Influence of Hydrothermal Synthesis Conditions and Device Configuration on the Photoresponse of UV Sensors Based on ZnO Nanorods

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Abstract— Zinc oxide nanorods in the form of powder or nanostructured films were synthesized by the hydrothermal method using aqueous solutions. Once the synthesis time was completed, the reaction vessel was naturally cooled or submitted to a quenching process. X-ray diffraction, Scanning Electron Microscopy and Transmission Electron Microscopy techniques were employed to characterize the crystallinity, morphology and dimensions of the nanorods as well as their growth direction. Suspensions of powder material were spin-coated over oxidized silicon substrates in order to obtain nanostructured layers. Photodetectors based on in-situ grown and spin-coated films were prepared in order to evaluate the influences of cooling rate and device configuration on the UV sensing characteristics. Spin-coated layers showed an excellent performance (current change by more than four orders of magnitude), better than nanorods grown directly on the substrate during the hydrothermal process. For both configurations, the sensitive layers built from quenched samples exhibited enhanced UV photoresponses when compared to the naturally cooled ones.

Index Terms— cooling rate, hydrothermal synthesis, nanorods, UV photodetectors, zinc oxide.

I. INTRODUCTION

THE increasing progress in nanotechnology led to the fabrication of a wide range of semiconducting oxide 1D nanostructures having great prospects for application in optoelectronic devices [1]-[5]. Among the materials studied in one-dimensional form, zinc oxide (ZnO) is one of the most exploited semiconductors not only due to the simplicity of its processing, but also due to its intrinsic properties, like the wide direct band-gap (3.37 eV), large exciton binding energy (60 meV) and excellent chemical and thermal stability, allowing its use in a large number of devices [5]. In this context, one promising nano-scale sensing device is based on nanostructured zinc oxide for application in ultraviolet photodetectors. Due to large surface-to-volume ratios and Debye length comparable to their dimensions, UV light sensors based on single nanowires, nanostructured oriented layers or nanostructure/film combinations show good sensitivity, short response and recovery times and high internal gain [6]-[10] when compared to bulk or thin film devices. Moreover, their low dimensions make possible their incorporation in binary switches, imaging devices and components for light-wave communications [10].

For nanoscale synthesis, the control of morphology, size uniformity, growth direction and degree of crystallinity are decisive factors due to their influence in the functionality of the nanostructures and their application [4]. Methods such as hydrothermal synthesis [11]-[13], chemical vapor deposition [14], [15], thermal evaporation [16], [17] and sputtering [18], [19] were used for obtaining semiconducting oxides in powder and nanostructured film forms.

Among the chemical methods, hydrothermal synthesis is often used because it allows the control of grain size, morphology and degree of crystallinity by simple changes in the experimental conditions. The ZnO morphology is determined by a great number of variables such as substrates, counter-ions, different pH conditions and catalyst layers [20]-[22]. However, the cooling rate of the system after the hydrothermal process has been systematically overlooked by the researchers, judging by the absence in the literature of more studies on its control and effects [11].

In this work we undertake an evaluation of the effect of cooling rate during hydrothermal synthesis over the morphology, degree of crystallization and, consequently, the UV photoresponse of sensors based on ZnO nanorods. Nanostructured ZnO layers obtained by two different methods (in-situ growth and spin-coating of nanorod suspensions) were studied in order to determine the influence of device configuration on the performance of UV photodetectors.

II. EXPERIMENTAL PROCEDURE

Oxidized silicon substrates coated with Ti/Zn thin films were used for the hydrothermal synthesis of nanostructured layers composed of ZnO nanorods. For this purpose, room temperature RF and DC sputtering depositions were performed

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in order to obtain a 15 nm Ti adhesion layer and 50 nm of Zn thin film, respectively. The zinc film easily oxidizes in the early stages of hydrothermal processing, working as a seed layer for nanostructured ZnO growth.

