In situ sputtering rate measurement by laser interferometer applied to SIMS analyses.

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Abstract. The heterodyne laser interferometer installed on the new Cameca SC-Ultra SIMS apparatus permits an in situ depth evaluation during depth profiling. The aim of this work is an investigation of the laser interferometer advantages and limitations for profiling of dopants in silicon. The laser depth calibration has been compared with the one determined by measuring the crater final depth with a stylus mechanical profilometer. Some experimental conditions where this approach can provide accurate results have been identified and confirm the usefulness of this device.

INTRODUCTION

The depth calibration in Secondary Ion Mass Spectrometry (SIMS) has been usually carried out ex situ by measuring the final crater depth by a mechanical profilometer and then converting the sputtering time to depth supposing a constant erosion rate. In the new magnetic sector SIMS Cameca SC-Ultra [1] an in situ laser interferometer device [2-4] has been introduced and it can give the possibility to evaluate point by point the crater depth during the sputtering process. In the first instance this tool should correlate every secondary ion signal data point with its own depth. In this way it should be possible to reduce the relative shift of the profiles due to delay time in the sequential acquisition of different species typical of magnetic sector mass spectrometers. Moreover this laser system could overcome the issue of sputtering rate changes during multilayer analysis: the thickness of every opaque layer (30% reflectivity required) could be easily measured [3, 4]. Recently large variations of sputtering rate have been detected also in silicon depth profiling by oxygen sub-keV oblique incidence beam or by glancing cesium beam [5, 6]; therefore an in situ sputtering rate evaluation could turn out very useful in such technologically interesting samples as ultra shallow boron and arsenic implants. Finally the in situ depth measurement allows saving time in data processing by providing the depth scale calibration without the need of a final crater evaluation. In order to check the capabilities of this device we pointed our attention to some particular analytical conditions used to investigate dopant depth distributions in silicon. We investigated both cesium and oxygen primary beam analyses. In deep analyses we experienced inaccuracies, perhaps due to induced roughening of the bottom surface and to contribution of crater walls to laser reflection. In shallow depth profiles the effect of the native oxide influenced strongly the depth calibration: in fact the silicon oxide is transparent to the wavelength of the laser (λ=633 nm) and an initial delay in depth measurement has been noticed in some cases. Moreover we confirmed the instantaneous sputtering rate oscillations with a regular λ/4 period already reported by De Chambost et al. [4]; the origin of this noise is still under investigation. Nevertheless even in such cases an accurate measurement of the average sputtering rate in silicon is possible and this turns out to be an undeniable saving of time.

EXPERIMENTAL

The laser interferometer system installed on the SC-Ultra apparatus produces two parallel and orthogonally polarized beams, with two frequency components, collimated on the sample surface; a first spot is focused in the crater bottom whereas a reference spot is reflected from the outside surface. A piezoelectric z-
positioning system combined with an autofocus device allows the right sample alignment. The distance between the two spots is about 200 \( \mu \text{m} \) from each other. The wavelength of the heterodyne laser (\( \lambda = 633 \) nm) ensures a theoretical resolution of 0.62 nm; the double beam reflection geometry produces a phase shift periodicity of \( \lambda/4 \) between the laser head and the detector. During the sputtering the phase shift of the two beams recombined on the detector causes a frequency shift of the detector signal that is converted to a path length difference [2]. Therefore depth and sputtering rate can be determined point by point at each instant.

The mechanical profilometer used for depth calibration is a KLA Tencor P15, where the vertical movement of the stylus arm is sampled by a capacitive sensor. The nominal vertical resolution of that instrument is 0.5 \( \AA \); the repeatability is better than 8 \( \AA \).

Oxygen SIMS analyses have been performed at 3 keV, 1 keV and 500 eV of impact energy; the respective incidence angles are 65°, 76° and 68° (with reduced extraction voltage for 500 eV) with respect to the surface normal. The cesium primary beam was at 1 keV and 500 eV impact energies with 45° and 44° incidence angles and we collected secondary ions. In each measurement the sputtering crater width was set at a value compatible with laser spot positions and size, i.e. smaller than 300 \( \mu \text{m} \) and larger than 40 \( \mu \text{m} \) in order to avoid sputtering near the reference spot or adverse reflections from crater edges.

To investigate roughness induced effects, such as sputtering rate variations and shifting of depth scale, we performed analyses on a boron delta-doped Si multilayer. The sample was grown by Reduced Pressure Chemical Vapor Deposition (RP-CVD) at a temperature of 725°C [7] and it was then characterized by TEM. It 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. On this sample the oxygen measurements have been carried out with and without an oxygen leak (chamber pressure 9.6x10^{-6} Torr).

The thickness of oxide on silicon surface has been measured by X-ray Photoelectron Spectroscopy (XPS) through the experimental intensity ratio of the oxidic and metallic Si 2p peaks. XPS spectra were obtained by a SCIENTA 200 equipped with a AlK\( \alpha \) monochromatic x-ray source and hemispherical electron energy analyzer.

\[ y = 0.99 x - 4.55 \]
\[ R = 0.990 \]

O\( _2 \)\(^{+} \), 1.0 keV @ 46°, vacuum

\[ y = 0.99 x - 4.55 \]
\[ R = 0.990 \]

Cs\(^{+} \), 1.0 keV @ 46°, vacuum

FIGURES 1-3. Final crater depths measured by laser interferometer vs. depth determined by stylus mechanical profilometer, for oxygen at 3 keV, 1 keV and for cesium at 1 keV respectively. Best linear fit is also reported.
RESULTS AND DISCUSSION

