fermi edge of a clean silver sample. The overall accuracy of the fitting procedure was determined to be ± 20 meV.

The results are given in Table I, taking into account the correction arising from work function lowering by Schottky effect as mentioned in Section II. The correction factor is estimated from the following equation :

$$\Delta\Phi = -e\left(\frac{eE}{4\pi\epsilon_0}\right)^{1/2}, \quad (2)$$

and yields a lowering term of 98 meV. The estimated work function values exhibit a systematic negative shift (-100 meV) relative to literature data [10]. The value extracted from the threshold of a spectrum taken over the whole image is also 100 meV below that expected for polycrystalline copper with random grain orientation. Therefore, the observed shift is not due to a local effect due the choice of particular grains, and may be due to defects created all over the surface by the Ar⁺ sputtering. Indeed, it has been shown that a little number of defects can lower the work function by as much as 300 meV [12].

Nevertheless, these results confirm the initial qualitative assignment of the crystallographic orientations of grain 1, 2 and 3, and are consistent with independent analysis using Electron Back-Scattering Diffraction. They highlight the benefit of VUV spectromicroscopy with nanoESCA to extract local electronic informations in the sub-micron range.

IV. CONCLUSIONS

With nanoESCA, a novel energy-filtered PEEM instrument, we have imaged micro-sized polished copper grains using UV (4.9 eV) and VUV (21.1 eV) radiation. Reconstructed VUV microspectra directly show up varia-

tions in the local work function of grains having $\langle 111 \rangle$, $\langle 100 \rangle$ or $\langle 110 \rangle$ crystalline orientations. This experiment opens new possibilities in the local characterization of surfaces with laboratory instruments in the decanano-metric range with high energy resolution.

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