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Protocol

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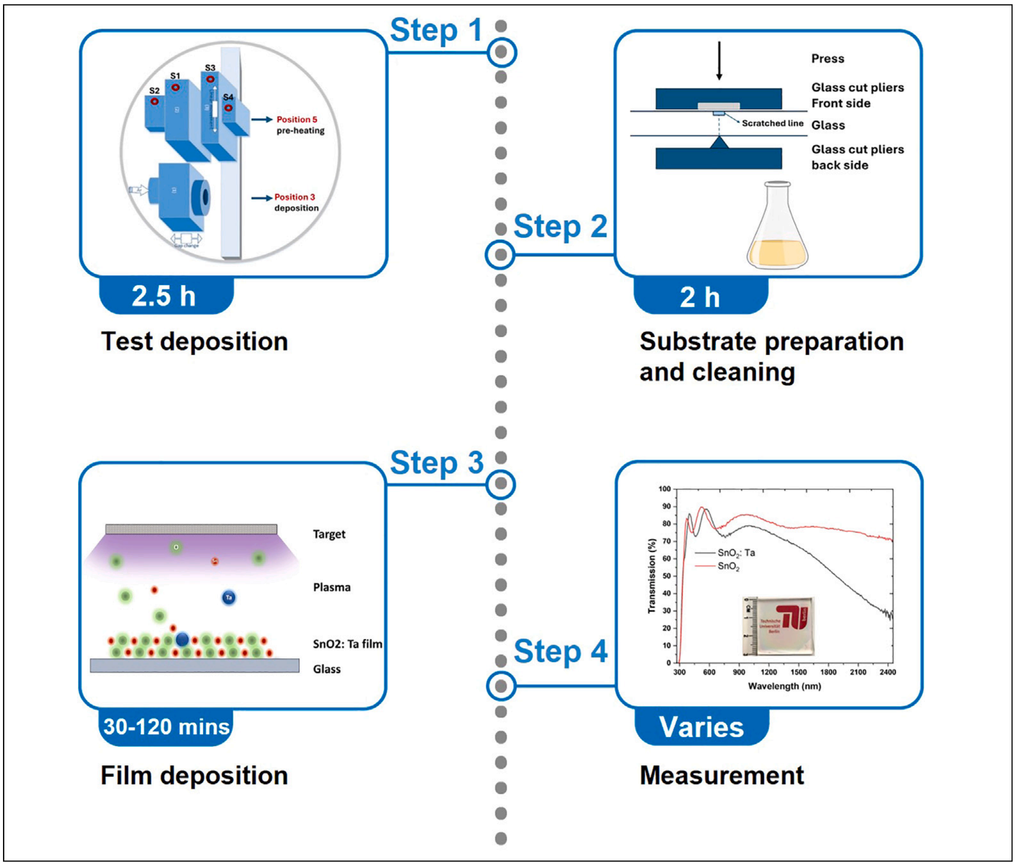

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Highlights

Steps for deposition of film on fresh uncorroded float glass substrate

Instructions for hollow cathode gas flow sputtering (GFS) technology

Guidance on identifying Ta doping effect on SnO_2 film property

Steps for optical and electrical property measurement procedure

Transparent conductive Ta-doped SnO_2 (SnO_2: Ta) thin film with low surface roughness, low resistivity, and high carrier concentration is one potential alternative of commercial transparent conductive oxides (TCOs). Here, we present a protocol for fabricating tin oxide films by hollow cathode gas flow sputtering technology. We describe steps for preparing and cleaning substrate, and film deposition process on the fresh uncorroded float glass substrate. We then detail procedures for measuring the optical and electrical properties of the film.

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Protocol

Protocol for depositing transparent conductive Ta-doped SnO_2 film by hollow cathode gas flow sputtering technology

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SUMMARY

Transparent conductive Ta-doped SnO_2 (SnO_2: Ta) thin film with low surface roughness, low resistivity, and high carrier concentration is one potential alternative of commercial transparent conductive oxides (TCOs). Here, we present a protocol for fabricating tin oxide films by hollow cathode gas flow sputtering technology. We describe steps for preparing and cleaning substrate, and film deposition process on the fresh uncorroded float glass substrate. We then detail procedures for measuring the optical and electrical properties of the film.
For complete details on the use and execution of this protocol, please refer to Huo et al.[1]

BEFORE YOU BEGIN

The protocol below describes the specific steps to deposit undoped SnO_2 and Ta doped SnO_2 (SnO_2: Ta) films on glass substrate by hollow cathode gas flow sputtering (GFS) technology under pulsed mode with different substrate temperature. This protocol could be used as a reference or guideline of other thin film materials deposited by hollow cathode gas flow sputtering technique.

