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Javohir Sh. XUDOYQULOV,
PhD student at the National University of Uzbekistan, Tashkent
E-mail: xjavokhir1799@gmail.com ORCID: 0009-0005-4223-8863
Shavkat U. YULDASHEV,
Head of Laboratory, Center for Nanotechnology Development, National University of Uzbekistan
Azamat O. ARSLANOV,
Lecturer at the Faculty of Physics, National University of Uzbekistan
Jamoliddin X. MURODOV,
PhD student at the Center for Nanotechnology Development, National University of Uzbekistan
Noiba U. BOTIROVA,
PhD student at the Center for Nanotechnology Development, National University of Uzbekistan
Ra’no Sh. SHARIPOVA
PhD student at the Center for Nanotechnology Development, National University of Uzbekistan

Based on the review of R. Nusretov, Senior Researcher, Uzbekistan–Japan Innovation Center.

SOL–GEL, SPIN COATING USULI YORDAMIDA OLINGAN NANOSTRUKTURALI NIO YUPQA PLYONKALARI: STRUKTURAVIY VA OPTIK XOSSALARI

Annotatsiya

NiO yupqa plyonkalari SiO_2 taglikda sol–gel va spin coating usuli yordamida o‘stirildi. Plyonkalarning strukturaviy xossalari rentgen difraksiyasi (XRD) usuli orqali tahlil qilinib, kubik kristall panjaraga ega bo‘lgan polikristall NiO fazasi hosil bo‘lgani tasdiqlandi. Kristallit o‘lchami va mikrodeformatsiya Williamson–Hall usuli yordamida aniqlanib, o‘rtacha kristallit o‘lchami taxminan 48 nm va mikrodeformatsiya 1.5×10^{-3} ga teng ekanligi aniqlandi. Sirt morfologiyasi skanerlovchi elektron mikroskopiya (SEM) yordamida o‘rganildi, optik xossalari esa UV–Vis spektroskopiyasi orqali tadqiq qilindi. Taqiqlangan zona kengligi Tauc grafigi yordamida aniqlangan. Olingan natijalar shuni ko‘rsatadiki, tayyorlangan NiO yupqa plyonkalari funksional qurilmalarda qo‘llash uchun mos strukturaviy va optik xossalarga ega.

Kalit so‘zlar*:* *sol–gel, NiO, rentgen difraksiyasi, yupqa plyonka, annealing harorati.*

НАНОСТРУКТУРИРОВАННЫЕ ТОНКИЕ ПЛЕНКИ NIO, ПОЛУЧЕННЫЕ МЕТОДОМ СОЛ–ГЕЛЬ С ИСПОЛЬЗОВАНИЕМ ЦЕНТРИФУГИРОВАНИЯ: СТРУКТУРНЫЕ И ОПТИЧЕСКИЕ СВОЙСТВА

Аннотация

Тонкие пленки NiO были получены на подложках SiO_2 методом сол–гель с использованием центрифугирования (spin coating). Структурные свойства пленок были исследованы методом рентгеновской дифракции (XRD), что подтвердило формирование поликристаллической фазы NiO с кубической кристаллической решеткой. Размер кристаллитов и микродеформация были определены с использованием метода Уильямсона–Холла, при этом средний размер кристаллитов составил около 48 нм, а микродеформация — 1.5×10^{-3}. Морфология поверхности была изучена с помощью сканирующей электронной микроскопии (SEM), а оптические свойства исследованы методом УФ–видимой спектроскопии. Оптическая ширина запрещенной зоны была определена с использованием метода Така (Tauc plot). Полученные результаты показывают, что синтезированные тонкие пленки NiO обладают подходящими структурными и оптическими характеристиками для потенциального применения в функциональных устройствах.

Ключевые слова*:* *сол–гель, NiO, рентгеновская дифракция, тонкие пленки, температура отжига.*

NANOSTRUCTURED NIO THIN FILMS FABRICATED BY SOL–GEL SPIN COATING: STRUCTURAL AND OPTICAL PROPERTIES

Annotation

NiO thin films were deposited on SiO_2 substrate using the sol–gel spin coating method. The structural properties were analyzed by X-ray diffraction (XRD), confirming the formation of polycrystalline NiO with a cubic phase. The crystallite size and microstrain were evaluated using the Williamson–Hall approach, yielding an average crystallite size of approximately 48 nm and a microstrain of 1.5×10^{-3}. Surface morphology was examined by scanning electron microscopy (SEM), and the optical properties were investigated using UV–Vis spectroscopy. The optical band gap was determined using the Tauc plot method. The obtained results indicate that the prepared NiO thin films exhibit suitable structural and optical characteristics for potential applications in functional devices.

Key words*: Sol-gel, NiO, X-ray diffraction, thin film, annealing temperature.*

Introduction. Nickel oxide (NiO) is a p-type wide band gap semiconductor that has attracted considerable attention due to its excellent chemical stability, optical transparency, and electrical properties. Owing to these characteristics, NiO has been widely investigated for applications in optoelectronic devices, photocatalysis, gas sensors, and electrochromic systems [1,2].

