

Submission ID #: 71082

Scriptwriter Name: Pallavi Sharma

Project Page Link: <https://review.jove.com/account/file-uploader?src=21680058>

Title: Improving Thermoelectric Properties of Bi₂Te₃ Thin Films by Manganese Co-Sputtering

Authors and Affiliations:

Angella Th'ng¹, Nurfarhana Ahmad Musri¹, Puvaneswaran Chelvanathan¹, Mohd Sukor Su'ait¹, Nadhrah Md Yatim², Assayidatul Laila Nor Hairin³, Ubaidah Syafiq¹

¹Solar Energy Research Institute (SERI), Universiti Kebangsaan Malaysia

²Faculty of Science and Technology, Universiti Sains Islam Malaysia

³Department of Manufacturing and Materials Engineering, Kulliyah of Engineering, International Islamic University Malaysia

Corresponding Authors:

Ubaidah Syafiq

(ubaidahsyafiq@ukm.edu.my)

Angella Th'ng

(p153906@siswa.ukm.edu.my)

Email Addresses for All Authors:

Nurfarhana Ahmad Musri

(farhanamusri@gmail.com)

Puvaneswaran Chelvanathan

(cpuvaneswaran@ukm.edu.my)

Mohd Sukor Su'ait

(mohdsukor@ukm.edu.my)

Nadhrah Md Yatim

(nadhrah@usim.edu.my)

Assayidatul Laila Nor Hairin

(assayidatul_laila@iiium.edu.my)

Ubaidah Syafiq

(ubaidahsyafiq@ukm.edu.my)

Angella Th'ng

(p153906@siswa.ukm.edu.my)

Author Questionnaire

1. Microscopy: Does your protocol require the use of a dissecting or stereomicroscope for performing a complex dissection, microinjection technique, or something similar? **No**

2. Software: Does the part of your protocol being filmed include step-by-step descriptions of software usage? **No**

3. Filming location: Will the filming need to take place in multiple locations? **Yes**

400 m

4. Testimonials (optional): Would you be open to filming two short testimonial statements **live during your JoVE shoot**? These will **not appear in your JoVE video** but may be used in JoVE's promotional materials. **No**

Current Protocol Length

Number of Steps: 28

Number of Shots: 50

Interviews

Videographer: Obtain headshots for all authors available at the filming location.

INTRODUCTION:

~~REQUIRED: Summarize your research focus and the main questions it aims to answer.~~

- 1.1. **Angella Th'ng:** My research examines the changes in thermoelectric performance of manganese-doped bismuth telluride thin films deposited through controlled co-sputtering.
 - 1.1.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera *Suggested B roll: 3.7.1*

~~Describe the research gap or limitation in existing methods and how this protocol addresses them.~~

- 1.2. **Angella Th'ng:** Existing methods fail to adequately examine the impact of manganese doping on the thermoelectric performance of bismuth telluride thin films. This protocol addresses this gap through systematic investigation.
 - 1.2.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera

CONCLUSION:

~~What does this protocol allow researchers to study or measure?~~

- 1.3. **Angella Th'ng:** This protocol allows researchers to co-sputter manganese dopant on bismuth telluride film and evaluate how dopant input level changes thermoelectric performance.
 - 1.3.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera

~~Describe the most important consideration or challenge when performing this protocol.~~

- 1.4. **Angella Th'ng:** The key challenge of this protocol is to maintain stable plasma and sputtering conditions to incorporate manganese reliably and consistently.

- 1.4.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera. *Suggested B-roll: 2.8.2*

~~How can future studies build on this work? Describe the questions or research directions that can be investigated next.~~

- 1.5. **Angella Th'ng:** Future studies can be built by optimizing thermoelectric properties post-synthesis and fabricating a thin-film thermoelectric generator for device-level testing.
- 1.5.1. INTERVIEW: Named talent says the statement above in an interview-style shot, looking slightly off-camera

Videographer: Obtain headshots for all authors available at the filming location.