The hollow cathode gas flow sputtering system (GFS) operates as a remote plasma tool. The sputtering is performed in Ar and the reactive gas is introduced in the afterglow of the hollow cathode plasma, where the sputtered particles are transported by forced convection. There for, in contrast to reactive magnetron sputtering, no transition mode process stabilization is necessary for deposition of high quality transparent conductive oxides (TCOs) films.[2]

Glass tin and air side detecting

Timing: 30 min

1. Identify tin and air side of fresh uncorroded float glass substrate.

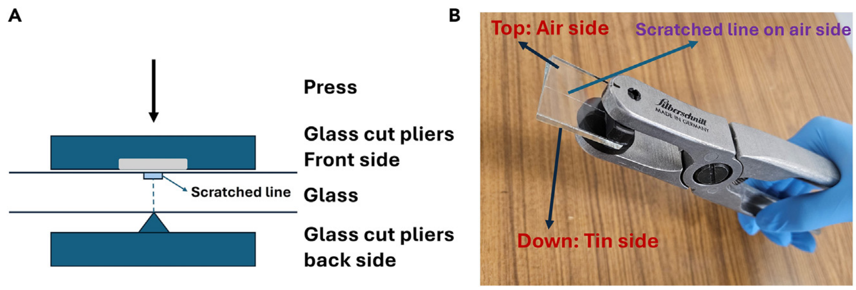

Figure 1. Glass cutting
(A) Schematic of glass cutting with glass cut pliers.
(B) Sample cutting.

a. Put glass on a flat surface.
b. Operate tin side detector following manual.
c. Make a mark '×' on the tin side with diamond marker.

Note: Protect glass surface and avoid scratch left on both surfaces.

Glass cut

Timing: 1 h

2. Cut glass substrate to desired dimension.
   a. Put glass tin side on bottom.
   b. Cut 10 cm × 10 cm × 3 mm glass to 3 cm × 3 cm × 3 mm.
   c. Make a mark '×' on tin side of each 3 cm × 3 cm × 3 mm glass with diamond marker.
   d. Separate glass to small pieces glass following the scratch line with glass cut pliers.

⚠ CRITICAL: It is necessary to put tissues on the bottom of glass to protect the surface during scratching. Put glass surface with scratched line on the center of round part with bulge when using glass cut pliers, the position of glass is shown in Figure 1.

Substrate cleaning

Timing: 90 min

3. Glass substrate cleaning.
   a. Clean glass with ethanol by ultrasonic cleaner, 15 min, room temperature.
   b. Clean glass with isopropanol by ultrasonic cleaner, 15 min, room temperature.
   c. Clean glass with deionized water by ultrasonic cleaner, 15 min, room temperature.
   d. Drying glass surface by N_2 gun.

Note: Use sample holder during cleaning process to protect glass surface.

4. Store glass substrate in sample box under air.

⚠ CRITICAL: Sample box should be cleaned before store substrate. Avoid touching substrate surface during the whole cleaning process.

KEY RESOURCES TABLE

REAGENT or RESOURCE	SOURCE	IDENTIFIER
Chemicals, peptides, and recombinant proteins		
Ethanol	MicroChemicals	CAS Nr.: 64-77-5
Isopropanol	MicroChemicals	(VLSI)CAS Nr.: 67-63-0
Deionized water	Clean room laboratory	0.055 μS/cm
Software and algorithms		
EVA	Bruker	V5.1
Other		
Glass	Saint-Gobain Glass	PLANICLEAR 30 cm × 30 cm
Ultrasonic cleaner	BANDELIN SONOREX	RK 102 H
Tin side detector	TS1420	Line-powered
Glass cut pliers	Silberschnitt	BO 700.0A
Sn target	Avaluxe International GmbH	ID 40 mm, OD 50 mm, L 70 mm
Ta target	Avaluxe International GmbH	ID 40 mm, OD 50 mm, L 5 mm
Hollow cathode gas flow sputtering system	Fraunhofer Institute for Surface Engineering and Thin Films	KL-GFS-RQ50.LCG.TA40
Four-point probe	Veeco Instruments Inc.	FPP-5000
Hall measurement	Lake Shore Cryotronics	M91
UV/VIS/NIR spectrometer	PerkinElmer	Lambda 1050+
Atomic force microscopy (AFM)	Spectral Instruments	NTEGRA II
Scanning electron microscope (SEM)	Zeiss	DSM 982 GEMINI
Wavelength dispersive X-ray spectroscopy (WDXS)	Hitachi	S-2700
X-ray diffraction	Bruker	D8 Discover

MATERIALS AND EQUIPMENT

The glass substrate cleaning process with ultrasonic cleaner should be operated in fume hood in chemical lab. The Sn and Ta targets should be stored under vacuum. The experiment program is shown in Figure 2.

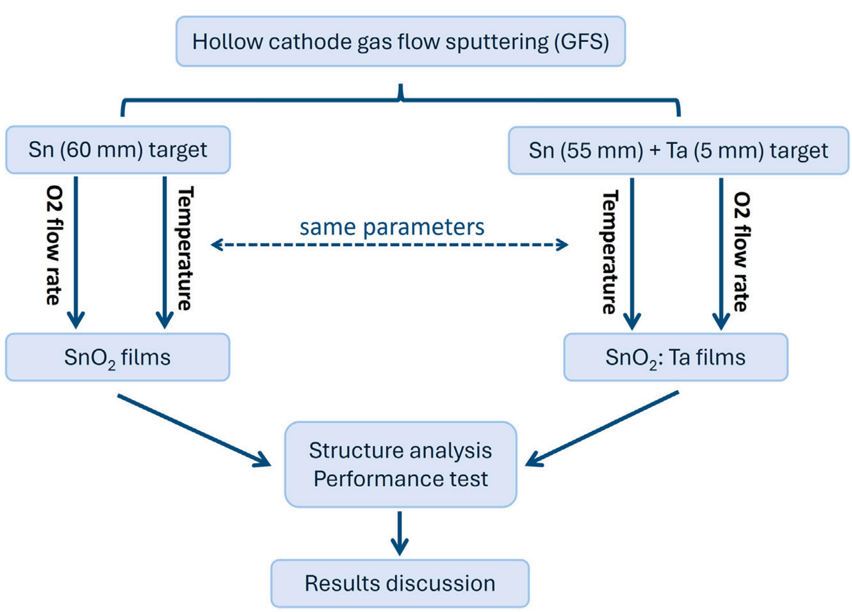

Figure 2. Experiment program

Note: Check quantity and quality of materials before experiment to get better perception in considering the same steps of experiments.

