ε-Ga 2 O 3 epilayers as a material for solar-blind UV photodetectors

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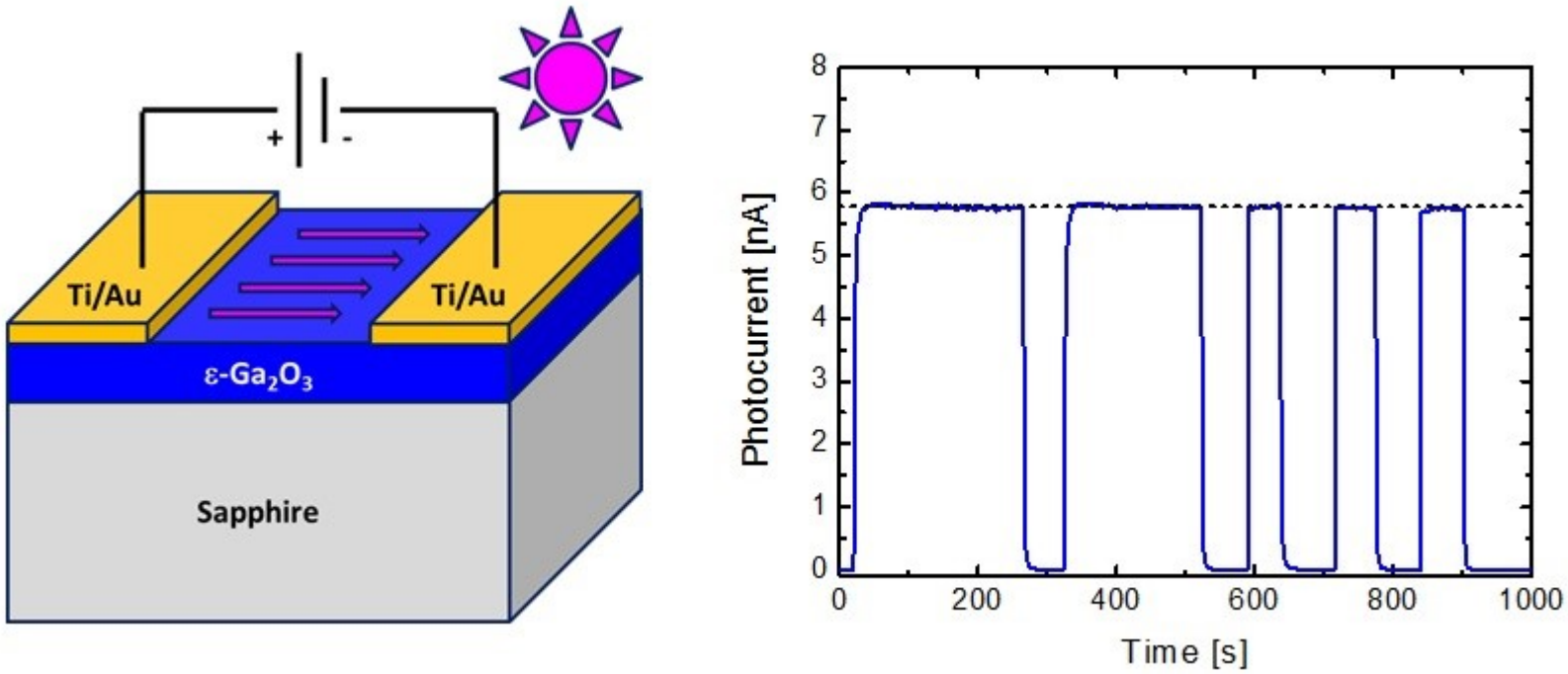
Abstract: Electrical and optical properties of undoped single-phase ε-Ga2O3 epitaxial films prepared by MOCVD are reported. It is shown that this still unexplored polymorph of gallium oxide possesses wide bandgap and very high dark resistivity, thus allowing the design and fabrication of solar-blind UV photodetectors. Simple cost-effective photoresistors, fabricated by direct deposition of the epilayers on c-oriented sapphire substrates, exhibited good performance. The physical properties and the photoresponse of ε-Ga2O3 make this material very interesting in view of novel applications.

Graphical Abstract



Highlights

- Epilayers of ε-Ga_2O_3 are employed for the first time as UV photodetectors.
- Fabricated photoresistors are sensitive to UV (270 nm), stable and reliable.
- Their properties compare well with those of the β polytype of Ga_2O_3.
- Bandgap and deep levels are investigated by optical absorption/cathodoluminescence.

ε-Ga_2O_3 epilayers as a material for solar-blind UV photodetectors

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Abstract

Electrical and optical properties of undoped single-phase ε-Ga_2O_3 epitaxial films prepared by MOCVD are reported. It is shown that this still unexplored polymorph of gallium oxide possesses wide bandgap and very high dark resistivity, thus allowing the design and fabrication of solar-blind UV photodetectors. Simple cost-effective photoresistors, fabricated by direct deposition of the epilayers on *c*-oriented sapphire substrates, exhibited good performance. The physical properties and the photoresponse of ε-Ga_2O_3 make this material very interesting in view of novel applications.

