

МАГНЕТРОННОЕ ОСАЖДЕНИЕ ПРОЗРАЧНЫХ ПЛЕНОК AZO ИЗ КОМПОЗИЦИОННЫХ И ОДНОЭЛЕМЕНТНЫХ МИШЕНЕЙ**Д.В. Колодко, А.В. Казиев, Д.Г. Агейченков, А.В. Тумаркин,
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Для создания прозрачных проводящих тонкопленочных покрытий на основе оксида цинка, легированного оксидом алюминия, ($ZnO:Al_2O_3$, AZO) на полимерных (акриловых) подложках были рассмотрены различные способы магнетронного осаждения. В первом методе использовался один магнетрон с композиционной мишенью $ZnO:Al_2O_3$. Магнитная конфигурация магнетрона имела возможность внешней регулировки положения магнитов для управления потоком ионов на подложку. Была установлена корреляция между электрическим сопротивлением сформированной пленки AZO и потоком ионов на подложку при осаждении. Однако, минимальное удельное сопротивление покрытий AZO, полученных этим методом, не опускалось ниже значения $300 \text{ Ом}/\square$. Было показано, что возможности регулирования процесса осаждения прозрачной пленки AZO значительно расширяются за счет использования двух независимых магнетронов с одноэлементными металлическими мишенями Zn и Al. Был предложен новый метод многостадийного нанесения покрытия, в котором слой ZnO формировался путем распыления металлической мишени Zn в газовой смеси Ar/O₂, а легирующий слой осаждался путем распыления металлической мишени Al в атмосфере аргона. Доля легирующей примеси в полученной структуре AZO контролировалась путем изменения продолжительности циклов осаждения слоев ZnO и Al. Такой подход обеспечил гораздо более гибкий контроль состава покрытия. Полученные образцы продемонстрировали высокие функциональные свойства, обладая низкими поверхностным сопротивлением $10 \text{ Ом}/\square$ и удельным сопротивлением $3 \times 10^{-6} \text{ Ом} \times \text{м}$ в сочетании с высокой оптической прозрачностью, что подтверждает потенциал этих полупроводниковых покрытий для использования в оптоэлектронных устройствах.

Ключевые слова: AZO, прозрачных проводящий оксид (ППО), магнетронное распыление, поверхностное сопротивление, ионный поток

MAGNETRON DEPOSITION OF TRANSPARENT AZO FILMS FROM COMPOSITE AND SINGLE-ELEMENT TARGETS

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Different magnetron deposition approaches have been considered to fabricate transparent conductive coatings based on aluminum-doped zinc oxide (ZnO:Al₂O₃, AZO) on polymer substrates. In the first method, a single magnetron with a compound ZnO:Al₂O₃ target was used. The magnetron's magnetic configuration was adjusted to control the ion flux to the substrate. A correlation was established between the electrical resistance of the formed AZO film and the ion flux. The minimum sheet resistance of AZO coatings obtained by this approach was as low as 300 Ω/□. The tunability of the process has been shown to be greatly enhanced by using two independent magnetrons with single-element Zn and Al targets. A novel multilayer coating method was proposed where ZnO layer was formed by sputtering a metallic Zn target in Ar/O₂ gas mixture while the alloying layer was deposited by sputtering a metallic Al target in a pure argon atmosphere. The dopant ratio in the resulting AZO structure was controlled by varying the duration of ZnO and Al layer deposition cycles. This approach provided flexible control over the coating's composition. The corresponding samples demonstrated high functional properties, with a surface resistance of 10 Ω/□ and a resistivity of 3×10⁻⁶ Ω×m coupled with high optical transparency, confirming the coatings' potential for use in optoelectronic devices.

