Soft X-ray reflectometry for the inspection of interlayer roughness in stacked thin film structures

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ABSTRACT

A key element of semiconductor fabrication is the precise deposition of thin films. Amongst other aspects, the quality of interfaces between different materials plays a crucial role for the success of further process steps. We here present soft X-ray reflectometry measurements on stacked thin film samples of silicon and silicon-germanium in various concentrations as they are produced for complementary field-effect transistor (CFET) applications. Synchrotron-based, angle- and energy-resolved broadband reflectance data sets can be modeled using a matrix-method approach that describes reflection, absorption, and diffuse scattering off the interfaces. This method is often used to determine the optical constants of materials in the EUV spectral region as parameters of the fitting procedure. We here show that the method is equally well suited to investigate roughness and layer intermixing between different deposited materials. These roughness parameters alongside the actual thicknesses of the individual layers also result from the physical modelling of the measured data. The method is inherently non-destructive and very sensitive, down to approximately 50 nm depth and as such gives valuable information. To further qualify our findings, we compare the data to scanning transmission electron microscopy (STEM) and energy dispersive X-ray spectroscopy (STEM-EDX) to give insight into the atomic structure at the interfaces.

Keywords: EUV reflectometry, STEM-EDX, SiGe, layer intermixing

1. INTRODUCTION

As the semiconductor industry progresses to more complex and smaller transistor designs, the accompanying metrology must constantly refine existing methods and develop new ones to keep up with the rapid development. One of the foundations for further manufacturing steps is precise thin-film deposition. The determination of the layer thicknesses for thin-film structures is typically done using X-ray reflection (XRR), optical methods like ellipsometry, or TEM, but the study of buried interfaces still remains challenging. For devices with shrinking dimensions, the role of interfaces becomes more and more important since they determine the performance to a large extent, therefore also the importance of interface metrology is rising. The next step in the device evolution is the CFET, which is considered for beyond 1 nm technology nodes.[1] It starts with the epitaxial deposition of a complicated SiGe/Si multilayer-stack with at least two different germanium concentrations (Figure 1a). This work compares our studies of blanket layer stacks as used for CFET devices using broadband, angular-resolved soft X-ray/EUV reflectometry and scanning transmission electron microscopy combined with spectroscopic mapping (STEM-EDX). Both methods can determine the layer structure of a sample and quantify the extent of interdiffusion layers at the interfaces between different materials. Reflectometry is non-destructive, but model-based and requires a large sample area, while STEM-EDX needs lamella cuts of the sample. We find remarkable agreement between both methods and discuss their applicability, advantages and disadvantages.

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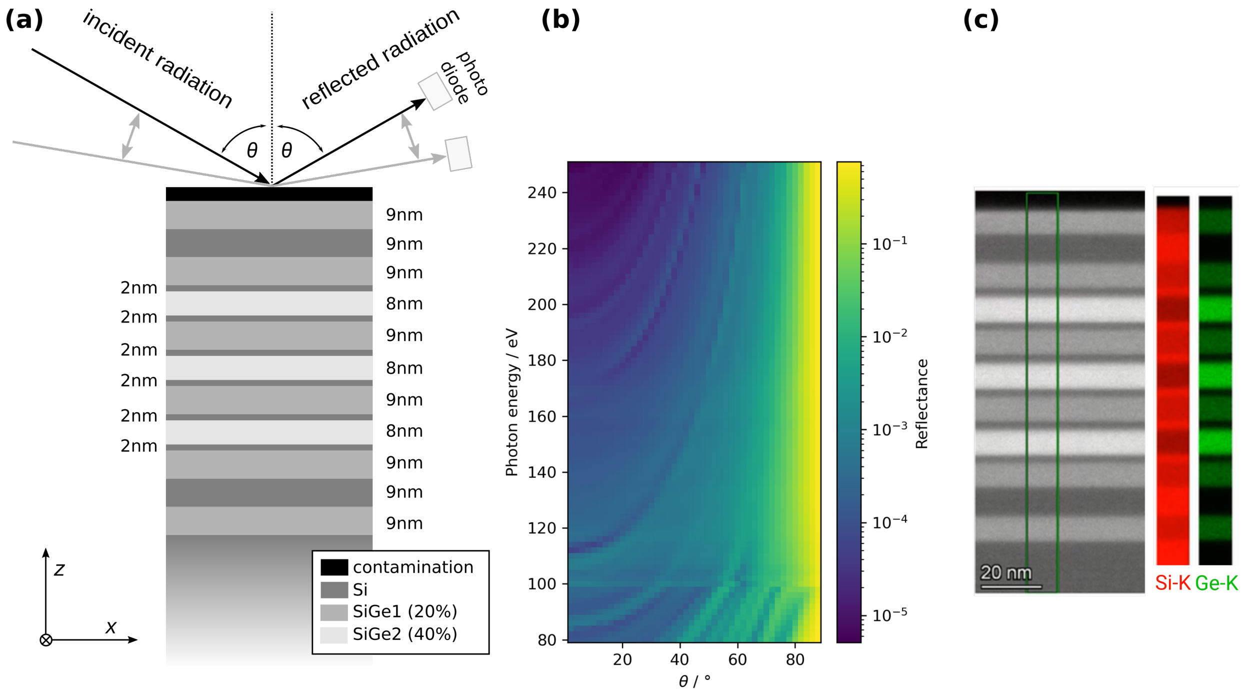

