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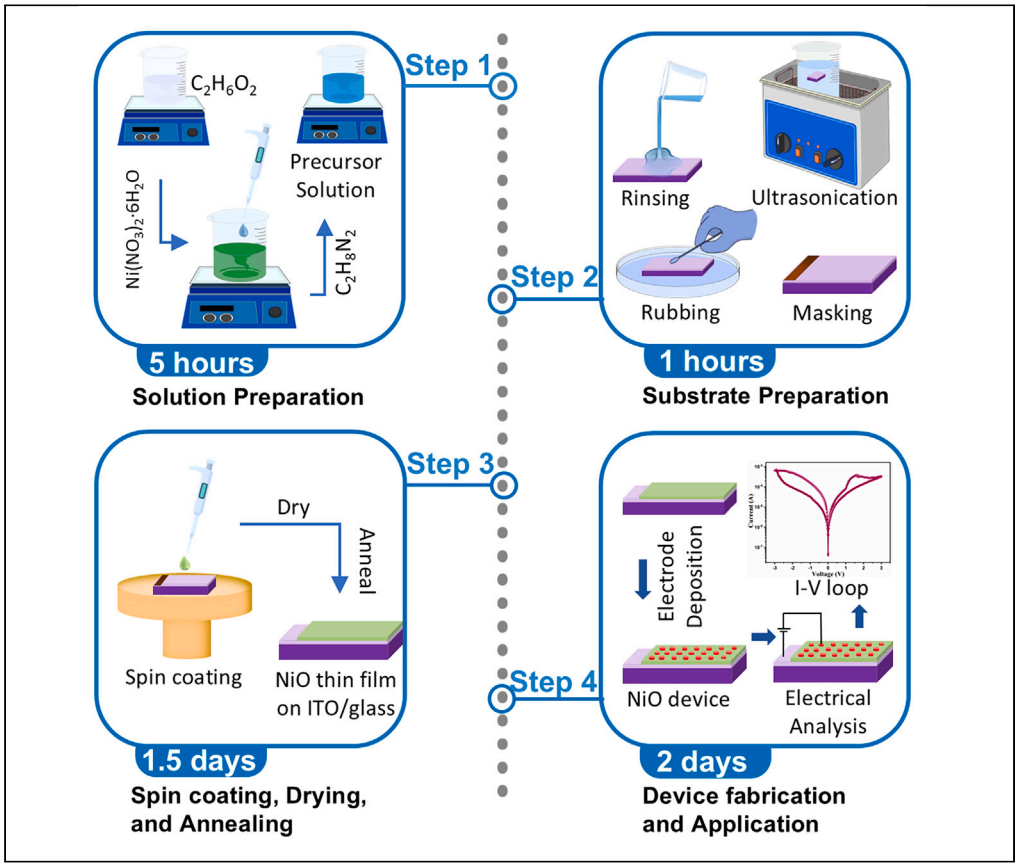
Protocol for fabricating optically transparent, pinhole-free NiO thin film for memory devices using the spin-coating technique



Fabricating visibly transparent and uniform metal oxide thin films is a significant challenge. Here, we present a protocol for the fabrication of NiO thin films. We detail the essential steps for substrate preparation. We then describe the optimized steps for spin coating, considering various parameters such as the concentration of the precursor solution, drying temperature, annealing temperature, and spin-coating speed.

Publisher's note: Undertaking any experimental protocol requires adherence to local institutional guidelines for laboratory safety and ethics.

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Highlights

Instructions for preparing the precursor solution for subsequent deposition

Step-by-step protocol to spin coat NiO thin film

Protocol facilitates the deposition of film of a thickness of ~110 nm

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Protocol for fabricating optically transparent, pinhole-free NiO thin film for memory devices using the spin-coating technique

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SUMMARY

Fabricating visibly transparent and uniform metal oxide thin films is a significant challenge. Here, we present a protocol for the fabrication of NiO thin films. We detail the essential steps for substrate preparation. We then describe the optimized steps for spin coating, considering various parameters such as the concentration of the precursor solution, drying temperature, annealing temperature, and spin-coating speed.