Keywords: AZO, transparent conductive oxide (TCO), magnetron sputtering, sheet resistance, ion flux

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INTRODUCTION

Transparent conducting oxides (TCOs) are a special class of materials that combine high optical transmittance and sufficient electrical conductivity. This unique combination of properties has enabled modern thin-film technologies and a wide range of optoelectronic devices, from transparent *p-n* junction diodes and light-emitting diodes to solar cells and flat panel displays [1-3]. Among the materials in this group, thin-film zinc oxide-based compounds, including aluminium-doped zinc oxide (ZnO:Al₂O₃, or AZO) occupy special place and are widely used in optoelectronic devices such as solar cells, displays, and touchscreens [4-12]. The popularity of such coatings is due to its low electrical resistance and high optical transparency coupled with relatively low cost. Moreover, due to the favourable combination of electrical and optical properties, AZO is considered a promising material for the creation of energy-efficient windows and a variety of sensors. AZO is particularly valuable for the development of next-generation flexible electronics, as it demonstrates good environmental stability and mechanical flexibility. Intensive research aimed at improving the properties of this material is currently ongoing: new doping strategies are being developed, and the deposition procedures are optimized.

When depositing coatings on transparent polymer substrates (plastics), specialized technical solutions are required to prevent thermal damage to the material. In particular, pulsed magnetron power supply systems (e.g. high-power impulse magnetron sputtering, HiPIMS) are used, or the magnetrons are operated in low-power modes with frequency-modulated DC or RF power sources [13-22].

In the practice of AZO magnetron deposition, several choices of the target materials can be considered. The first and the most popular one involves sputtering a sintered ceramic ZnO:Al₂O₃ or a compound Zn/Al target containing a predetermined percentage of aluminium dopant [23-32]. The second method uses two separate magnetrons – the one with Zn or ZnO target, and the other with metallic Al target [33-40]. In this method, the dopant (Al) and main compound (ZnO) contributions are controlled by varying the magnetron discharge parameters.

An alternative approach to AZO coating deposition also exists, involving the sputtering of two separate metal targets (Zn and Al) in an argon-oxygen working gas mixture [41, 42]. This method is technologically complex, as it requires precise regulation of the fluxes of zinc, aluminium, and oxygen species. On

the other hand, this method enables the full control of coating's composition, which governs conductivity and transparency.

In this study, we investigate, analyze and compare the processes of AZO film deposition by magnetron sputtering of a compound AZO and single-element Zn/Al targets in a common experimental setup and investigate the ways of minimizing the coating's resistivity.

EXPERIMENTAL SETUP

A dedicated confocal sputter deposition system (Fig. 1) comprising a set of Magneto-3GABS type circular magnetrons (Pinch LLC, Moscow, Russia) was used to fabricate AZO coatings on the polymer substrates. In the first experimental series, a single magnetron was used with a sintered compound AZO target (98 wt.% ZnO + 2 wt.% Al₂O₃, Goodwill Metal Tech Co. Ltd., Beijing, China). In the second experimental series, two magnetrons were used. One of them had Al target (99.99% pure, Girmet LLC, Moscow, Russia), and the other one had Zn target (99.975% pure, Girmet LLC, Moscow, Russia) installed. Each target was circular with a diameter of 76.2 mm and thickness of 3 mm.

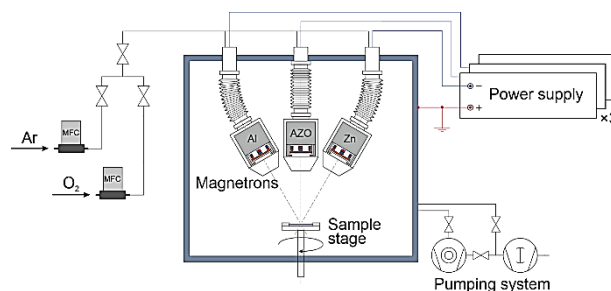


Fig. 1. Confocal magnetron sputtering system

Рис. 1. Конфокальная магнетронная распылительная система

The magnetrons had the option of changing magnetic unbalancing degree by adjusting the configuration of the magnet packs [43]. In the experiments with compound target, the unbalancing degree was varied to investigate the dependence of coating properties on the ion flux at the substrate. In the experiments with single-element targets, the magnetic configuration was fixed.

The magnetrons were powered by APEL-M-5PDC-800 DC power supplies (Applied Electronics, LLC, Tomsk, Russia). All procedures were conducted at a power level of 100 W in DC power stabilization mode.

The ultimate residual pressure in the vacuum chamber was $1 \cdot 10^{-4}$ Pa. During the deposition, the working gas pressure was kept constant at 0.1 Pa for

both the experiments with pure Ar and Ar/O₂ gas mixtures. The gas mixture composition was precisely controlled using Bronkhorst El-Flow digital gas flow controllers. In the experiments with metallic targets, the Ar/O₂ flow ratio was set to 2/1.

