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Practical Considerations in Quantitative Nanoscale Energy-Dispersive X-ray Spectroscopy (EDX) and Its Application in SiGe

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In advanced semiconductor technology, epitaxial SiGe is used in P-channel Field-Effect Transistor (PFET) as source/drain. It serves as either a strain inducer or a "container" for B dopants, either of which plays a vital role for Complementary Metal Oxide Semiconductor (CMOS) device. Understanding the structure and composition of SiGe can provide crucial information for process development and device performance enhancement.

At present, the Ge atomic concentration (%Ge) in SiGe is determined by Secondary Ion Mass Spectrometry (SIMS), High-resolution Rutherford Backscattering Spectrometry (HRBS), Low energy Electron induced X-ray Emission Spectrometry (LEXES) and/or X-ray Diffraction (XRD) on large SiGe structures of hundreds of microns in size. For small SiGe structures at the nanometer scale, like those in source/drain in CMOS device, Energy-dispersive X-ray spectroscopy (EDX) in the Transmission Electron Microscope (TEM) is used. EDX offers exceptional advantages in quantification, including high spatial resolution, good precision and accuracy. This quantitative EDX technique is based on the Cliff-Lorimer ratio method for a two-element SiGe system: $\frac{C_{Ge}}{C_{Si}} = k_{GeSi}\frac{I_{Ge}}{I_{Si}}$ and $C_{Ge} + C_{Si} = 100\%$, where C represents the weight percentages, I represents X-ray intensities, and k is termed the Cliff-Lorimer factor. Here k is not a constant, but a sensitivity factor, which varies with electron microscopes, accelerating voltages, samples and so forth.

This work presents our efforts in understanding the variables that influence EDX quantification results and in developing a standard methodology to quantify %Ge using EDX in the TEM for semiconductor research and development. A standard $Si_{1-x}Ge_x$ blanket test sample (Figure 1) was characterized by HRBS and XRD, and found to be $Si_{0.664}Ge_{0.336}$ with a relative error smaller than 1%. It is then used to calibrate k factor. Effects of sample tilting, sample thickness, electron voltage and electron fluence on quantitative EDX were evaluated. As shown in Figure 2, tilting off the zone axis greatly affects the quantification if the K-line is used, but not as much if the L-line is used. After 2 degree tilting around either [001] axis or [110] axis of SiGe, %Ge reaches a plateau, indicating that electron channeling effect is minimized. [1] Table 1 shows the impact of sample thickness on %Ge determination. As expected for a typical TEM sample that is usually thinner than 80nm, sample thickness effects (i.e. X-ray absorption and fluorescence) can be neglected. It is well known that electron radiation damage in TEM can significantly change the physical and/or chemical state of the tested sample. [2] Figure 3(a) demonstrates that after an electron fluence of 2.54E+09 nm^{-2} the Si-K intensity of the sample seems to decrease due to electron radiation damage. Figure 3(b) shows that reducing electron energy can effectively diminish the electron radiation damage on SiGe sample. The critical electron fluence on $Si_{0.664}Ge_{0.336}$ at 120kV is found to be 3.46E+10 nm^{-2} as compared with 2.54E+09 nm^{-2} at 200kV, allowing for longer acquisition time and statistically more accurate %Ge determination. Using the standard methodology, namely a thin sample tilted by >2° away from zone axis, low electron energy, below critical electron fluence and a calibrated k factor, %Ge

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of a typical SiGe source/drain diamond structure [3] can be obtained in Figure 4. In addition, EDX measurement on the zone axis of the SiGe sample can be performed by setting electron beam in precession mode. [4] The established methodology of quantitative nanoscale EDX technique can be applied to other material systems in semiconductor industry, such as TiSi, NiSi, AsSi etc.

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[5] Acknowledgements: The authors thank John Bruley (IBM), Yun-Yu Wang (IBM), Jinghong Li (IBM), Alexandre Pofelski (STMicroelectronics) and Germain Servanton (STMicroelectronics) for valuable discussions.

