

## DEPTH SCALE CALIBRATION OF SIMS DEPTH PROFILES BY MEANS OF AN ON-LINE CRATER DEPTH MEASUREMENT TECHNIQUE

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**Abstract :** The depth scale calibration of a SIMS depth profile requires to determine the sputter rate used for the analysis from the crater depth measurement. An in-situ crater depth measurement system based on the heterodyne laser interferometer has been developed. Experimental results demonstrate crater depths can be measured from nanometers to micrometers range with an accuracy better than 5% in different matrices and a repeatability of 1%.

**Keyword :** depth profile, depth scale calibration, laser interferometer, crater depth. Application examples for SiGe samples are presented.

### 1. Introduction

The data quantification of SIMS depth profile consists in converting qualitative data *Intensity vs. Time* into quantitative data *Concentration vs. Depth*. The accuracy of this quantification depends on the knowledge about the sputtering and ionization processes occurring during SIMS measurements. For depth profiles through homogeneous samples, constant sputtering and ionization yields are commonly assumed with a rather good confidence. But when more complex samples have to be analyzed, such simple models cannot describe the actual physical processes and reliable quantification requires more sophisticated data interpretation. For the depth scale calibration, 2 different methods can be distinguished:

- i) Off-line determination of the sputtering rate (SR) either from a reference sample or from crater depth measurements. These techniques lead to precise scale calibration provided that the instrument is stable and the sample structure are consistent with a constant SR assumption. Note off line technique slows down the data quantification process.
- ii) In-situ and continuous crater depth record for the profile measurement [1]. Such a real time SR monitoring offers a calibration precision which is not sensitive either to the instrument stability or to any SR change because of the sample structure. Moreover it is an on-line technique and therefore shortens the quantification procedures.

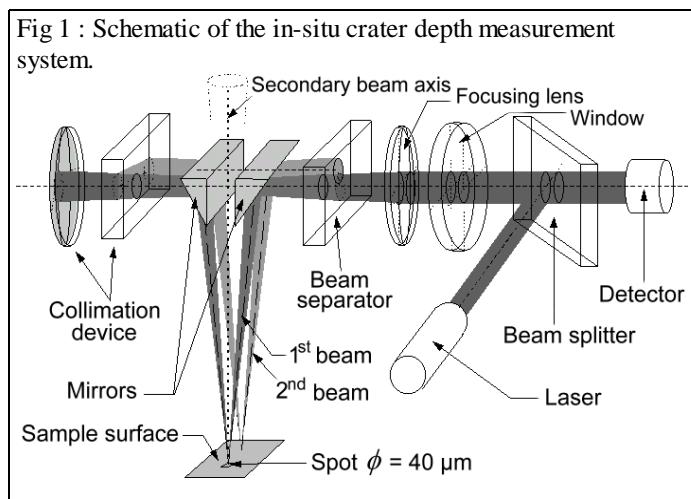
This paper presents results of depth calibration of SIMS depth profiles by means of a laser interferometer developed by CAMECA. The basic principle of the experimental set-up is described as well as the advantages and the limits of this technique are discussed in terms of accuracy and repeatability. Applications to SiGe alloys are also presented.

### 2. Experimental

The laser interferometers used for this work were mounted on CAMECA IMS Wf instruments. The basic principle of the optical system of the interferometer is shown in Figure 1. The heterodyne laser head produces a laser beam consisting of two coaxial laser beams with two very close frequencies (frequency shift of  $10^{-8}$  order) with an orthogonal polarization. Part of this beam is sent to a photo-detector fitted into the laser head to be used as a reference signal (REF).

At the output of the laser head, the laser beam is split in two beams according to their polarization and both are focused at the sample surface in spot with a gaussian diameter of 40  $\mu\text{m}$ . One beam is focused at the bottom of the crater and the other at the sample surface. The distance between the two spots is 200  $\mu\text{m}$ . After a double reflection on the target, the two

beams are recombined to form the return signal (RTN) sent to the detector of the interferometer. If the two targets (sample surface and bottom crater) are not moving relative to each other, the two signals (REF and RTN) have a stable phase shift. When the relative position of the targets changes, the monitoring of the phase shift allows to record the depth variation of the bottom crater. The double beam reflection geometry yields to a period phase shift corresponding to a path difference of  $\lambda/4$ . Thus, with a laser wave length of 633 nm and a phase resolution of 1/256, the theoretical resolution is 0.62 nm which has been experimentally checked on a bench test. The measurable crater depth range spreads from nanometers to micrometers. The two beams concept makes the system very little sensitive to mechanical vibration and thermal dilatation. Note that this design introduces some constraints :