The experimental setup included a rotating sample stage. Unless explicitly noted, the substrates were 50×50×3 mm plates made from transparent acrylic plastic.

The surface resistance (R_s) of the produced coatings was evaluated with an in-house realization of van der Pauw method [43]. The specific resistivity (ρ) was then calculated from R_s according to the formula:

$$\rho = R_s \times d, \quad (1)$$

where d is the film thickness.

The film thickness was determined in the reference experiments where the deposition was made onto substrates pre-patterned with a mask. After deposition, the mask was removed, and the step height was measured using a Veeco Dektak 150 surface profiler. This approach provided a calibrated thickness value based on the profiler's vertical resolution. In the reported experiments, the film thickness was in the range 150-250 nm.

In a separate experiment, an array of flat collecting ion probes was used to measure the radial ion current distribution in the substrate region (not shown).

RESULTS AND DISCUSSION

Deposition with compound AZO target

Using a compound target limits the number of parameters that can be varied to adjust the coating properties. One of the most significant factors influencing the performance of semiconductor coatings is the ion bombardment during the film growth process. In its turn, the ion flux to the substrate is governed by the magnetic field configuration [44-50]. The magnetrons used in this work were capable of varying the ion flux to the substrate by changing the magnetic field configuration. For this purpose, the central magnet of the magnetron was moved with respect to the outer one. Therefore, the magnet displacement Δz served as a tuning parameter.

Varying the magnetic configuration at a fixed discharge power leads to changes in the discharge current and voltage. This is illustrated in Fig. 2, which shows the dependence of the discharge voltage and current on the magnetic field configuration. Increasing the unbalancing degree stimulates discharge current growth and decrease of the discharge voltage.

For the compound target, the effect of magnetic system unbalancing on discharge parameters and the ion current to the substrate was studied with radial resolution.

To measure radial ion current distribution profiles, an array of flat ion collecting probes with an area of 1 cm² each, spaced 30 mm apart, was used. The probes were mounted on a metal strip attached to the sample stage.

The dependence of the total ion current on the radial distance to the magnetron symmetry axis for different magnetic field configurations is shown in Fig. 3.

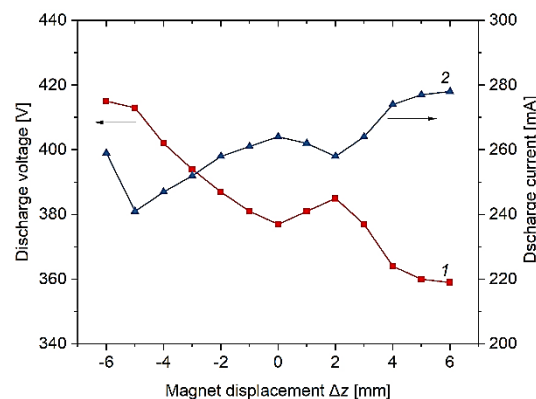


Fig. 2. Dependences of discharge voltage (1) and current (2) on the magnet displacement

Рис. 2. Зависимости напряжения (1) и тока разряда (2) от смещения магнита

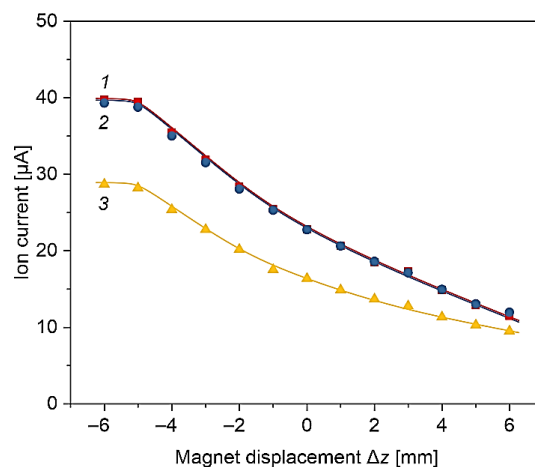


Fig. 3. Dependence of the ion current on the magnetic field configuration, measured at different radial distances: 0 cm (1), 3 cm (2), and 6 cm (3)

Рис. 3. Зависимость ионного тока от конфигурации магнитного поля, измеренная на разных радиальных расстояниях: 0 см (1), 3 см (2) и 6 см (3)

We observe a nearly linear relationship between the ion current and the distance between the magnets, which is maintained during radial movement. This means that the developed tunable magnetic system allows for smooth variation of the ion current to the substrate, thereby affecting the properties of the resulting coatings.