Protocol

***Videographer:** Please always record an audio slate while filming each shot mentioned in the shotlist.*

2. Pre-deposition Preparation for Thin Film Deposition

Demonstrator: Angella Th'ng

- 2.1. To begin, place the prepared and cleaned soda lime glass substrates on the rotating substrate holder to evaluate deposition stability during target conditioning [1]. Close the chamber door and initiate pump-down [2]. Apply gentle pressure around the chamber door at the start to ensure proper sealing [4] and confirm the pressure drop on the gauge [5].
 - 2.1.1. WIDE: Talent placing the test substrates on the rotating substrate holder inside the sputtering chamber. ***Videographer:** Please obtain CU shot as well for this shot.*
 - 2.1.2. Talent closing the chamber door and initiating pump-down on the sputtering system.
 - 2.1.3. Talent applying gentle pressure around the chamber door seal.
 - 2.1.4. Shot of the chamber pressure dropping on the system gauge.
- 2.2. ~~Monitor the chamber pressure using the system vacuum gauge integrated with the sputtering system controller [1].~~ Continue pumping until the chamber pressure reaches less than or equal to 5×10^{-3} Torr [1].
 - 2.2.1. ~~Show the chamber pressure reading on the sputtering system vacuum gauge.~~ **NOTE: This shot can be omitted because the footage was captured after the chamber pressure had already reached below the required value.**
 - 2.2.2. Show the chamber pressure reaching less than or equal to 5×10^{-3} Torr on the controller display.
- 2.3. Switch on the cooling system and set the temperature to 288 Kelvin [1] to ensure continuous circulation of recirculated deionized water through both sputter guns prior to plasma ignition [2].
 - 2.3.1. Talent switching on the cooling system and setting the temperature to 288 kelvin on the controller.
 - 2.3.2. Shot of recirculated deionized water flowing through the cooling lines connected to both sputter guns.
- 2.4. Introduce high-purity argon gas [1] and allow it to stabilize for approximately 2 to 5 minutes, or until it remains steady at greater than or equal to 3.9 standard cubic centimeters per minute [2]. **NOTE: The VO is edited**

- 2.4.1. Talent introducing argon gas into the sputtering chamber at 4 standard cubic centimeters per minute.
- 2.4.2. Show the stabilized argon gas flow reading at greater than or equal to 3.9 standard cubic centimeters per minute. **NOTE: The argon flow controller was changed before filming, so the displayed flow value differs from the previous system. During filming, the controller display did not show 4 sccm directly. The argon condition was adjusted based on the new controller setting and confirmed by the chamber pressure response.**
- 2.5. Set the substrate rotation to 10 rpm in a single fixed direction and maintain continuous rotation throughout pre-sputtering and deposition [1]. Power on the radio frequency power supply and the matching-network controller [2].
 - 2.5.1. Talent setting the substrate rotation speed to 10 revolutions per minute on the controller. **NOTE: The substrate rotation controller uses a numbered dial from 0 to 9. During filming, the dial was set to "1," which corresponds to 10 rpm for this sputtering system**
 - 2.5.2. Talent powering on the radio frequency power supply and the matching-network controller.
- 2.6. Adjust the matching-network settings for both radio frequency guns using the Minimum or Maximum function until the Load value and the Tune value reach 50 watts [1]. Switch the controller mode from Manual to Auto matching [2].
 - 2.6.1. Show the matching-network controller while adjusting the Load and Tune values to 50 watts using the Min/Max function.
 - 2.6.2. Show the controller mode being changed from Manual to Auto matching.
- ~~2.7. Minimize reflected power before plasma ignition, keeping it at or below 5 watts. [1].~~
 - ~~2.7.1. Show the reflected power decreasing on the system controller before plasma ignition, and show the reflected power stabilized at less than or equal to 5 watts.~~ **NOTE: Not filmed. Information is merged in 2.8 due to repetitive information**
- 2.8. Set the radio frequency power to 50 watts on the bismuth telluride gun and 10 watts on the manganese gun before plasma ignition. Minimize reflected power before plasma ignition, keeping it at or below 5 watts [1]. Ignite the plasma for both guns and confirm stable plasma by observing a steady glow maintained for approximately 1 to 2 minutes without visible flicker or sudden pressure spikes [2].
 - 2.8.1. Show the radio frequency power settings of 50 watts for the bismuth telluride gun and 10 watts for the manganese gun. *Video editor: Highlight the FWD value for radio frequency power and REF value for reflected power*
 - 2.8.2. Talent ignites the plasma for both sputter guns with a shot of stable plasma glow inside the sputtering chamber.