1. Introduction

Oxides are materials with very high potential for novel generations of electronic devices, especially for sensing and energy applications. Among the oxide compounds, sesquioxides (In_2O_3, Ga_2O_3, Al_2O_3, and their ternary alloys) have attracted much attention as they present a wide band gap that ranges from 3.7 eV for In_2O_3 to 8.9 eV for Al_2O_3, and lend themselves for many applications, such as power diodes and transistors, gas sensors, and UV photodetectors. Most technological and scientific research activities have so far been focused on β-Ga_2O_3 [1, 2] (monoclinic lattice), as it is the thermodynamically stable polymorph and easy to prepare in form of single crystals [3], thin epilayers [4], and nanostructures [5]. However, β–Ga_2O_3 generally presents anisotropic physical properties, and the single crystals are known to be prone to cleavage, which of course poses serious problems at the time of device manufacturing. For these reasons, there is a growing interest also on metastable polytypes, where metastable means that they tend to convert to β phase upon high-temperature annealing. These polytypes present a crystallographic structure of much higher symmetry (cubic or hexagonal) and are appealing since they do not exhibit the anisotropy typical of the β phase.

Recent work of some of the present the authors on heteroepitaxial growth of Ga_2O_3 demonstrated the coherent growth of ε–Ga_2O_3 on sapphire [6]. This polytype was seen to be stable when

thermally annealed in oxygen atmosphere up to 700 °C, thus allowing the fabrication of devices that work at room or higher temperature. The availability of a hexagonal phase of Ga_2O_3, with good crystallographic matching to gallium and aluminum nitride, and stable up to relatively high temperatures, is also intriguing in view of developing novel nitride-based devices. Indeed, they may take advantage of the more favorable symmetry and epitaxial relationships with respect to the monoclinic phase.

The ε-phase was first observed in the middle of the last century in β-contaminated precipitates that had resulted from thermal decomposition of $Ga(NO_3)_3$ [7], and a recent study showed that this phase belongs to the high symmetry hexagonal system with space group $P6_3mc$ [8]. The study was however carried out on samples that contained more phases than pure ε, which obviously poses some limits to the accuracy of lattice parameter determination.

Single-phase ε-Ga_2O_3 layers were obtained for the first time by Oshima *et al.* by halide vapor phase epitaxy (HVPE) on GaN, AlN, and β-Ga_2O_3 substrates [9], and a little later by Boschi et al. [6] and Xia et al. [10] by means of Metal Organic Chemical Vapour Deposition (MOCVD), respectively on c-oriented sapphire and 6H-SiC.

Unfortunately, a detailed analysis of the ε–Ga_2O_3 physical properties and of its potential use in devices is still lacking, which is the principal motivation of the present paper. Here, we shall first report on the results of optical and electrical investigations of ε–Ga_2O_3 films on c-oriented sapphire, and then show an application of this material as photo-detector for deep-UV radiation. The produced devices demonstrated very good performance in terms of photoresponse, stability, and reproducibility. The UV-response is adequately fast and reliable to render this "unusual" Ga_2O_3 phase a subject of great interest for future applications.

2. Electro-optical investigation and photodetector characteristics

The samples studied and processed in this work were grown by MOCVD on *c*-oriented 2-inches sapphire substrates heated at 650 °C under the growth conditions reported in detail in ref. [6].

Trimethylgallium (TMG) and ultrapure water, stored in dedicated stainless steel bubblers kept at 2 °C and 30 °C, respectively, were employed as precursors. The ratio of the reagent partial pressures in the growth chamber (p_{H_2O} / p_{TMG}) was >1000. The films were transparent and smooth, as checked by SEM (see Fig 1). Previous XRD and AFM investigations, reported in ref. [6], on the same films used for the present work, confirm that they are single-phase ε-Ga_2O_3 and present a maximum roughness of ± 3 nm, although the typical roughness RMS is 1.5 nm. A later investigation by X-ray and high-resolution TEM, performed on our high-quality epilayers, showed that the hexagonal ε phase actually consists of ordered 120°-rotational twin domains 5-10 nm in size. These nano-domains have an orthorhombic structure with *Pna21* space group symmetry, the so-called κ-Ga_2O_3 [11].