To study the influence of ion current distribution across the substrate on the coating parameters, the

surface resistance of the resulting AZO films was measured as a function of the radial distance to the magnetron's symmetry axis. For this purpose, a 150×26×1 mm glass strip was used as a substrate instead of acrylic plate. The radial profiles of sheet resistance R_s of the films deposited using different magnetic field configurations are shown in Fig. 4a. Fig. 4b demonstrates the correlation between the sheet resistance and the ion current derived from the data in Fig. 3 and Fig. 4a.

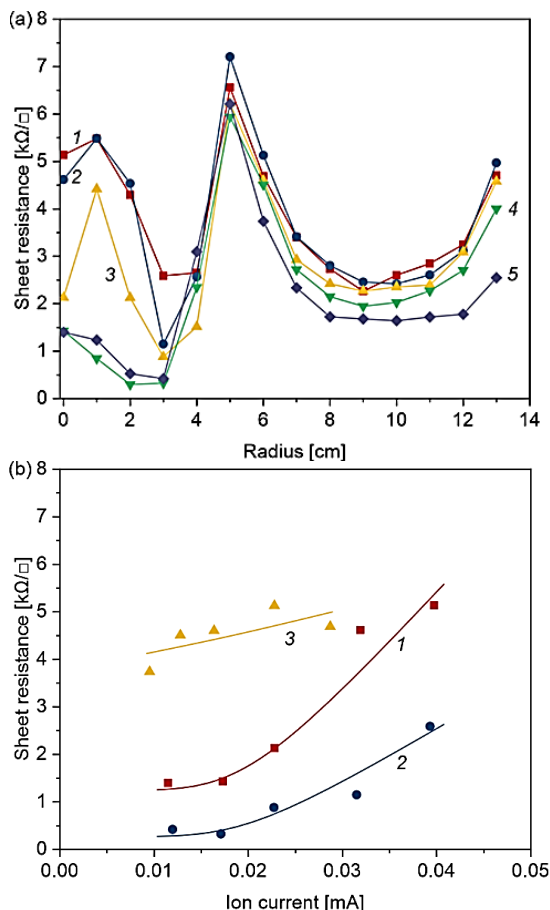


Fig. 4. Sheet resistance of the AZO films deposited in different magnetic configurations: (a) radial profiles for $\Delta z = -6$ mm (1), -3 mm (2), 0 mm (3), 3 mm (4), 6 mm (5); (b) dependences on the ion current at $r = 0$ mm (1), 3 mm (2), 6 mm (3)

Рис. 4. Поверхностное сопротивление пленок AZO, осажденных в разных магнитных конфигурациях: (а) радиальные профили при $\Delta z = -6$ мм (1), -3 мм (2), 0 мм (3), 3 мм (4), 6 мм (5); (б) зависимости от ионного тока при $r = 0$ см (1), 3 см (2), 6 см (3)

Fig. 4 demonstrates that the ion current has a significant impact on the coating properties. Varying the magnetic configuration has a particularly strong and non-monotonic effect on the surface resistance in the region spanning to $r \sim 5$ cm, which is the magnetron's main operating zone (Fig. 4a). The region of minimum coating resistance, however, closely matches the target's racetrack region ($r \sim 2$ cm). From Fig. 4b, one

might notice that for a fixed radius, R_s shows rapid growth with ion current provided $r < D/2$, where D is the target diameter (7.62 cm).

Deposition with single-element Zn and Al targets

Using separate magnetrons with metallic targets to deposit an oxide coating with complex composition requires fine tuning of a large number of process parameters. Here, a summary of the deposition conditions resulting in highly conductive transparent AZO coatings is given.