⚠ CRITICAL: Ethanol and Isopropanol are hazardous chemical due to their inflammability. One should wear protective glass, gloves and clothing to operate glass cutter due to the splash of glass.

Alternatives: The cleaning process with other chemicals, for example acetone and other special glass cleaning solutions, can be used to clean substrate surfaces.[3] The glass cutter can be replaced by other cutting methods, for example laser cutter.[4] The general chemicals used for substrate cleaning process in this protocol can be replaced by those from other sources. The instruments applied for film property characterization applied in this protocol can be replaced by those which have similar functions.[3,5–7] For example, another ultraviolet visible near infrared (UV/VIS/NIR) spectrometer, four-point probe and hall measurement setup. The experiment program can be changed by other experiment program with other sputtering system which has similar functions. For example, replace O_2 flow rate series by Ar flow rate series to study the influence of plasma conditions on film properties, replace *in situ* substrate temperature series by post annealing series.

STEP-BY-STEP METHOD DETAILS

Test deposition

⏲ Timing: 2.5 h

Test deposition should be done before the first sample deposition, to make sure the device runs under normal conditions. The following steps describe the process of preparation work before experiment starts, it works as a tiny device debugging. Figure 3 shows the overview of hollow cathode gas flow sputtering (GFS) system windows control center (WinCC), and Figure 4 shows schematic of film deposition process.

Note: Wear glove during sample preparation and inside chamber operation process. Avoid touching film surface.

⚠ CRITICAL: The following steps can be only operated when the gases (Ar, N_2 and O_2), cooling water and electricity supply normally in the sputtering lab, and there is no machine defect. After the whole procedure finished, test sample to check if sheet resistance stays in the expected range. If it is in the expected range, then continue the following protocol steps, if not, contact engineer to find out issues.

1. Turn on gas valve.
   a. Turn on the main gas valve.
   b. Turn on Ar, N_2 and O_2 valve.
2. Turn on cooling machine.
3. Click button 1 and button 2, vent chamber and vacuum line by filling with N_2.
4. Open chamber door, the structure of hollow cathode gas flow sputtering (GFS) system vacuum chamber is shown in Figure 5A.
5. Insert target.

Note: The total length of target is 60 mm $\pm$ 0.1 mm. If the target is a combination of several pieces, make sure each piece contact with other well, there is no clear space between every target piece. The shape of the target is shown in Figures 5B and 5C.

