Reflectance Measurements on Clean Surfaces for the Determination of Optical Constants of Silicon in the EUV/Soft-X-Ray Range

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INTRODUCTION

The response of a given material to an incident electromagnetic wave is described by the energy dependent complex index of refraction $n = 1 - \delta + i\beta$. In the extreme ultraviolet (EUV)/ soft x-ray spectral region, the need for accurate determination of n is driven by activity in areas such as synchrotron based research, EUV/x-ray lithography, x-ray astronomy and plasma applications. Various methods are used in order to determine the optical constants δ, β such as reflectance measurements, angle dependent electron yield, transmission measurements, interferometry and ellipsometry. In this work, the method of angle dependent reflectance is evaluated and implemented in order to obtain the optical constants of Si in the region around the $L_{2,3}$ edge (99.8 eV). Silicon is among the materials of particular importance for practical applications in the EUV/soft x-ray range, due to its implementation as filter and spacer material in multilayer mirrors, for energies below the $L_{2,3}$ edge. The refractive index of Si in this energy range has been investigated by previous experimenters [1,2,3] using various methods. The discrepancies among their data arise mainly due to the surface quality of the samples used for measurements and the inherent difficulties of each method. Angle dependent reflectance measurements have the advantage that both δ and β may be deduced experimentally, and thus provide an important test of the tabulated values [4] of δ generated using the Kramers-Kronig relations. In addition, measurements may be performed on bulk samples without the need to fabricate the free standing thin films required for transmission measurements.

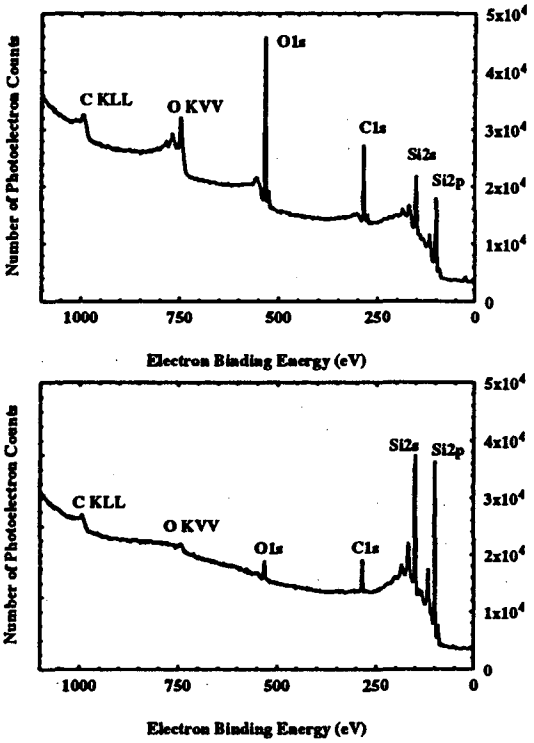

Figure 1. XPS survey scan results for a Si(111) wafer, "as received" (top) and after H-passivation (bottom).

EXPERIMENT

The samples investigated include crystalline silicon ((111) and (100) orientations) and amorphous silicon. The reliability of reflectance data depends on the quality of the sample surface. The most serious sources of error are: 1) surface roughness, 2) the presence of native oxide and organic contamination on the sample surface. For these reasons, particularly smooth samples were used and a cleaning treatment was applied to the sample surfaces, prior to the measurements. The cleaning procedure includes UV cleaning (for organic contamination removal) and subsequent HF:ethanol dipping of the samples, which results in H-passivated surfaces [5,6]. The samples were investigated before and after cleaning was applied, using the X-ray Photoelectron Spectroscopy

(XPS) method, in order to determine the effectiveness of the H-passivation technique, as shown in Fig. 1.

Two instruments were used for the reflectance measurements on the H-passivated silicon samples: 1) The EUV/soft X-ray reflectometer is described in detail in [7]. The instrument uses a laser produced plasma source and a spherical grating monochromator to produce continuously tunable radiation in the range 30<E<300 eV. 2) Beamline 6.3.2 at the ALS is described in [8]. This beamline is using radiation from a bending magnet in the energy range 50<E<1000 eV. The optics consist of a plane grating monochromator, a reflectometer chamber (10^{-8} Torr base pressure) and refocusing mirrors to provide a small spot on the sample. Data were collected in the form of reflectance curves (R vs. angle of incidence) for a number of energies in the region 50-180 eV. The reflectance data vs. angle at each energy were fitted in order to obtain the optical constants δ, β by means of a least squares fitting algorithm, using the Fresnel equation.

RESULTS

It is shown that the method of least squares fitting in order to derive the optical constants δ, β from experimental data is reliable only in regions where β is sufficiently below δ, while it is producing results with large uncertainties in the energy range where $\beta \geq \delta$, as shown in Fig. 2. The effect of the β/δ ratio in the fitting of a reflectance curve has a purely mathematical nature, thus, the above reliability criteria should apply to the fitting of reflectance data from any material in any energy range. However, the exact value of β/δ which marks the boundary between reliable and uncertain fitting should depend on the particular experimental conditions and therefore it has to be determined separately in each case.

In this experiment, reliable data are obtained in the range 50 - 90 eV (below the silicon $L_{2,3}$ edge), corresponding to $\beta/\delta \leq 0.5$. It is shown that crystalline Si exhibits structure from 60 to 80 eV, also observed in previous transmission data. Furthermore, the fitted values for δ are compared to the tabulated values [4]. It is demonstrated through the sum rule that the tabulated values in [4] should overestimate δ because of missing oscillator strength in the absorption coefficient data used. This effect is verified by the present reflectance results for δ.

The above discussion suggests that least squares fitting of reflectance data may not be a suitable method for the determination of the refractive index in certain energy regions; different techniques should be explored in the regions where the fitting algorithm becomes problematic. Particularly for silicon, there is a need for improved measurements in the region above the $L_{2,3}$ edge, where the available absorption data are poor and the reflectance method fails to provide reliable results.

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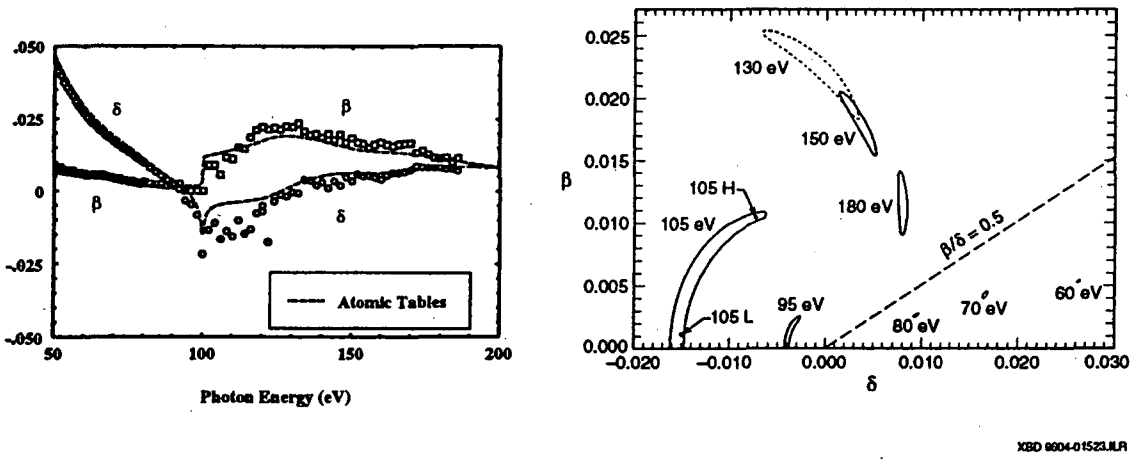

Figure 2. Left: The optical constants δ (○), β (□), for a Si(100) sample measured at the ALS beamline 6.3.2. The values for δ, β (dash) from the atomic tables [4] are also shown. Right: Contour plots of the least squared error generated with the data on the Si(100) sample. The energies shown are 60, 70, 80, 95, 105, 130, 150 and 180 eV in a $\delta - \beta$ axis system. Each contour area is enclosing the optical constant pairs fitted by the least squares algorithm within $\pm 1\sigma$ of their true values. When the contour areas become large, then the fitting is uncertain. This is true for the energies above the edge in this experiment.

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