Zinc oxide nanostructures were grown using aqueous solutions of zinc nitrate ($Zn(NO_3)_2{\bullet}6H_2O$, 98% purity - Sigma-Aldrich) and hexamethylenetetramine (HMTA, 99% - Sigma-Aldrich) as precursors. The reagents were dissolved in distilled water at room temperature, with the 0.1M HMTA solution being slowly added to the zinc nitrate solutions (0.055M or 0.0275M) under continuous stirring. The resulting transparent solution was placed in a pressure vessel and the substrates were suspended in the solution using Teflon tape, with the zinc-coated surface facing the bottom of the flask. The hydrothermal process was performed by immersing the reaction chamber in a hot Vaseline bath at 110ºC for 6 hours, under continuous stirring. The pressure vessel was then removed from the hot bath and either allowed to cool down naturally (in air), or immersed in a room temperature Vaseline bath in order to increase the heat-transfer rate. The nanostructured precipitates were washed several times with distilled water and ethanol and dried in an oven at 75ºC.

Suspensions made from the powders obtained in the hydrothermal procedure previously described were used for preparing spin-coated films over oxidized silicon substrates. The parameters used in this process were: 300 rotations per minute (rpm) velocity and 15 seconds spinning time. For each sample, four spin-coated layers were sequentially deposited, with a drying process at 75ºC for 5 minutes being applied after each layer. A final treatment of these samples was performed in a tubular furnace at 500ºC for 30 minutes. Table I summarizes composition, experimental conditions and nomenclature for both the in-situ grown and spin-coated samples.

Table I: Composition of solutions, processing methods and sample names.

Sample name	Zinc nitrate concentration (mol/l)	Cooling method	Film preparation
Z5NI	0.055	natural	*in-situ*
Z5QI	0.055	quenched	*in-situ*
Z2NI	0.0275	natural	*in-situ*
Z2QI	0.0275	quenched	*in-situ*
Z5NS	0.055	natural	spin-coating
Z5QS	0.055	quenched	spin-coating
Z2NS	0.0275	natural	spin-coating
Z2QS	0.0275	quenched	spin-coating

X-Ray Diffraction scans (XRD, Rigaku Rint 2000) were performed in order to assess the crystalline structure of the nanostructured deposits and their orientation. The influence of the cooling process over the morphology and dimensions of the nanorods was studied by Field Emission Scanning Electron Microscopy (FE-SEM, Zeiss Supra 35). High Resolution Transmission Electron Microscopy (HR-TEM, Jeol JEM 2100) and Selected Area Electron Diffraction (SAED) were performed on individual nanorods, naturally cooled or quenched, in order to determine the growth direction and the influence of the cooling rate over the degree of crystallinity and morphology of the nanostructures.

In order to carry out the electrical measurements, 150 nm thick and 172 μm diameter sputtered platinum electrodes were deposited onto the in-situ grown (Fig. 1a) and spin-coated films using a shadow mask (Fig. 1b). The distance between the electrodes was 200μm. The UV response of the devices was registered at room temperature and atmospheric pressure using an Hg vapor lamp. The measurements were performed with a Solartron SI1287A electrochemical interface, applying different light intensities.

The two different architectures allow us to study the UV response of devices built from nanorods obtained in the same synthesis process. By comparing the measurements performed on the in-situ grown nanorods with the spin-coated ones, the effect of the continuous layer present in the conduction path (see Fig. 1a) can be assessed. A similar study has been reported by Kumar et. al. [23], but in their work the comparison was made between thick ZnO films with or without the presence of ZnO nanoneedles at the surface. In either case, the continuous film was always present, while in our study, the continuous layer is part of one conduction path, being absent in the other one (Fig. 1b). This should allow a clearer picture regarding the role of the continuous layer on device performance.

