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Controlled hydrothermal synthesis of ZnO nanorods for high-performance gas sensors

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Abstract. Controlled synthesis of ZnO nanorods with tailored crystallinity and morphology is of great importance for the development of high-performance gas sensors. In this study, ZnO seed layers were deposited on glass substrates by RF magnetron sputtering and subsequently annealed at 400 °C to improve crystallinity and surface uniformity. Hydrothermal growth of ZnO nanorods was then carried out under different precursor concentrations (0.01 M:0.01 M and 1 M:1 M zinc acetate/HMTA) to investigate the effects of solution chemistry on nanorod formation. X-ray diffraction (XRD) revealed a dominant (002) peak in all samples, confirming c-axis oriented growth, while scanning electron microscopy (SEM) demonstrated that higher precursor concentrations yielded longer, densely packed nanorods with well-defined hexagonal cross-sections. The results highlight that both seed layer annealing and precursor concentration strongly influence the structural and morphological evolution of ZnO nanorods. Optimized synthesis conditions lead to vertically aligned, highly crystalline nanorods with increased aspect ratios, which are expected to enhance gas adsorption and electron transport, making them highly suitable for resistive-type gas sensing applications.

Keywords: ZnO, nanorods, gas sensor, MOS, hydrothermal method

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Introduction

The demand for efficient, selective, and low-cost gas sensors has grown significantly in recent years due to increasing concerns over environmental pollution, industrial safety, and public health monitoring [1–3]. Among the different sensing platforms, metal oxide semiconductor (MOS)-based sensors are particularly attractive because of their simple device architecture, low fabrication cost, high sensitivity, and compatibility with microelectronic integration [4–6].

Within this class, zinc oxide (ZnO) has emerged as one of the most promising materials owing to its wide direct band gap (~3.37 eV), high electron mobility, chemical and thermal stability, and ease of tailoring into diverse nanostructures [7,8]. ZnO nanorods (NRs), in particular, offer unique advantages for gas sensing applications due to their one-dimensional (1D) morphology, high aspect ratio, and large surface-to-volume ratio, which enhance surface reactivity and adsorption/desorption kinetics [9,10]. The direct electron transport pathways along the c-axis of ZnO nanorods further minimize grain boundary resistance, resulting in faster charge carrier mobility and, consequently, improved response and recovery characteristics in resistive-type gas sensors [11].

Various approaches have been developed to synthesize ZnO nanorods, including chemical vapor deposition (CVD), pulsed laser deposition (PLD), sol–gel, and hydrothermal growth [12–14] . Among these, the hydrothermal method stands out as a low-temperature, scalable, and cost-effective route that enables the growth of vertically aligned, single-crystalline ZnO nanorods with well-controlled morphology [15,16]. Importantly, the hydrothermal process allows precise control over synthesis parameters such as precursor concentration, pH, temperature, and reaction time, all of which strongly influence the crystallinity, aspect ratio, and surface activity of ZnO nanorods [17,18]. Such tunability makes the hydrothermal route highly suitable for tailoring ZnO nanostructures toward enhanced gas sensor performance.

Therefore, this work focuses on the controlled synthesis of ZnO nanorods via the hydrothermal method and evaluates their structural, morphological, and gas-sensing properties. The insights obtained here contribute to the design of cost-effective, scalable ZnO nanostructures optimized for next-generation gas sensor applications.