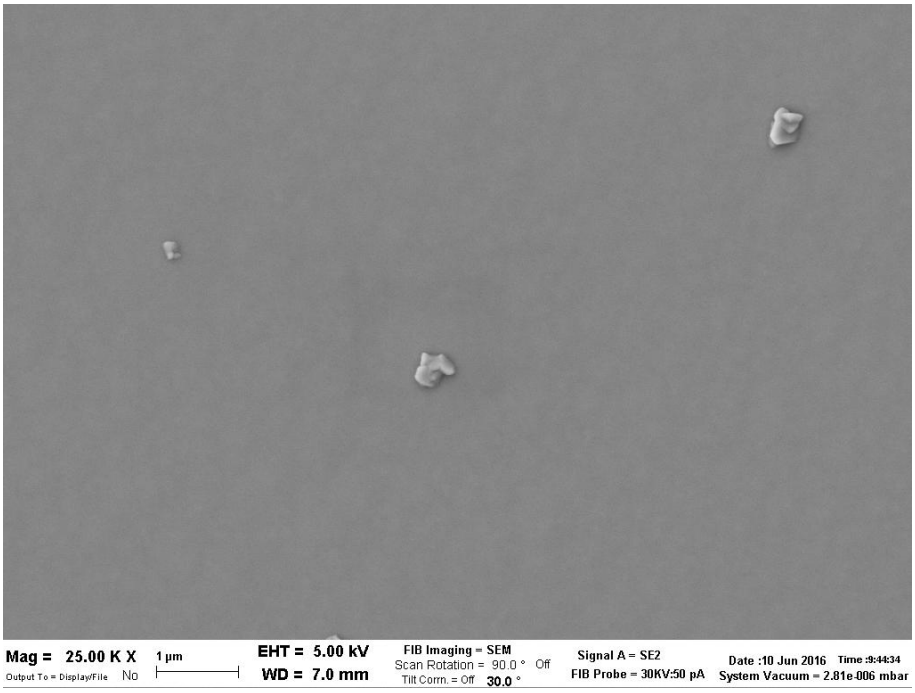

Fig. 1: SEM image for a representative ε-Ga_2O_3 film.

One-face planar-geometry metal–semiconductor–metal was employed for electro-optical characterization. The electrodes, consisting of Au (250 nm) / Ti (20 nm) bilayer, were deposited through a metal mask by thermal evaporation, followed by 10 minutes annealing at 500 °C. The reproducibility of the Au/Ti contact deposition was verified on a large number of thin films,

confirming the robustness of the fabrication procedure. Typically, thin film large-area photodetectors, with a distance of 2 mm between electrodes, were fabricated and measured in this study. As shown below, electrodes were ohmic over the wide range of applied biases and very stable in time and after several temperature cycles.

The photoresponse of ε-Ga_2O_3 thin films was studied in air at fixed temperature (25°C) and constant humidity. The experimental apparatus for Photocurrent Spectroscopy (PCS) consisted of a light source system ORIEL Mod. 66882 suitably screened and focused with an ORIEL Xenon – 300 W lamp, neutral filters, a monochromator CornerStone 130TM 1/8 m Mod. 74000 covering the range 200 ÷ 1600 nm (wavelength resolution of 3 nm). The chopped monochromatic light was focused on the free surface of the ε-Ga_2O_3 film between two Au/Ti electrodes. The spectral distribution of the light beam was measured using a Hamamatsu Photonic Multi-Channel Analyzer PMA-11 and a correction factor was introduced for all the measured spectra, taking into account the spectral distortion of metal contacts, measured for a reference Au/Ti layer of the same thickness deposited on quartz.

The mean irradiance on the active surface of sample detectors (i.e. outside the metal contacts) in the spectral region 200 ÷ 400 nm was around 1 mW cm^{-2}. The photon flux was varied intercepting the light beam with optical neutral filters having optical density in the range 0.1 ÷ 8.0. Voltages between 0 and 200 V were applied to the contacts by means of a Source-Meter Keithley Mod. 2400. Measurements of the dark current at a function of temperature in the range 20-300°C showed that the film conductance increases by three orders of magnitude with the temperature. The corresponding Arrhenius plot is linear and presents an activation energy of 0.695 eV (Fig. 2). This effect is possibly related to the thermal ionization of relatively deep electronic levels located at about 0.7 eV below the conduction band. Similar electronic states were previously detected by DLTS in β-Ga_2O_3 single crystals [12], although their nature was not clarified.

