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### Imaging by atomic force microscopy of the properties difference of the layers covering the facets created during SIMS analysis

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#### ABSTRACT

Atomic force microscopy (AFM) is used in tapping mode in order to study the roughness created in the crater bottom during secondary ions mass spectrometry (SIMS) analysis in silicon, using  $O_2^+$  primary ions without flooding. Previous studies of the chemical composition of the facets created during the analysis have lead to the conclusion that the facets oriented toward the  $O_2^+$  beam during the ionic bombardment were close to SiO<sub>2</sub> in composition, while the facets hidden from the beam were covered with a sub-stoichiometric oxide SiO<sub>x</sub> (with x < 2). We show that the AFM phase contrast during tapping mode observation of the facets reflects this composition difference, revealing a sharp contrast between the facets. The observed contrast may arise from the different chemical composition of the facets, leading to a different energy dissipation of the tip/sample system over Si and SiO<sub>2</sub> due to the different properties of the materials (hardness, adhesion, etc.). As a comparison, an observation of a surface covered with SiO<sub>2</sub> and Si (SiO<sub>2</sub> deposed with a 90 nm or 4 nm thickness, and partially removed from a Si surface) shows the same kind of contrast.

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#### 1. Introduction

The analysis by the secondary ions mass spectrometry (SIMS) remains a mandatory step for the study of the devices of the microelectronics, the characterization of the junctions, and a very popular method for chemical depth profile analysis of materials. This analysis is limited by parasitic physical phenomena among which is included the formation of roughness in the crater bottom. The development of a severe roughness at the crater bottom remains one of the main obstacle from attaining optimal depth resolution. The latter was improved by means of a reduced primary ions energy [1,2] and of deconvolution algorithms [3–5].

The morphology and the dynamic of appearance of the roughness have been studied by several authors [6–8]. It has been demonstrated that cones, ripples or waves appear in the crater bottom after a certain critical depth. The section of the waves is not sinusoidal but rather triangular [9]. Previous work suggests a direct relation between surface composition of the crater bottom and the development of surface roughness. The composition of the facets has been studied by X-ray photo-electron spectroscopy (XPS)

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http://dx.doi.org/10.1016/j.apsusc.2014.04.054 0169-4332/© 2014 Elsevier B.V. All rights reserved. [7]: regions of pure Si, SiO<sub>2</sub> and suboxides have been observed. Elst et al. [6] have studied the difference of oxidation state between two facets of rippled topography: the side oriented toward the beam is completely oxidized whereas the facets located at the other side are very slightly oxidized. Another method to detect the difference of properties of the oxides covering the facets of the ripples in the SIMS crater bottom was used by Gautier et al. [10]. We have shown that the AFM (atomic force microscope) used in conductive-AFM mode (C-AFM, also called tunneling AFM (TUNA) mode) detects a clear difference between the facets. We has reached the conclusion that when a rather strong electric field is applied between the tip and the sample, one side of the ripples (facets hidden from the beam) exhibits a far more important leakage current than the other (facets facing the beam) at a given fixed voltage, suggesting that the oxide formed on the facet hidden from the beam is not a stoichiometric  $SiO_2$  and may be a poorer insulator or a thinner oxide.

In this paper, we present AFM images which tend to confirm the fact that when facets are formed during SIMS analysis, the material which covers both sides of the ripples (the side oriented toward the beam and the other) does not have the same physical and mechanical properties. For this purpose, we use the AFM operated in phase contrast tapping mode in order to get a contrast arising from a different tip/sample interaction over areas of the sample of different nature.

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#### 2. AFM tapping mode phase imaging

Since its introduction in 1993, tapping mode imaging has become one of the most common imaging mode for the topography using AFM. The cantilever supporting the tip is driven at a given alternating voltage  $V_d \cos(\omega t)$ , and vibrates with certain amplitude A. When the tip is far from the surface, A is called the free amplitude  $A_0$  and the motion of the cantilever is sinusoidal:  $A_{(t)} = A_0 \cos(\omega t + \emptyset)$ . When the tip brought into contact with the sample surface, the motion is still sinusoidal at first approximation [11], and the amplitude of the cantilever is damped. The topography image is made by imposing a constant vibration amplitude during the surface scan. It is also possible to record the phase Ø of the cantilever oscillation during the scan, which has been demonstrated to provide information on localized AFM probe/sample interactions and especially on the energy dissipated by the tip/sample system during tapping mode operation [11–14]. In phase contrast, the phase shift of the cantilever oscillation, relative to the signal sent to the cantilever's piezo driver, is simultaneously monitored and recorded. This phase shift is very sensitive to local variations in the material properties [15].

The phase contrast is not straightforward to interpret and much work has still to be done in order to fully understand its origins and interpretations. The phase is very sensitive to variations in many material properties like local stiffness, viscoelastic properties and variation in chemical composition. This is due to the interaction force that exists between the tip and the surface that contains chemical and viscoelastic information about the sample [16,17]. Contrast in phase images can be strongly dependent on the magnitude of the tip sample interaction force. Strong contrast between different regions can be obtained in phase images even for topographically flat surfaces. Changes in phase angle during the scan may arise from many distinct phenomena among which the changes in surface properties like adhesion and friction. In many samples, large adhesion differences are caused by differences in the thickness of the adsorbed water layer, which is a function of hydrophobicity and hydrophilicity [18]. However, the sample curvature can also be regarded as an indirect source of phase contrast [13,19] because the modification of the sample curvature modifies the tip environment and thus the tip-sample interaction. This particularity has been used to enhance the contrast of tapping mode topographic images, especially in the areas of the sample with a



Fig. 1. Phase contrast images AFM of a SIMS crater bottom (2 µm × 2 µm) for different values: Up: scan angle. Middle: scan speed. Bottom: amplitude set-point.

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