BEFORE YOU BEGIN

Spin coating is a simple deposition technique that can produce uniform films as thin as a few nanometers with simple instrumentation.[1,2] In the spin coating process, a precursor solution is dispensed onto a substrate. Upon spinning, centrifugal forces propel the solution outward to the edges of the substrate, expelling the excess solution and resulting in the formation of a thin, uniform film. This process consists of four distinct stages: (1) deposition, (2) spin-up, (3) spin-off, and (4) evaporation, as depicted in Figure 1. This method is preferred over other deposition techniques for its simplicity, cost-effectiveness, and uniform coverage, even with minimal equipment.[3,4] These qualities make spin coating particularly suitable for depositing NiO thin films on glass or ITO/FTO-coated glass substrates for applications where uniformity is critical for performance. The following protocol outlines the deposition of NiO thin films, which can be used in applications such as resistive switching devices, solar cells, gas sensing, and water splitting.[5–8] Although other techniques of deposition can be used, choosing the appropriate deposition method depends on several factors, including the specific application, ease of operation, cost, and safety.

It is important to conduct the entire procedure under proper safety conditions. Users should review the safety data sheets (SDS) for all chemicals involved, ensure accurate labeling of chemicals and chemical waste, and adhere to established safety protocols for handling and disposal.

KEY RESOURCES TABLE

REAGENT or RESOURCE	SOURCE	IDENTIFIER
Chemicals, peptides, and recombinant proteins		
Nickel (II) nitrate hexahydrate	Sigma-Aldrich	CAS Number: 13478-00-7
Ethylenediamine, AR	Central Drug House	CAS Number: 107-15-3

(*Continued on next page*)

Continued

REAGENT or RESOURCE	SOURCE	IDENTIFIER
Ethylene glycol, AR	Central Drug House	CAS Number: 107-21-1
Isopropyl alcohol, AR	Sisco Research Laboratories Pvt. Ltd.	CAS Number: 67-63-0
Acetone (99%)	Sisco Research Laboratories Pvt. Ltd.	CAS Number: 67-64-1
ITO-coated glass substrate	Advanced Process Technology Pvt. Ltd, Pune	None
Labolene	Thermo Fisher Scientific	None
Polyimide tape	Schofic	None
Other		
Analytical balance	Sartorius BSA224S-CW	https://mkp.gem.gov.in/compact-analytical-balance/sartorius-compact-analytical-balance-with-5-year-warranty/p-5116877-94788351122-cat.html
Hot plate with magnetic stirrer	Tarsons Digital Spinot	https://tarsons.com/product/spinot-digital-magnetic-stirrer-hot-plate/
Ultrasonic cleaner		
Spin coater	Spektrospin	https://www.spektroninstruments.in/spin-coater.html#portable-spin-coater
Muffle furnace		
Profilometer	Lyncee Tec	https://www.lynceetec.com
X-Ray diffraction system	Rigaku Technologies, Japan	https://rigaku.com/products/x-ray-diffraction-and-scattering/xrd/smartlab
Keithley 4200A-SCS Parameter analyzer	Tektronix	https://www.tek.com/en/products/keithley/4200a-scs-parameter-analyzer

MATERIALS AND EQUIPMENT

Reagent/ Chemical	Final concentration	Amount
Ethylene glycol	N/A	1 mL
Nickel nitrate hexahydrate	1 M	290.79 mg
Ethylene diamine	1 M	67 μL
Final Precursor Solution	**1 M**	1.067 mL

(Final Precursor Solution, storage condition: 25°C, max. 1 month)

STEP-BY-STEP METHOD DETAILS

Solution preparation

Timing: 5 h

This section outlines the steps to prepare the precursor solution for subsequent deposition, see Figures 2A–C. Among the different concentrations of precursor solution, we consider 1 M for nickel nitrate hexahydrate as a reference.