Methodology

ZnO seed layers were deposited onto pre-cleaned glass substrates via radio-frequency (RF) magnetron sputtering using a high-purity ZnO target. The deposition was performed under a working pressure of 10 mTorr with a mixed argon/oxygen gas flow of 15/15 sccm and an RF power of 60 W, yielding a film thickness of approximately 100 nm. To enhance the crystallinity and adhesion of the seed layer, the as-deposited films were subsequently annealed in air at 400 °C for 2 hours. For the hydrothermal growth process, precursor solutions of different concentrations were prepared. In the low-concentration case (0.01 M:0.01 M), 73.3 mg of zinc acetate dihydrate ($Zn(CH_3COO)_2{\cdot}2H_2O$) and 56.1 mg of hexamethylenetetramine (HMTA, $C_6H_{12}N_4$) were dissolved in 40 mL of deionized (DI) water and stirred for 45 minutes to achieve a homogeneous mixture. For the high-concentration solution (1 M:1 M), 7.46 g of zinc acetate dihydrate and 5.55 g of HMTA were used under identical preparation conditions. The annealed, seed-layer-coated substrates were vertically immersed in the precursor solution and placed in a Teflon-lined stainless-steel autoclave, where hydrothermal growth was conducted at 100 °C for 3 hours. Upon completion, the samples were removed, rinsed thoroughly with DI water to eliminate residual precursors, and dried at room temperature. The crystallographic characteristics of the seed layers and nanorods were examined using XRD and energy-dispersive X-ray spectroscopy (EDS), while SEM was employed to investigate surface morphology and cross-sectional features. These analyses provided insights into the crystallinity, orientation, and morphology of the ZnO nanorods synthesized under the described conditions.

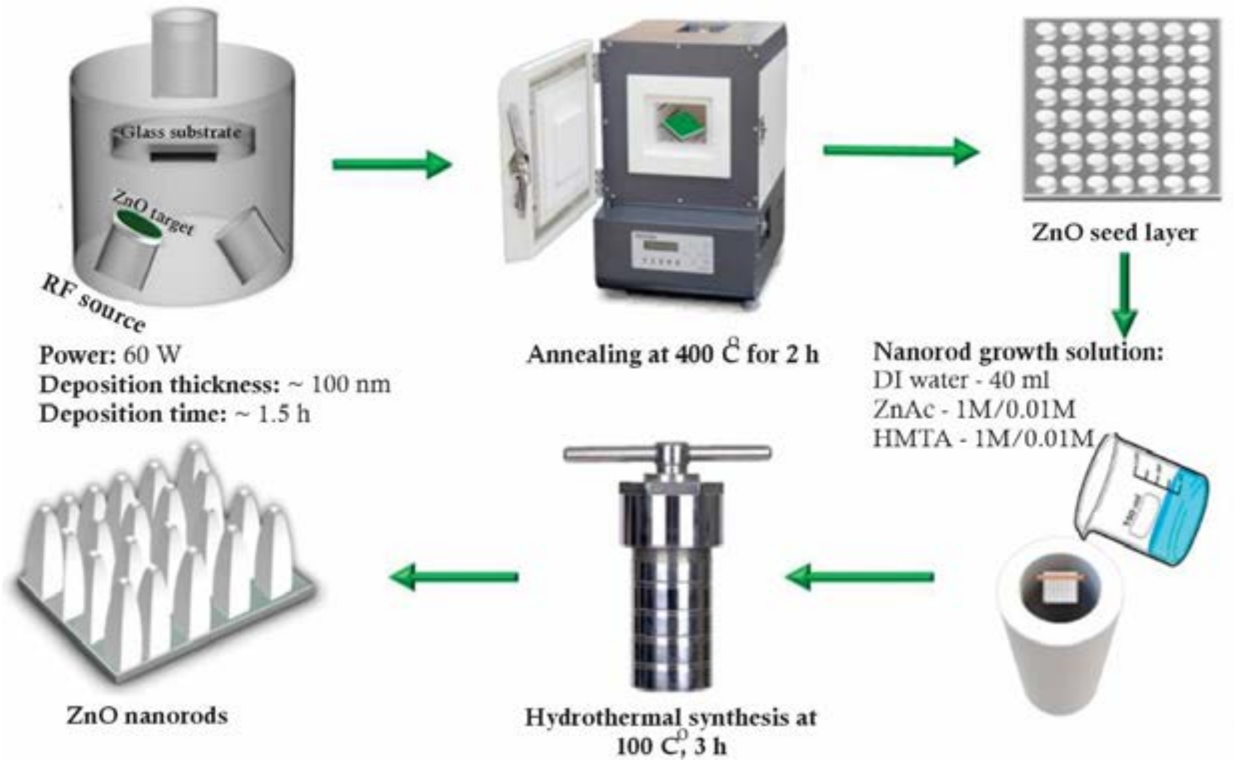

Figure 1. Schematic illustration of synthesis procedure

Results and discussions

The crystallinity and orientation of the ZnO seed layer were first examined by XRD, as illustrated in Figure 1. The pattern revealed a sharp and intense diffraction peak at (002), characteristic of the hexagonal wurtzite ZnO structure (JCPDS Card No. 00-005-0664). The dominance of the (002) reflection indicates strong preferential orientation along the c-axis, which is known to promote the vertical alignment of ZnO nanorods during hydrothermal growth [19]. Such orientation is particularly beneficial for gas sensing applications, as c-axis aligned structures provide direct electron pathways and minimize grain boundary scattering.