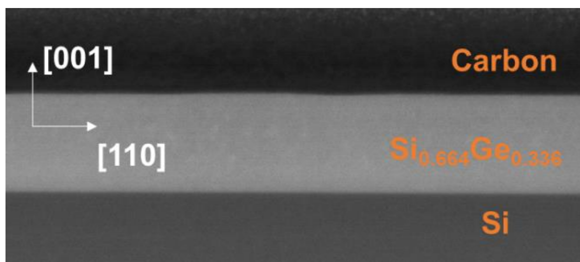


Figure 1. HAADF-STEM micrograph of the standard SiGe blanket test sample. It has [110] in-plane direction and [001] out-of plane direction.

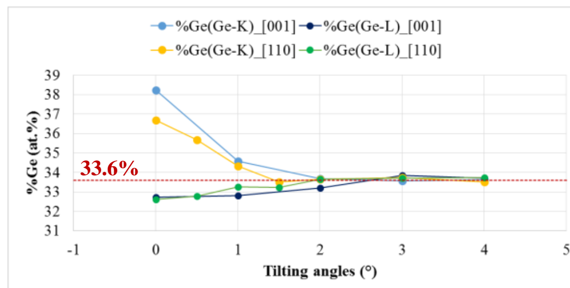


Figure 2. %Ge as a function of sample tilting angles. Due to electron channeling effect, %Ge measured close to the zone axis strongly depends on tilt, and can be off by up to 20% if the K-line is used. This effect is much smaller if the L-line is used.

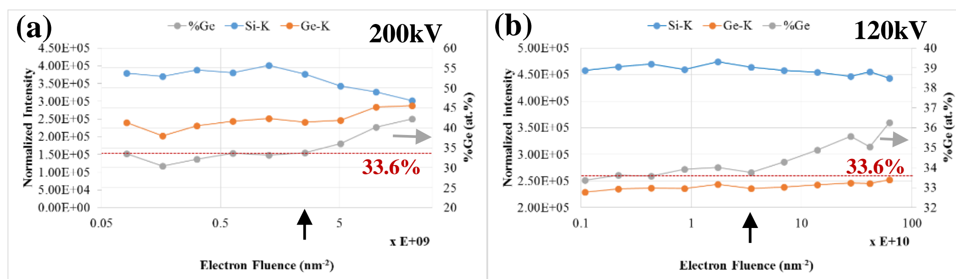
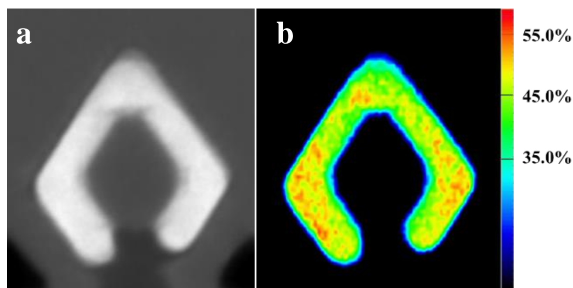


Figure 3. %Ge and normalized intensity as a function of electron fluence at (a) 200kV and (b) 120kV, respectively. Critical electron fluence (as arrowed) on $Si_{0.664}Ge_{0.336}$ is found to be 2.54E+09 nm^{-2} for 200kV, and 3.46E+10 nm^{-2} for 120kV.



SiGe sample thickness (nm)	68	80	100	350
%Ge (Ge-K) (at.%)	33.4 ± 0.15	33.8 ± 0.12	34.4 ± 0.11	37.0 ± 0.09
%Ge (Ge-L) (at.%)	33.6 ± 0.04	33.5 ± 0.04	33.5 ± 0.08	30.2 ± 0.08

Table 1. %Ge as a function of sample thickness.

Figure 4. (a) HAADF-STEM micrograph and (b) %Ge map of a SiGe diamond structure. The %Ge varies from 48.7% to 55.6% within this diamond with an average %Ge at 51.0%.

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