1. Prepare 1 M precursor solution using nickel nitrate hexahydrate.
   a. Add 1 mL of ethylene glycol to a clean, dry glass vial.
   b. Add 290.79 mg of nickel nitrate hexahydrate to the vial containing ethylene glycol.
   c. Stir the mixture using a magnetic stirrer at 500 rpm for 30 min at 30°C, until a clear green solution is formed.
   d. To the green solution, add 67 μL of ethylene diamine (ensuring an equal molar ratio of nickel nitrate hexahydrate and ethylene diamine).
   e. Stir the mixture using the magnetic stirrer at 500 rpm for 4 h at 60°C.

⚠ CRITICAL: Do not increase the temperature to more than 150°C, otherwise it will increase the viscosity of the solution, making it difficult to uniformly coat.

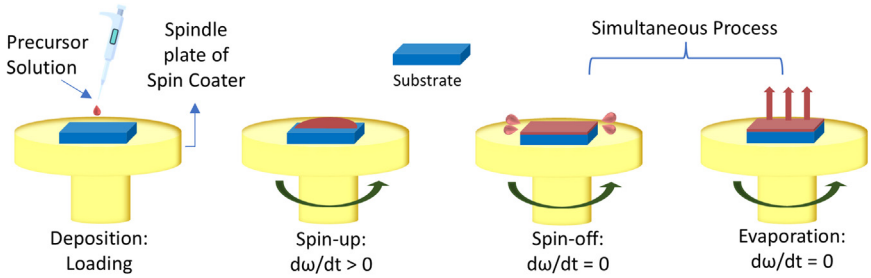

Figure 1. Stages of spin coating process

Substrate cleaning

Timing: 1 h

This step ensures that substrates are thoroughly cleaned to remove contaminants, enhancing the adhesion and uniformity of the film.

2. Clean the ITO-coated glass substrates.
   a. Rinse the ITO-coated glass substrate with warm deionized water (~40°C).
   b. Rub the surface with a cotton swab dipped in 2% v/v Labolene in H_2O (Figure 2E).
   c. Rinse thoroughly with warm deionized water.
   d. Immerse the substrate in acetone within an ultrasonic bath for 10 min (Figure 2F).
   e. Immerse the substrate in Isopropyl Alcohol within an ultrasonic bath for 10 min.
   f. Dry using nitrogen flow, or any source of dust-free air.
3. Mask the ITO-coated glass substrates.
   a. Check the conducting side using a multimeter (Figure 2G).
   b. Mask a small area of the substrate with polyimide tape (Figure 2H).

⚠ CRITICAL: Avoid touching substrates with bare hands before or after washing. Oils and grease will contaminate the substrate.

Note: If a nitrogen gun is unavailable, use a hair dryer or rubber syringe bulb as a dust blower and dryer. Skip the polyimide tape if an uncoated area is not required.

Spin-coating deposition

Timing: 20 min

In this step, a solution is applied onto the prepared substrate and spread into a thin, even film through spin-coating (Figure 2I). This method allows for control over the thickness and homogeneity of the film by adjusting spin speed and time.

4. Spin coating on the substrate.
   a. Place the substrate on the spin coater platform.
   b. Turn ON the vacuum.
   c. Set the operating parameters as: rpm to 3000, time 30 s, and ramp rate of 1000 rpm/s.
   d. Using a micropipette, dispense 10 μL of the precursor solution onto the center of the spinning substrate. After 15 s, add another 10 μL of the solution onto the same spinning substrate.
   e. Once the program is finished, turn off the vacuum, and place the coated substrate in a petri dish.