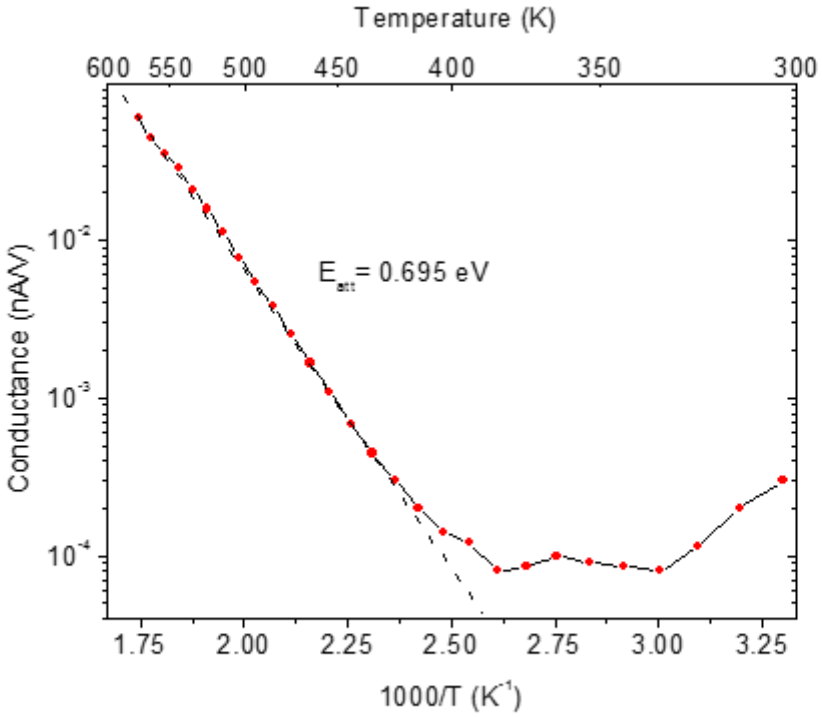

Fig. 2: Arrhenius plot for the dark current in the temperature range 20 ÷ 300 °C.

The photoconductivity spectrum (PCS) for a representative thin film detector is shown in Fig. 3, along with the corresponding optical absorption spectrum. All investigated samples practically showed the same behavior.

Let us examine some aspects of the photocurrent and absorption spectra shown in Fig. 3: though similar, they present some remarkable differences. This can however be explained by considering that photocurrent depends on the concentration and lifetime of photo-carriers, i.e. on the number of photogenerated carriers that actually reach the electrodes. These two entities are not independent of each other, as, for instance, a higher concentration of traps can actually produce more photo-carriers, but also shorten their lifetime; it is the competition between coexisting phenomena that ultimately determines how many carriers do reach the electrodes.

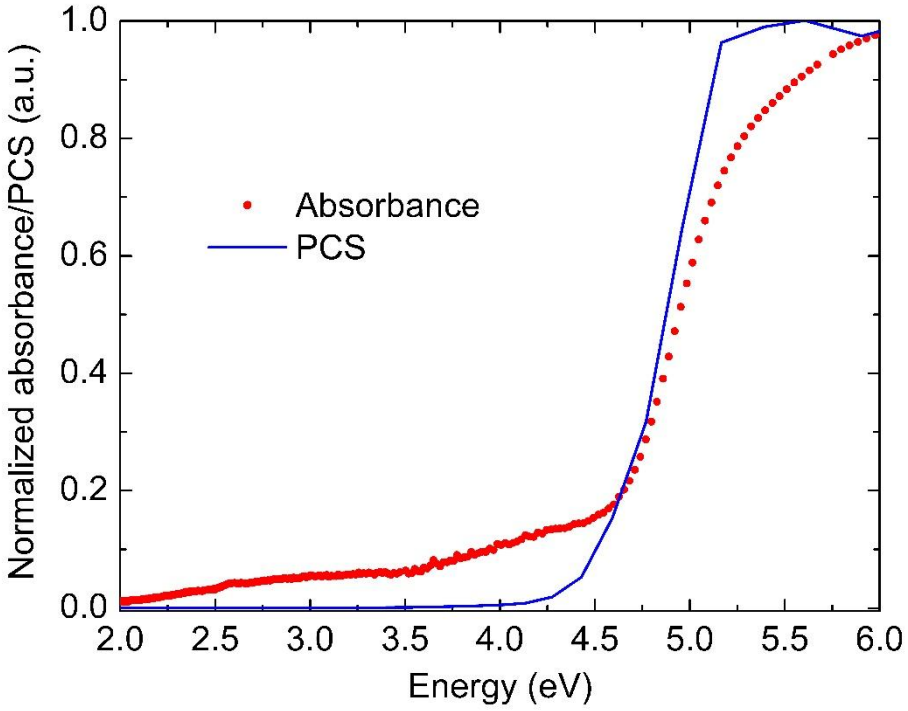

Fig. 3: Spectral photocurrent and optical absorption edge (normalized curves).

The normalized PCS curve of Fig. 3 shows a relatively mild onset at about 4.2 eV that becomes much sharper at about 4.6 eV. The onset of photoconductivity occurs below the estimated bandgap and is ascribed to the presence of energy levels connected with point defects or complexes, as already observed for β-Ga_2O_3 [13, 14]. The optical absorption spectra, recorded at room temperature by using a Varian 2390 spectrophotometer and a 500 μm thick sapphire substrate as reference, look somewhat different: a non-negligible absorption is present already at 2 eV, and it increases monotonically up to band edge. The long tail, extending from the absorption edge down to 2.1 eV, may include other components in addition to excitation of photocarriers from deep levels, for example light absorption by defects at substrate/film interface, etc. Photocurrent values in the same spectral region, far from bandgap edge, are too low to be appreciable in the same linear plot of Fig. 3. It is to be noted that the photogenerated current is low even when the absorbance is non-negligible.