Various techniques have been employed for the deposition of NiO thin films, including sputtering, chemical vapor deposition, and pulsed laser deposition. However, these methods often require complex equipment and high processing costs. In contrast, the sol–gel spin coating technique offers significant advantages such as low cost, simplicity, compositional control, and the ability to produce uniform large-area thin films [3].

Despite extensive studies, the structural quality and optical properties of NiO thin films strongly depend on synthesis parameters such as precursor composition, deposition conditions, and annealing temperature. In particular, controlling crystallite size, lattice strain, and surface morphology remains a key challenge for optimizing the performance of NiO-based devices.

In this work, NiO thin films were prepared using the sol–gel spin coating method on Si/SiO_2 substrates. The structural, morphological, and optical properties of the films were systematically investigated using X-ray diffraction (XRD), scanning electron microscopy (SEM), and UV–Vis spectroscopy [4]. Special attention was given to the evaluation of crystallite size and microstrain using the Williamson–Hall method, as well as the determination of optical band gap using the Tauc plot approach [5,6].

Research Methodology, NiO thin films were prepared using the sol–gel spin coating technique. Nickel acetate tetrahydrate ($Ni(CH_3COO)_2 \cdot 4H_2O$) was used as a precursor, while 2-methoxyethanol served as the solvent and ethanolamine (MEA) was employed as a stabilizing agent. The solution was magnetically stirred at 60°C for 2 hours to obtain a homogeneous and stable sol. A total volume of 10 ml of the prepared solution was used for film deposition. The films were deposited onto SiO_2 substrates using a spin coater at a rotation speed of 3000 rpm for 30 seconds. After deposition, the films were subjected to thermal treatment at 400°C for 2 hours in air atmosphere to achieve the formation of NiO thin films [7]. The structural, morphological, and optical properties of the films were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), and UV–Vis spectroscopy, respectively [8-10].

Analysis and results. The structural, morphological, and optical properties of the NiO thin films annealed at 400°C for 2 hours were systematically investigated, and a strong interdependence between these characteristics was observed.

The X-ray diffraction pattern of the prepared films (Fig. 1) reveals distinct diffraction peaks corresponding to the (111), (200), (202), (311), and (220) planes, which are in good agreement with the standard cubic phase of NiO. The absence of any additional peaks indicates that the films are phase-pure and free from secondary phases or impurities, confirming the effectiveness of the sol–gel synthesis route.

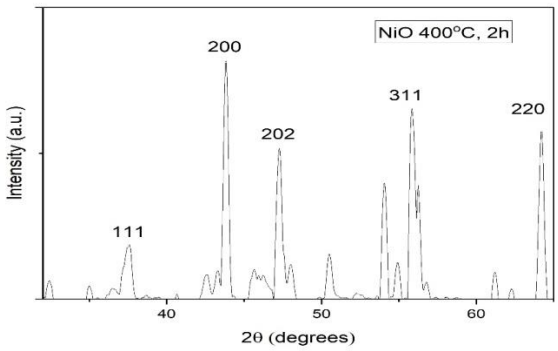

Fig. 1. *X-ray diffraction (XRD) pattern of NiO thin film deposited on SiO_2 substrate and annealed at 400°C for 2 hours, showing characteristic peaks of cubic NiO structure.*

A noticeable feature of the diffraction pattern is the relatively higher intensity of the (200) peak compared to other reflections, indicating a preferred orientation along this plane. This preferential growth can be attributed to the minimization of surface and interface energies during the film formation process. In sol–gel derived films, such orientation is often associated with controlled nucleation and anisotropic grain growth during annealing.

In addition to phase identification, the microstructural parameters were evaluated using the Williamson–Hall method. The calculated values (Table 1) indicate an average crystallite size of approximately 48 nm and a microstrain of 1.5×10^{-3}.

$$\beta\cos\theta = \frac{k\lambda}{D} + 4\epsilon\sin\theta \qquad (1)$$

The obtained crystallite size confirms the nanocrystalline nature of the films. At this scale, the surface-to-volume ratio becomes significant, which can influence both structural stability and optical transitions. The relatively low microstrain value suggests minimal lattice distortion, indicating that the annealing process effectively reduces internal stresses and improves crystal quality.

Structural parameters of NiO calculated using the Williamson–Hall method, **Table1**

Number	(hkl)	2θ (°)	β (rad)	βcosθ	4sinθ	D, average(nm)	$\varepsilon(\times 10)^{-3}$
1	111	37.2	0.00559	0.00530	1.28	48	1.5
2	200	43.3	0.00489	0.00454	1.47		
3	202	48.5	0.00524	0.00478	1.64		
4	311	55.4	0.00576	0.00509	1.86		
5	220	62.8	0.00506	0.00432	2.08		

The sharpness and intensity of the diffraction peaks further support the conclusion that the films possess good crystallinity. This improvement in crystallinity can be attributed to enhanced atomic diffusion and grain growth at the annealing temperature of 400°C [11-13].