Figure 1. Sample material and central measurements. (a) The sample consists of several layers of silicon and silicon-germanium as used for CFET devices. Reflectometry measurement principle: monochromatic radiation in the soft X-ray regime is reflected off the sample surface and detected by a photodiode as a function of the angle of incidence θ and the photon energy. (b) Angle-resolved reflectance data in the photon energy range of 80 eV ... 250 eV and from grazing incidence to near-normal. (c) STEM-EDX data of the sample: dark field signal and EDX data for the silicon and germanium K-edge.

2. MEASUREMENTS

Reflectometry measurements were performed at the soft X-ray beamline[2,3] in the laboratory of the Physikalisch-Technische Bundesanstalt (PTB) at the synchrotron radiation facility BESSY II in Berlin. The raw measurement data is presented in Figure 1b, with an average relative measurement uncertainty 0.8%. We used a transfer matrix approach[4–7] to calculate the reflectivity of a specific sample as a function of its geometrical parameters. This model was then used for a fit to the experimentally obtained data, which yielded the layer thicknesses and a value σ describing interface roughness and interlayer diffusion.[8] STEM, STEM-EDX and High Resolution STEM-EDX micrographs were acquired at ThermoFisher Scientific by means of a Spectra Ultra Transmission Electron Microscope. The system was equipped with a monochromated X-FEG (not excited), a piezo stage, a PantherSTEM™ detector, an UltraX™ EDX detector. The STEM-EDX signal was processed through ThermoFisher Scientific's Velox™ software. In this environment the STEM-EDX map is quantified over an X by Y window by using an empirical model consisting in a 3 parameter Bethe Heitler function that is fit across the entire measured spectrum. From this data, the layer thicknesses and the interlayer roughness parameters were extracted using a model fit, based on error functions.

3. RESULTS AND DISCUSSION

In Figure 2, we present the geometrical parameters of the sample stack as determined by soft X-ray reflectometry and STEM-EDX. Figure 2a shows the film thicknesses and their correlation and we find an excellent agreement between the two methods, which has also been verified on a second sample (data not shown). This shows the general applicability of both, STEM-EDX and soft X-ray reflectometry to the problem. Both methods have advantages and disadvantages. STEM-EDX is a destructive method, since a lamella must be cut out of the