2.9. Once stable plasma was achieved at both guns, pre-sputter both targets for 15 minutes to remove surface contamination and native oxides after stable plasma is achieved [1].

2.9.1. Talent performing pre-sputtering with stable plasma inside the chamber.

2.10. Remove the test substrates after pre-sputtering [1].

2.10.1. Talent removing the test substrates from the rotating substrate holder.

3. Co-Sputtering Deposition Procedure for Mn-Doped Bi₂Te₃ Thin Films

3.1. Before loading the substrates for the deposition run, remove dust from the chamber interior and the sample-holder region using a clean rubber blower [1]. Keep the viewing window protected with a clean petri dish lid for plasma observation [2].

3.1.1. Talent removing dust from the chamber interior and sample-holder region using a clean rubber blower.

3.1.2. Talent placing a clean petri dish lid over the viewing window.

3.2. Inspect the targets to ensure that they are properly seated, centered, and in good condition [1]. Verify that each target is firmly clamped and properly centered within the sputter gun [2].

3.2.1. Talent inspecting the sputtering targets inside the chamber.

3.2.2. Talent checking that the targets are firmly clamped and centered within the sputter guns.

3.3. Place the cleaned substrates symmetrically near the outer region of the rotating holder at approximately 5 to 10 millimeters from the holder center [1]. Align the longer side of each substrate parallel to the holder edge [2]. Fix the target-to-substrate distance at 55 millimeters for both sputter guns [3]

3.3.1. Talent placing the cleaned substrates symmetrically on the rotating holder near the outer region.

3.3.2. Close-up shot of the substrates aligned parallel to the holder edge.

3.3.3. Talent confirming the 55-millimeter target-to-substrate distance for both sputter guns.

3.4. Pump down the chamber and continue evacuation for at least 5 hours or until the base pressure stabilizes at the target value [1]. Confirm base-pressure stability when the pressure drift remains less than or equal to 5% over approximately 10 minutes [2] and the base pressure reaches 5×10^{-5} Torr or lower [3]. **NOTE: The VO is edited as per the shots**

3.4.1. Talent initiating chamber pump-down for the deposition run.

3.4.2. ~~Show the chamber pressure decreasing during evacuation.~~ **NOTE: NOTE filmed. This shot may appear similar to earlier pressure-gauge footage because the**

same pressure gauge display was used throughout the sputtering process.

- 3.4.3. Show the stabilized base-pressure reading with less than or equal to 5% drift over approximately 10 minutes.

Added shot: Show the vacuum gauge at a base pressure of 5×10^{-5} Torr or lower.

- 3.5. Stabilize the cooling system at 288 kelvins after reaching base pressure [1]. Set the argon flow to 4 standard cubic centimeters per minute and allow it to stabilize [2]. Maintain substrate rotation at 10 rpm [3].

- 3.5.1. Talent setting the cooling-system temperature to 288 kelvin.

- 3.5.2. Show the argon gas flow stabilized at greater than or equal to 3.9 standard cubic centimeters per minute. **NOTE: Due to the new controller display, the setting shown during filming may differ from the nominal 4 sccm value.**

- 3.5.3. Show the substrate rotation speed set to 10 revolutions per minute.

- 3.6. Confirm the matching-network settings for both guns and enable Auto matching on the controller [1].

- 3.6.1. Show the settings for both sputter guns and enable auto matching

- 3.7. Initiate the bismuth telluride gun at 70 watts for easier plasma ignition [1]. Increase the power by 5 watts every 10 seconds until reaching 100 watts [2]. Start the deposition timer only after the bismuth telluride power readback remains at 100 watts for approximately 30 seconds and plasma stability is confirmed [3].

- 3.7.1. Show the bismuth telluride gun initiated at 70 watts on the power controller.

- 3.7.2. Show the gradual increase of the bismuth telluride power in 5-watt increments until reaching 100 watts.

- 3.7.3. Show the stable 100-watt power readback and the deposition timer being started.