The reported samples were processed at a magnetron discharge power of 100 W. The discharge voltage of 508 V was typical for the Al target magnetron operated in pure argon. For the sputtering of an almost completely oxidized Zn target in a working gas ratio of $\text{Ar}/\text{O}_2 = 2/1$, typical Zn target magnetron discharge voltage was 408 V.

The precise control of the deposition process was implemented by cycling the magnetron operation. In each cycle, the ZnO deposition was followed by Al deposition, meaning the magnetrons were operated one at a time. Therefore, the magnetrons were not influencing each other, which is particularly important keeping in mind that highly reactive species produced in the discharges might have resulted in the unwanted surface contamination of the neighbor magnetron. Hence, using the separate ZnO/Al deposition allowed controllable doping of the coating surface with Al.

For all selected samples, we varied the following parameters: the number of processing cycles (one cycle is ZnO deposition, followed by Al deposition); the total time of Al deposition t_{Al} ; the total time of ZnO deposition t_{ZnO} . All variable parameters can be taken into account by introducing a characteristic value of the $t_{\text{Al}}/t_{\text{ZnO}}$.

The sheet resistance of the coatings was measured by the van der Pauw method, and resistivity was calculated using Eq. (1). The dependences of surface resistance and resistivity of AZO coatings on the ratio $t_{\text{Al}}/t_{\text{ZnO}}$ are shown in Fig. 5.

Sample with a $t_{\text{Al}}/t_{\text{ZnO}}$ ratio exceeding 0.33 exhibited a notable reduction in optical transparency accompanied by a significant increase in electrical resistance. These samples were excluded from the graphical representation. Optimal electronic and optical properties were achieved for samples within $t_{\text{Al}}/t_{\text{ZnO}}$ range of 0.26 to 0.31 . For these coatings, the minimal R_s values were $\sim 10 \Omega/\square$, and the corresponding $\rho \sim 3 \cdot 10^{-6} \Omega \cdot \text{m}$.

When comparing two samples with identical $t_{\text{Al}}/t_{\text{ZnO}}$ ratios, the films fabricated using a higher num-

number of deposition cycles with shorter cycle times consistently demonstrated lower sheet resistance. This suggests that the deposition process influences the film's microstructure, potentially enhancing charge carrier mobility [11]. Besides, a pronounced anti-reflection effect was observed when ZnO layer was deposited on top of Al.

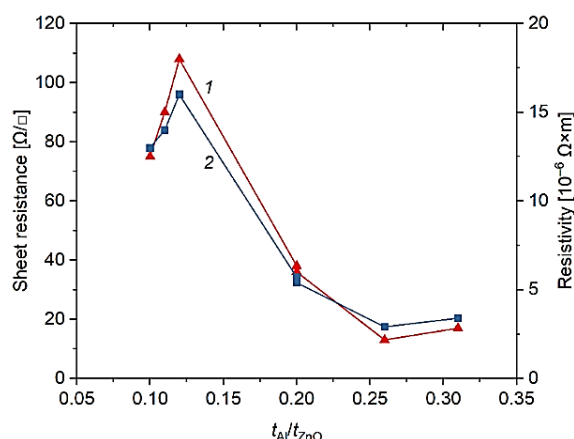


Fig. 5. The dependences of sheet resistance (1) and resistivity (2) of AZO films on the ratio t_{Al}/t_{ZnO}

Рис. 5. Зависимости поверхностного сопротивления (1) и удельного сопротивления (2) пленок AZO от отношения t_{Al}/t_{ZnO}

CONCLUSIONS

The electronic properties of AZO coatings can be greatly enhanced by fine tuning the deposition procedure. In particular, using separate magnetrons with single-element targets has been shown to enable much more flexible deposition process control than conventional compound AZO target sputtering approach.

Adjusting the ion current to the substrate during the deposition has a strong effect on the coating properties, demonstrating rapid growth of film resistance with ion current. However, tuning it requires a specialized hardware such as a variable magnetic field magnetron, which is not so common and more expensive than conventional setups.

Effective minimization of AZO coating's resistivity can be achieved by organizing the deposition in a cycled manner when forming ZnO layer and doping it with Al are separated in time. The coatings prepared using this approach have sheet resistance as low as $10 \text{ } \Omega/\square$, and resistivity $\sim 3 \times 10^{-6} \text{ } \Omega \cdot \text{m}$.

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