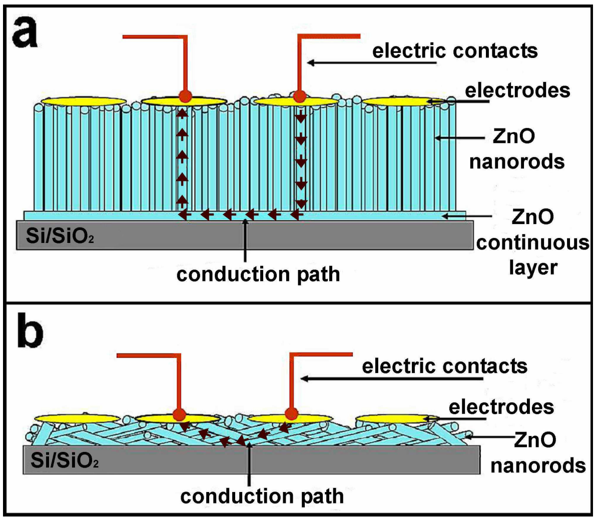

Fig. 1. The configuration of the UV sensors based on (a) in-situ grown and (b) spin-coated ZnO nanorods.

III. Results and Discussion

Quenching heat-transfer between a solid and a liquid is notoriously difficult to quantify, since it depends on extrinsic factors such as sample shape, liquid volume, sample and liquid temperatures, fluid circulation, etc. In the absence of available data for cooling rates in hydrothermal processes, we quote typical heat-transfer coefficient values for the quenching of steel samples in different fluids: water (1400Wm^{-2}K^{-1}), oil (270 Wm^{-2}K^{-1}) and air (21 Wm^{-2}K^{-1}) [24]. Based on this data, we expect cooling rates around ten times higher for Vaseline quenching, when compared to the natural cooling in air.

Fig. 2 shows the FE-SEM images of the naturally cooled (Fig. 2a) and quenched (Fig. 2b) rods obtained from the most concentrated solution, samples Z5NI and Z5QI, respectively. The naturally cooled rods have very few surface defects, tapering along the length, many of them exhibiting pointed tips (Fig. 2a) with a length of approximately 5 μm.

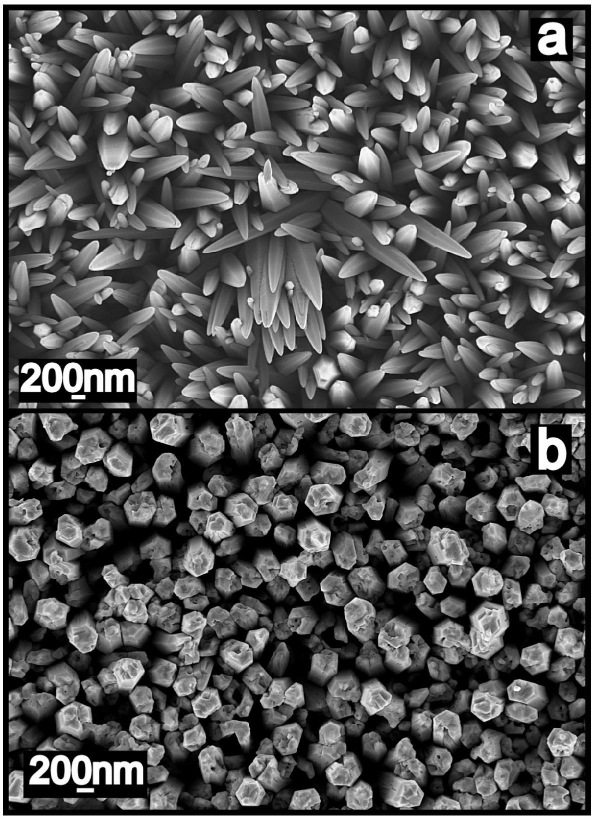

Fig. 2. FE-SEM images of the samples (a) naturally cooled and (b) submitted to quenching, obtained from the most concentrated solution (Z5NI and Z5QI, respectively).

Looking at the quenched sample (Z5QI) it is obvious that the changes in the cooling rate greatly affected the morphology and length of the rods. The FE-SEM images (Fig. 2b) indicate that the rapidly cooled nanostructures exhibit large hollow tips of approximately 250 nm, having half the length of the Z5NI nanostructures (2.5 μm), with many imperfections and a poorly defined hexagonal cross section. In both cases, the nanostructures exhibit a very similar, high substrate coverage density. Comparing these two samples, it is clear that for the quenched one the crystallization process was drastically reduced once the reactor was immersed in the room temperature Vaseline bath. The discontinuity in the thermal energy supply lead to a decrease in the rate of dissolution/recrystallization during the cooling stage, typical of the hydrothermal process [25], [26]. As a consequence, there was a decrease in the length and in the degree of crystalline perfection for the quenched nanorods, implying a higher number of defects.