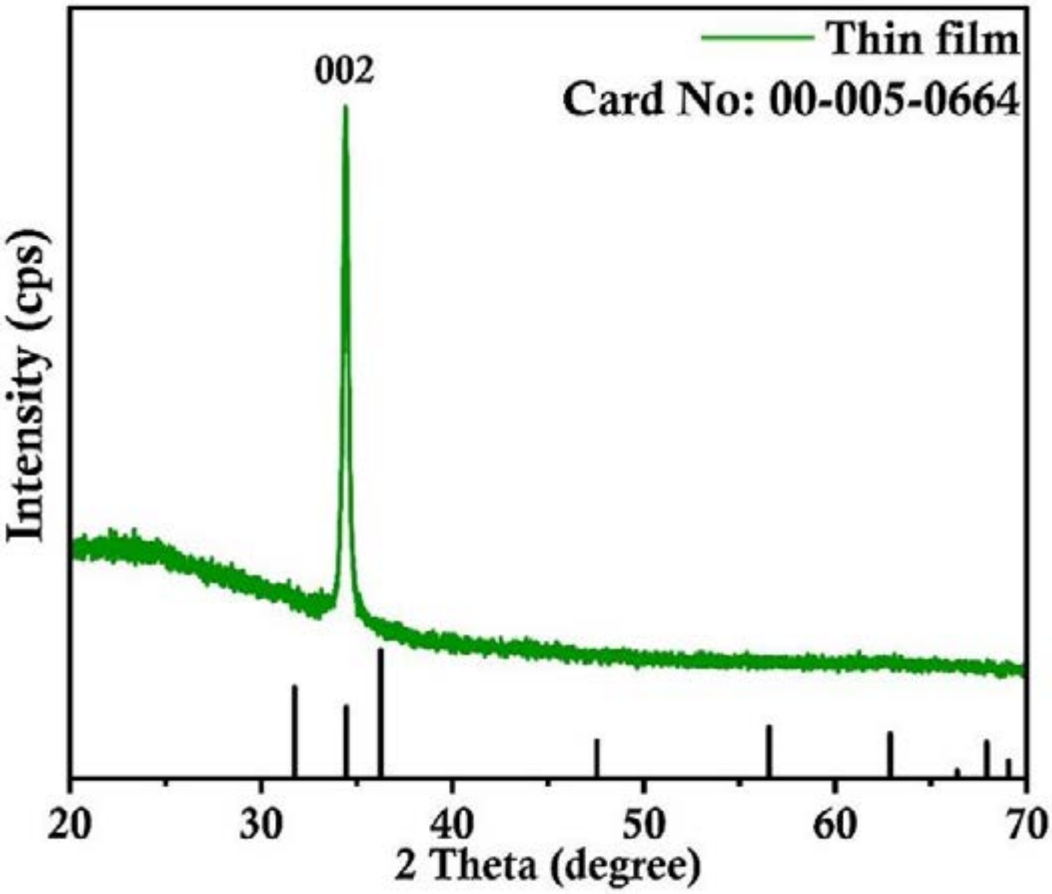

Figure 2. XRD pattern of ZnO thin film seed layer deposited by RF magnetron sputtering, showing a dominant (002) diffraction peak corresponding to hexagonal wurtzite ZnO (JCPDS Card No. 00-005-0664)

The surface morphology of the seed layer, observed by SEM in Figure 2, further highlights the effect of annealing. The as-deposited seed layer exhibited a relatively smooth surface with poorly defined grains, whereas the film annealed at 400 °C for 2 h showed more distinct and uniformly distributed grains. Thermal treatment is known to reduce lattice defects and enhance surface uniformity, thereby providing favorable nucleation sites for subsequent nanorod growth [7,8]. These improvements in seed layer quality are consistent with reports that annealing significantly enhances the alignment and density of ZnO nanorods produced by hydrothermal methods.

