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# Topography induced by sputtering in a magnetic sector instrument: an AFM and SEM study

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#### Abstract

Due to the sensitivity, the good depth resolution and the great interest in ultra shallow profile, secondary ion mass spectrometry (SIMS) is one of the prime techniques used in the semiconductor industry. Low impact energy beams are required to profile shallow distributions. Since  $Cs^+$  beam sputtering can cause morphological artifacts as well as  $O_2^+$  beam does, a detailed study is required to understand development and limiting analytical conditions. In this work we analyzed the effect of low energy  $Cs^+$  primary beam incident at 68° and 78° on different silicon samples. By using atomic force microscopy (AFM) and scanning electron microscopy (SEM) we underline their reliability and correlate the morphological effects to the SIMS analytical parameters and samples characteristics.

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# 1. Introduction

Secondary ion mass spectrometry (SIMS) is based on ion sputtering process. By removing atoms layer by layer we can get a satisfactory depth distribution analysis.  $Cs^+$  and  $O_2^+$  incident beams are usually utilized to obtain depth distributions of electronegative and electropositive elements, respectively. Nowadays low impact energy and glancing angles are recommended to obtain the adequate depth resolution for semiconductor ultra shallow characterization [1]. However, in some conditions ion bombardment can produce a change of surface topography on the crater bottom, causing problems in quantitative analysis and

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depth resolution deterioration. The induced morphology by ion sputtering requires detailed characterization. Roughness formation induced by  $O_2^+$  beam was widely investigated [2–7] underlining the strict correlation between ripples formation, incidence angle, and samples material. Less work has been done on topography change during Cs<sup>+</sup> depth profile analysis [8–11]. Most of these works are based on quadrupole SIMS with delta layered silicon sample. They underline the correlation with incident angle and impact energy. Wider analyses on different samples are necessary.

SEM and AFM are the most used techniques to analyze silicon crater bottom. Their qualitative and quantitative analyses contribute to compose a complete description of the corrugation development. In this work topographic irregularities induced by low impact energy SIMS are studied, showing the

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dependence on sputtering condition. SEM technique limit is outlined. SIMS transient effect on matrix signal is correlated to roughness trend.

#### 2. Experimental

The investigation was performed on three types of substrate. The first one is of a monocrystalline Si substrate terminated with native oxide. The second type of sample is composed by boron delta-doped Si multilayers CVD grown [9]. This sample, characterized by TEM, presents a first set of five deltas 5.8 nm spaced, with the first delta distant 16.4 nm from the surface, and a second deeper set of five deltas 17.8 nm spaced. The last one is 10 nm of silicon oxinitride  $(SiO_xN_y)$  grown on silicon.

SIMS depth profiles were collected by a magnetic sector CAMECA SC-ULTRA using 1 keV Cs<sup>+</sup> primary ion beam rastered over an area of  $250 \,\mu\text{m} \times 250 \,\mu\text{m}$ . The sputtering time has been varied stopping the analysis at four different crater depths. Each series of four craters has been obtained at two different impact angles:  $78^{\circ}$  and  $68^{\circ}$ .

The final crater depths have been measured by a mechanical stylus profilometer Tenkor P15 in order to determine the sputtering rate necessary to calibrate the depth axis.

After depth profiling, the topography of the sputtered crater bottoms was explored by AFM using a UniSolver from NT-MDT. The system is equipped with a scanner head  $(10 \,\mu\text{m} \times 10 \,\mu\text{m} \times 2 \,\mu\text{m})$  for high resolution measurements. AFM images were collected over  $1 \,\mu\text{m} \times 1 \,\mu\text{m}$  areas using tapping mode.



Fig. 1. SEM (a, b) and AFM (c) crater bottom images: 13 keV (a) and 7 keV (b, c)  $Cs^+$  primary ion beam. (d) shows a cross section referred to dashed line in (c).



Fig. 2. Waviness (left y-axis) and ripple height (right y-axis) vs. primary energy beam; associated  $1 \mu m \times 1 \mu m$  AFM images are shown. Grey arrow points out cesium ion beam direction.