Fig. 3 shows the HR-TEM images of nanorods broken from the naturally cooled (Fig. 3a) and quenched samples (Fig. 3b).

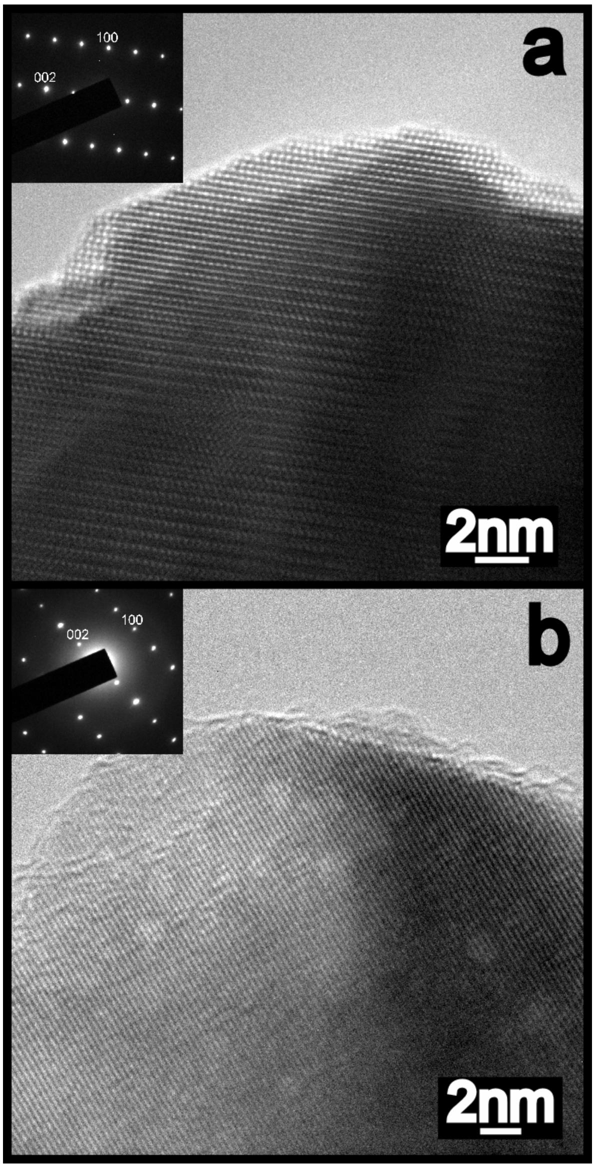

Fig. 3. HR-TEM images of the (a) naturally cooled and (b) quenched nanorods composing the Z5NI and Z5QI samples, respectively. The insets present the SAED patterns taken on these nanostructures.

As can be noticed in Fig. 3a, the naturally cooled rods have a hexagonal structure characteristic of wurtzite ZnO. On the other hand, the quenched nanorod exhibits clusters imbedded in a thicker amorphous coating layer surrounding the crystal (Fig. 3b), indicating a higher degree of defects when compared to the naturally cooled nanorod. The SAED patterns for the naturally cooled (Inset Fig. 3a) and the quenched rods (Inset Fig. 3b) indicate the growth direction as being [001] in both cases.

Fig. 4 shows the XRD patterns of the Z2NI (Fig. 4a) and Z2QI (Fig. 4b) nanorod arrays. All the peaks present in the diagrams are indexed to the wurtzite crystalline structure of zinc oxide (JCPDS card no. 36-1451). The higher intensity obtained for the (002) diffraction peak, when compared to the relative intensity of the (101) plane that is usually correlated to the maximum intensity peak of ZnO, indicates the anisotropic growth of the nanorods in the [001] direction, perpendicular to the substrate, confirming the HR-TEM results. The less intense, wider peaks of the Z2QI sample indicate a smaller crystal diameter for this sample when compared with the naturally cooled one (Z2NI). The XRD patterns are characteristic for all the nanorods grown directly on the substrate.