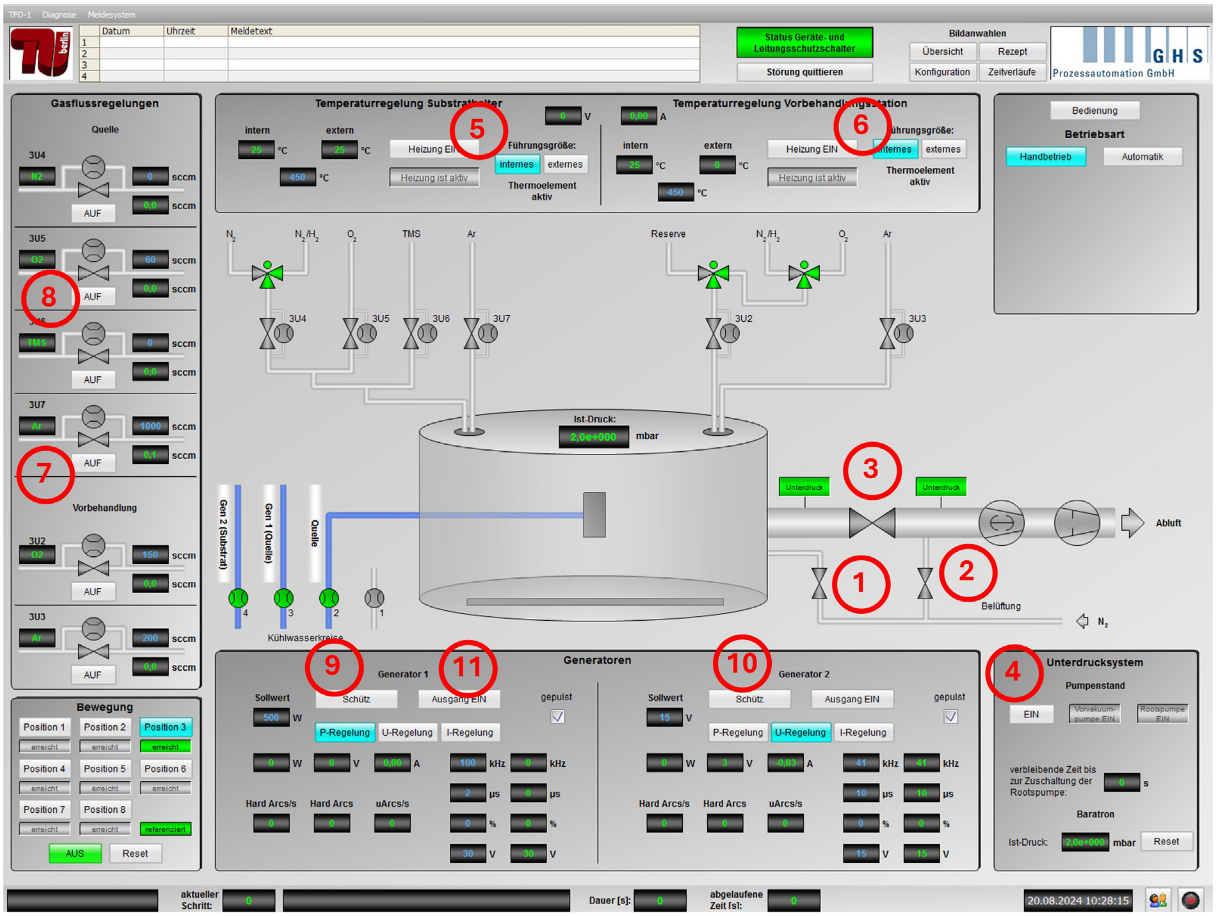

Figure 3. Hollow cathode gas flow sputtering (GFS) system

Hollow cathode gas flow sputtering (GFS) system windows control center (WinCC) overview. Button 1: vent chamber. Button 2: vent vacuum line. Button 3: open/close main vacuum valve. Button 4: start vacuum pumping. Button 5: Substrate holder temperature control. Button 6: Pretreatment temperature control. Button 7: Ar flow rate control. Button 8: O_2 flow rate control. Button 9: Generator 1 main switch. Button 10: Generator 2 main switch. Button 11: GFS Plasma control.

6. Insert substrate.

Note: For room temperature and heated substrate temperature under 150°C, substrate could be fixed by heat resistant tape. For heated substrate temperature over 150°C, substrate should be fixed with matched substrate holder.

⚠ CRITICAL: Wear fresh gloves to move and insert substrate, avoid touching the sample surface which the film will be deposited on. After sample is fixed, use N2 gun clean sample surface to avoid substrate contamination on surface.

7. Adjust the distance between target and substrate.
8. Move substrate holder to position 5.
9. Close chamber door.
10. Click button 3 and button 4, vacuum the chamber until 2 × 10^{-2} mbar.
11. Turn on heater power, click button 5 and button 6 to start heating process.

Note: Heating process is designed in advance before every experiment.

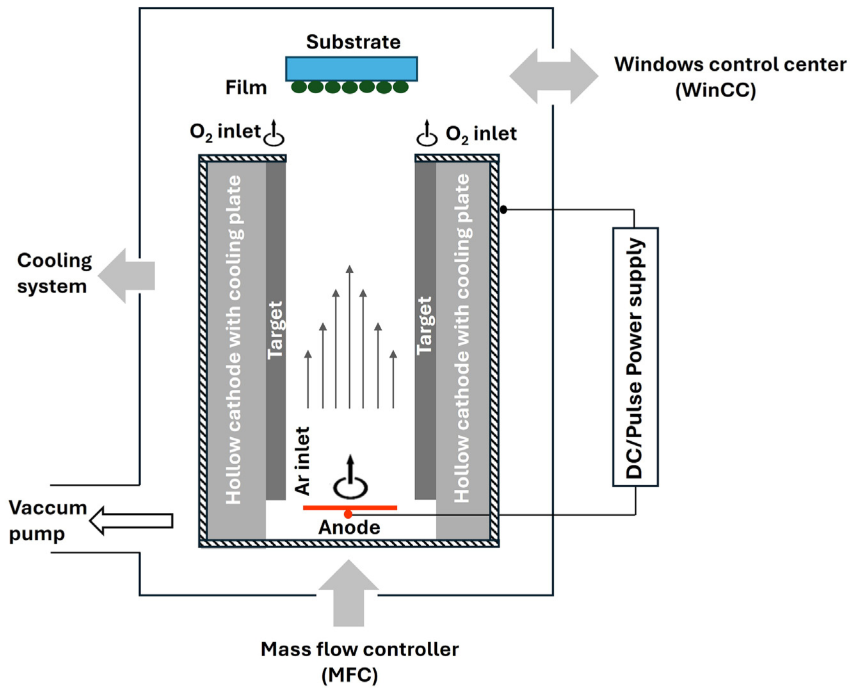

Figure 4. Schematic illustration of hollow cathode gas flow sputtering deposition process

12. Click button 5 and button 6 again to end heating process.
13. Click button 9 and button 10, fill in Ar and reactive gas O_2 to chamber.

Note: After filling in Ar and O_2, wait the vacuum value stays stable.

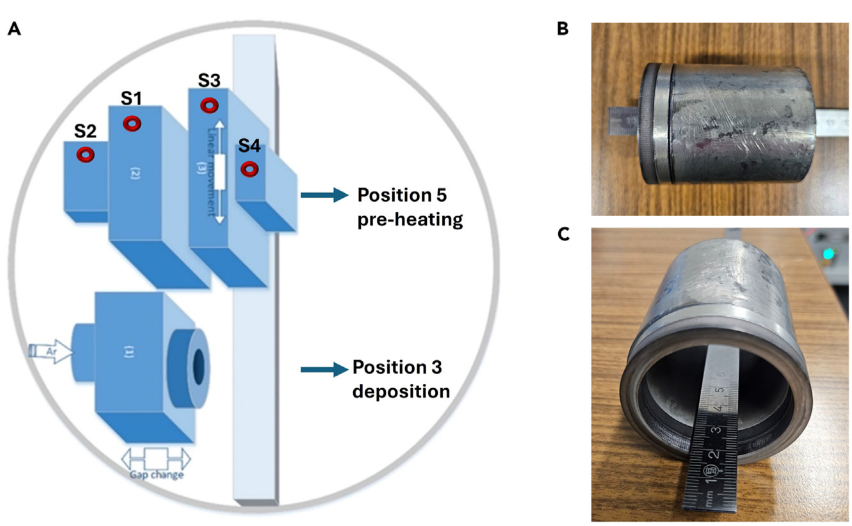

Figure 5. Hollow cathode gas flow sputtering system and target

Figure reprinted with permission from Huo et al., 2024.[1]