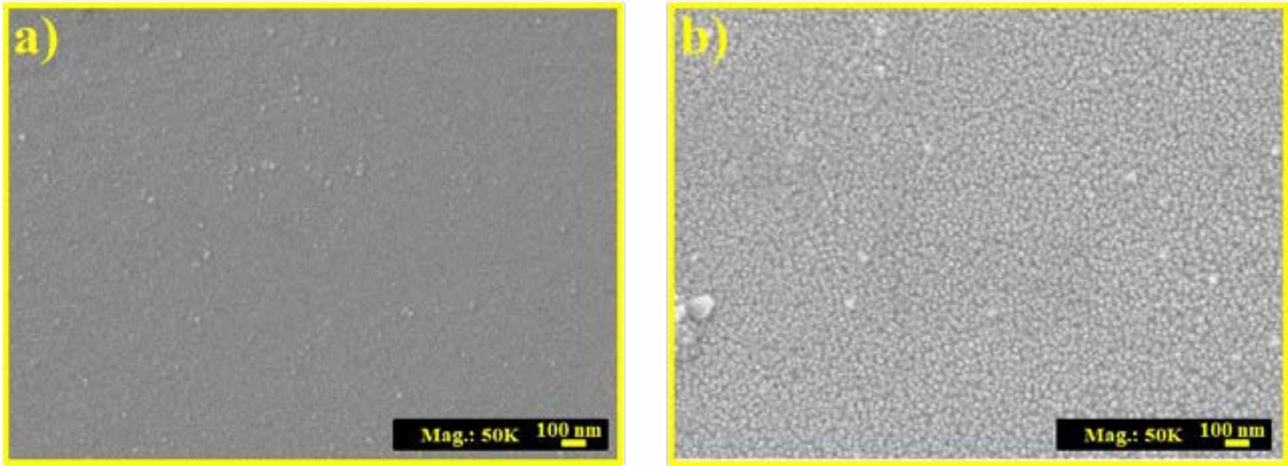

Figure 3. SEM images of ZnO seed layers deposited by RF magnetron sputtering: (a) as-deposited (without annealing) and (b) annealed at 400 °C for 2 h

The influence of precursor concentration on ZnO nanorod crystallinity is evident from the XRD results shown in Figure 3. Both low (0.01 M:0.01 M) and high (1 M:1 M) precursor concentrations produced nanorods with a dominant (002) peak, confirming that c-axis growth was maintained in both cases. However, the high-concentration sample displayed a much stronger and sharper (002) peak, indicating superior crystallinity and larger crystallite size. This suggests that higher precursor concentrations increase the availability of Zn2+ ions, thereby enhancing nucleation rates and promoting anisotropic crystal growth along the c-axis.

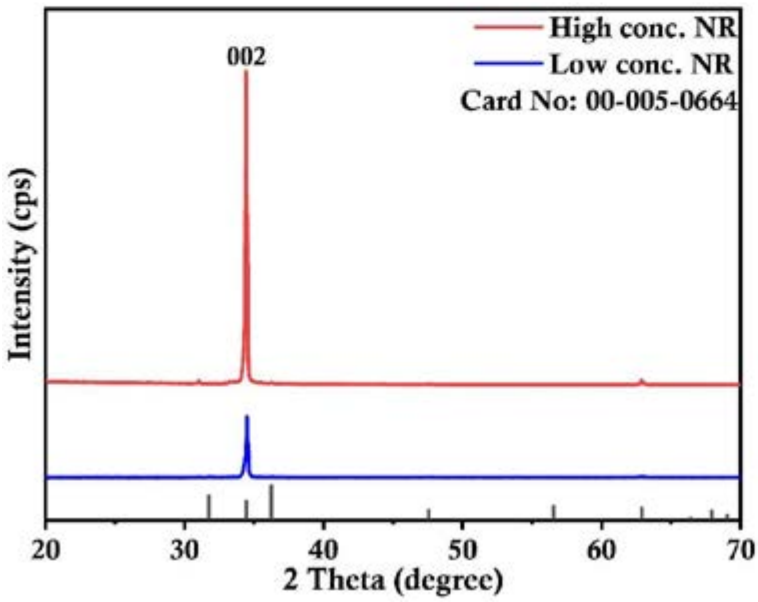

Figure 4. XRD patterns of ZnO nanorods grown under different precursor concentrations: (blue) low concentration (0.01 M:0.01 M) and (red) high concentration (1 M:1 M)