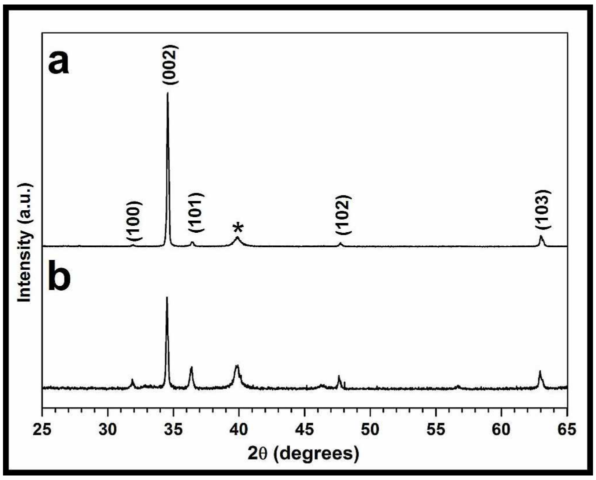

Fig. 4. X-Ray diffraction patterns of (a) Z2NI and (b) Z2QI samples. The peaks marked with the * symbol are due to the platinum electrodes.

When the zinc nitrate molar concentration was halved, thinner and shorter nanorods were obtained (Fig. 5). This can be attributed to a decrease in the Zn^{2+} concentration, being consistent with the results reported in the literature by other researchers [20], [22]. Fig. 5a presents the spin-coated layer based on the powder obtained from the 0.0275M zinc/0.1M HMTA solution and naturally cooled (Z2NS). The sample exhibits good substrate coverage, with some agglomerates formed during the drying process of the solvent used for powder suspension. The rods are interconnected, with diameters in the 500nm to 1μm range and several microns in length (Inset Fig. 5a). The Z2QS sample, obtained from quenching the less concentrated zinc solution, preserves some of the characteristics of the Z2NS sample, like good substrate coverage, interconnected nanostructures and agglomerated regions (Fig. 5b). The Inset of Fig. 5b shows that the rods composing this sample are thinner and shorter than the ones from the Z2NS sample.

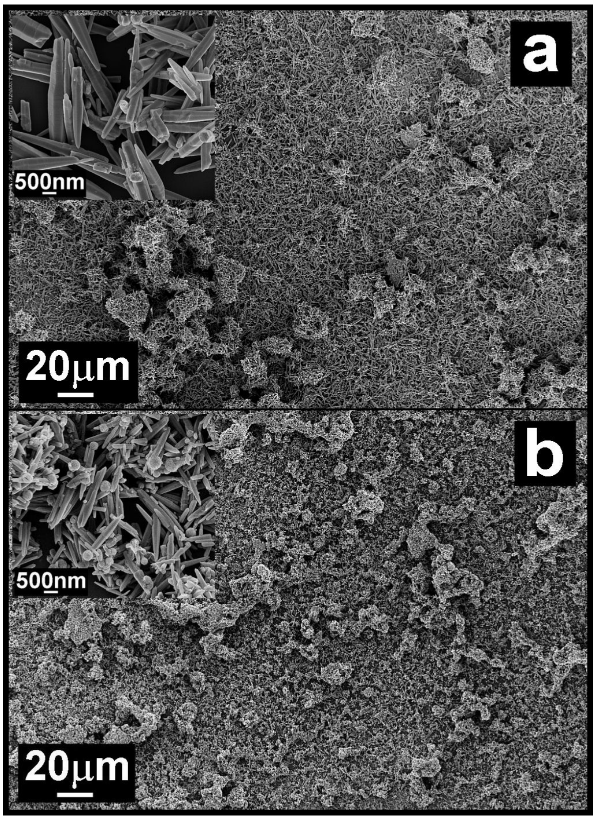

Fig. 5. FE-SEM images of the spin-coated samples obtained from the powders submitted to (a) natural cooling (Z2NS) and (b) quenching (Z2QS) using the 0.0275M zinc/0.1M HMTA solution.