The surface morphology of the films, as shown in Fig. 2, provides additional insight into the growth mechanism. The SEM image reveals a dense and uniform surface composed of closely packed nanograins. The absence of cracks or pinholes indicates good film continuity and strong adhesion to the substrate.

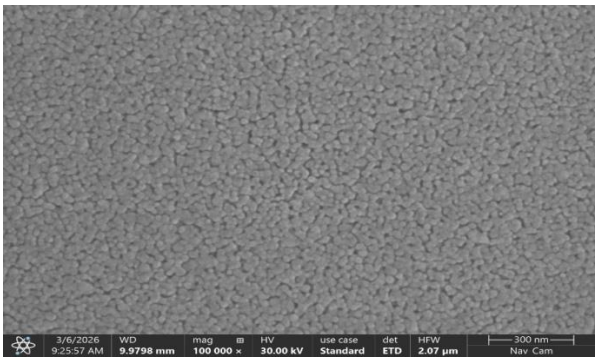

Fig. 2. SEM image of NiO thin film annealed at 400°C, illustrating a uniform, dense, and crack-free surface morphology composed of nanostructured grains.

The uniform grain distribution suggests that the nucleation process during spin coating was homogeneous across the substrate surface. This is a key advantage of the sol–gel method, where solution-based deposition promotes even coverage. The observed slight agglomeration of grains is likely due to coalescence during annealing, where smaller particles merge to form larger grains in order to reduce the overall surface energy. Importantly, the grain size observed in SEM is in good agreement with the crystallite size obtained from XRD analysis (~48 nm). This correlation indicates that the grains are either single crystalline or composed of a small number of coherently scattering domains, which contributes to improved structural integrity.

The optical properties of the NiO thin films were analyzed using UV–Vis spectroscopy, and the corresponding Tauc plot is shown in Fig. 3. The linear behavior of the $(\alpha h\nu)^2$versus $h\nu$plot suggests that the optical transitions in the prepared films are of direct allowed type.

$$(\alpha h\nu)^2 = A(h\nu - Eg) \qquad (2)$$

By extrapolating the linear region, the optical band gap was determined to be approximately 3.52 eV. This value falls within the typical range reported for NiO thin films and confirms its wide band gap semiconducting nature [13-15].

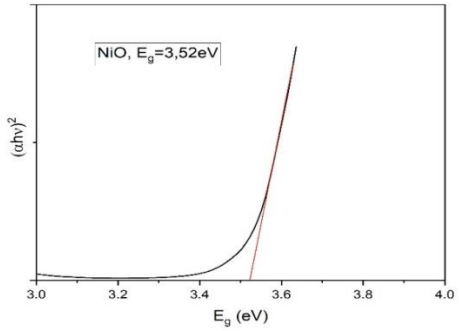

Fig. 3. *Tauc plot $(\alpha h\nu)^2$versus photon energy $(h\nu)$for NiO thin film used to determine the optical band gap.*

The optical band gap is strongly influenced by the microstructural properties of the films. The nanocrystalline size and low microstrain reduce the density of localized defect states within the band gap, resulting in a sharper absorption edge and a well-defined optical transition. In addition, the uniform and compact morphology observed in SEM minimizes light scattering and enhances optical homogeneity.

Overall, the results demonstrate a strong correlation between the structural, morphological, and optical properties of the NiO thin films. The nanocrystalline structure with low lattice distortion leads to uniform surface morphology, which in turn results in stable and well-defined optical properties. This interdependence highlights the importance of controlling synthesis parameters in order to optimize the performance of NiO thin films for advanced applications [16,17].

Conclusion. In this study, NiO thin films were successfully fabricated on SiO_2 substrate using the sol–gel spin coating method. The structural analysis confirmed the formation of crystalline NiO with a cubic crystal structure and a preferred orientation along the (200) plane. The Williamson–Hall analysis revealed an average crystallite size of approximately 48 nm and a low microstrain value (1.5×10^{-3}), indicating the formation of nanocrystalline films with good structural quality and minimal lattice distortion.

The morphological investigation demonstrated that the films possess a dense, uniform, and crack-free surface composed of closely packed nanograins. This uniform morphology reflects controlled nucleation and growth during the deposition process and contributes to the overall stability of the films. Optical analysis showed that the NiO thin films exhibit a wide band gap of approximately 3.52 eV, confirming their semiconducting nature. The observed optical behavior is strongly correlated with the structural properties, where the nanocrystalline size and low defect density result in well-defined optical transitions.

Overall, the results indicate that the sol–gel spin coating method provides an effective and reliable route for producing high-quality NiO thin films with controlled structural, morphological, and optical properties. The strong correlation between these characteristics highlights the importance of optimizing synthesis parameters. The prepared films demonstrate significant potential for applications in optoelectronic and other functional thin-film devices.

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