(A) The structure of hollow cathode gas flow sputtering system vacuum chamber: source (1), pre-heating (2), and substrate holder (3). Position 5 is used for preheating, position 3 is used for film deposition. S1 is pretreatment position (VBS)_intern sensor, S2 is pretreatment position (VBS)_extern sensor, S3 is substrate holder (SH)_ intern sensor, S4 is substrate holder (SH)_extern sensor.

(B) Top view of target.

(C) Side view of target.

14. Click buttons 9, 10 and 11 in sequence, to generate plasma and start pre – sputtering process.
15. Move substrate holder to position 3.
16. Start depositing process with designed parameters.
17. To end the deposition process, click on button 11, 10, and 9 in sequence.
18. Click button 8 and button 7 to turn off the Ar and O_2 gas flow.
19. Click button 4 and button 3 to stop vacuum chamber.
20. Wait substrate temperature cooling down to be below 70°C, click button 1 and button 2, open chamber door, and take sample out.

Note: Please follow the sputtering lab security rules carefully. When it is necessary, wear the mask when opening the chamber to take sample out to avoid detrimental hazardous situation.

21. Close chamber door.
22. Click button 3 and button 4 to vacuum system again.
23. After chamber vacuum pressure reach 10^{-2} mbar range, click button 3 and button 4 turn vacuum system off.
24. Turn heater power off, turn cooling system off, close all gas valves.
25. Measure sheet resistance of test sample.

Deposition SnO_2 film

Timing: 30–120 min (per sample, varies by deposition parameter)

The follow steps describe the deposition process of SnO_2 film on the fresh uncorroded float glass substrate based on the procedure of "Test deposition". Five series with different substrate temperature are designed for SnO_2 film deposition: I: room temperature; II: 50°C; III: 110°C; IV: 270°C; and V: 400°C.

Note: Wear glove during sample preparation and inside chamber operation process. Avoid touching film surface.

26. Design heating process, as is shown in Figures 6A–6D.

Note: Room temperature deposition does not need heating process. In this protocol, substrate temperature is defined by the temperature detected by substrate holder (SH)_extern sensor, which is marked with red dot in the Figure 6. The corresponding input value to heating system is marked with black dot, which are detected by pretreatment position sensor (VBS)_intern sensor and substrate holder (SH)_intern sensor.

⚠ CRITICAL: From 200°C heater should be heated step by step every 100°C to protect whole heating system not to overwork.

27. Insert Sn target.
28. Insert glass substrate, adjust the distance between target and substrate to 4 cm.

Note: Make sure glass substrate is fixed stable and at the center of substrate holder.

29. Pump chamber pressure to 2×10^{-2} mbar.
30. Move sample to position 5 and start heating process which is shown in Figure 6.

Note: Skip step 31 when do room temperature deposition, directly following the deposition parameters described in step 32 to prepare films.