Considering that the bandgap width is a most crucial parameter in order to define the working range of a semiconductor photodetector, we made an assessment of the bandgap of ε-Ga_2O_3 by carrying out absorbance measurements on a large set of films with thicknesses in the range 175 nm ÷ 2 μm. In all cases, the onset due to band-band absorption started at about 4.6 eV and the measured absorption coefficients were in good agreement with those extrapolated from the data of Oshima *et al* [9]. However, attempts to apply the standard Tauc plot ($(\alpha h\upsilon)^2$ vs. $h\upsilon$) for the analysis of the experimental absorbance failed, as the extrapolated absorption edges changed with the layer thickness, i.e. the thinner the layer the higher its apparent bandgap. Furthermore, they all shifted above 5 eV. Obviously, this fact cannot have a physical meaning, since the energy gap of a given material is an intrinsic property and it may not depend on the specimen geometry.

The impossibility of finding a unique bandgap from the Tauc plots suggests that some precaution has to be taken when dealing with absorption in very thin films, as previously pointed out for amorphous silicon [15]. It also indicates that an extended theoretical and experimental investigation on the band structure of this polytype is needed, aimed at clarifying if the band is direct or indirect. Nevertheless, for the purpose of the present work, we succeed to reconcile the experimental absorbance of ε-Ga_2O_3 layers of variable thickness by simply analyzing the absorption coefficients as a function of photon energy. This analysis, based on the set of films with thickness between 175 nm and 2 μm, showed that the optical bandgap of all tested ε-Ga_2O_3 films converges between 4.6 eV and 4.7 eV, hence not so different from that observed for β-Ga_2O_3 [16, 17].

The current-voltage curves, carried out in dark and under illumination at different wavelength, are shown in Fig. 4 and confirm a linear behavior over a wide range of applied biases. Remarkably, Au/Ti contacts were easily fabricated, with good ohmic characteristics. The good linearity of contacts was verified for temperatures up to 300 °C and for incoming light intensities at 250 nm up to about 1.8 mW/cm^2 (inset of Fig. 4).

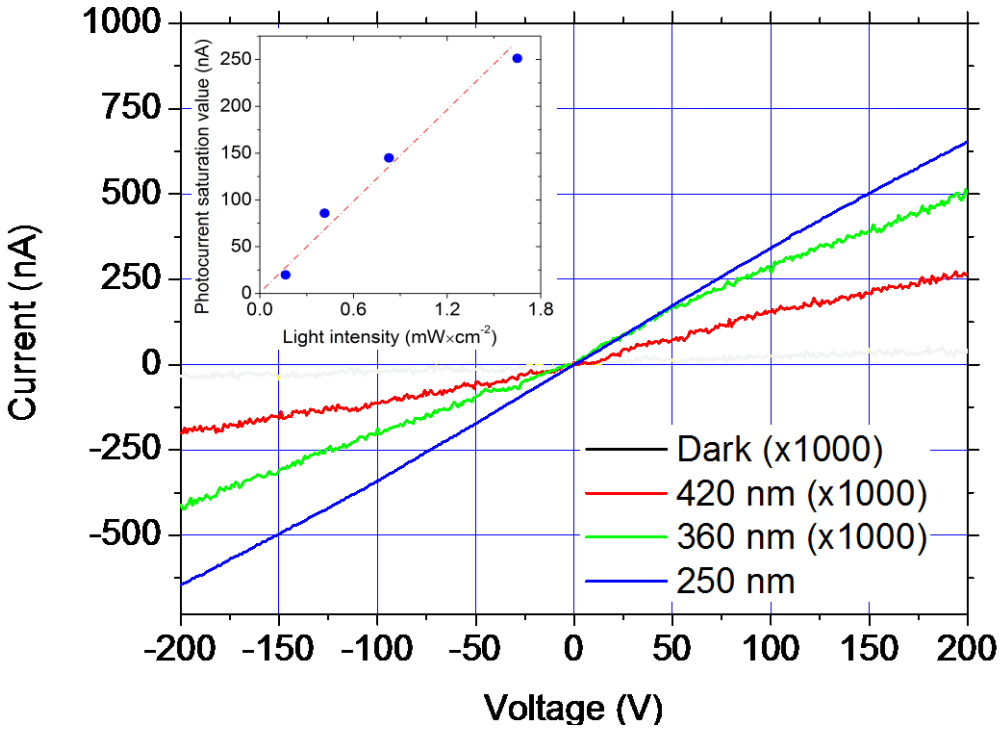

Fig. 4: current-voltage curves in dark and under illumination at different wavelengths. Inset: photocurrent saturation values at fixed applied voltage (75V) as a function of the light intensity (250 nm).