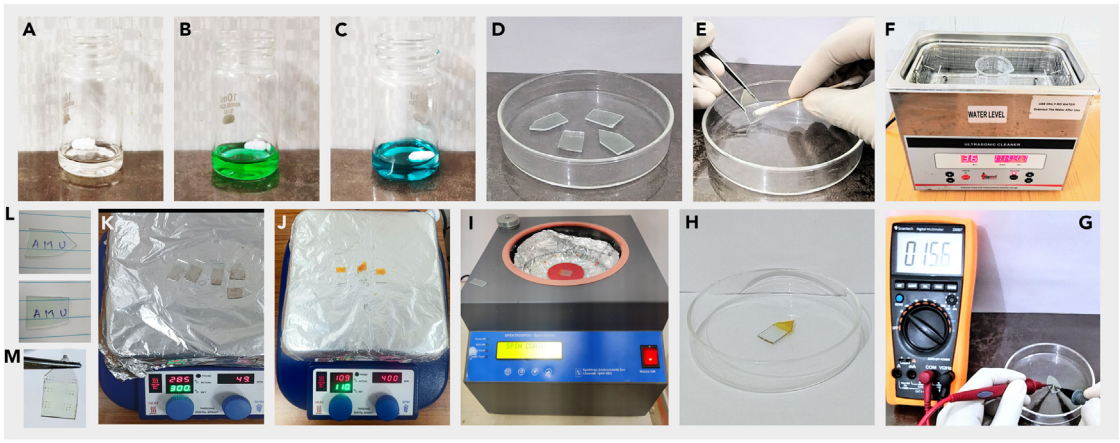

Figure 2. NiO spin coating, from substrate preparation to successful deposition
(A–C) Precursor solutions.
(D–H) Substrate Preparation.
(I) Spin coater for deposition.
(J and K) Drying of the deposited film.
(L) Film after annealing.
(M) Final device obtained after top electrode deposition.

⚠ CRITICAL: Ensure the substrate is cleaned thoroughly before spin coating to avoid any problem with film uniformity and coverage.

Note: A thickness of 110 nm was achieved through the values stated in this protocol. Rpm and time can be adjusted to get the thickness desired by the user. The volume of solution can also be increased if the coverage of the substrate is less. The spin coating speed and time can be adjusted depending on the viscosity of the solution.

Drying

Timing: 3 h 45 min

This step involves drying the spin-coated film to remove solvents gradually, helping to stabilize the film's structure and improve its quality. Proper drying is essential to prevent defects such as cracking or wrinkling.

5. Drying the thin films.
   a. Place the coated substrate onto a pre-heated hot plate at 110°C for 10 min (Figure 2J).
   b. Turn off the hot plate. Once the coated substrate has cooled down to room temperature, place it in a petri dish and remove the polyimide tape carefully using forceps.
   c. Place the coated substrate back onto the hot plate. Turn on the hot plate and set the temperature to 300°C.
   d. Now heat the substrate at 300 °C for 30 min, to get a grayish-black film (Figure 2K).
   e. Turn off the hot plate and allow the coated substrate to cool down before proceeding to the next step.