- 3.8. Deposit the films for 30 minutes while maintaining stable plasma conditions during the deposition process [1].

- 3.8.1. Talent monitoring the sputtering system during the 30-minute deposition process.

- 3.9. Using the same parameters, repeat the deposition steps for manganese at radio-frequency powers of 10 watts and 15 watts, using fresh substrates [1].

- 3.9.1. Talent loading fresh substrates for subsequent manganese power conditions.

4. System Shutdown, and Post-Deposition Sample Handling

- 4.1. Initiate shutdown through the system controller by switching off the radio frequency output first [1]. Reduce the argon flow to 10 to 11 standard cubic centimeters per minute while keeping the cooling system active until the chamber is vented and the

sputter guns return to near-ambient temperature [2].

4.1.1. Show the radio frequency output being switched off on the controller.

4.1.2. Show the argon flow reduced to 11 standard cubic centimeters per minute while the cooling system remains active. NOTE: The new argon flow controller did not display 0 mL/min during filming. The lowest displayed value was approximately 10–11 mL/min, so the filmed shot shows the argon flow being reduced from 22 mL/min to approximately 10–11 mL/min.

4.2. Confirm that the turbomolecular pump has stopped completely and the speed reading is 0 before venting the chamber [2].

4.2.1. Show the turbomolecular pump status indicator on the controller.

4.3. Vent the chamber slowly using dry nitrogen through the vent valve to allow gradual pressure equalization and minimize particle disturbance [1].

4.3.1. Talent slowly opening the vent valve to introduce dry nitrogen into the chamber for venting.

4.4. Open the chamber only after the pressure returns to approximately atmospheric pressure at approximately 760 Torr, and the chamber door can be released without resistance [1].

4.4.1. Talent opening the sputtering chamber after confirming atmospheric pressure conditions.

4.5. Wear a mask and gloves during sample removal to reduce contamination and exposure to fine particles [1]. Remove the deposited samples from the chamber [2].

4.5.1. Talent wearing a mask and gloves before handling the samples.

4.5.2. Talent removing the deposited samples from the chamber.

4.6. Transfer the samples into a clean petri dish [1]. Store the samples in a vacuum storage container and perform characterization within 24 to 72 hours after deposition [2].

4.6.1. Talent placing the deposited samples into a clean petri dish.

4.6.2. Talent placing the petri dish into a vacuum storage container.

4.7. After sample removal, rough-pump the chamber for 10–15 minutes to reach the required pre-TMP pressure, minimizing exposure to ambient air and moisture [1].

4.7.1. Talent initiating rough pumping of the chamber after sample removal.

Results

5. Results

5.1. X-ray diffraction patterns confirmed that all samples retained the rhombohedral bismuth telluride phase across the manganese power series [1].

5.1.1. LAB MEDIA: Figure 4.

5.2. The dominant diffraction peak shifted toward higher 2 theta values with increasing manganese input [1]. The crystallite size generally decreased from 14.23 nanometers at 0 watts to 8.35 nanometers at 15 watts, consistent with peak broadening at higher manganese power [2].

5.2.1. LAB MEDIA: Figure 5.

5.2.2. LAB MEDIA: Figure 5. *Video editor: Highlight the wider peak shape of the 15 watt manganese curve compared with the narrower 0 watt curve.*

5.3. Surface field-emission scanning electron microscopy images demonstrated clear changes in film microstructure with increasing manganese input [1].

5.3.1. LAB MEDIA: Figure 6.

5.4. The 0-Watt control film showed larger polygonal grains with distinct grain boundaries [1], while the 5-Watt Manganese-doped film exhibited a more compact surface with finer grains [2].

5.4.1. LAB MEDIA: Figure 6. *Video editor: Highlight 6A.*

5.4.2. LAB MEDIA: Figure 6b. *Video editor: Highlight 6B.*

5.5. Films deposited at 10 Watt and 15-Watt Manganese power appeared denser, with fewer intergranular gaps than the 0-Watt and 5-Watt samples [1].

5.5.1. LAB MEDIA: Figure 6. Video editor: *Highlight 6c and D.*

5.6. Elemental composition analysis confirmed that manganese incorporation increased with increasing manganese target power [1].

