EFTEM and EELS SI: tools for investigating the effects of etching processes for III-V MOSFET devices

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Abstract. High quality oxides layers are now available for MOSFETs on GaAs. For successful devices, suitable process schemes are required. In this paper we show an investigation of an etching process on a GaAs/Ga₂O₃/GGO dielectric gate stack. This investigation has been carried out using EFTEM and EELS SI. EFTEM provides a quick analysis on the structure while EELS SI offers much better resolution and the possibility to quantitatively characterize the material.

1. Introduction
Electron energy loss spectroscopy (EELS) is the analysis of the energy distribution of electrons that have passed through the specimen and interact inelastically with it [1]. It is a powerful technique that provides information on elemental composition and electronic structure in a particular area of the material. There are two different approaches to get an elemental map of a particular area in the material. The first method is to combine the EELS with scanning transmission electron microscopy (STEM) to give a spectrum image (SI) where an EELS spectrum is collected at each point in the scan. The second method is to use energy filtered transmission electron microscopy (EFTEM). EFTEM provides rapid elemental maps to identify unknown elemental distributions and give some information on the composition. SI imaging gives very detailed information on the local chemistry and composition with much higher sensitivity than EFTEM. However, the acquisition time can be much longer so that it must be possible to identify the area of interest. Thus the use of EFTEM in combination with SI is a very powerful approach with EFTEM identifying areas that require detailed analysis by SI.

Here this approach is applied to an investigation of an ion etching process on GaAs/Ga₂O₃/GGO where GGO is gallium gadolinium oxide with nominal composition GdₓGa₀.₄₋ₓO₀.₆. The oxides form the dielectric gate stack for use in III/V MOSFETs. A thin template layer of Ga₂O₃ unpins the Fermi level and a thicker layer of GGO provides low leakage.

The aim of this study is to develop a way to study the effects of etching processes on Ga₂O₃/GGO dielectric gate stacks. This obviously represents a very important issue for fabrication purposes. Electrical properties in MOSFET devices are sensitive to the elemental distribution in the dielectric gate stack, the nature of the interfaces across the channel and the oxide region, and the actual architecture. Hence a good understanding of the local composition and chemistry is required in order to make successful devices. The type of analysis presented here is important in optimising the
performance of III-V MOSFET devices. This has led to the fabrication of world leading GaAs based MOSFET devices at Freescale Semiconductor, Inc. [2] and in Glasgow [3].

Material growth was carried out by MBE on a 4” semi-insulating GaAs substrate, using a dual chamber system. The Ga$_2$O$_3$/GGO gate stack consists of three layers; a crystalline Ga$_2$O template layer, which unpins the GaAs surface, an amorphous Ga$_2$O$_3$ layer, which protects the interface from Gd migration, and an amorphous GGO layer, which controls leakage current [4]. A key step in the fabrication process is the etching process. This is important in order to define the regions where the gate and the ohmic contacts will be placed. It is extremely important that dielectric gate stack layer under the gate is preserved from the etching process and completely removed from all the other parts.

~80nm silicon nitride (SiN) is used as a mask for the etching process. After the etching, a further ~80nm of SiN is deposited to provide a protective cap layer for the surface during TEM specimen preparation. Fig. 1 shows the ideal schematic structure after the etching process.

2 Experimental methods

TEM specimens were prepared by conventional cross-sectioning involving cutting, grinding, dimpling and ion milling using a GATAN PIPS. EFTEM images were acquired utilising a Gatan GIF 2001 spectrometer attached to a TEM FEI Tecnai T20 equipped with a LaB$_6$ source. Compositional maps were made using both the three windows method and the jump ratio method. In the former, images are recorded using two pre-edge and one post-edge window. The pre-edge images are used to subtract the background in the post-edge window, assuming a power law background of the form $A E^{-r}$, where A and r are fitting parameters and $E$ is the energy loss. The jump ratio method takes the ratio between one post-edge and one pre-edge window and is useful where the background shape prior to the edge is perturbed. However, it requires the edge to have a significant intensity above the background.