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wafer. Furthermore, it only represents a very small fraction of the sample and cannot make statements about the average sample quality, which can be good or bad, depending on the situation. Soft X-ray reflectometry, on the other hand, is a non-destructive method, but it requires a relatively large sample area due to the increased beam footprint at grazing incidence. It can only provide layer thickness and roughness on a model-based reconstruction process, which either also has to fit the optical constants, or needs precise knowledge of the optical constants of the materials in question. Figure 2b compares the retrieved interface parameters σ_i. We find that both methods show the same trend over the layer stack and that their values compare well, although STEM-EDX retrieves generally higher values of σ than reflectometry. In the theory of reflectometry, σ describes light scattered off the interfaces, where ideally only refraction and reflection appears. It enters theory as a reduction factor of the Fresnel reflection amplitudes, called Debye-Waller factor or Névot-Croce factor.[5,8] Within this theory, the parameter σ describes a combination of real interface roughness and interlayer intermixing, but for the specular reflex, that is detected in reflectometry, the two contributions cannot be discriminated. In the TEM analysis, we found no significant interface roughness but a pronounced interlayer mixing, so we conclude that also for reflectometry, the interface parameter σ must be dominated by interlayer mixing instead of interface roughness. Therefore, σ describes the width of the intermixing region between two materials for both experimental methods.

The labels in Figure 2b indicate the interfaces from top to bottom, such that ”Si / SiGe1” refers to an interface where a silicon layer which was deposited on top of a silicon-germanium layer. Two trends in σ are remarkable and give insight into the details of the layer structure. First we see that SiGe on top of Si gives sharper interfaces (lower σ) than Si on top of SiGe. Second, the interfaces containing SiGe1 are generally sharper than those, containing SiGe2.

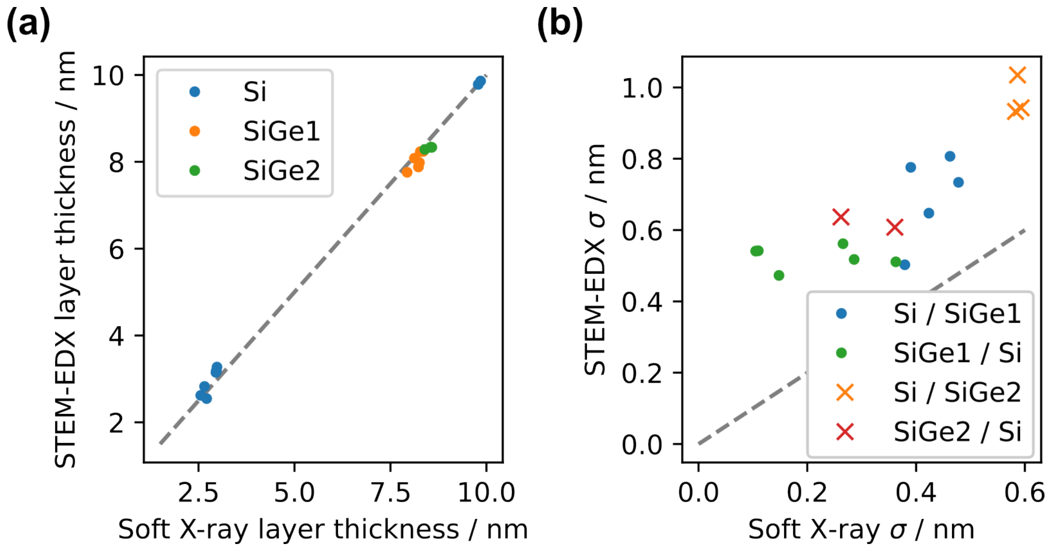

Figure 2. Comparison of STEM-EDX-based and soft X-ray-based determination of layer thicknesses and interlayer roughness parameters. In (a) it is shown that the film thicknesses correlate very well for all layers. In (b) it is visible that the layer intermixing σ, determined through STEM-EDX follow the same trend as those, determined through reflectometry, but feature slightly higher values.

4. SUMMARY

We have presented a comparative study on blanket layer stacks, as used for CFET devices. The samples feature sub 10 nm thick layers of two variants of silicon-germanium. From the sample wafer, TEM-lamellas were cut for extensive STEM-EDX characterization and other parts were used for soft X-ray reflectometry. We showed that both methods can determine the different layer thicknesses of the complex layer stack and provide values for the magnitude of their interlayer intermixing regions. We found that both methods agree on the measured numbers, but STEM-EDX results in slightly higher roughness parameters than soft X-ray reflectometry. The advantages and disadvantages of both methods were discussed and compared and the investigated sample system was found to be an ideal basis for such a comparison.

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