5.6.1. LAB MEDIA: Table 3. *Video editor: Highlight the "Mn (at.%)” column showing the increase from 0.00 to 0.59 across the power values.*

5.7. All films exhibited a negative Seebeck coefficient, confirming n-type conduction behavior, and the magnitude of the Seebeck coefficient generally increased with temperature for all samples [1].

5.7.1. LAB MEDIA: Figure 7. *Video editor: Follow the plotted lines from left to right and emphasize the marked points on the right side.*

5.8. The highest Seebeck coefficient magnitude was observed for the 15-watt film, reaching

minus 162.08 microvolts per kelvin at 523 kelvins [1].

5.8.1. LAB MEDIA: Figure 7. *Video editor: Highlight the pink 15-watt manganese curve*

5.9. All films exhibited semiconducting transport behavior, where resistivity decreased with increasing temperature [1].

5.9.1. LAB MEDIA: Figure 8.

5.10. The maximum power factor was achieved for the 5-watt film, reaching approximately 529.33 microwatt per meter per kelvin squared at 523 kelvins [1].

5.10.1. LAB MEDIA: Figure 9. *Video editor: Highlight the red 5 watt manganese curve*

5.11. Although the 15-watt film exhibited the highest Seebeck coefficient magnitude, the increased resistivity reduced the power factor to 300.59 microwatt inverse meter inverse kelvin squared [1].

5.11.1. LAB MEDIA: Figure 9. *Video editor: Highlight the pink 15-watt manganese curve*

1. Sputtering
Pronunciation link: <https://www.merriam-webster.com/dictionary/sputter>
IPA: /'spʌtərɪŋ/
Phonetic Spelling: sput·er·ing
2. Deposition
Pronunciation link: <https://www.merriam-webster.com/dictionary/deposition>
IPA: /,depə'zɪʃən/
Phonetic Spelling: dep·uh·zish·uhn
3. Substrates
Pronunciation link: <https://www.merriam-webster.com/dictionary/substrate>
IPA: /'sʌbstreɪt/
Phonetic Spelling: sub·strayt
4. Soda Lime Glass
Pronunciation link: <https://www.merriam-webster.com/dictionary/soda-lime%20glass>
IPA: /'soudə 'laɪm glæs/
Phonetic Spelling: soh·duh lym glas
5. Vacuum
Pronunciation link: <https://www.merriam-webster.com/dictionary/vacuum>
IPA: /'vækju:m/
Phonetic Spelling: vak·yoom
6. Deionized
Pronunciation link: <https://www.merriam-webster.com/dictionary/deionize>
IPA: /di'arəˌnaɪzd/
Phonetic Spelling: dee·eye·uh·nyzd
7. Argon
Pronunciation link: <https://www.merriam-webster.com/dictionary/argon>
IPA: /'ɑ:rgən/
Phonetic Spelling: ar·gon
8. Radio Frequency
Pronunciation link: <https://www.merriam-webster.com/dictionary/radio%20frequency>
IPA: /'reɪdiəʊ 'fri:kwənsi/
Phonetic Spelling: ray·dee·oh free·kwuhn·see
9. Matching Network
Pronunciation link: <https://www.merriam-webster.com/dictionary/network>
IPA: /'nɛtˌwɜ:k/
Phonetic Spelling: match·ing net·wurk
10. Reflected Power
Pronunciation link: <https://www.merriam-webster.com/dictionary/reflected>
IPA: /rɪ'flɛktɪd/
Phonetic Spelling: ruh·flek·tid
11. Plasma
Pronunciation link: <https://www.merriam-webster.com/dictionary/plasma>
IPA: /'plæzmə/
Phonetic Spelling: plaz·muh

