OPTICAL ANALYSIS METHOD FOR QUALITY CONTROL OF MICROFLUIDIC DEVICES BASED ON ZINC-OXIDE NANOWIRE ARRAYS

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Zinc Oxide Nanowires (ZnO-NWs) gained a lot of interest due to their diverse and unique semiconductive, optical, and piezoelectric properties. ZnO-NWs has been used in different applications such as nanogenerator of electricity [1], chemical sensors [2], photovoltaic cells [3] and recently in water purification [4]. In all those applications, performance is directly related to the actual properties of the ZnO-NWs. The latter properties can be investigated in detail using Scanning Electron Microscopy (SEM), X-Ray Diffraction and High Resolution Transmission Electron Microscopy for the purpose of characterization and optimization of the growth process.

However, once ZnO-NWs are integrated within a microfluidic device, there is a need for a much simpler characterization technique for checking *in situ* the quality and uniformity of ZnO-NWs during and after their growth. In this work, we take advantage of the strong dependence of the ZnO-NWs properties on their dimensions, density and possible contamination, considered here as quality indicators. We also note that those indicators can be obtained by optical measurement of effective thickness (d_{eff}), effective refraction index (n_{eff}) and light absorption, respectively, of the ZnO-NW array, which is considered here as a thin film layer (Fig.1). We propose herein a simple and time-saving method measuring all those parameters in the same experiment, based on the reflection spectral response in the Ultraviolet (UV), Visible (Vis) and Near-Infra-Red (NIR) range.

The purity of the ZnO growth is observed through absorbance, while, ZnO-NWs density (n_{eff}) and height can be obtained from the reflection response, which reveals interference patterns in the ZnO-NWs layer. The optical path ($d_{eff} \times n_{eff}$) is retrieved by the Free Spectral Range (FSR).

The ZnO-NWs are synthesized using the hydrothermal method [5]. Typical results for different growth times are shown in Fig. 2. A top view of the synthesized NWs array grown over silicon is shown in Fig. 1 together with illustrative schematic of the synthesized NWs cross-section at different positions. Denser and longer NWs are synthesized on the edge of the chip while the density and length decrease moving away from the edge. The n_{eff} of the ZnO-NWs layer is in-between air and ZnO refractive indices, based on the ZnO-NWs density. The measured absorbance (normalized to silicon) is shown in Fig. 3 for ZnO growth for 2 hours (2h) at different positions. Absorbance cut-off is observed around 370 nm corresponding to the ZnO bandgap absorption. The ripples correspond to the reflection response of the ZnO thin film where the smallest FSR observed at the edge relates to the denser ZnO-NWs growth (higher n_{eff}) compared to the other two regions. Fig. 4 relates to a 3h growth where some residual Zn(OH)_x masking the reflection response at the edge in addition to the expanded absorption. Besides density, the purity of the synthesized ZnO can be evaluated by the absorbance cut-off (Fig. 5) for the 4h growth where the purest ZnO is synthesized at the center of the chip.

Word count: 496

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Fig. 1. (Top) Illustrative schematic of ZnO-NWs growth cross sections at different positions over a flat silicon substrate and (bottom) over a structured silicon substrate, typical of a microfluidic device.



Fig. 2. Typical cross-section-view and top-view SEM photos of ZnO-NWs of different NWs densities and different height due to different growth time. The ZnO-NW arrays can be seen as a thin film layer of different effective refraction index depending of NWs density (ratio of ZnO/air).



Fig. 3. Measured UV-Vis-NIR absorption spectra of ZnO-NWs synthesized for growth time of 2 hours at different positions showing effect of the NWs density in the interference pattern.



Fig. 4. Measured UV-Vis-NIR absorption spectra of ZnO-NWs synthesized for growth time of 3 hours at different positions showing effect of the NWs density and $Zn(OH)_x$ residuals absorption near the chip edge.



Fig. 5. Measured UV-Vis-NIR absorption spectra of ZnO-NWs synthesized for growth time of 4 hours at different positions showing effect of $Zn(OH)_x$ residuals absorption near the chip edge.