Advanced structural characterization of Gallium Oxide by transmission electron microscopy

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Intrinsic properties of Ga_2O_3, like very wide bandgap (Eg > 4.5 eV) and high critical breakdown field (8 MV/cm) higher than SiC or GaN have, makes it a promising candidate for high power electronics, suitable for fabrication of solar-blind detectors for UV-C radiation (wavelength below 280 nm).

Beside the stable monoclinic β, other polymorphs (α, γ, κ(ε), δ) are relevant candidates for applications. The most crucial step in developing devices is the structural characterization of the polymorphs: understand their detailed crystal structure, growth mechanism, thermal stability and phase transformations up to 900°C, and the structural relationship between polymorphs at the atomic scale. In the case of more polymorphs it is lacking or less known. Atomic scale study of these can be achieved by conventional and scanning transmission electron microscopy combined with elemental mapping and image simulations. In situ TEM annealing is an adequate method to study transformations alive.

Recently we carried out a comprehensive study of Ga_2O_3 epilayers grown at low temperature (650 °C) using high-resolution TEM and X-ray diffraction (XRD) in order to investigate the real structure at the nanoscale. Initial XRD measurements showed that the films were of the so-called ε phase; *i.e.* they exhibited hexagonal *P63mc* space group symmetry, characterized by disordered and partial occupation of the Ga sites. Our work clarifies the crystal structure of Ga_2O_3 layers deposited at low temperature at the nanoscale: TEM investigations revealed that the Ga atoms and vacancies are not randomly distributed, but actually possess ordering, with (110)-twinned domains of sub-10 nm size. Each domain has orthorhombic structure with P*na*21 space group symmetry, referred to as κ-Ga_2O_3.

The γ polymorph is considered to be of defect spinel structure, however several crystallographic questions are still opened (e.g. ordering of vacancies/Ga, occupancy of more than two possible sites, symmetry, domain structure), which strongly affects its macroscopic properties and phase transformations. It is important to understand the crystallographic background of the polymorphism of Ga_2O_3 for their formation mechanism.

Until now, five polymorph transformations were studied by us applying *ex-situ* and *in situ* TEM heating measurements. Our first experiments were done on MOCVD grown κ-Ga_2O_3 layers. The κ → β transformation was crystallographically described *in situ* in the microscope under vacuum, and using differential scanning calorimetry. The κ →γ transformation was also studied with *ex-situ* heating measurement on a κ-Ga-oxide thin film, where the role of O_2 fugacity of heating measurements on transformation of κ structure was discovered and the interfaces between κ /β and κ /γ were modelled.

Our second group of TEM experiments were carried out on radiation induced phase transformation of β-Ga_2O_3. First, the β → γ phase transformation was crystallographically described. Later the structural and textural development of the γ→ β transformation was monitored by ex situ heating (series of samples at different temperatures from RT up to 1100°C) and in situ TEM annealing experiments. The experimental data were excluded by simulations using JEMS software applying multislice approach for TEM image simulations.

Recently, we investigated the crystallization process of RF sputtered amorphous Ga-oxide thin film in situ in TEM.

By this the talk we would like to show that conventional and advanced transmission electron microscopy with in situ annealing measurements and simulation is an effective and appropriate method in the study of the polymorphs of Ga_2O_3 and their temperature stability in order to show future possible applications.