12. Bismuth
 Pronunciation link: <https://www.merriam-webster.com/dictionary/bismuth>
 IPA: /'bɪzməθ/
 Phonetic Spelling: biz·muth
13. Telluride
 Pronunciation link: <https://www.merriam-webster.com/dictionary/telluride>
 IPA: /'tɛljəˌraɪd/
 Phonetic Spelling: tel·yuh·ryd
14. Manganese
 Pronunciation link: <https://www.merriam-webster.com/dictionary/manganese>
 IPA: /'mæŋɡəniːz/
 Phonetic Spelling: mang·guh·neez
15. Oxides
 Pronunciation link: <https://www.merriam-webster.com/dictionary/oxide>
 IPA: /'ɑːksaɪd/
 Phonetic Spelling: ok·syd
16. Co-Sputtering
 Pronunciation link: No confirmed link found
 IPA: /koʊ 'spʌtərɪŋ/
 Phonetic Spelling: koh sput·er·ing
17. Mn-Doped
 Pronunciation link: No confirmed link found
 IPA: /ɛm ɛn doʊpt/
 Phonetic Spelling: em·en doapt
18. Bi₂Te₃
 Pronunciation link: No confirmed link found
 IPA: /baɪˈzɪməθ təˈlʊraɪd/
 Phonetic Spelling: by·zuh·muth tuh·lur·ide
19. Evacuation
 Pronunciation link: <https://www.merriam-webster.com/dictionary/evacuation>
 IPA: /ɪˌvækjuˈeɪʃən/
 Phonetic Spelling: ih·vak·yoo·ay·shuhn
20. Turbomolecular
 Pronunciation link: <https://www.howtopronounce.com/turbomolecular>
 IPA: /ˌtɜːrbəʊməˈləkjələr/
 Phonetic Spelling: tur·boh·muh·lek·yuh·ler
21. Nitrogen
 Pronunciation link: <https://www.merriam-webster.com/dictionary/nitrogen>
 IPA: /'naɪtrədʒən/
 Phonetic Spelling: ny·truh·juhn
22. Rhombohedral
 Pronunciation link: <https://www.howtopronounce.com/rhombohedral>
 IPA: /ˌrɑːmboʊˈhiːdrəl/
 Phonetic Spelling: rom·boh·hee·druhl

23. Diffraction
Pronunciation link: <https://www.merriam-webster.com/dictionary/diffraction>
IPA: /dɪˈfrækfən/
Phonetic Spelling: dih·frak·shuhn
24. Crystallite
Pronunciation link: <https://www.howtopronounce.com/crystallite>
IPA: /ˈkrɪstələɪt/
Phonetic Spelling: kris·tuh·lyt
25. Field-Emission
Pronunciation link: <https://www.howtopronounce.com/field-emission>
IPA: /fiːld ɪˈmɪʃən/
Phonetic Spelling: feeld ih·mish·uhn
26. Microstructure
Pronunciation link: <https://www.merriam-webster.com/dictionary/microstructure>
IPA: /ˈmaɪkroʊˌstrʌktʃər/
Phonetic Spelling: my·kroh·struk·cher
27. Polygonal
Pronunciation link: <https://www.merriam-webster.com/dictionary/polygonal>
IPA: /pəˈlɪɡənəl/
Phonetic Spelling: puh·lig·uh·nuhl
28. Intergranular
Pronunciation link: <https://www.howtopronounce.com/intergranular>
IPA: /ˌɪntərˈɡrænjələr/
Phonetic Spelling: in·ter·gran·yuh·ler
29. Elemental
Pronunciation link: <https://www.merriam-webster.com/dictionary/elemental>
IPA: /ˌɛləˈmentəl/
Phonetic Spelling: el·uh·men·tuhl
30. Seebeck
Pronunciation link: <https://www.howtopronounce.com/seebeck>
IPA: /ˈziːbek/
Phonetic Spelling: zee·bek
31. Semiconducting
Pronunciation link: <https://www.merriam-webster.com/dictionary/semiconducting>
IPA: /ˌsemɪkəˈndʌktɪŋ/
Phonetic Spelling: sem·ee·kun·duk·ting
32. Resistivity
Pronunciation link: <https://www.merriam-webster.com/dictionary/resistivity>
IPA: /ˌriːzɪˈstɪvəti/
Phonetic Spelling: ree·zih·stiv·uh·tee
33. Thermoelectric
Pronunciation link: <https://www.merriam-webster.com/dictionary/thermoelectric>
IPA: /ˌθɜːmoʊɪˈlektrɪk/
Phonetic Spelling: thur·moh·ih·lek·trik