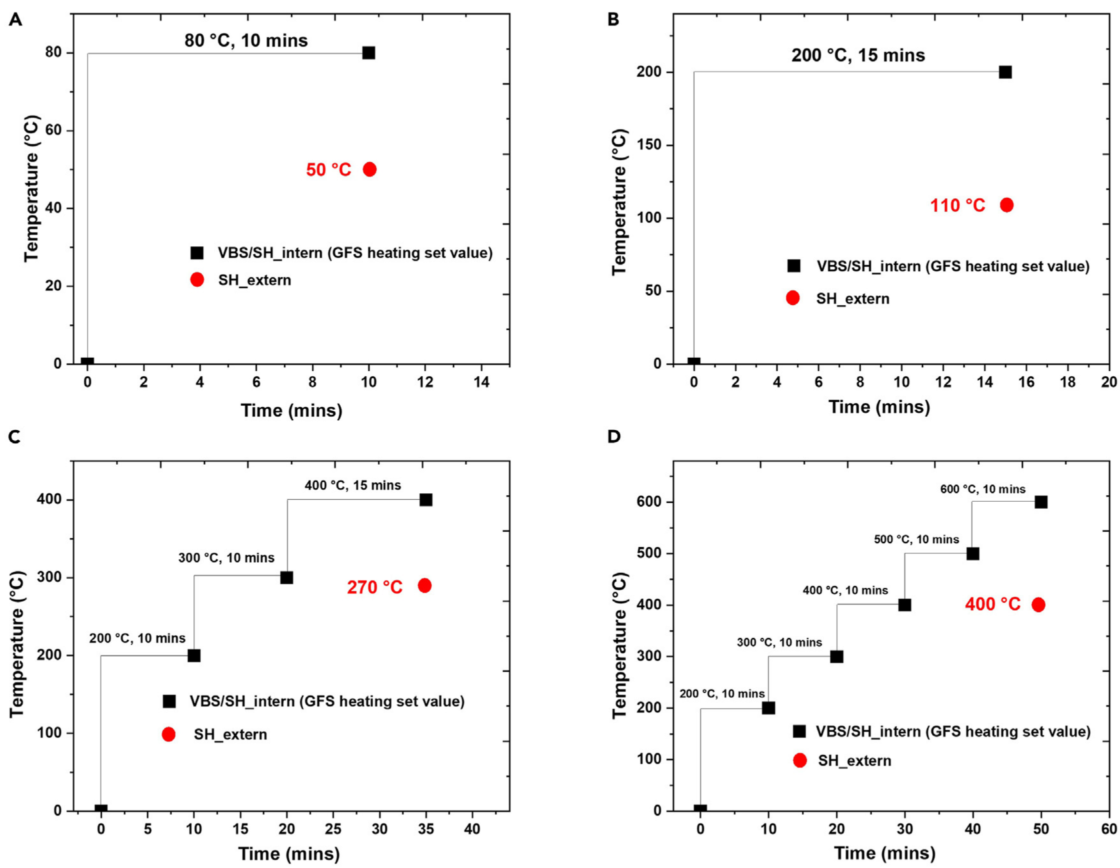

Figure 6. Heating process of different substrate temperature

Heated substrate holder temperature detected by substrate (SH)_extern sensor is marked with red dot in the figure.

(A) 50°C.

(B) 110°C.

(C) 270°C.

(D) 400°C.

31. After heating process completed, move sample to position 3.
32. Start deposition process.
    a. Ar flow rate is 1000 sccm.
    b. O_2 flow rate is 5–45 sccm.

    Note: The step of O_2 flow rate is 5 sccm.

    c. Operating pressure during sputtering is held at 3.2×10^{-1} mbar.
    d. Operating power is 250 W.
    e. Pre- sputtering time is 5 min.

    Note: Pre – sputtering step here is to clean target surface.

    f. Pulse mode reverse voltage is 100 V, frequency is 100 kHz, time is 2 μs.
    g. Deposition time is 60 s to deposit ~350 nm SnO_2 film, as deposition rate is 6 nm/s.
33. Repeat step 17 to step 20, until take sample out.

Note: When it is necessary, wear the mask when opening the chamber to take sample out to avoid detrimental hazardous situation.

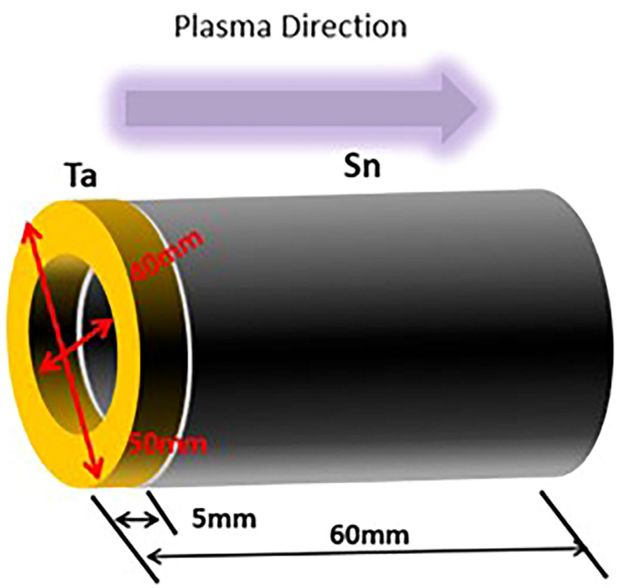

Figure 7. 3D Ta doping ring and Sn target used for SnO_2: Ta films deposition

34. Insert next substrate, repeat step 30 to step 34 to prepare the second sample.
35. Repeat step 35 for multiple times and complete the depositions for all samples which are designed for temperature series and O_2 flow rate series.
36. Repeat step 21 to step 24 to finish deposition.

Deposition SnO_2: Ta film

Timing: 30–120 min (per sample, varies by deposition parameter)

The follow steps describe the deposition process of SnO_2: Ta film on the fresh uncorroded float glass substrate based on the procedure of "Deposition SnO_2 film". Five series with different substrate temperature are also designed for SnO_2: Ta film deposition: I: room temperature; II: 50°C; III: 110°C; IV: 270°C; and V: 400°C.

Note: Wear glove during sample preparation and inside chamber operation process. Avoid touching film surface.

CRITICAL: To compare SnO_2: Ta films with SnO_2 film, expect the target is different, other parameters should keep same with SnO_2 film. Substrate heating process is same as the heating process of SnO_2 film which is shown in Figure 6.

37. Insert Ta target, the 5 mm doping ring.

Note: Make sure Ta ring is at the beginning of plasma direction, the 3-dimension (3D) Ta ring and plasma direction is shown in Figure 7.

38. Insert Sn ring.
39. Repeat steps from 28 to 36 to complete the deposition of SnO_2: Ta films.

Measuring optical and electrical property of the films

Timing: depends on measurement method

The following steps describe the process of measurements of optical and electrical properties.

Note: For all measurement wear gloves to move sample and avoid touching sample surface.

40. Apply a UV/VIS/NIR spectrometer to measure total transmittance and reflectance of prepared films.
    a. To measure the total transmittance, put sample at the front window of the integrating sphere, and put white standard at the back window of integrating sphere.
    b. To measure the reflectance, put sample at the back window of integrating sphere.

⚠ CRITICAL: Calibration should be done before transmittance and reflectance measurement.