The performance of the ε-Ga_2O_3 UV detector is shown in Fig. 5. The photogenerated signal shows good stability over a long time (a few hundred seconds). After multiple on/off illumination cycles, the photodetector maintained an identical response, demonstrating robustness and good reproducibility. The noise affecting the current output under illumination was estimated to be well below 1%.

The photocurrent transients for on/off operations was also analyzed: the transient following the switch-on may be well described by a single exponential with time constant of 2.5 s, whereas the switch-off shows a bi-exponential behavior (τ_1 = 0.4s and τ_2 = 2.6s, inset of Fig. 5). Photocurrent reaches saturation over longer times (typically few tens of seconds), but with a behavior that depends on the previous illumination of the sample (see repeated on/off in Fig. 5) and on the wavelength of incident photons.

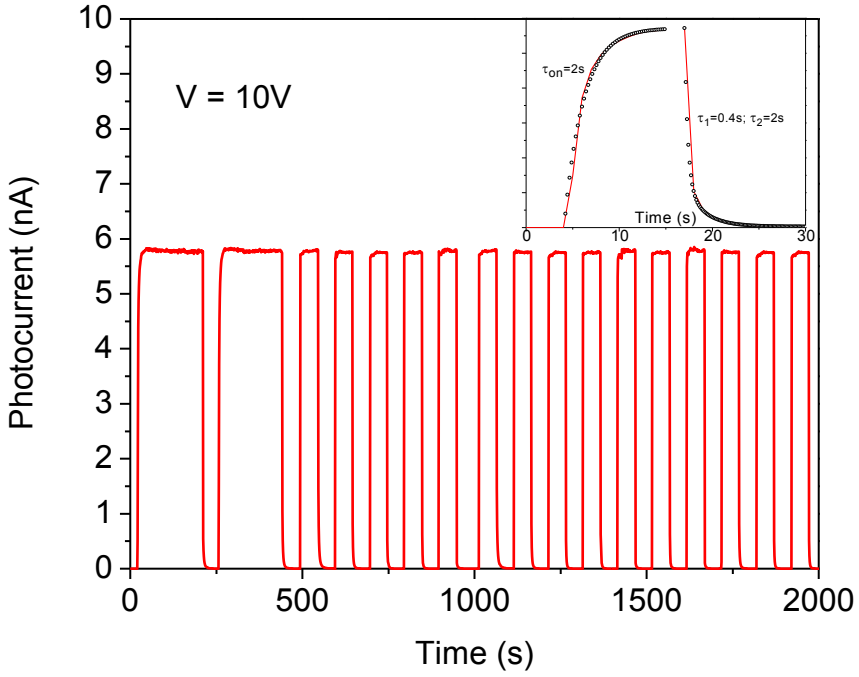

Fig. 5: Time-dependent photoresponse to UV light (270 nm) under an applied bias voltage (V = 10V). Switching on/off with different intervals of time confirms a good stability even over long times. Inset: best fit for the switching on (τ_{on} ~ 2 s) and off (τ_1 ~ 0.4 s and τ_2 ~ 2 s, see text for details).

This behavior, already reported by other authors [5,18], could be ascribed to surface states that strongly influence the photocurrent process, and that obviously modify their action according to penetration depth and intensity of incoming light, as well as (previous) ionization status of the levels. All these features, in particular the difference of photocurrent for illumination below/above bandgap of about three orders of magnitude (Fig. 4), make ε-Ga_2O_3 a very promising material for solar-blind detector applications. Its photo-responsivity indeed compares well with that of β-Ga_2O_3, a proven material for deep-UV detection [2, 18, 19].

In order to check the distribution of deep electronic states as a function of the film thickness, in particular when approaching the film surface and substrate/film interface, we performed some cathodo-luminescence spectroscopy measurements by means of a commercial Gatan monoCL system, mounted onto an S360 Cambridge Scanning Electron Microscope. The system was

equipped with grating and a multi-alkali photomultiplier sensitive in the range 350 ÷ 830 nm (3.6 ÷ 1.5 eV). The spectra were collected with a constant power density.

In Fig. 6a we reported the CL spectra for a representative ε-Ga_2O_3 film as a function of three different accelerating voltage, i.e. electron penetration depth of 50, 150 and 460 nm. The measurements were repeated on different samples to assess the reproducibility of the growth results. The Gaussian deconvolution of the spectra provides three broad bands centered at 2.34 eV (530 nm), 2.67 eV (464 nm), and 2.75 eV (450 nm), respectively (see inset of Fig. 6a, for the spectrum corresponding to penetration of 460 nm). With the same bands, but with different specific intensities, it was possible to fit other spectra as well. No band-band emission was detected, which is common also for other Ga_2O_3 polymorphs.