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These structural differences are corroborated by SEM images, as depicted in Figure 4. At low precursor concentration, the nanorods appeared short, sparsely distributed, and irregular in shape. In contrast, nanorods synthesized from the high-concentration solution were vertically aligned, densely packed, and exhibited well-defined hexagonal cross-sections with higher aspect ratios. The observed morphology can be attributed to increased supersaturation of Zn^{2+} ions in the growth solution, which accelerates unidirectional growth and yields longer, more uniform nanorods [20]. Such vertically aligned and high-aspect-ratio nanorod arrays are highly desirable for resistive-type gas sensors, as they maximize the active surface area for gas adsorption and provide efficient electron transport pathways [21].

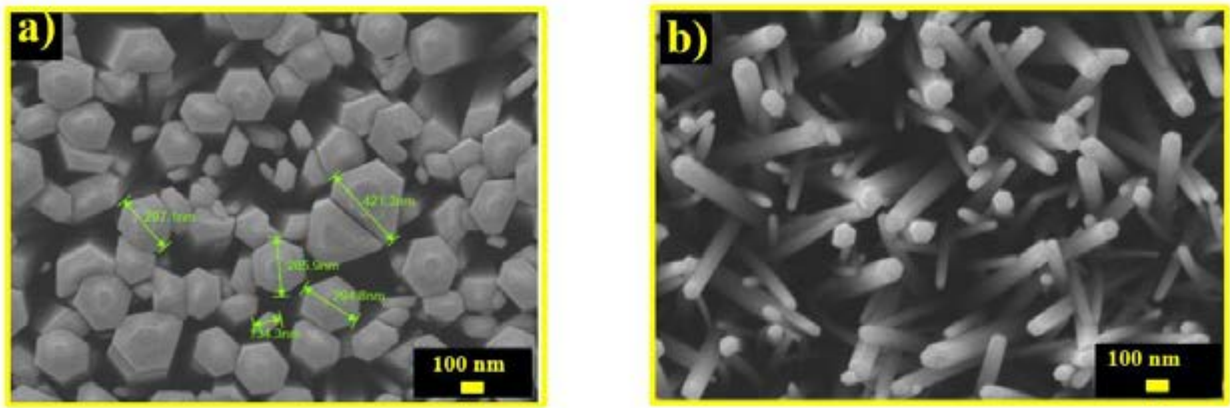

Figure 5. SEM images of ZnO nanorods synthesized by the hydrothermal method at different precursor concentrations: (a) low concentration (0.01 M:0.01 M), showing shorter and less densely packed nanorods with hexagonal facets, and (b) high concentration (1 M:1 M)

Taken together, these findings demonstrate that both the annealing of the ZnO seed layer and the adjustment of precursor concentration critically influence the structural and morphological evolution of hydrothermally grown ZnO nanorods. Optimized processing conditions lead to vertically aligned, highly crystalline nanorods with dense packing and enhanced aspect ratios, properties that are directly linked to improved sensing performance in gas sensor applications.

Conclusion

In conclusion, ZnO nanorods were successfully synthesized by a hydrothermal method using sputtered ZnO seed layers, and the effects of seed layer annealing and precursor concentration were systematically investigated. The XRD results confirmed preferential c-axis orientation in all samples, while SEM analysis revealed significant improvements in nanorod alignment, density, and aspect ratio under optimized conditions. Annealing of the seed layer at 400 °C enhanced grain uniformity and provided well-defined nucleation sites, whereas higher precursor concentrations promoted anisotropic crystal growth, resulting in vertically aligned nanorods with superior crystallinity. These findings demonstrate that precise control over seed layer

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properties and growth solution parameters is crucial for tailoring ZnO nanorod morphology. The optimized nanostructures are particularly promising for gas sensor applications, where high crystallinity, vertical alignment, and large surface area-to-volume ratios are essential for achieving high sensitivity, fast response, and stable performance.