41. Choose Bragg Brentano geometry to do XRD measurement.
42. Put sample on test plate, measure edge and center film position sheet resistance by using four-point probe, calculate the average value as the film sheet resistance R_{sh} (Ω/ □), mark the films which are conductive.
43. Follow step 2, cut conductive samples to 1.5 cm × 1.5 cm to hall measurement. The needles contacts four corners edge of sample. When contact well, change the direction of magnet to get film resistivity, mobility and carrier concentration.
44. Go for scanning electron microscope (SEM) to study the morphology and cross section of the films.

Note: To ensure sample could be reused and remeasured after SEM measurement, do SEM without any coating on sample surface.

EXPECTED OUTCOMES

The operating pressure of sputtering system is at 3.2×10^{-1} mbar with inputting different O_2 flow rate from 5 sccm to 45 sccm. For all experiments, the time needed to pump chamber pressure to background 2×10^{-2} mbar is similar. The sample under room temperature is not conductive, with the increasing of substrate temperature, film turns to be conductive. The conductivity of the film is changing with different O_2 flow rates. If the film looks dark at the lowest O_2 flow rate at 5 sccm, it should turn to be transparent by eyes after increasing the O_2 flow rate to next level. Since the deposition time is controlled at 60 s, the thickness of all films is between 300 and 400 nm. The doping effect could be seen at least in one temperature series, by observing the conductivity of Ta doped SnO_2 film is higher than that of SnO_2 film. With crystallized film, which is detected by X-ray diffraction (XRD) measurement, the growing structure shows like collum grown crystal, which is shown in Figure 8A. Transmittance of SnO_2 and SnO_2: Ta is shown in Figure 8B. The peak positions in XRD pattern of crystallized films are matched with references, which are shown in Figure 8C. The host material structure of SnO_2 is not changed by Ta doping since the atom ratio of Ta in SnO_2: Ta film is small, 2.11% in SnO_2: Ta film with 270°C substrate temperature, corresponding 1.608% of C, 69.316% of O, 0.47 of Ar, 26.496% of Sn. And Ta doping influences the film crystallinity and grown structure. Diffractive (110), (200) and (211) peaks move to a smaller 2θ value could be observed under different oxygen flow rate, due to the radius of Ta5+ is smaller than that of Sn^{4+}, which is shown in Figures 8D–8F. The diffraction peaks of the thin films differ under different oxygen flow rates comes from the correlated different plasma conditions with high plasma density in GFS technology and reactive gas oxygen during deposition process, which could influence the film grown structure. Roughness of the deposited film at 25 sccm oxygen flow with different substrate temperature is listed in Table 1.

Note: For oxides other than SnO_2, oxygen flow rate and other deposition parameters could be established based on the literature overview work from other researchers and groups, and different trial experiments following the experiment steps which are describe in this protocol.

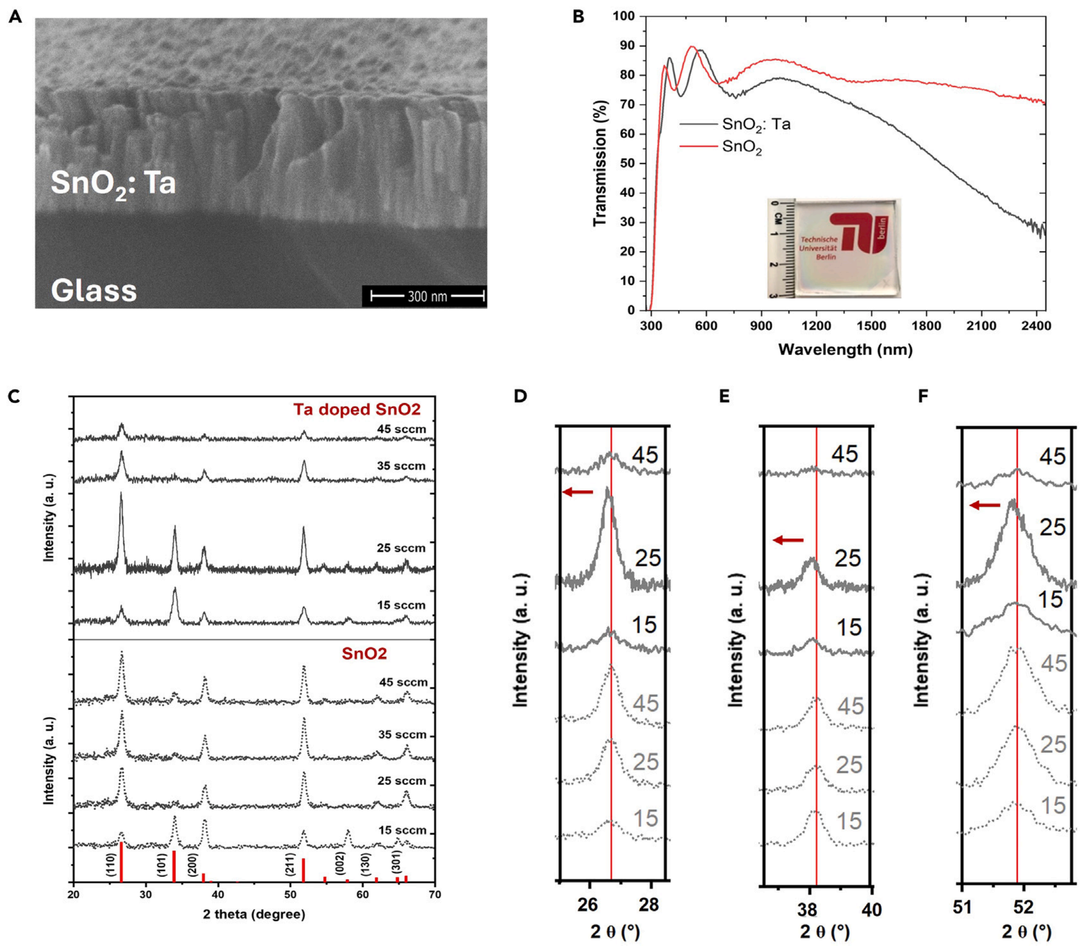

Figure 8. The deposited successful tin oxide film

(A) Collum structure of tin oxide film, scale bar is 300 nm.