The CL spectra were deconvoluted using a standard Levenberg-Marquardt algorithm to minimize the Chi-Square. To avoid any possible artifacts, we applied constraint on only the full width at half maximum (w) and set the peak position (xC) and amplitude (A) as free fitting parameters. We imposed a w maximum equal to 0.5 eV. As a result of the fitting, all peak positions were affected by an error of 0.01 eV, lower than the error from the spectral resolution of the system (5 nm). The uncertainty in A and w determination was estimated to be about 5%. The origin of these emission bands is not known yet, but the comparison with literature data for the more studied β-Ga_2O_3 suggests that the three main bands could be connected with intra-gap donor-acceptor pairs due to point defects [20, 21].

As mentioned above, the electron penetration depth was varied between 50 to 450 nm by varying the accelerating voltage between 2.5 and 10 keV. This allowed a depth-resolved CL analysis and the study of the radiative centers distribution along the film cross-section, as previously made for other materials [22]. The volumes affected by generation and recombination mechanisms at different accelerating voltage were estimated from Montecarlo simulations performed with the CASINO software [23].

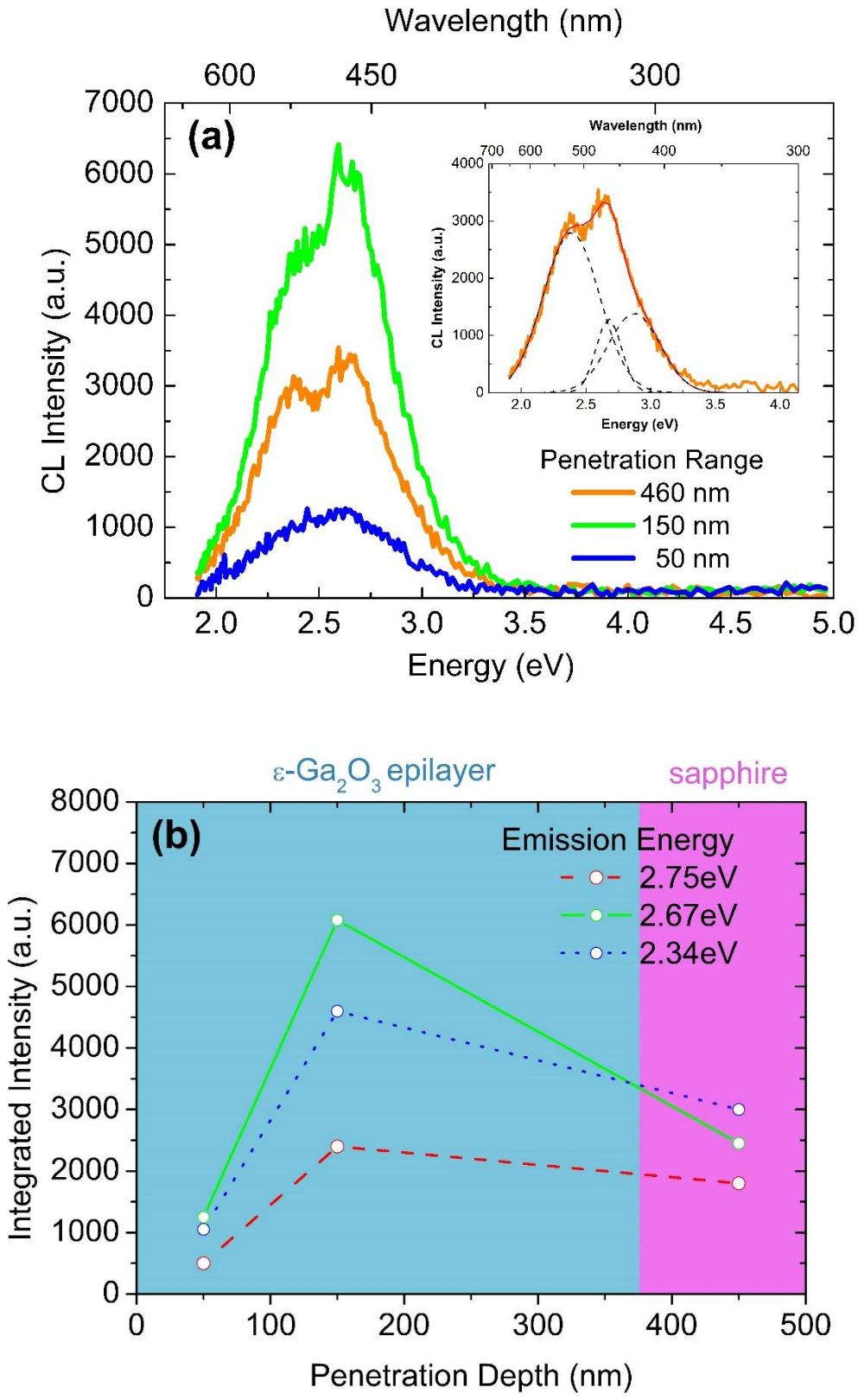

Fig. 6: **a)** Depth-resolved CL analysis at fixed injection power (10^{-5} W) for three different electron penetration ranges. Inset: gaussian deconvolution of the 10 keV CL spectrum; **b)** Integrated CL intensity as function of the electron penetration depth in a 380 nm thick.

