Analysis of steam oxidation of crystalline Si$_x$Ge$_{1-x}$ using AFM and CABOOM
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It has been shown in literature that the oxidation rate of SiGe in wet oxidation is 2 to 3 times higher than Si due to the weaker Si-Ge bonds compared to the Si-Si bonds [1]. In this study we investigate the atmospheric steam oxidation rate of Si$_x$Ge$_{1-x}$ over a wide temperature (600°C, 700°C, 800°C), and composition range (x=0.05 to 0.4 in steps of 0.05) for times t=40, 60, 80, 100 and 120 min. The low temperatures have been chosen to restrict Ge segregation during oxidation. To allow a consistent comparison, one heterostructure, consisting of 8 SiGe layers with a 5% Ge increase in each layer, has been grown. The small step in Ge concentration, combined with thick layers (~200nm) ensures unstrained, crystalline layers. SIMS measurements demonstrate a negligible composition error for 0<x<25% but increasing to up to ±2% for the higher Ge concentrations.

The sample preparation technique for each oxidation time and temperature is as follows (see fig.1):
1) Bevel a 3mm x 5mm heterostructure sample under a shallow angle (1°). We have used chemical-mechanical beveling as proposed in [2] for good linearity along the bevel and bevel smoothness.
2) Oxidize the beveled surface in steam at a particular temperature and time. Oxidize a pure Si sample as control at the same time.

Fig 1: Surface preparation for oxidation analysis. Note that the angle in the figure is exaggerated.

As can be seen in fig 1 (right), each layer will generate a different oxide thickness that is directly related to the Ge concentration in the layer. Using AFM, the steps in the oxide thickness compared to the oxidized Si substrate can be measured after the shallow bevel angle is removed by leveling the bevel on the substrate. In order to calculate the oxide thickness on each layer in the heterostructure, we assume that 56% of the total oxide thickness is lying upon the original surface while 44% is lying below the surface, resulting in the following relationship:

\[
t_{ox}(x) = t_{ox}(0) + 1.78 \Delta t
\]

with \(t_{ox}(x)\) the oxide thickness of the SiGe layer with Ge concentration \(x\), \(t_{ox}(0)\) the oxide thickness on the substrate and \(\Delta t\) the measured thickness step between both. \(t_{ox}(0)\) is determined by ellipsometry on the Si control sample, oxidized during the same oxidation process.

The results of the measurements are summarized in fig.2. The figures show the oxide thickness for different Ge concentration and for different oxidation times at resp. 600°C, 700°C, and 800°C from top to bottom. For increasing oxidation times, an increasing oxide thickness is measured for both the 700 and 800°C oxidations. For 600°C this relationship seems invalid. This is due to measurement errors on small oxide thicknesses as those occurring for 60 min oxidations. This in confirmed by the CABOOM technique [3] that shows a small oxide thickness increase (20nm) over the whole Ge concentration range. The nearly constant character of the oxide thickness versus Ge concentration at low temperatures is consistent with the slow oxidation process that does not increase the oxidation rate of SiGe, cfr. dry oxidation.

Fig 2: The oxide thickness versus Ge concentration for \(T=600, 700\) and 800°C at different oxidation times 120, 100, 80, 50, 40 min. At 600°C no 40min oxidation is shown.

Based on CABOOM, it can be seen that no Ge segregation occurs for all the 600°C and 700°C oxidations up to 100min. For 800°C, Ge segregation becomes apparent for Ge concentrations above 20%. This is illustrated in fig.3.

Fig 3: Left CABOOM for 800°C, 120min, right for 600°C, 120min. Color gradients, indicating segregation, occur for the high T oxidation.

In conclusion, we have measured the oxide thickness of SiGe under steam oxidation for different Ge concentrations, times and temperatures using beveling and AFM. CABOOM is used to determine measurements errors and segregation effects.

References: