Gallium nitride is a widely used material in optoelectronic devices like LEDs. The LED structures are mostly grown by epitaxy onto sapphire substrates, but, for cost reasons, silicon is currently being studied as an alternative candidate. Transmission electron microscopy (TEM) is a powerful technique to understand the growth mechanisms of nitrides on silicon and to assess the structural quality of the epitaxial layers. For that purpose, preparing high quality cross section samples is key. The use of ion beam related techniques is well known for creating implantation and structural defects. Wedge mechanical polishing offers very satisfying results but is also quite challenging when dealing with samples composed of materials with different mechanical properties or chemical reactivity like GaN and Silicon. The problem is even more critical when the grown structure is made of different nitride materials like AlN and GaN and/or when the Si substrate is non-monolithic like for instance silicon on insulator (SOI).

The aim of this work is to present a dedicated sample preparation technique that we have developed and implemented to obtain samples from GaN-AlN heterostructures grown onto Si and SOI with good quality on large areas, typically tens of microns. The desired samples need to be as thin as possible without preparation damages, in order to make comprehensive (S)TEM studies: from high resolution to obtain interface images at the atomic scale, to larger views allowing to understand the dislocation behavior, at the micrometer scale.

The process is based on a wedge polishing method. It consists in polishing the sample with a very low angle (1-2°), in order to obtain an area on the wedge thin enough for TEM observations. The samples studied in this work were grown at CNRS / CRHEA laboratory, then prepared with an Allied Multiprep polishing tool, and observed on a FEI Titan Cs probe corrected TEM using a 300 kV tension in STEM mode, at the NanoCharacterization PlatForm (PFNC) in CEA Grenoble.

The detailed process for the preparation of nitride materials on bulk silicon and SOI based substrate with different oxide thicknesses will be presented. Typical TEM images of GaN/AlN epitaxial stacks on Si are presented in figures 1 and 2, highlighting the benefits of the described preparation technique at all scales of the TEM investigations for heterogeneous samples, and this in the different TEM modes (STEM, HRTEM, diffraction contrast).
Fig. 1: STEM HAADF image of GaN/AlGaN/AlN heterostructure, in [11-20] zone axis. A field of view of several square microns is obtained.

Fig. 2: High resolution STEM HAADF image of GaN/InGaN quantum well heterostructure, in [11-20] zone axis. Lattice resolution is obtained without any artefact induced by sample preparation.