Surface roughness is presented in the form of roughness (rq) and mean square deviation (ra).

### 3. Results

SEM analyses were performed using a Jeol JMS 6100 microscope.

SEM analysis seemed a very useful approach to reveal SIMS induced ripples. In several works



Fig. 3. Roughness behaviour of the three samples (ra: mean square deviation and rq: roughness) splitted for different incident angle:  $68^{\circ}$  (above) and  $78^{\circ}$  (below).

[1,12–14] SEM images are used to characterize SIMS crater bottom. In Fig. 1a and b for example, SEM images on 13 and 7 keV Cs<sup>+</sup> sputtered sample successfully describe the ripples formation. Viceversa SEM approach seems not reliable to analyze ripples formed by low energy (1 keV or below) ion beam since decreasing primary ion energy beam leads to a reduction of ripple height, whereas AFM analysis is still able to determine quantitatively height and ripple waviness. In Fig. 1c and cross-section (Fig. 1d), AFM image exhibits well defined rippled surface, about 1–2 nm high, 70–80 nm width.

Present work is devoted to characterize samples below this roughness range. Just for a comparative purpose we show in Fig. 2 the roughness trend versus  $Cs^+$  ion beam energy considering also two crater bottom sputtered at higher impact energy on first sample. The analytical conditions required (in a magnetic sector mass spectrometer angle and impact energy are strictly correlated) for a deep profile (13 keV, 58°; 7 keV, 58°) induce wide and deep structures. On the other hand we can note that decreasing impact energy down to 1 keV and using 78° impact angle, analytical condition suitable for shallow profile, both roughness and ripple waviness are reduced.

Roughness behaviour related to the three samples is shown in Fig. 3 where data are splitted for  $68^{\circ}$  and  $78^{\circ}$ analyses. Data sequence points out a clear increase in roughness according to a crater depth increase. We notice slight differences between  $68^{\circ}$  and  $78^{\circ}$ . At  $78^{\circ}$ impact angle all samples are aligned on the same behaviour, whereas at higher impact angle, data show more spreading. A grazing angle is supposed to be more sensitive to the surface oxide thickness and silicon structure.

Fig. 4a shows the SIMS profile on boron deltadoped Si sample. Near the surface, matrix signal exhibits a slight variation. This could be caused by a reduction of the sputtering rate that also affect boron signal with a small shift on deltas position due to roughness formation. On silicon native oxide (figure not shown) although less pronounced, the same behaviour is observed. Moving on depth profile, larger roughness variations are observed for more grazing angles causing a more dramatic delta shift.

Regarding third sample, 10 nm silicon-oxinitride, as seen in Fig. 3, roughness behaviour at  $68^{\circ}$  impact angle is similar to others. On the contrary at  $78^{\circ}$ 



Fig. 4. (a) SIMS profile obtained on delta sample by 1 keV  $Cs^+$  beam at two different angles. Black triangles indicate delta positions as determined by TEM analysis. Star symbols show where SIMS analyses were stopped and consequently analyzed by AFM. (b) SIMS profile obtained on oxinitride sample by 1 keV  $Cs^+$  beam at two different angles.

grazing angle, data present more spread caused by a bad point found at 6.9 nm. A clear correspondence is present in SIMS measurements (Fig. 4b) where we note an unexpected matrix signal variation. It is probably caused by a sputtering rate variation due to a compositional variation.

# 4. Conclusion

SIMS roughness evolution in the upper layer of the three different substrates, interesting from semiconductor technology point of view, has been studied under  $Cs^+$  bombardment. Ripple dimensions and density are function of sputtering energy and angle, in particular ripple waviness decreases following energy reduction. The results show that ripples formation starts in the early stage of sputtering process. Roughness evolution induced by 1 keV and 68° sputtering beam seems independent from sample matrices. Viceversa roughness induced by 78° beam shows more spreading values caused by a major sensitiveness on the surface condition. In deeper profile, roughness induced by 78° beam is bigger than roughness obtained by using beam at 68°.

Future work will be devoted to determinate physical principles of the morphology development.

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