(B) Transmission of SnO_2 and Ta doped SnO_2 film. Insert is the photo of prepared film.

(C) XRD pattern of crystallized tin oxide films.

(D) (110) peak shift.

(E) (200) peak shift.

(F) (211) peak shift.

LIMITATIONS

This protocol should be applied carefully since there are limitations. The deposition parameters for tin oxide films, for example, target power, O_2 flow rate, substrate temperature, and deposition time, etc. for the equipment in this protocol may be not applicable for other sputtering equipment with similar functions. Due to the limitation of the equipment, only one sample in this protocol could be deposited one time. With high temperature substrate deposition, heating and cooling process takes time. There is also the limitation of the kind of glass substrate used in this protocol. The tin oxide film structure and property may not be applicable for other substrates.[8]

TROUBLESHOOTING

Problem 1

Plasma cannot be generated successfully. After filling in Ar, switch on generator, plasma cannot be seen through the chamber window, there is no power, voltage and current value in WinCC window (steps 15, 33 and 39).

Table 1. AFM measurements on a 1 μm × 1 μm large area for samples deposited at 25 sccm oxygen flow

Sample	Roughness (RMS)
SnO_2 @ 270°C	6.02 nm
SnO_2 @ 400°C	4.08 nm
SnO_2: Ta @ 270°C	3.01 nm
SnO_2: Ta @ 400°C	2.32 nm

Potential solution

Use handheld vacuum cleaner to clean anode surface and check the insertion of target.

For one piece 60 mm target, make sure the target surface and anode surface are in one plane. There is no gap between the cover and anode and cathode (target), which is shown in Figure 9.

For several integrated targets, make sure each pieces contact well with others.

For the thin target ring (< 5 mm), insert slowly and carefully to forbid the rotating in anode.

Problem 2

Heating curves shows not correctly or stay at 0 during heating process in WinCC window (steps 11 and 30).

Potential solution

Check the contact of sensor cable at substrate holder and chamber wall. Substrate holder (SH)_extern sensor cable at substrate holder back side maybe is damaged due to the movement of substrate holder or the incorrectly operation of the position changing of substrate holder to fix and take sample out.

Problem 3

Target edge color is different with the color in target middle position by visual. (e.g., step 5).

Potential solution

Sand target internal surface with sandpaper, remove the particles on target surface. Target surface status could be changed due to long time deposition. Besides pre-sputtering, remove target surface by physical and mechanical way is also possible to make sure the target is under metallic status.

Problem 4

Film is not deposited on the center of the substrate. (step 28).

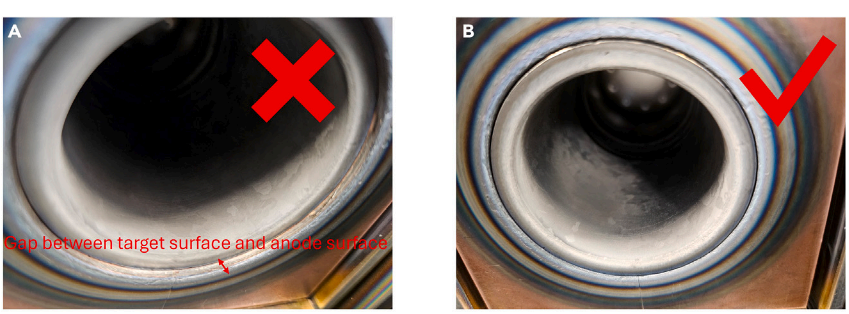

Figure 9. Target installation
(A) Wrong installation.
(B) Correct installation.

Potential solution

Make sure sample is stuck in the substrate holder at the matched position.

RESOURCE AVAILABILITY

Lead contact

Further information and requests for resources should be directly to and will be fulfilled by the lead contact, Bernd Szyszka (bernd.szyszka@tu-berlin.de).

Technical contact

Questions about the technical specifics of performing the protocol should be directed to and will be fulfilled by the technical contact, Fangfang Huo (fangfang.huo@tu-berlin.de) and Bernd Szyszka (bernd.szyszka@tu-berlin.de).

Materials availability

This study did not generate new unique reagents.

Data and code availability

This study did not generate/analyze datasets/code.

ACKNOWLEDGMENTS

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AUTHOR CONTRIBUTIONS

F.H. conducted the experiments, analyzed data, and wrote the manuscript. B.S. supervised the whole work and provided experiment resources. M.H. cut glasses and samples and provided sputtering lab support. B.B.O.S. provided XRD lab support and AFM studies. N.A. and R.M. provided sputtering lab support. C.W., P.G., and B.S. contributed to manuscript editing.

DECLARATION OF INTERESTS

The authors declare no competing interests.

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