The EELS SI was carried out in a TEM FEI TECNAI TF20 with a Schottky field emission gun operated at 200kV and with STEM capability. It was equipped with a Gatan Enfina EELS spectrometer. The Enfina system allows the utilisation of the spectrum imaging mode, in the scanning transmission electron microscope (STEM). This system presents several advantages in terms of sensitivity and, most importantly, the possibility to record the entire EELS spectrum at each pixel. The choice of the acquisition parameters for a spectrum image represents a compromise to achieve optimum conditions in terms of signal/noise, spatial and energy resolution. Two different energy sets have been used. For the analysis of the redeposed layer on the sidewall and the modified layer on the substrate, the high-energy setting has been used. In this case we have been able to quantitatively characterize these areas [5]. Spectra covering the energy range from 480eV to 1800eV were acquired at a dispersion of 1eV/channel using a convergence semi-angle of 9mrad and a collection semi-angle of 22mrad aperture. Thus the O K-edge, the Gd N$_{4,5}$-edges, the Ga L$_{2,3}$-edges and the As L$_{2,3}$-edges are included in the same data-set with a good signal to noise ratio at relatively low electrons dose. For the investigation of the interface between the mask and cap layer, the fast beam switch system (FBS) was used [6]. This allowed the acquisition of the low loss and core loss at each pixel using the same experimental conditions. The two spectra can be spliced together and an accurate energy calibration obtained for the Si L$_{2,3}$-edges. This allows the threshold of 103.7eV for the edges from the SiN to be distinguished from that of 106.7eV for the edges from the "SiO$_2". In this case, the zero loss and the core loss data covered the energy range from −150eV to 520eV and 80eV to 750eV respectively, both with 0.5eV/channel dispersion and were acquired with a convergence semi-angle of 9mrad and collection semi-angle of 13.5mrad.

3. Results

The bright field image (Fig. 2) shows structures resulting from the etching. In particular, there is an amorphous layer on the GaAs substrate and a redeposited layer on the side wall resulting from the
sputtering process which occurs during an etching process. In this specimen, Ga, As, Gd, O, N and Si are present. Therefore elemental maps were recorded in order to qualitatively characterize the specimen. The three window method was used for the Si L_{2,3}-edges at 99eV, the Gd N_{4,5}-edges at 140eV, the Ga L_{2,3}-edges at 1115eV and O K-edge at 532eV respectively. The jump ratio method was used for the As L_{2,3}-edges at 1323 eV and to show up the difference between thresholds of the Si L_{2,3}-edges in SiN (103.7eV) and in "SiO₂" (106.7eV).

Figs 3a, 3b, 3c and 3d show the Si, Gd, Ga and O elemental maps respectively. The mask and the cap layers are essentially made by SiN and the redeposited layer on the sidewall appears to be made from Gd, Ga and O. It is very hard to understand from these elemental maps how the elements are distributed across the redeposited layer. The O elemental map (Fig 3d) shows that O decorates all the SiN interfaces. This is due to the presence of "SiO₂," which appears to form at the surfaces of the SiN. Figs. 3e and 3f show the As and "SiO₂" jump ratio maps respectively. The As is uniformly distributed in the GaAs substrate, as expected, and is also present in the modified layer on the substrate. According to these EFTEM results, this modified layer is composed of O, As and Ga. The Si jump ratio map in Fig. 3f tends to highlight the areas containing "SiO₂" rather than SiN but clearly creates other contrast.

Nonetheless, this map indicates the oxidation of the SiN surfaces.

The EELS SI technique gives more detail than EFTEM. The regions marked 1, 2 and 3 on Fig. 4 correspond to regions where SI were recorded. Fig. 5 shows the edge intensities across the modified layer in region 1 of fig. 4. They are normalised to their maximum values. Here there appears to be O in the substrate but it is likely to be oxidation of the surface of the TEM sample. The O concentration rises steeply across the modified layer until it reaches the "SiO₂" at the interface with the SiN. In addition there is a low concentration of residual Gd in the modified layer not seen in the EFTEM map in Fig. 3b. The atomic% of all the elements has been obtained for the region in layer that presents the highest Gd concentration. Fig. 6 shows the edge intensities across the redeposited layer in region 2 of fig. 4. They are normalised to their maximum values. The material near the SiN
mask is Gd rich and comes from the etching of the GGO layer while the material near the SiN cap is Ga rich having come from the substrate once the GGO is etched away. The O is uniformly distributed across this layer rising at the edge where the SiN layer is oxidised. Atomic fractions are given for the points where the Gd and the Ga intensities peak.

Fig. 8 which shows the Si L_{2,3}-edges across the interface between the SiN mask and the SiN cap in region 3 of Fig. 4. Both the threshold and shape change because of the "SiO_2" layer at this interface.

4. Conclusions
By combining EFTEM and EELS SI, it has been possible to characterize the effects of an etching process on a GaAs/Ga_2O_3/GGO dielectric gate stack. EELS SI is a powerful technique for the characterization of materials at the nanoscale but it is time consuming. EFTEM provides a rapid indication of the elemental distribution but with lower sensitivity and resolution than EELS SI. The analysis shows that the etching leaves an amorphous layer on the substrate and this contains approximately equal amounts of Ga and As with some residual Gd. There is also an increasing O content towards the surface. Redeposition occurs on the sidewall giving initially Gd and then Ga rich areas as first the GGO and then the substrate is etched. This layer contains oxygen throughout. All the interfaces with the SiN show oxidation to “SiO_2,” and all the SiN contains some O.

5. References
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