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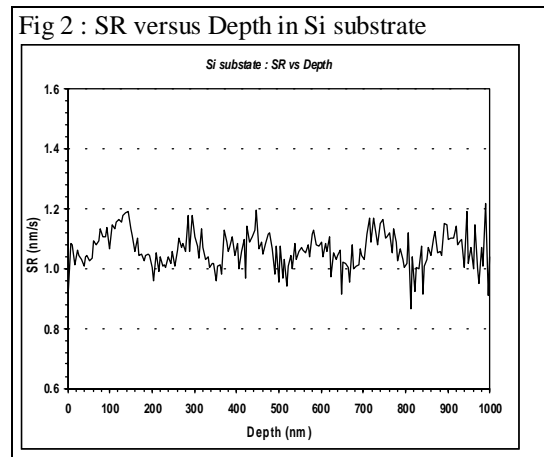
- i) The distance and the size of the laser spots on the target require to use a raster size smaller than 300  $\mu\text{m}$  (in order to avoid the overlap between the crater and the reference spot) and larger than 125  $\mu\text{m}$  (in order to keep negligible crater edge contribution on the measurement spot).
- ii) The crater depth is a mean value measured over the 40  $\mu\text{m}$  of the laser spot size.
- iii) The sample surface and the bottom crater must have a reflectivity of at least 30%.
- iv) Crater in transparent materials cannot be measured. Note that in the case of a thin transparent layer on the top of an opaque substrate, the system still works but the two laser beams are no longer reflected at the surface but at an apparent surface located between the surface and the interface transparent/opaque materials. The exact position of this apparent surface is unknown (it depends on the relative reflection coefficient and refractive index) and this introduces an error on the crater depth smaller than the transparent layer thickness.

The craters were sputtered with  $\text{O}_2^+$  and  $\text{Cs}^+$  primary ions at different impact energies. The accuracy of the laser interferometer has been evaluated by comparison with measurement performed with a surface profilometer DEKTAK 3ST daily calibrated with a step reference sample of 473 nm. This calibration procedure had been previously qualified with 3 magnification standards (issued by Taylor Hobson, NAMAS certified) of 2330nm, 270nm and 29nm, respectively. This qualification has allowed to establish a 3% accuracy for the surface profilometer results over this depth range.

# 1 Results and Discussion

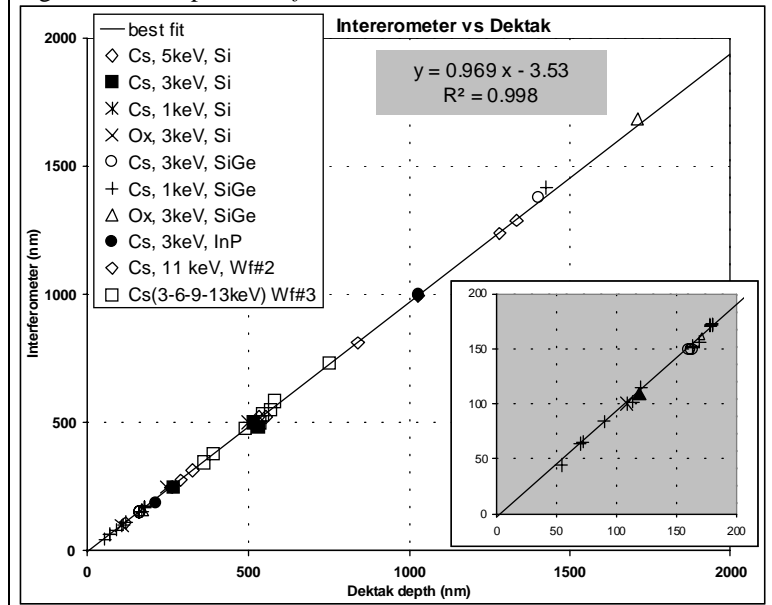
## 1.1 Accuracy

A first source of error on the depth measurement is the non-linearity of the electronic device measuring the phase shift over a cycle equal to  $\lambda/4$  (~160 nm). A second possible source of error is the uncertainty on the laser beam polarization which can induce a “cross talk” effect between the two laser beams. These two sources of error induce a periodic error on the measured sputter rate (Figure 2) with a period very close to  $\lambda/4$  and a peak to peak variation around 15%. Note that the sputter rate is the derivative of the depth vs. time curve. Therefore, a modulated sputter rate of  $\lambda/4$  period and a peak to peak variation of k% corresponds to an absolute error on the depth of  $k \times \lambda / (4 \times 2\pi)$ , i.e. ~3.8nm.



Craters of different depths have been sputtered in different types of samples : Si, InP, SiGe on 3 different instruments. The crater depth was measured with the laser interferometer during the crater sputtering and then with the surface profilometer. The results are plotted in Figure 3. The laser interferometer provides a crater depth lower than the surface profilometer. The equation of the straight line fitting on the data points is  $y = 0.98x - 7$ . No physical explanation of this 7 nm offset can be yet proposed and it is still under investigation. However, correcting the data for this offset reduces the mean depth accuracy (relative to the surface profilometer) from 5.4% down to 0.5% over the investigated depth range (50-1700nm).

Fig 3 : Crater depth, Interferometer vs. Dektak



## 1.2 Repeatability and Reproducibility

These two important analytical features have been determined by measuring the depth of a Poly\_Si/Si interface. The measurements were performed for Cs<sup>+</sup> primary ion bombardment and the interface position was defined as the center of the width half maximum of the oxygen peak at the

Table 1 : Repeatability and reproducibility test results

Test	Depth (nm)	1 $\sigma$ (%)
Short term repeatability	289	0.5
Long term repeatability	292	1.2
Reproducibility	-instrument#1: 297 -instrument#2: 292	n.a.

138 848 913

interface. Three types of tests have been completed : series of measurements on the same instrument with fixed experimental conditions (short term repeatability, 8 measurements over 2 hours), series of measurements on the same instrument with different experimental conditions on various days (long term repeatability, 4 measurements series over 2 days ), comparison of measurement performed on two different instruments (reproducibility). The results are reported in Table 1. These results demonstrate a short term precision better than 0.8% while the long term repeatability and the reproducibility are respectively 1.2 and 1.7%.

### 1.3 Depth calibration of multi matrix sample : application to SiGe

The  $\text{Si}_{1-x}\text{Ge}_x$  system makes difficult the SIMS analysis quantification [2] because the erosion rate varies as a function of [Ge] and this variation depends on the nature of the primary ions. The present laser interferometer has been evaluated for SiGe sample analysis. The sample consists 9 different layers with a [Ge] spreading from ~5 to 20 at%. The Ge profile has been measured under  $\text{O}_2^+$  and  $\text{Cs}^+$ . The depth calibrated profiles based on the in-situ crater depth measurement data are plotted in Figure 4 . They show a perfect overlay of the two profiles demonstrating that this depth scale calibration method allows to easily compare data obtained under  $\text{O}_2^+$  and  $\text{Cs}^+$  bombardment. Actually, figure 5 presents the SR variation as a function of [Ge] determined for the two profiles plotted in Figure 4. These results do not support the expected continuously increasing SR with the [Ge]. Moreover measurements performed on a thinner SiGe layer structure (layer of 20nm thickness) would indicate that thin SiGe layers are, to some extent, a semi transparent material for the laser beam inducing an apparent bottom crater shift when going through an interface Si/SiGe. In conclusion, the laser interferometer offers a good measurement repeatability for SiGe samples but the physical processes controlling the accuracy needs to be more investigated.

### References

- [1] J. Kempf, Proceedings of the 2<sup>nd</sup> international conference on SIMS, 1979, page 97, Springer-Verlag.  
 [2] M.S. Denker and al., Proceedings of the 12<sup>th</sup> international conference on SIMS, 1999, page 639, Elsevier Science B.

Fig 4 Ge profiles measured under  $\text{O}_2^+$  and  $\text{Cs}^+$  primary ions in  $\text{Si}_{1-x}\text{Ge}_x$  multi-layers sample.

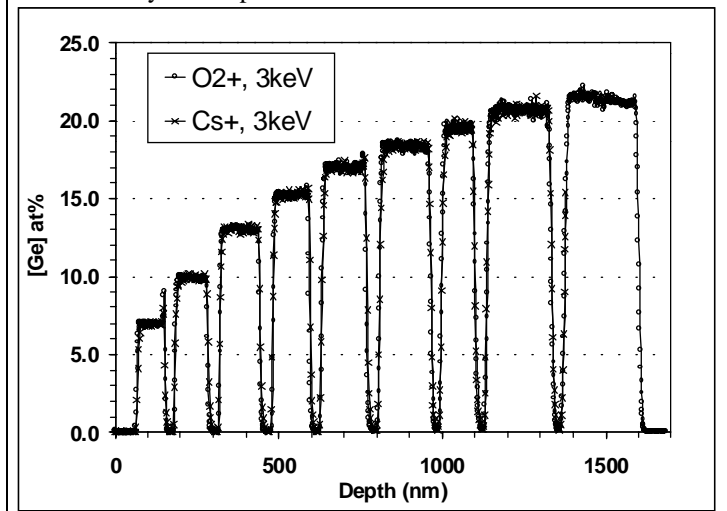


Fig 5 : SR variation as a function of [Ge] as determined from the laser interferometer data.