Guo et al. [22] performed a very detailed study of the effect of experimental parameters (catalytic layer, temperature, time, etc.) over the growth and morphology of ZnO nanostructures, but no reference was made to the cooling process used. The FE-SEM results on Fig. 5, once again, indicate that the cooling rate is a very important parameter, playing an important role in the crystallization process and therefore having a significant effect on rod morphology.

Fig. 6 presents the UV response of the naturally cooled and quenched nanorods composing the nanostructured films grown directly in solution (Fig. 6a) and spin-coated (Fig. 6b), using a 5V potential difference and a 2.4mW/cm^2 light intensity.

As reported in the literature, the UV photoconductivity in ZnO is basically the result of two processes: one surface-related and another bulk-related [23]. The surface-related photoconductivity is due to the adsorption and desorption of chemisorbed oxygen at the surface of the ZnO nanorods, which generates a carrier-depletion layer, while the bulk related process is associated with the release, upon UV illumination, of the electrons trapped by deep defect states [27], [28].

The quenched samples obtained in-situ (blue and green lines in the on-line version of Fig. 6a) exhibit a significantly higher UV photoresponse when compared with the naturally cooled ones (red and violet lines on-line, Fig. 6a). The enhanced sensitivity (defined as the ratio of current values measured under illumination and in the dark) can be attributed to the existence of a higher amount of defects at the surface and in the bulk of the quenched nanorods, increasing both contributions to the UV response. When compared to the results obtained by Bera et al. [27], the response of these sensors is relatively slow, due to the thicker nanorod diameters and continuous layers in contact with the substrate.

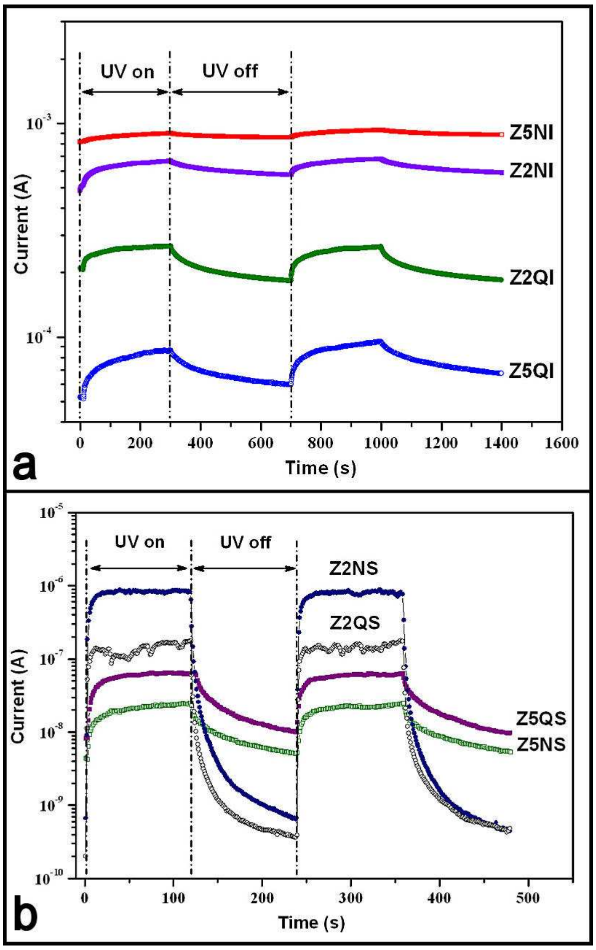

Fig. 6. UV photodetection response of the sensors based on (a) ZnO grown directly in solution or (b) spin-coated from powder suspensions. The parameters used for these measurements were 2.4mW/cm^2 UV intensity and 5V.