A first set of analyses has been carried out on several implants in silicon, in order to obtain a calibration curve of the laser measured depth. Comparing the final crater depths evaluated by laser with those measured by mechanical profilometer it was possible to estimate the linearity of the calibration curve, hence the accuracy of this technique; this linear fitting procedure was separately repeated for both oxygen and cesium primary beams and the results are reported in Figures 1-3. In both cases the best fit is achieved by a line with slope close to 1 and a negative offset that depends on the primary species and impact energy; the offset is larger for Cs and for higher energy. This behavior could arise from an initial delay of the laser measured depth because of the transparency of surface oxide to the wavelength used. In fact it is important to note that the stylus profilometer measures the difference between the surface and the real crater bottom whereas the laser interferometer reveals the difference between two reflecting points. This tool can not measure any significant values until the interface between oxide and silicon has not been reached. XPS measured oxide thickness (average value for analyzed sets of samples) is 2.5 nm and therefore it could really be an influencing factor of the laser response. Nevertheless the presence of a thin oxide can not by itself explain the offset because it depends also on the used analytical conditions and not simply on samples. Figure 4 shows the example of in situ measured depth against primary ion dose in analyses performed at 1.0 keV with Cs⁺ and O₂⁺ beams. In the inset the laser delay is evident and the first measured values by interferometer are negative: this fact suggests that the origin of the offset could be a convolution of several factors: the presence of oxide, the strong modification in surface composition induced by the primary beam and the consequent variation of the surface optical reflectivity, the ion mixing at the interface native oxide/substrate. Further investigations are necessary to understand these factors.

In Figure 5 we had confirmation of a periodic noise with depth of the laser measured sputtering rate, already noted by de Chambost et al.[4], despite the high primary beam stability. There is an amazing coincidence between this periodicity (estimated measuring the distances between peaks and valleys) and λ/4 value, i.e. exactly 158.7 nm. The resulting maximum deviations of the laser depths from the real depth values estimated by a linear fit are 1.8 nm, in good agreement with peak to peak variations of 3.8 nm given by de Chambost. Consequently the relative error caused by laser noise is negligible, lower than 1%, for craters deeper than 180 nm. Therefore in deep profiling it is suggested to sputter at least 200 nm and then apply a constant sputtering rate as the mean value on the whole range.

By performing analyses on boron delta layers, we identified a second regime of measurement in which the laser interferometer shows noticeable advantages, despite its periodic noise. In fact it is known that oxygen sub-keV oblique beam causes a strong sputtering yield variation in the early nanometers of the profile [5], hence profiles are shifted towards the surface if a constant Sputtering Rate (SR) is used. The analysis of delta doped multilayer samples allows observation of the SR variations by comparing the
delta intervals, but in depth profiling of samples of technological interest we have to face the lack of references to correct the depth scale. Instead we observed a clear advantage in applying the in situ sputtering rate in the first range of the profiles, well before the first half period of $\lambda/4$ (80nm).

In Figure 6 we report boron depth profiles obtained on a multilayer sample; the dashed curve represents a profile with depth calibrated by using a constant SR whereas the continuous one by using laser point by point measured values. The analysis has been carried out with a 0.5 keV oxygen beam and by introducing an oxygen leak. The dashed curve turns out shifted toward the surface as expected whereas the agreement of laser calibrated delta positions with TEM values is quite good for the first six deltas; in fact the interferometer was able to detect the variation of SR that occurred at the start of the analysis. The $\lambda/4$ periodic oscillation of the laser measured SR could introduce some artifacts but until 50-60 nm the induced error is relatively lower compared to the one caused by supposing a constant erosion rate. Therefore the laser signal can provide useful information at the early stages of oxygen sub-keV analysis and this turns out to be a very powerful tool in depth profiling shallow implants. The accuracy of the interferometer has also been tested by repeating the analysis of the same delta doped sample by using a 1 keV cesium primary beam and monitoring SiB (Figure 7). In this case we did not observe any initial SR variation and the delta positions turn out well after a rigid depth shift of 2 nm. This shift, lower than the one determined by the linear fit of fig. 3 (4.5 nm), could be determined by

![FIGURE 6. Boron depth profiles obtained on delta doped RP-CVD grown sample by O$_2^+$ primary beam at 0.5 keV of impact energy. The dashed curve was calibrated in depth by a constant sputtering rate whereas the continuous curve by the in situ measured depth. In situ sputtering rate is also reported.](image)

**FIGURE 7.** Boron depth profiles obtained on delta doped RP-CVD grown sample by using a 1 keV Cs$^+$ primary beam and monitoring $^{28}\text{Si}^{11}\text{B}$. In situ evaluated sputtering rate is also reported.

### CONCLUSIONS

The in situ depth measurement tool installed on the new Cameca SC-Ultra has been evaluated as an useful system to calibrate the depth axis in silicon depth profiling. We tested its applicability on analyses carried out with both cesium and oxygen primary beams at energy ranging from 3 to 0.5 keV. We identified two approaches for two different regimes. In deep depth profiling one faces the periodic noise of laser measured sputtering rate. Nevertheless the use of averaged SR gives an error on depth less than 1% if the total sputtered depth is greater than 200 nm. Therefore it is not necessary to measure the final crater depth by a stylus profilometer with the consequent advantage also in saving time.

In the shallow depth profiling regime we found the great advantage of measuring variation of SR when we use an oblique incidence sub-keV oxygen primary beam. Also for this difficult task a more accurate depth calibration is obtainable. In some cases a starting delay of laser measurement could affect the depth calibration and therefore the in situ depth has to be adjusted in the initial 1-2 nm region.
ACKNOWLEDGMENT

The authors would like to thank Philippe Holliger and Frederic Laugier of CEA-Leti, Grenoble France, for providing the RP-CVD grown multilayer samples.

This work was supported by European Project IMPULSE, IST-2001-32061.

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