Annealing

Timing: 25–30 h

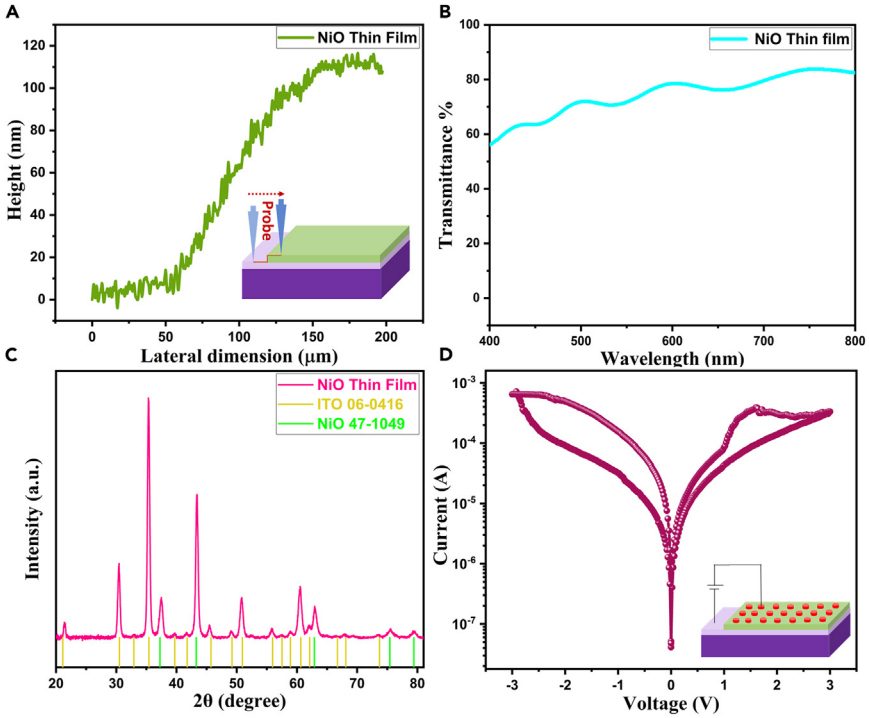

Figure 3. Structural, optical, and electrical characterization of NiO thin Films and Fabricated device

(A) Height vs. lateral distance scan showing the thickness of NiO film, inset shows the schematic for thickness measurement using a profilometer.

(B) Transmission spectrum of NiO thin film.

(C) X-ray diffraction pattern of NiO film on ITO-glass substrate.

(D) Current-voltage scan showing resistive switching for the fabricated device.

This step involves annealing the dried, spin-coated film to improve crystallinity and remove residual organic components from the film.

6. Annealing the thin films.
   a. Place the coated substrate into a muffle furnace.
   b. Turn the furnace on and set the temperature to 400°C with a ramp rate of 5°C per minute.
   c. Anneal at 400°C for 60 min.
   d. Turn off the furnace.
   e. Leave the coated substrate in the furnace for 20–24 h, allowing the temperature inside the furnace to return to room temperature before it is safe to remove the coated substrate. Once cooled, take out the coated substrate.
   f. A green-colored film is obtained (Figure 2L).

EXPECTED OUTCOMES

NiO thin films were deposited on ITO-coated glass substrates using spin coating, resulting in a uniform thickness suitable for resistive switching applications. This spin-coated film is advantageous compared to films obtained through other deposition methods, as it is cost-effective and ideal for laboratory-scale research.

The thickness of the deposited film was measured through a profilometer. The obtained height vs. length scan is shown in Figure 3A. The thickness of the film was ~110 nm. Instead of a sharp rise, we observe a gradual increase in height. The inset of Figure 3A shows the schematic of the profilometer

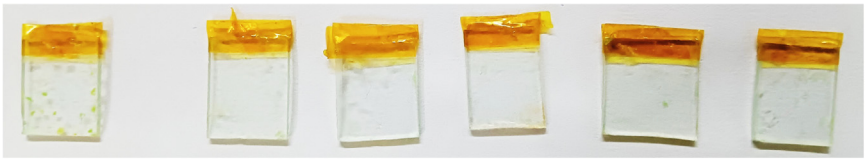

Figure 4. Films of failed deposition

measuring the thickness of the film. Using this protocol, films with thicknesses in the range of 100–150 nm can be achieved. The transmittance spectrum obtained through UV-Vis analysis is shown in Figure 3B, with a maximum of 80% transmittance in the visible range.