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Authorship contribution statement

A.Tanirbergen: Conceptualization, Methodology, Investigation, Data curation, Formal analysis, Visualization, Writing – original draft. Y.Shynybekov: Investigation, Methodology, Data curation, Writing – review & editing. A.Mentabyeva: Methodology, Resources, Supervision, Writing – review & editing. B. Soltabayev: Conceptualization, Supervision, Project administration, Funding acquisition, Writing – review & editing.

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Жоғары өнімді газ сенсорлары үшін ZnO нанотаяқшаларын бақыланатын гидротермиялық синтездеу

Андатпа. Кристалдық құрылымы мен морфологиясы реттелген ZnO нанотаяқшаларын синтездеу жоғары өнімді газ сенсорларын дамыту үшін аса маңызды. Бұл зерттеуде ZnO тұқымдық қабаты шыны астарға RF-магнетронды бүрку әдісімен өсіріліп, кейін 400 °C-та аннелденіп, кристаллдық сапасы мен беткі біркелкілігі арттырылды. Кейін ZnO нанотаяқшалары гидротермиялық әдіспен әртүрлі прекурсор концентрацияларында (0.01 M:0.01 M және 1 M:1 M мырыш ацетаты/ГМТА) синтезделді. Рентгендік дифракция әдісі (XRD) барлық үлгілерде (002) доминантты шыңды көрсетті, бұл c-ось бойымен бағытталған өсуді дәлелдеді. Сканирлеуші электрондық микроскопия (SEM) прекурсор концентрациясы артқан сайын ұзағырақ, тығыз орналасқан және жақсы анықталған алтыбұрышты қимасы бар нанотаяқшалардың түзілетінін көрсетті. Нәтижелер тұқымдық қабатты аннелдеу мен прекурсор концентрациясының ZnO нанотаяқшаларының құрылымдық және морфологиялық эволюциясына айтарлықтай әсер ететінін көрсетті. Оптималды синтездеу жағдайлары жоғары кристаллды, тік бағытталған және жоғары аспектілік қатынасқа ие нанотаяқшаларды алуға мүмкіндік береді. Мұндай қасиеттер газ адсорбциясын және электрон тасымалдауды күшейтіп, оларды резистивті типті газ сенсорларына қолдануға өте қолайлы етеді.

Түйін сөздер: ZnO, нанотаяқшалар, газ сенсоры, металл оксиді жартылай өткізгіштері (MOS), гидротермиялық әдіс

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Контролируемый гидротермальный синтез наностержней ZnO для высокоэффективных газовых сенсоров

Аннотация. Контролируемый синтез наностержней ZnO с регулируемой кристалличностью и морфологией имеет ключевое значение для разработки высокоэффективных газовых сенсоров. В данной работе зародышевые слои ZnO были осаждены на стеклянные подложки методом

РФ-магнетронного распыления и затем отожжены при 400 °C для улучшения кристалличности и однородности поверхности. Далее проводился гидротермальный рост наностержней ZnO при различных концентрациях прекурсоров (0.01 M:0.01 M и 1 M:1 M ацетат цинка/ГМТА) для изучения влияния химии раствора на формирование наноструктур. Рентгеновская дифракция (XRD) выявила доминирующий пик (002) во всех образцах, что подтверждает рост вдоль c-оси. Сканирующая электронная микроскопия (SEM) показала, что более высокие концентрации прекурсоров приводят к формированию длинных, плотно упакованных наностержней с чётко выраженным гексагональным сечением. Полученные результаты демонстрируют, что как отжиг зародышевого слоя, так и концентрация прекурсоров существенно влияют на структурную и морфологическую эволюцию наностержней ZnO. Оптимизированные условия синтеза позволяют получать вертикально ориентированные, высококристалличные наностержни с увеличенным аспектным отношением, что усиливает адсорбцию газа и транспорт электронов, делая их перспективными для применения в резистивных газовых сенсорах.

Ключевые слова: ZnO, наностержни, газовый сенсор, МОП (металлооксидные полупроводники), гидротермальный метод

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