The sensors based on spin-coated layers exhibit short response/recovery times (defined as the times needed for reaching 90% of the final current values) and high sensitivity (Fig. 6b). As one can observe, the quenched spin-coated Z5QS layer (Fig. 6b) presents a better performance when compared to the naturally cooled one (Z5NS in Fig. 6b). Its response/recovery times are of 35s and 93s, respectively and its sensitivity is 6.2, while the Z5NS sample presents response/recovery times of 67s and 100s, respectively and a sensitivity of 4.75. The Z2NS/Z2QS set (Fig. 6b) show an even better performance, when compared with the Z5NS/Z5QS set, due to a smaller crystal diameter which reduces the bulk contribution to the final response. When exposed to UV radiation, these devices present a change in the electrical current of more than three orders of magnitude. The sensitivities are of 880 for the Z2QS and 1250 for the Z2NS sensors. The response time is 25s for the Z2NS and 12s for the Z2QS sample, while their recovery times are 108s and 96s, respectively. The most striking differences in photoresponse are clearly apparent when comparing the results obtained with different sensor geometries (curves in Fig. 6a and Fig. 6b). The sensing performances of the devices made by spin-coating are superior, both in terms of sensitivity and response time. This general trend applies to all the synthesis conditions used in this study. This can be attributed to the different orientation of the rods in relation to the light source and, more importantly, to the fact that in the spin-coated samples the current path is exclusively made of nanorods, while on the in-situ grown samples a significant part of the current transport occurs in the continuous film in contact with the substrate.

Fig. 7 presents the photoresponse of the Z2NS sensing device submitted to different UV light intensities. Even for a small intensity of 0.1mW/cm^2, the Z2NS sample presents a good sensitivity, the nanostructured film exhibiting a change in the electrical current of more than one order of magnitude (red line in the on-line version). As expected, with the increase in light intensity, the sensors show an increase in the photocurrent, leading to a response three times more intense for the highest intensity used.

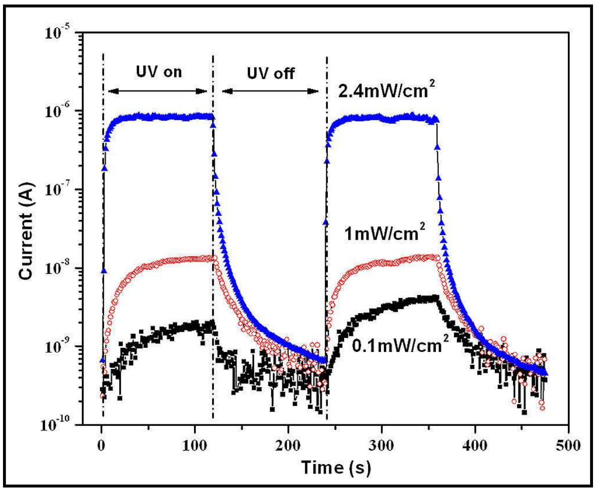

Fig. 7. The photoresponse to different applied UV light intensities for the Z2NS devices based on spin-coated layers. For testing the response to different light intensities, a potential difference of 5V was used.

IV. CONCLUSION

ZnO nanostructured films were grown directly from solution by hydrothermal synthesis or spin-coated from suspensions based on powder products. During the synthesis process, the cooling rate and zinc concentration were varied for observing their influence over the morphology and crystallinity of the developed nanostructures. XRD patterns and TEM analyses showed that the nanorods grew oriented in the [001] direction in all cases, being perpendicular to the substrate when grown directly in the reaction vessel. FE-SEM images indicated that the cooling rate is an important parameter to be controlled due its influence on the crystallinity and morphological characteristics of the nanostructures. HR-TEM proved that the decrease in the crystallization process during quenching lead to the formation of an amorphous layer at the surface of the nanorods. The UV response of the spin-coated layers is improved when compared to the ones grown directly during hydrothermal synthesis, both in terms of sensitivity and response time. Moreover, the quenched samples exhibit higher sensitivity to UV light than the naturally cooled ones. The results indicate that nanostructures with different morphologies, crystalline levels and properties can be obtained by controlling the cooling rate applied during the hydrothermal process.

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