# ЭЛЕКТРИЧЕСКАЯ РАЗМЕРНАЯ ОБРАБОТКА МАТЕРИАЛОВ

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# THE IMPACT OF HIGH ENERGY ION IRRADIATION UPON CO GAS SENSITIVITY OF NANOSTRUCTURED GAN EPILAYERS

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#### Introduction

GaN is a high temperature material with excellent potential for use in various fields including high power electronics, light emitting devices, and sensors. It exhibits pronounced chemical stability and radiation hardness, the latter property being considerably enhanced by nanostructuring of the compound by means of photoelectrochemical (PEC) etching techniques [1]. Recently we found that nanostructuring is an effective tool for improving the sensitivity and selectivity of GaN-based gas sensors [2]. This paper reports the investigation of the influence of high energy heavy ion irradiation and subsequent annealing on CO gas sensitivity of photoelectrochemically nanostructured GaN epilayers

#### **Experimental details**

The unintentionally doped GaN layers used in our experiments were grown by low-pressure metalorganic chemical-vapor deposition (MOCVD) on (0001) *c*-plane sapphire substrates. The thickness of the layers was 2.7  $\mu$ m and the concentration of free electrons was of about 10<sup>17</sup> cm<sup>-3</sup>. Ti/Au ohmic contacts of concentric annular design were deposited by lift-off on the GaN epilayers. Subsequently the samples were subjected to PEC etching in 0.1 M of KOH solution for 20 min at room temperature under illumination of a 350W Hg lamp focused on a spot of 5 mm in diameter. The obtained morphology represents a uniform distribution of conical nanostructures, as illustrated in fig. 1. The origin of these nanostructures is related to threading dislocations inherent to GaN epilayers grown on lattice-mismatched substrates [3]. The electrochemically treated samples were irradiated at room temperature by 166 MeV Xe<sup>+23</sup> ions at doses 10<sup>10</sup>, 10<sup>11</sup> and 10<sup>12</sup> cm<sup>-2</sup>. The irradiation was carried out on the IC-100 cyclotron at the Joint Institute for Nuclear Research in Dubna, Russia. After ion irradiation and preliminary gas sensitivity exploration, the GaN samples were subjected to rapid thermal annealing in N<sub>2</sub> atmosphere for 1 min. For the gas response investigation the samples were mounted in a cell where the CO gas concentration was varied from 200 to 2000 ppm, whereas the sample temperature was increased from 180 to 280°C.

### **Results and discussion**

The relative sensitivity S was calculated using the equation:

$$S = \frac{R_{N2} - R_{CO}}{R_{N2}} \times 100\%$$

where  $R_{N2}$  is the electrical resistance in nitrogen as a carrier gas, while  $R_{CO}$  is the resistance in CO gas environment.

The sensitivity of PEC etched GaN layers to CO concentration was found to be linear before ion irradiation. The gas sensitivity as a function of sensor temperature is illustrated in fig. 1. The sensor response time was in the range of 2-3 sec, whereas the recovery time did not exceed 10 sec at  $280^{\circ}$ C.

After ion irradiation, the gas sensitivity was reduced to 17 % at the dose of  $10^{10}$  cm<sup>-2</sup>, and even to 6 % for the samples subjected to ion irradiation at the dose of  $10^{12}$  cm<sup>-2</sup> (fig. 2). This corresponds to the diminution of the sensitivity by at least ten times in comparison with the sensitivity of non-irradiated samples

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(fig. 1). The drastic reduction of the sensitivity is expected to be caused by the large number of lattice defects induced by ion irradiation in the sensitive layer of GaN which is located at the base of conical structures.



Fig. 1. The morphology of PEC nanostructured GaN and gas response towards 200, 1000 and 2000 ppm of CO before  $Xe^{+23}$  ion irradiation. 1 – 2000 ppm CO; 2 – 1000 ppm CO; 3 – 200 ppm CO



Fig. 2. The gas response of PEC nanostructured GaN toward 1000 ppm of CO for 3 doses of Xe irradiation. The initial three points represents data for the samples before annealing.  $1 - \text{Dose } 10^{10}$ ;  $2 - \text{Dose } 10^{11}$ ;  $3 - \text{Dose } 10^{12} \text{ cm}^{-2}$ ; CO = 1000 ppm; Ts = 280 C

Fig. 3. The influence of Xe irradiation and rapid thermal annealing on recovery time of the nanostructured GaN based gas sensor. 1 - after Xeirradiation;  $2 - after tr. an. 500 \ C; 3 - after tr. an.$ 700  $\ C; 4 - after tr. an. 800 \ C; CO = 1000 ppm$ 

An effective method for curing the defects induced by high energy ion irradiation is thermal annealing [4]. The annealing-induced restoration of the crystal lattice is particularly effective for nanostructured semiconductor materials. The results of our investigation prove to confirm this trend. From the data illustrated in fig. 2 it can be seen that for the highest dose of  $10^{12}$  cm<sup>-2</sup> the rapid thermal annealing at 800°C leads to 50 % restoration of the sensitivity parameter in comparison with the non-irradiated samples.

The high energy Xe irradiation influences not only the sensitivity parameter, but also the recovery time of the gas sensor. In our case, the response time was in the range of 2–3 sec before and after  $10^{12}$  cm<sup>-2</sup> Xe irradiation, while the recovery time was found to be in the range of 35–36 sec after this high dose irradiation and of about 22 sec after subsequent rapid thermal annealing at 800°C (fig. 3).

### Conclusions

Host defects created in nanostructured GaN epilayers by high energy Xe<sup>+23</sup> ion irradiation lead to considerable deterioration of the CO gas sensitivity which is caused probably by irradiation-induced changes in the surface properties of GaN nanostructures. It should be noted that the mechanism of high energy particle interaction with nanostructured GaN require further investigation since the observed increase in the conductivity of these layer after irradiation could involve different specifics. Nevertheless, the results

presented in this report are promising from the point of view of potential applications of nanostructured GaN in gas sensor electronics operating in high energy ion irradiation environment.

Acknowledgements. One of the authors (O. Volciuc) would like to thank the NATO Science for Peace and Security Programme for the financial support under Reintegration Grant N 983328. The financial support from the Supreme Council for Research and Technological Development of Moldova is also acknowledged.

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Received 10.05.10

## Summary

Photoelectrochemically nanostructured GaN epilayers were found to exhibit good sensitivity towards CO in the temperature range from 180 to 280°C. We show that subjection of nanostructured GaN samples to 166 MeV  $Xe^{+23}$  ion irradiation causes considerable reduction of the gas sensitivity, while post-irradiation rapid thermal annealing results in sensitivity restoration, the effect being dependent upon the dose of irradiation and annealing temperature. A 50 % restoration of the relative sensitivity is demonstrated after rapid thermal annealing for 1 min at 800°C in samples irradiated by  $Xe^{+23}$  ions at a dose of  $10^{12}$  cm<sup>-2</sup>.