Characterization of the thin film was performed using X-ray diffraction (XRD) to confirm the structural properties of the deposited layer. The observed XRD peaks as shown in Figure 3C, matched with ICDD 47–1049, confirming the successful deposition of NiO on the ITO-coated glass substrate.[9] The additional peaks observed in the XRD spectra were matched with the ICDD reference card 06–0416, confirming the presence of indium tin oxide (ITO). After the confirmation of the successful deposition of NiO through XRD, the top electrodes of aluminum were deposited by a thermal evaporator using a shadow mask at a rate of 0.2 Å/s in the vacuum of 5×10^{-6} Torr. For the obtained device (Figure 2M), the electrical characterization was done using a Keithley 4200 SCS. The current-voltage (I-V) measurements demonstrated bipolar resistive switching behavior as shown in Figure 3D, confirming the film's applicability in resistive memory devices.

This protocol provides reproducible results when performed under the specified conditions, making it a reliable approach for fabricating thin films for various electronic applications.

LIMITATIONS

While the described protocol offers a reliable method for thin film deposition, several limitations need to be considered. Dust particles in the laboratory can settle on the substrate, causing pinholes and uneven films, so deposition should occur in a clean environment. During deposition, agglomeration may occur, leading to defects in the thin film. To minimize agglomeration, ensure that the precursor solution is well-mixed and free from contaminants. Before use, the solution can also be filtered through a PVDF micron filter to remove any precipitates that may cause nucleation and agglomeration. Additionally, the sol-gel solution may precipitate after prolonged storage (~30 days). To address this, either a freshly prepared solution should be used for deposition, or the stored solution should be filtered through a PVDF micron filter to maintain the precursor solution's quality. Viscosity also affects film uniformity—high viscosity can cause uneven coating, while low viscosity may result in insufficient thickness. Through this protocol, spin coating at speeds below 3000 rpm tends to produce thicker films, while speeds greater than 3000 rpm typically result in thinner films. However, thinner films may face issues such as pinholes, cracking, and poor adhesion due to the rapid solvent evaporation.

TROUBLESHOOTING

Problem 1

The substrate does not appear to be clean.

Potential solution

The grease and oils on the substrate may come from touching it with bare hands. Use gloves while touching/holding the substrate. Use Labolene or any detergent to wash off the contaminants from the surface, so that it is completely clean.

Problem 2

The precursor solution is viscous and difficult to coat evenly.

Potential solution

Prepare a fresh precursor solution.

Several factors could contribute to increasing the viscosity of the solution.

- The stirring temperature during preparation exceeded 150°C.
- The solution was stirred for longer than 4 h, even at 100°C.
- The solution was stored or aged for an extended period under varying temperatures and laboratory conditions.

Problem 3

The film is not even, there is a splash-type coating on the film, see Figure 4.

Potential solution

There are three potential causes for the film exhibiting blotches and splashes. First, verify the cleanliness of the substrate. If the substrate is not clean, refer to problem 1. If the substrate is clean, check if the precursor solution was heated to a temperature below 150°C. If it was not, refer to problem 2. If the solution was properly heated, adjust the rotational speed (rpm) and spinning time. Environmental factors, such as temperature and humidity, can influence the precursor solution, potentially altering deposition parameters. Consequently, the rpm and time should be adjusted to compensate for these changes.

RESOURCE AVAILABILITY

Lead contact

Further information and requests for resources and reagents should be directed to and will be fulfilled by the lead contact, Adiba Adiba (adibaeshaal@gmail.com).

Technical contact

Technical questions on executing this protocol should be directed to and will be answered by the technical contact, Franco Mayanglambam (franco@iitg.ac.in).

Materials availability

No new reagent was generated through this protocol.

Data and code availability

The authors declare that the data supporting the findings of this study are available within the article. All other data are available from the lead contact upon reasonable request.

ACKNOWLEDGMENTS

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AUTHOR CONTRIBUTIONS

Conceptualization and visualization, A.A. and T.A.; investigation and analysis, A.A. and F.M.; writing, A.A.; review and editing, A.A. and F.M.

DECLARATION OF INTERESTS

The authors declare no competing interests.

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