Fig. 6b reports the integrated intensity of the three individual emission bands as function of the electron penetration depth for the same sample of Fig. 6a. The maximum intensity of the three bands always occurs corresponding to acceleration voltages of 5 kV (penetration depth of about 150 nm), that means in the middle of the epilayers, away from surface and interface. The lower CL emission close to the external surface is likely related to higher density of non-radiative defects with respect to the bulk of the films. At 10 keV acceleration voltage, the generation-recombination volume surely included the interface with sapphire, and part of the substrate, so that a direct comparison with the emission intensity at lower acceleration voltages is not possible. However, it can reasonably be argued that the concentration of non-radiative defects musty be higher than in the central portion of the epilayer.

The physical picture provided by the different measurements is rather complicated, however it is possible to reconcile all results considering a band diagram like the one reported in Fig. 7.

Actually, the deep donor at about 0.7 eV below conduction band should be the dominant defect, in order to pin the Fermi level and to account for the experimental thermal activation energy of dark current. On the other hand, the three levels (most probably families of levels) with energies ranging from 2.3 to 2.75 eV below the conduction band may well account for the CL emission, for the pronounced absorption tail, and the weaker increase of photo-conductivity for photons of energy higher than 3.0 eV. In this model, we reasonably assumed a negligible contribution of holes to the photocurrent, due to their very low mobility. It should also be noted that the schematic bandgap of Fig 7 does not include the non-radiative deep levels. Actually, the results presented in Fig. 6b may be interpreted in two ways: either considering a maximum concentration of the radiative levels at 2.3-2.75 eV in the middle of the layer section, or a minimum concentration of non-radiative killer centers in the same region of the sample. The present data do not allow removing this ambiguity, neither to clarify the role that radiative and non-radiative deep levels play in terms of the photocurrent relaxation following light switch-off. This interesting question will be certainly addressed in the near future.

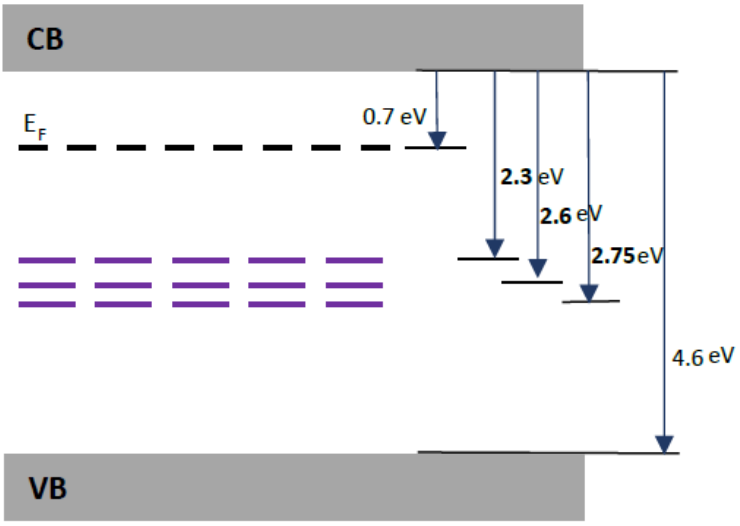

Fig. 7: schematic band diagram for ε-Ga_2O_3 thin films.

3. Conclusions

In summary, undoped single-phase ε-Ga_2O_3 epitaxial films prepared by MOCVD were investigated by photoconductivity, optical absorption and cathodoluminescence. It was observed that this "unusual" gallium oxide polymorph possesses very high dark-resistivity and a bandgap of 4.6-4.7 eV, i.e. comparable with that of β-Ga_2O_3, thus allowing the design and fabrication of solar-blind UV photodetectors. Simple photoresistors, fabricated by alloying ohmic contacts on the epilayers grown on insulating sapphire substrates, exhibited a three orders of magnitude photocurrent difference upon below/above bandgap illumination and relatively fast on/off switching times. The CL measurements revealed a broad blue emission peaked at about 2.6 eV, whose intensity changed along the film cross-section and was maximum away from surface and substrate interface. The band was deconvoluted into three narrower bands, very probably connected with native deep levels. The full set of experimental results may be explained by a band diagram including a deep donor located at 0.7 eV below the conduction band, responsible for the thermal activation of the dark current, and three deeper levels that account for the CL radiative emission and the tails of the absorption and PC spectra. Such levels (and possibly other non-radiative levels) are probably related to the transients

following the light on/off of the ε-Ga2O3 based photodetector. The physical properties and the photo-response of ε-Ga_2O_3 make this particular phase very interesting in view of novel applications.

Acknowledgements

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